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General synthetic approach towards annelated 3a,6-epoxyisoindoles by tandem acylation/IMDAF reaction of furylazaheterocycles. Scope and limitations.

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ABSTRACT

An efficient and versatile one-pot synthesis of 3,6a-epoxyisoindoles annelated with oxazine, oxazole, thiazine, thiazole, pyrimidine fragments and with their benzoannelated analogues is presented. The method is based on tandem *N*-acylation/intramolecular cycloaddition (the intramolecular Diels-Alder reaction of furan, IMDAF) reaction between α , β -unsaturated acid anhydrides and α -furyl substituted azaheterocycles. The latter can be easily prepared by condensation of diverse furfurals and 1,2- or 1,3-*N*,*X*-binucleophiles (aminoalcohols, aminothiols, diamines). The observed IMDAF reaction is stereoselective: *exo*-adducts are formed exclusively with large prevalence of one of the diastereoisomers. In most cases, the condensation/*N*-acylation/IMDAF reaction sequence may be carried out *via* a one pot domino protocol. The scope and limitations of the proposed approach are thoroughly investigated. The obtained Diels-Alder adducts are attractive and useful substrates for further transformations. Fused isoindoles can be prepared from them in one step by aromatization of the 7-oxabicyclo[2.2.1]heptene ring. Other transformations, including halogenation, ring cleavage, and Wagner–Meerwein skeletal rearrangement, are also demonstrated. The spatial structures of the obtained compounds have been established by *X*-ray diffraction analyses.

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1. Introduction

Furyl amines are common building-blocks for the preparation of nitrogen-containing heterocyclic compounds and are often utilized for the formation of indole rings in particular. Electrophilic recyclization¹ and intramolecular [4+2] cycloaddition reactions² are used for this purpose. Intramolecular Diels-Alder reactions of furfurylamines towards isoindoles, on the other hand, have been investigated less comprehensively. The present work is aimed at demonstrating the possibility of construction of diverse isoindole-containing heterocyclic systems using this method.





Fig. 1. Target objects.

A vast amount of research and many publications have been devoted to the synthesis and chemical transformations of with isoindoles³ and isoindoles annelated various azaheterocycles: 1,3-oxazolidine,⁴ 1,3-oxazinane,^{4a-c,4f,4g,5} 1,3thiazolidine,^{4a,5d,5f,6} 1,3-thiazinane,^{5f,6b,6d,6e,7} pyrimidine.5b,5f,8 Similar epoxyisoindoles (Figure 1) have been studied significantly less well,^{5a,9} and there is only sparse and not always reliable information^{9b,10} (see Results and Discussion) on 3a,6epoxyisoindoles annelated with the above mentioned heterocyclic fragments. Given the limited number of available methods of preparation of partially hydrogenated isoindoles, as well as the high synthetic potential of the 7-oxabicyclo[2.2.1]heptane (heptene) fragment and its immediate availability via IMDAF from furans bearing unsaturated substituents in the side chain,¹¹ we propose an effective and versatile method of synthesis of epoxy-containing [1,3]oxazolo-, [1,3]oxazino-, [1,3]thiazolo-, [1,3]thiazino[2,3-a]isoindoles and pyrimido[2,1-a]isoindoles, as well as their benzoannelated analogues (Figure 2).



Fig. 2. General approach to the synthesis of isoindoles using IMDAF reaction

We demonstrate that such epoxy-containing adducts are useful precursors for further transformations, one of them being a onestep approach to fused isoindoles. The only known general approach towards isoindoles annelated with various heterocycles was proposed by a Hungarian research group^{4a,4c,5a,5b,5d,5e,6a,6c,8d,9a} led by P. Sohár and is based on condensation of 2-oxocarbonic acids with various 1,2-(1,3-, 1,4-)aminoalcohols (thioalcohols and diamines). In many cases the authors were able to achieve satisfactory yields of target heterocycles, however, in general, the key step of the transformation – *retro*-Diels-Alder reaction required high temperature (over 170 °C) and often led to mixtures of isomeric products. Also, the Pedrosa group in their 1998–2000 publications^{10a-10c} utilized a similar IMDAF-based approach for the preparation of optically active decahydroisoquinolines and tetrahydroisoindolines. Synthetic approaches to heterocycle-fused isoindoles were recently reviewed.¹²

It should be noted also, that a large number of heteroannelated isoindoles possess biological activity¹³; some representative examples are given in Figure 3. Excellent activities against grampositive bacteria of compounds A $(X = O)^{14a}$ and B^{14b} and an activity against gram-negative bacteria of A (X = O, S, NH)^{14a} have been reported. Compound C has been proposed to protect shoots against adverse effects of 2,4sunflower dichlorophenoxyacetic acid herbicide.¹⁵ Sub-structures D and E are useful as antiviral drugs.^{6e,16} Particularly, some thiazolo[2,3alisoindol-5-ones \mathbf{D}^{6e} and 10b-phenyl-1,3,4,10btetrahydropyrimidino[2,1-a]isoindol-6(2H)-one¹⁶ inhibited human immunodeficiency virus reverse transcriptase with an IC_{50} of 2.2×10^{-6} M. Heterocycles **F** increase the blood level of GLP-1 (glucagon-like peptide 1) and are useful as remedies for diabetes, preventives for chronic complications of diabetes, and antiobesity agents.17

We could not find any information regarding bioactivity of heterocycle-annelated epoxyisoindoles and their derivatives.



Fig. 3. Selected examples of bioactive isoindoles annelated with various five- and six-membered *O*,*S*,*N*-heterocycles

This work is a development of our approach towards annelated isoindoles and isoquinolines *via* epoxyisoindoles by $IMDAF^{18}$ and proposes a short preparative methodology for design of isoindoles fused with two heteroatom containing fiveand six-membered heterocyclic fragments and their benzoannelated analogues. The approach is based on substituted furfurals and involves a sequence of transformations including condensation/ acylation/ intramolecular [4+2] cycloaddition. Subsequent aromatization of the cycloaddition adducts may lead to fused isoindoles (Figure 2).

Given that the general strategy of synthesis of isoindoles using the IMDAF reaction has already been widely utilized,¹⁹ we believe that the experimental simplicity of the method, availability of starting materials and the synthetic diversity of the final products will promote wide application of the presented approach to preparation of fused epoxyisoindoles.

2. Results and discussion

2.1. Epoxyisoindoles fused with oxazoles and oxazines

Schiff bases **1**, the products of condensation of aromatic aldehydes (including furfural) with 1,2- and 1,3-aminoalcohols, are known to exist in tautomeric equilibrium with 1,3-oxazolidines^{20a-f} and 1,3-oxazinanes **2**, respectively.^{20a,20c,21,22} The equilibrium position depends on the solvent, temperature and on the nature of the Ar-2 substituent. When ethanolamine is treated with aromatic aldehydes, the equilibrium usually shifts towards the open chain form – azomethine. The presence of a bulky substituent in the 1,2-aminoalcohol leads to a shift towards the

ring form – up to 70% of 2-aryl-1,3-oxazolidines can be observed. 2-Phenyl-1,3-oxazinanes are usually more stable than the corresponding chain tautomers: the mixture may contain up to 98% of the former.²² However, the tautomeric mixture of products of condensation of furfural with 1,3-aminopropanol contains only 23% of the cyclic form – 2-furyl-1,3-oxazinane (**2a**)^{20a} (Scheme 1). Also, direct *N*-acylation of imines has been demonstrated.^{20g-j} Nevertheless, the presence of the ring form **2** in the tautomeric mixtures allows access to epoxyisoindoles-containing heterocycles *via* tandem acylation/IMDAF reaction¹⁸ (Figure 2), which has been the main topic of our research in recent years.



Scheme 1. Synthesis of 8,10a-epoxy[1,3]oxazino[2,3-a]isoindoles 4 and their 7-carboxy derivatives 3.

First, we investigated the reaction of aliphatic aminoalcohols with substituted furfurals. The condensation of 3-aminopropanol with $5-R^1$ -furfurals occurred at room temperature in the presence of a dehydrating agent (MgSO₄) and resulted in formation of the equilibrium $1 \leftrightarrows 2$ mixtures in almost quantitative yields. The position of the ring-chain tautomeric equilibrium $1 \leftrightarrows 2$ was determined by the ratio of the ¹H NMR integral intensities of the N=CH (8.0-8.3 ppm, chain form) and H-2 (5.2-6.0 ppm, ring form) protons for reaction mixture solutions in CDCl₃. In most cases, the reaction mixture contained 12-27% of the ring tautomer 2. The rate of formation of the tautomeric mixtures depends on the electronic effects of the substituent R^{1} : the reaction of 5-nitrofurfural was complete in 10 min at 0 °C and it took about 1 day at room temperature for the condensation to proceed in case of 5-arylfurfurals. We failed to carry out the condensation of 5-methoxyfurfural,²³ bearing a strongly electrondonating methoxy group, with propanolamine in these conditions.

The next step of the proposed protocol is the acylation of furfurylamines (ring tautomers 2) with α,β -unsaturated acid anhydrides. As was shown before,^{11a,18,19} the reaction of furfurylamines with anhydrides and chlorides of α,β -unsaturated acids does not stop at the *N*-acylation products (similar to 2*) formation step, instead, it is immediately followed by intramolecular *exo*-Diels–Alder cyclization with the furan ring (IMDAF). In general, the last step of this transformation requires heating up to 80–110 °C, but in a recent short communication^{18a} we showed, that reaction of 2-furyl-1,3-oxazinane 2a with maleic anhydride occurs at room temperature, which allows performing the transformation of furfurals to 8,10a-epoxy[1,3]oxazino[2,3-*a*]isoindole 3a in a single synthetic procedure (Scheme 1).

From a preparative point of view, the procedure is exceptionally simple. A solution of maleic anhydride and the tautomeric mixture $1 \Rightarrow 2$ in CH₂Cl₂ was stirred at room temperature for 4 h – 2 days. The slightly colored adducts **3** were isolated by filtration and were used for most practical purposes without further purification. Oxazino[2,3-*a*]isoindoles (**3**) can also be obtained by carrying out the acylation/[4+2] cycloaddition in acetone (room temperature, 4–24 h). In this case the obtained samples were more than 98% pure. Only the adduct obtained by acylation of 2-(5-phenyl-2-furyl)-1,3-oxazinane (**2f**) could not be isolated by simple filtration, probably due to the low content of the ring form **2f** (14%) in the tautomeric equilibrium mixture **1f**=**2f**, and the low crystallization propensity of the product. Therefore, column chromatography was used to isolate the adduct **3Af**.

The reaction of Schiff bases 1a-c with acryloyl chloride, a weaker dienophile compared to maleic anhydride, led to 8,10a-epoxy[1,3]oxazino[2,3-a]isoindoles 4a-c via a similar acylation/cycloaddition domino reaction but in somewhat lower yields (Scheme 1). Similar to the case of maleic anhydride, the intermediate *N*-acyl derivatives (analogous to 2^*), were neither detected nor isolated.

The IMDAF reaction in both cases was essentially stereoselective:^{11a} only *exo*-adducts were formed with a large prevalence of the **3A**, **4A** diastereoisomers. Individual isomers **3A** were easily isolated by crystallization from an *i*-PrOH/DMF mixture (individual diastereoisomers **3A**, **4A** were used for further transformations). Only for product **4c** ($R^1 = Br$) a low diastereoselectivity was observed – the **4Ac/4Bc** ratio was 64/36.

Similar IMDAF reaction conditions were used to obtain adducts discussed below (Schemes 5, 8, 9, 11, 12, 15, 16, 18) and the experimental data are summarized in Table 1.

We tried to determine the relative configuration of the H-10b proton in the adducts **3c,d** (for samples with the highest content

of the minor isomer **3B**) on the basis of 1D nOe experiments. When the H-10b proton in **3Ac,d** was irradiated, small changes in the integrated signal intensity were observed in H-6a. Somewhat higher but comparable intensities were observed for the minor isomers **3Bc,d**. Therefore, we employed a more reliable structural determination method for these adducts (see Scheme 13 and corresponding text below).

The sensitivity of the condensation/acylation/cycloaddition tandem reaction to steric hindrance by substituents in the α position to the nitrogen atom of the amino binucleophile was then probed (Scheme 2). Readily available 4-amino-4-methylpentan-2-ol²⁴ was selected as a model compound. Due to the Thorpe-Ingold effect,²⁵ the tautomeric equilibrium in this case completely shifted towards the ring form - oxazinane 5 (furyl and Me-6 occupy equatorial positions in the six-membered cycle). The ring tautomer 5 readily reacted with maleic anhydride at 25 °C in ether, but the reaction led to formation of a large amount of byproducts. Diastereomers 6 were isolated in rather low yields (Scheme 2) with the 6A/6B ratio of ~ 3/1. When acetone, benzene or dichloromethane were used as reaction solvents, the diastereoselectivity was higher by comparison with ether, but the overall yield decreased (for instance, a single isomer 6A was isolated in 12% yield after 1 h reflux in benzene). Thus, the bulky α -4,4-dimethyl fragment in oxazinane 5 led to lower yields of target compounds caused by formation of side products.



Scheme 2. Bulky 1,3-aminopropanol in the tandem reaction







Schiff bases 7 readily reacted with maleic anhydride or $CH_2=CH-COCl/NEt_3$ at room temperature in acetone, dichloromethane or benzene. However, instead of crystalline products, only dark viscous poorly soluble in acetone or dichloromethane oils were formed. ¹H NMR spectra of the reaction mixtures revealed complex mixtures of products with no signals of the target 10b-methyloxazino[2,3-*a*]isoindoles. When acryloyl chloride was used, peaks responsible for the presence of the alkenyl fragment ($C_{(q)}=CH_2$) in adducts similar to **11** (see Scheme 4 below) were observed. In this case, the separation of the reaction products was not performed.

After investigation of the reaction of 1,3-aminoalcohols and furfurals towards oxazino[2,3-*a*]isoindoles, we turned to exploration of transformations of 1,2-aminoalcohols, expecting to obtain oxazolo[2,3-*a*]isoindoles similar to **10** (Scheme 4).



Reagents and conditions: (a) CH(OEt)₃/TsOH (cat.); (b) CH₂=CH-COCl/NEt₃/CH₂Cl₂; (c) CHCl=CH-COCl/NEt₃/CH₂Cl₂; (d) PhMe/Δ/1.5–4 h; (e) MeOH/rt/1 d.

Scheme 4. IMDAF reaction of 3-acryloyl-2-(2-furyl)-2-methyl-1,3-oxazolidines (**8**) reported earlier by M. Jung and L. Street^{10d-f} (top in frame) and the results of this work.

A literature review revealed a successful intramolecular [4+2]cvcloaddition of 3-acryloyl-2-(2-furyl)-2-methyl-1,3oxazolidines 9 (Scheme 4) as a step of a newly developed route dihydroxyhexahydrobenzofuran unit in avermectins to (macrocyclic lactones with potent antihelmintic and insecticidal properties).^{10d-10f} 7,9a-Epoxy[1,3]oxazolo[2,3-a]isoindol-5-ones 10 were isolated in these 3-step syntheses in moderate to good yields. Unfortunately, we could not reproduce the $8a \rightarrow 9a \rightarrow 10a$ sequence of steps as shown in Scheme 4. According to the ¹³C NMR data (δ_{C-N} 158.1 ppm), the product of condensation of 5methyl-2-acetylfuran with ethanolamine obtained was the chain azomethine, 2-[1-(5-methyl-2tautomer. furyl)ethylidene]aminoethanol (8Ba), which reacted with acryloyl chloride giving rise to a mixture of products, whose major component was (3-methylene-3a,6-epoxyisoindol-2yl)ethyl acrylate (11). According to ¹H NMR (4.75 and 4.72, two d, ${}^{2}J = 2.3$ Hz, C(3)=CH₂), the reaction mixture contained about 20% of compound 11, which was isolated by column chromatography in 15% yield (relative to the starting azomethine 8Ba).

A plausible mechanism of formation of the adduct **11** is presented in Scheme 4. In the one-step synthesis (1.5 eq of acryloyl chloride/ 2 eq of NEt₃/PhMe/ Δ , 4 h) deprotonation of the methyl group is probably facilitated by NEt₃. A more detailed

study of this interesting transformation is outside the scope of this work.

Perplexed by this outcome, we tried to carry out the condensation/acylation/[4+2]-cycloaddition transformation using the simplest reactants: unsubstituted furfural and 1,2-aminoalcohols – 2-aminoethanol and 2-amino-2-methylpropan-1-ol (Scheme 5). According to the NMR data (CDCl₃, $\delta_{CH=N}$ 151.6, $\delta_{CH=N}$ 8.07 ppm), the condensation product of 2-aminoethanol and furfural was azomethine 2-(2-furylmethylene)aminoethanol (**12a**) with no signals corresponding to the tautomeric 2-furyl-1,3-oxazolidine observed. The reaction of 2-amino-2-methylpropan-1-ol with furfural led (due to the Thorpe–Ingold effect) to a tautomeric mixture **12b**, consisting of 1,3-oxazolidine (**12Ab**) and imine (**12Bb**) in the ratio of 38/62.^{20a} The reaction of compound **12a** or tautomeric mixture **12b** with maleic anhydride gave rise to multicomponent reaction mixtures. The target cycloaddition adducts **13a,b** could not be isolated.

The reaction of aromatic *ortho*-aminoalcohol with constrained "*cis*"-conformation of the functional groups gave no positive results either. The only product of condensation of 2-aminophenol with furfural was aldimine -2-[(2-furylmethylene)amino]phenol (**12c**), which when reacted with maleic anhydride, gave *N*-maleinamide **14** instead of 2,4a-epoxyisoindolo[1,2-*b*][1,3]benzoxazole-1-carboxylic acid (**13c**).



Scheme 5. Attempted synthesis of 7,9a-epoxy[1,3]oxazolo[2,3-a]isoindoles 13

Thus, according to our results, furyl ketones and 1,2aminoalcohols cannot be used in the presented transformation for the synthesis of oxazino[2,3-*a*]isoindoles (**3**, **4**) and oxazolo[2,3*a*]isoindoles (**10**), respectively. Very limited information^{10a-c} was found on the chemical

Very limited information^{10a-c} was found on the chemical transformations of epoxy[1,3]oxazino[2,3-*a*]isoindoles **3**. Some possible directions of modification of the obtained isoindolic acids **3** are presented in Scheme 6. Depending on the reaction conditions, the oxazine ring can be preserved: oxidation of the olefinic fragment (diepoxides **15**) and esterification of the carboxylic group (esters **17**); or aromatization of the 7-oxabicycloheptene fragment can be carried out to obtain isoindolone **16**. We tried to perform halogenation of **3Aa** (NBS in CHCl₃, Br₂/NaOH in H₂O or MeOH; dioxane dibromide or Me₂N⁺HCOMe·Br₃⁻·Me₂NCOMe²⁶ in CHCl₃) but could not isolate any individual products. Probably, in this case, bromination of the double bond was complicated by a skeletal rearrangement (see Scheme 13 below).

Esters **17** are more attractive for further transformations than [1,3]oxazino[2,3-a]isoindole carboxylic acids **3**. The acids are powders with very low solubility in organic solvents (almost insoluble in CHCl₃ and Me₂CO), and high solubility in basic

aqueous solutions, which leads to difficulties for syntheses and low yields of the final products. The esters **17**, on the other hand, are soluble in organic solvents. Moreover, the esters **17a,b** crystallized in well-formed prisms. This allowed using X-ray analysis for determination of their structures, thus, the spatial configuration of the H-10b proton in both the esters **17** and the *major* isomers of the acids **3A** was unambiguously determined. It was shown (see Supporting information) that the proton H-10b in **3A, 15, 17, 18** is *cis*-oriented to the 8,10a-oxo bridge.

Unlike oxazino[2,3-*a*]isoindole carboxylic acids **3**, methyl ester **17a** was successfully transformed to *trans*-dibromide **18** with Me₂N⁺HCOMe·Br₃⁻·Me₂NCOMe.²⁶ This mild bromination agent gave better results than any other we tried (see above). Stereoselectivity of addition of bromine to the 7-oxaheptene fragment of the ester **17a** is governed by the steric factor: Br₃⁻ attacks the least hindered C-9 carbon atom (see Scheme 6) in the intermediate bromonium ion. This direction of bromination was established based on the spin-spin coupling constants of the H-8 – H-10 protons (${}^{3}J_{9,10} = 5.4$, ${}^{3}J_{8,9} = 2.2$ Hz) in the vicinal dibromide **18**. The moderate yields of the products **15**, **16**, **18**, presented in Scheme 6, are compensated by the ready availability

of the starting reagents and simplicity of the reaction and isolation procedures (see Experimental Section).



Reagents and conditions: (a) NaOH (10%), Δ , 2 h; (b) H₂O₂ (30%)/ HCO₂H/ CHCl₃, Δ , 12 h; (c) R²OH/H⁺, Δ , 5–25 h; (d) Me₂N⁺HCOMe·Br₃⁻·Me₂NCOMe/ CHCl₃ / Δ , 12 h; (e) BF₃·OEt₂/ Ac₂O, –5–25 °C, 24 h.

Scheme 6. Some examples of chemical transformations of 8,10a-epoxy[1,3]oxazino[2,3-*a*]isoindole-7-carboxylic acids **3Aa–d** and their methyl esters **17**

Benzoannelated 2-1,3-aminopropanols 2-(hydroxymethyl)aniline (Scheme 7) and (aminomethyl)phenol²⁷ (Scheme 8) can also be used as starting reagents for construction of the isoindole containing heterocycles. The condensation of o-aminobenzyl alcohol with 5- R^{1} -furfurals and follow-up reaction of the formed 2-(2-furyl)-1,3benzoxazines with maleic anhydride was performed in situ (Method A, see Experimental Section) in benzene (Scheme 7). 6b,9-Epoxyisoindolo[2,1-a][3,1]benzoxazine-10-carboxylic acids 20 were isolated as diastereomeric pairs 20A and 20B. Isomers 20A with the cis-oriented oxygen bridge and H-6a proton were more abundant and in these cases, the major components 20Aa and 20Ac were easily isolated by crystallization from an *i*-PrOH/DMF mixture. The presence of a substituent R^1 in the 5position of the furan ring decreased the yields of adducts **20b**,c.



Scheme 7. One-pot synthesis of 6b,9-epoxyisoindolo[2,1*a*][3,1]benzoxazine-10-carboxylic acids **20**

This transformation can also be carried out in two steps with isolation of the intermediate 2-furyl-1,3-benzoxazines (Method B). The yields of the adducts **20** obtained by methods A and B were comparable.

According to its ¹H NMR data, the ratio of tautomeric ring/chain forms in the equilibrium when furfural underwent condensation with *o*-aminobenzyl alcohol was ~ 78/22. The yields and the ratios of the isomers **20Aa** and **20Ba** for

cycloaddition reaction of this mixture with maleic anhydride in acetone or dichloromethane were similar. Esterification of carboxylic acid **20Aa** gave ester **21**, whose spatial structure was established by nOe. In the 1D nOe difference spectrum a low intensity peak, responsible for the spatial interaction of protons H-6a/H-10a (nOe η_{H-6a} {H-10a} < 3 %) was found. The nuclear Overhauser effect has an approximately similar value in the *major* isomer of acid **20Aa**. In the *minor* isomer **20Ba**, nOe is much higher on the same protons: η_{H-6a} {H-10a} 9.0 %. Based on these data and by analogy with esters **17**, *cis*-configuration of the proton H-6a relative to 6b,9-epoxy bridge was attributed to all *major* isomers **20A**.

The nucleophilicity of the nitrogen atom in the initial isomeric aminoalcohols (compare with Schemes 7 and 8) has no influence on the reaction. 2-(Aminomethyl)phenol,²⁷ when reacted with furfural and then anhydrides of α , β -unsaturated acids, underwent a similar transformation (Scheme 8). *Exo*-adducts of [4+2]-cycloaddition, 2,4a-epoxyisoindolo[1,2-*b*][1,3]benzoxazines (**22a**, **23**), were isolated with moderate yields as individual diastereomers. Adduct **22b** was isolated as a pair of isomers with a significant prevalence of the isomer **22Ab**. The relative configuration of the C-4b carbon in this series was established for isoindolo[1,2-*b*][1,3]benzoxazines **22a** and **23**, whose 2D ¹H NOESY NMR spectra showed only a weak cross-peak between the key protons H-4b/H-12a, but rather strong between H-4b/H-4 protons.



b][1,3]benzoxazines 22 and 23

Extension of the distance between the reactive termini of the binucleophile to four carbon atoms leads to an increased influence of the entropic factor and prevents tandem transformation. Condensation of 1,4-aminobutanol with furfural gave rise to azomethine, 4-[(2-furylmethylene)amino]butan-1-ol, with no trace of the tautomeric 2-(2-furyl)-1,3-oxazepane. When this Schiff base was treated with maleic anhydride in various conditions (from Me₂CO, 24 °C to PhMe, Δ , 1 h) a mixture of unidentifiable compounds was formed.

The domino condensation/acylation/cycloaddition reaction between 1,3-aminoalcohols, furfurals and anhydrides of α , β unsaturated carboxylic acids to give epoxylsoindoles **3**, **4**, **6**, **20**-**23** led us to pursue further studies of the scope of this transformation especially with regard to investigating whether sulfur and nitrogen analogues of 1,2- and 1,3-aminoalcohols could be involved in such transformation.

2.2. Epoxyisoindoles fused with thiazoles and thiazines

It is well known that 2-aminoethanethiols²⁸ and 3aminopropane-1-thiols^{6d,29} readily react with aromatic aldehydes at room temperature with no catalyst giving the condensation products in yields close to quantitative. The products exist only in the ring form owing to high nucleophilicity of the sulfur atom.^{6d,28,29} The condensation/acylation/cycloaddition sequence of steps carried out in one pot enabled us to obtain the target

9).

[1,3]thiazolo[2,3-a]isoindoles⁶ **24**, **25** in 48–67% overall yield from readily available 2-aminoethanethiol and furfurals (Scheme



Scheme 9. One-pot synthesis and some transformations of 7,9a-epoxy[1,3]thiazolo[2,3-a]isoindoles 24 and 25

Similarly to previous cases, the last step – the IMDAF reaction with maleic anhydride – is an *exo*-cycloaddition and gave a mixture of diastereoisomers 24A/24B with a prevalence of the diastereomers 24B (except 24b) having a *trans* relative configuration of the C-9b proton and the 7,9a-epoxy bridge. Two fold recrystallization of these isomer mixtures from EtOH or EtOH/DMF allowed to obtain the *major* isomers 24Ba and 24Ab in 30–40 % yield.

The cycloaddition of acryloyl chloride is more diastereoselective and led to isolation of a single isomer of adducts **25**.

Some chemical transformations of the functional groups in isoindoles 24 and 25a were performed. It was shown that transformations with conservation (ester 26) and cleavage (3a,6-epoxyisoindol-3-yl acetate 27) of the thiazole ring can be carried out. Unexpectedly, esterification of the individual diastereomers of acids 24Ba and 24Ab in boiling methanol led to the mixtures of diastereomers 26Aa,b/26Ba,b, probably due to retro-Diels-Alder decomposition of adducts 26B and follow-up recyclization to 26A (or vice versa as shown in Scheme 9). This statement is substantiated by the fact that a long-term reflux in methanol of the individual isomer 26Ba gave rise to the same mixture of 26Aa/26Ba.

It should be noted here that the only analogous compound was reported in a recent work^{9b} which described a reaction of an optically active derivative of lambertianate -2-(thiazolidinyl-2)-furyl-3 (**28**) with acryloyl chloride and maleic anhydride (Scheme 10).



Scheme 10. The cycloaddition of maleic anhydride to methyl 16-(thiazolidin-2-yl)lambertianate by Kharitonov and co-authors.⁹⁶ Only one of the two formed diastereoisomers **29** (in a 1:1 mixture) is presented in the scheme.

The authors^{9b} reported that the cycloaddition adduct 29 was formed in high yield and diastereoselectivity and stated, based on the 2D ¹H NOESY NMR analysis, that the H-9b proton in both of the diastereoisomers had a cis-orientation to 7,9a-epoxy bridge of the isoindole fragment. This conclusion does not agree with our results (Schemes 9, 12). In the 2D ¹H NOESY NMR spectra of the major isomer 24Ba, the major isomer 24Ab, adduct 25a, and ester 26Ba considerable cross-peaks are present. The features are responsible for spatial closeness of protons H-9b/H-5a. This spatial arrangement of the protons can be particularly clearly seen from the 2D ¹H NOESY NMR spectra of the 24Aa/24Ba and 24Ab/24Bb diastereomeric mixtures (the cross-peak between the H-9b/H-5a protons in isomers 24B is significantly more intensive than that in isomers 24A). Thus, the mentioned protons occupy the cis-position in the B series. This result is in agreement with the X-ray data for the rearrangement product 39b (see Scheme 13 below). It should also be noted here, that the isomers of 24-26 can be attributed to B (cis H-9b and H-5a) or A (trans H-9b and H-5a) configurations without using nOe, but based solely on their ¹H NMR spectra. The singlet peak of the H-9b proton for 24–26B is located in the lower field region (both in CDCl₃ and DMSO- $d_6 \delta$ 5.47–5.66 ppm) compared to the analogous peak in **24A**, **26A** (δ 5.16–5.23 ppm).



Scheme 11. One-pot synthesis of 8,10aepoxy[1,3]thiazino[2,3-*a*]isoindole 32 and its 7-carboxy derivatives 31

We would also like to note that a similar trend was observed for homologous compounds **31** and **32** (see Scheme 11), which could nominally be attributed to the **A** series (*trans* H-10b and H-6a), and whose H-10b proton is observed in the δ 5.04–5.26 ppm region.

3-Aminopropanethiol³⁰ reacted sequentially with furfural and anhydrides of α,β -unsaturated acids in a similar one-pot procedure (Scheme 11). No heating or use of dehydration agents were required for the formation of the intermediate 2-(furan-2yl)-1,3-thiazinanes 30. The latter can be introduced into the acylation/cycloaddition reaction with maleic anhydride or acryloyl chloride without isolation. In this case, the target [1,3]thiazino[2,3-a]isoindoles 31 and 32 were formed in moderate yields. According to the ¹H NMR data, the IMDAF reaction is diastereoselective. To our surprise, however, the configuration of H-10b in adducts 31, 32 was found to be the opposite to that of H-9b in their five-membered analogues 24, 25 (Scheme 9). No interaction between protons H-6a/H-10b was observed in the 2D ¹H NOESY NMR spectra of adducts **31a**, **32**, but in the same time cross-peaks H-2(ax)/H-10b(ax) and H-4(ax)/H-10b(ax) are present.

The behavior of 2-aminobenzenethiol in the studied transformations was similar to that of its aliphatic analogues, 2-aminoethanethiols. A one-pot synthesis of isoindolo[1,2-b][1,3]benzothiazoles **33**, **34** is presented in Scheme 12. In the case of 5-bromofurfural, the reaction led to resinification: the adduct **33c** was isolated only in 6 % yield. This may be caused by a competitive nucleophilic substitution of the bromine atom in 5-bromofurfural by sulfur in the first step, followed by polymerization of the intermediate compounds.



Reagents and conditions: (a) MgSO₄/CH₂Cl₂, rt, 3 h; (b) maleic anhydride/Me₂CO, 25 °C, 12 h; (c) CH₂=CH-COCI/NEt₃/PhMe, Δ , 4 h.

Scheme 12. Synthesis of 11-oxo-2,4a-epoxyisoindolo[1,2b][1,3]benzothiazoles 33-35

We were able to detect and isolate the intermediate *N*-acryloyl 2-furyl-1,3-benzothiazole 34^* in the course of the synthesis of isoindolo[1,2-*b*][1,3]benzothiazole 34^* : after 4 h heating under reflux in toluene, a mixture of compounds 34^* (15%) and 34 (20%) was isolated and separated by column chromatography. Longer reflux duration (22 h) allowed increasing the yield of 34 to 28 %.

Esterification of acid **33a** by methanol gave the corresponding ester **35** (Scheme 12). No isomerization similar to **26A** \Rightarrow **26B** (see Scheme 9) was observed for **35**. Similar to the congeneric compounds **24**, **25** (Scheme 9) and based on the 2D ¹H NOESY NMR spectrum of the most soluble tetracyclic compound **35**, we attributed a *trans*-orientation of the H-4b proton relative to the 2,4a-epoxy bridge (interaction H-4b/H-11a was observed) in isoindolo[1,2-b][1,3]benzothiazoles **33–35**.

The singlet peak of the H-4b proton in benzothiazoles 33-35 was observed in a significantly lower field (δ 6.52–6.99 ppm) compared to the analogous **24–26** (Scheme 9) and **31–32** (Scheme 11) compounds.

2.3. Wagner-Meerwein rearrangements of fused diepoxyisoindoles

Only some of the possible routes of modification of epoxy[1,3]oxazino[2,3-*a*]isoindole-7-carboxylic acids **3** showing their synthetic potential are presented in Scheme 6. Here we present another unusual transformation of the studied compounds. 7-Oxabicyclo[2.2.1]heptenes, 3,8-dioxatricyclo[3.2.1.0^{2.4}]octanes and their annelated derivatives are known to undergo sigmatropic rearrangements when treated with electrophilic agents, leading to significant and often unpredictable alterations in their carbon skeleton. ^{10e,18b,18d,31-33} In order to investigate the possibility of the Wagner–Meerwein type³¹ rearrangements, we selected the tetracycles **4a**, **17** and **25a,b** with different positions of the nodal hydrogen atom, H-10b and H-9b, respectively (Scheme 13).



Reagents and conditions: (a) $H_2O_2/HCO_2H/CH_2Cl_2$, rt, 16 h or m-CPBA/CHCl₃, rt, 4–14 h; (b) BF₃·OEt₂/Ac₂O, 0–25 °C, 1–2 h.

Scheme 13. Wagner–Meerwein rearrangement of diepoxides 36 and 38

Starting diepoxides **36**, **38** are readily available by the Prilezhaev reaction³¹ of the oxabicyclo[2.2.1]heptene fragment of isoindolones **4a**, **17a,b**, **25a,b** (both *m*-CPBA and HCO₃H can be used). When sulfur-containing compounds **25a,b** underwent this reaction, oxidation of the S(II) atom to sulfone took place, in addition to the oxidation of the double bond to the oxirane ring.^{34,35}

The cation-assisted skeletal rearrangements of hydrogenated 6,8a;7,8-diepoxyisoquinolines and 3a,6;4,5-isoindoles can occur in the presence of various electrophilic agents (I_2 /AgOAc, NBS/H₂O/DMSO, LA/Ac₂O),³⁵ but likely the best and the most predictable results are achieved when Ac₂O/BF₃·OEt₂ system is utilized. This reagent allowed to obtain the unusual target 4,6-epoxycyclopenta[*c*]pyridines (similar to **37**, **39**) in mild conditions with satisfactory yields and no by-products formation.

A plausible mechanism of catalytic action of $BF_3 \cdot OEt_2$ is presented in Scheme 14 for the transformation of diepoxide **38b** to 7,9-epoxycyclopenta[*d*][1,3]thiazolo[3,2-*a*]pyridine **39b**. Interestingly, the 1,3-thiazolidine and 1,3-thiazinane fragments were preserved in the presented reaction conditions.



Scheme 14. Plausible mechanism of the Wagner–Meerwein rearrangement of the diepoxyisoindole 38b

The NMR spectra of diacetates 37, 39 are rather complicated. In particular, it was difficult to establish the configurations of C-10a (for 37a,b) and C-9a (for 39a,b). X-ray analysis was performed for 37a and 39b in order to determine the spatial structure of the rearrangement products. In addition to the structures of compounds 37, 39, the X-ray data allowed us to unequivocally establish the structures of diepoxides 36, 38, and the starting isoindolones 4, 17, 25. A detailed X-ray description of structures 17a, 37a and 39b is given in the Supporting Information, here we only point out that the single crystals of 37a and **39b** are racemic, while the crystal of **17a** is chiral. Evidently, this substance crystallizes as a conglomerate and is capable of spontaneous enantiomeric separation by the Pasteur method. Nevertheless, it is impossible to determine unambiguously the absolute configuration of the asymmetric centers in the single crystal of 17a due to the absence of heavy atoms with Z > 14(Si) in its structure.

2.4. Epoxyisoindoles fused with imidazoles and pyrimidines

In this part of our work we investigated the possibility of introducing 1,2- and 1,3-*N*,*N*-binucleophiles into the reaction and extension of the described protocol to the synthesis of isoindolones, condensed with imidazolidine and hexahydropyrimidine rings (Figure 4).



Figure 4. Extension of the method to the synthesis of isoindoles annulated with azaheterocycles.

It is well known, that 1,3-*N*,*N*-binucleophiles with a constrained "*cis*"-conformation of the amino groups readily (and often in quantitative yields) undergo condensation with aromatic aldehydes. Among such nucleophiles (2-aminobenzyl)amine and 1,8-diaminonaphthalene are the most readily available and we selected them for further study.

It was shown earlier,³⁶ that when dissolved in ethanol at room temperature, 1,8-diaminonaphthalene and furfural (or 5-methylfurfural) lead to 2-(2-furyl)-2,3-dihydro-1*H*-perimidines (**40**). The target products **40** exist entirely in the ring form.³⁶ After solvent replacement, the standard acylation/cycloaddition

procedure was performed on the intermediate dihydro-2furylperimidines and adducts **41**, **42** were obtained in moderateto-high yields as mixtures of isomers **A** and **B** (Scheme 15). When toluene was used as the reaction solvent in the last step (10 min reflux) the yield of the mixture of products **41a** increased to 95%. However, the diastereoselectivity was lower in this case – the **41Aa/41Ba** ratio decreased from 89/11 to 54/46.



Scheme 15. Synthesis of 7b,10-epoxyisoindolo[2,1*a*]perimidinones 41 and 42

Spatial structures of the diastereoisomers **41A**, **41B** and **42A**, **42B** can easily be established by the 2D ¹H NOESY NMR spectra of their mixtures. The key nOe signals are interaction between protons H-7a/H-11a. In *minor* isomers **B** intensive cross-peaks were observed, whereas, in *major* isomers **A** these cross-peaks were hardly observable. *Major* isomers **41Aa** and **42B** were isolated as individual compounds by fractional recrystallization from *i*-PrOH/DMF mixture or by column chromatography, respectively.



Scheme 16. Synthesis of 2,4a-epoxyisoindolo[1,2b]quinazolines 44–46

A similar approach towards isoindolo[1,2-*b*]quinazolinones (44–46) is presented in Scheme 16. The intermediate tetrahydroquinazolines 43 also exist only in the ring form and undergo chemoselective acylation by anhydrides of α , β -unsaturated acids predominantly at the more nucleophilic nitrogen atom (N-3). The yields and ratio of the isomers 44Aa/44Ba depend strongly on the temperature. This was shown for reaction of quinazoline 43a (R¹=H) with maleic anhydride: in dichloromethane or acetone at 25 °C the isomers 44Aa/44Ba were formed in the 82/12 ratio (overall yields were 78 and 48%, respectively), whereas, when refluxed in toluene (10 min) a mixture with the 44Aa/44Ba ratio of ~50/50 (33 %) was formed.



Scheme 17. Evidence of epoxyisoindolo[1,2-*b*]quinazoline structure (**46**)

In the case of acryloyl chloride, a side product of double acylation **46** was isolated in an insignificant yield. It was established from its NMR data that the N-3 acetyl fragment was involved in the Diels-Alder reaction (Scheme 17). When diamine **43a** was acylated, dialkyl derivative **47** was probably formed, which could theoretically exist in equilibrium with adducts **46** and **46***. The 2D ¹H-¹³C HMBC NMR spectrum of isoindolo[1,2-*b*]quinazoline **46** displayed signals due to spin interaction H-10/C-12, which is impossible in the alternative product **46***. The structures of compounds **44** were established in a similar way. When isoindoloquinazolinone **45B** was treated with acryloyl chloride a product identical to amide **46** was formed.

Isomers of compounds **41**, **42**, **44**, **45** can also be attributed to either **A** or **B** series based only on the ¹H NMR shift of the nodal

proton H-7a (H-4b) in their spectra: δ 5.01–4.24 ppm for **A** and δ 5.28–5.64 ppm for **B** series.

Aliphatic diamines undergo more complicated transformations. The presented condensation/acylation/IMDAF approach was tested for reaction of propanediamine, furfural and maleic anhydride. A multicomponent reaction mixture was obtained instead of the expected amino acid **51**. Therefore we performed a detailed study of the first step (condensation) products.



Scheme 18. Attempted synthesis of epoxypyrimido[2,1*a*]isoindole 51

We found out that condensation of propane-1,3-diamine with furfural leads to an equilibrium mixture of mainly three products (**48–50**). NMR of the mixture in CDCl₃ unequivocally established their structures and relative abundances. These results are in a good agreement with earlier work.³⁷ So far, we could not find the suitable conditions (solvents and temperature range we have tried are CH₂Cl₂, Et₂O, PhMe and -70 °C - 110 °C) for a successful acylation of this mixture neither with maleic anhydride (Scheme 18) nor with acryloyl chloride. Viscous, dark brown multicomponent mixtures were formed in each case, which could not be separated into the components. Our preliminary attempts to perform the reaction with 1,4diaminobutylene, ethane- and benzene-1,2-diamines were not successful either.

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	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$										
Compound	\mathbb{R}^1	\mathbb{R}^2	R ³	\mathbb{R}^4	R ⁵	Х	n	A/B	Yield, % ^a		
3a	Н	CO ₂ H	H,H	H,H	H,H	0	1	94/6	60		
3b	Me	CO ₂ H	H,H	H,H	H,H	0	1	95/5	58		
3c	Br	CO ₂ H	H,H	H,H	H,H	0	1	83/17	42		
3d	Ι	$\rm CO_2 H$	H,H	H,H	H,H	0	1	80/20	55		
3e	NO_2	$\rm CO_2 H$	H,H	H,H	H,H	0	1	89/11	35		
3f	Ph	$\rm CO_2 H$	H,H	H,H	H,H	0	1	100/0	16		
3g	m-C ₆ H ₄ NO ₂	$\rm CO_2 H$	H,H	H,H	H,H	0	1	91/9	46		
3h	o-C ₆ H ₄ NO ₂	$\rm CO_2 H$	H,H	H,H	H,H	0	1	92/8	39		
4a	Н	Н	H,H	H,H	H,H	0	1	100/0	18		

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4b	Me	Н	H,H	H,H	H,H	0	1	100/0	19
4c	Br	Н	H,H	H,H	H,H	0	1	64/36	21
13a	Н	$\rm CO_2 H$	H,H	H,H	-	0	0	-	0
13b	Н	$\rm CO_2 H$	Me,Me	H,H	-	0	0	-	0
13c	Н	CO_2H	CH-CH=	CH-CH	-	0	0	-	0
20a	Н	CO_2H	CH-CH=CH-CH H,H		H,H	0	1	96/4	27
20b	Me	$\rm CO_2 H$	CH-CH=	CH-CH	H,H	0	1	57/43	23
20c	Br	$\rm CO_2 H$	CH-CH=	CH-CH	H,H	0	1	93/7	17
22a	Н	$\rm CO_2 H$	H,H	Н,Н СН-СН=СН-СН		0	1	100/0	34
22b	Me	$\rm CO_2 H$	H,H	CH-CH	=CH-CH	0	1	86/14	35
23	Н	Н	H,H	CH-CH	=CH-CH	0	1	100/0	25
24a	Н	$\rm CO_2 H$	H,H	H,H	-	S	0	15/85	49
24b	Me	$\rm CO_2 H$	H,H	H,H	-	S	0	67/33	48
25a	Н	Н	H,H	H,H	-	S	0	0/100	67
25b	Me	Н	H,H	H,H	-	S	0	0/100	57
31a	Н	$\rm CO_2 H$	H,H	H,H	H,H	S	1	100/0	48
31b	Me	$\rm CO_2 H$	H,H	H,H	H,H	S	1	100/0	46
32	Н	Н	H,H	H,H	H,H	S	1	100/0	26
33a	Н	$\rm CO_2 H$	CH-CH=CH-CH -		S	0	0/100	60	
33b	Me	$\rm CO_2 H$	CH-CH=CH-CH -		S	0	0/100	33	
33c	Br	$\rm CO_2 H$	- СН-СН=СН-СН		-	S	0	0/100	6
34	Н	Н	CH-CH=CH-CH -			S	0	0/100	28
41a	Н	$\rm CO_2 H$	CH-CH=CH-C-CH=CH-CH			NH	1	89/11	65
41b	Me	$\rm CO_2 H$	CH-CH=CH-C-CH=CH-CH			NH	1	54/46	94
42	Н	Н	CH-CH=CH-C-CH=CH-CH			NH	1	36/64	36
44a	Н	CO_2H	СН-СН=СН-СН Н,Н		NH	1	82/18	78	
44b	Me	$\rm CO_2 H$	CH-CH=	CH-CH	H,H	NH	1	100/0	39
44c	Br	$\rm CO_2 H$	CH-CH=	СН-СН=СН-СН Н,		NH	1	76/24	26
45	Н	Н	CH-CH=	СН-СН=СН-СН		NH	1	56/44	32
51	Н	$\rm CO_2 H$	H,H	Н,Н	H,H	NH	1	-	0
b	Н	$\rm CO_2 H$	Н,Н	H,H	H,H	NH	2	-	0
с	Н	$\rm CO_2 H$	CH-CH=	сн-сн=сн-сн -			0	-	0
d	Н	CO_2H	CH-CH=CH-C-CH=CH-CH			S	1	-	0
e	Н	CO ₂ H	СН-СН=СН-С-СН=СН-СН			0	1	-	0
					-	2	•		Ŭ

^aYields are given relative to the starting furfurals after two steps;

^bThe initial binucleophile is 1,4-diaminobutane;

^cThe initial binucleophile is 1,2-phenylenediamine;

^dThe initial binucleophile is 8-aminonaphthalene-1-thiol;

^eThe initial binucleophile is 8-aminonaphthalen-1-ol.

2.5. Attempted synthesis of naphtolo[1,3]oxazino- and naphtolo[1,3]thiazino[2,3-a]isoindoles

In order to show the synthetic potential of the method, in this work we used only two compounds as dienophiles: readily available maleic anhydride and acryloyl chloride. Given that in our previous works^{18a-d,31,38} we showed that a large range of dienophiles can be used in tandem acylation/IMDAF reaction (allyl halogenides, dihalogenomaleic, and citraconic anhydrides, as well as acryloyl, methacryloyl, crotonyl and cinnamoyl

chlorides and all were used as dienophiles), we believe that the two dienes are good representative reactants, demonstrating the advantages and limitations of the method. Similarly, we present only one example of aromatization of epoxyisoindole **3Aa** to isoindole **16** (Scheme 6), other examples of aromatization of 3,6a-isoindolones can be found in a recent publication.^{38e} It should be pointed out that aromatization of the 7-oxabicycloheptene fragment leads to elimination of all but one asymmetric centers in the molecules, thus, transforming

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diastereomeric mixtures of adducts **A/B** to single aromatization products (16).

As stated above, not every system we tried worked out successfully (for example, furyl ketones do not provide oxazinoor oxazoloisoindoles in these conditions), and we should mention here another unsuccessful attempt of the fused epoxyisoindole synthesis. For generality, we tried to involve two rather complex aminonaphthalenes in the tandem reaction (Scheme 19).



Scheme 19. Attempts to involve 8-aminonaphthalene-1-thiol and 8-aminonaphthalen-1-ol in the IMDAF reaction

8-Aminonaphthalene-1-thiol³⁹ and 8-aminonaphthalen-1-ol⁴⁰ react fairly readily with furfural at room temperature (according to TLC, the initial compounds disappear completely and a new spot appears in 1 h). But probably due to instability of the solutions of both 8-aminonaphthalenes and compounds **52**, we could not isolate them (the mixture components were turned dark brown (for X = O) or violet (X = S) in an attempt of concentration). The one-pot approach - without isolation of the condensation products **52** - failed as well: no solid products were observed in the reaction of **52** with maleic anhydride at 24 °C in acetone. But nevertheless, we believe that the proposed methodology may be extended to a wide range of *N*,*X*-binucleophiles. For instance, our preliminary attempts to utilize it for the reaction anthranilamide and methyl cysteine were successful and will be presented in a follow-up publication.

3. Conclusions

In summary, in this work we propose a general strategy for construction of epoxyisoindoles annelated with saturated 5- and 6-membered heterocycles. We have tested our approach on many available N,X-binucleophiles and showed that 1,3-aminoalcohols, 1,2- and 1,3-aminothiols, their benzoannelated analogues and some 1,3-diamines react with furfural and α , β -unsaturated acid anhydrides in a one-pot tandem reaction sequence leading to isoindolones condensed with oxazolidine, oxazinane, thiazolidine, thiazinane, pyrimidine and perimidine fragments, respectively. Some attractive transformations of the epoxyisoindoles were also demonstrated, including a one-step approach to condensed isoindolones and skeletal rearrangements of the oxabicyclo[2.2.1]heptenes fragment.

The proposed approach compares favorably with literature methods owing to the availability of starting reagents and simplicity of the experiment, allowing to obtain attractive polyfunctional building blocks with isoindole scaffolds, containing a large set of functional groups: carboxyl, amide, *N*, *O*-, *N*, *S*-acetal fragments, multiple bonds and epoxy bridges.

4. Experimental Section

5. Experimental Section

All commercially available reagents and solvents were used without further purification. Melting points were determined with

SMP30 apparatus within 0.5 °C accuracy and are uncorrected. IR spectra were obtained in KBr pellets for solids or in thin films for oils using an IR-Fourier spectrometer. NMR spectra were recorded on NMR spectrometers with working frequency 400 or 600 MHz for ¹H and 100.6 or 150.9 MHz for ¹³C, for 2-5% solutions in CDCl₃, DMSO-*d*₆, D₂O (for compounds **3b–d**, **15b**) or C₆D₆ (for **37a**) at ~ 27 °C. Traces of chloroform (¹H NMR δ 7.26 ppm and ¹³C NMR δ 77.16 ppm) or DMSO- d_5 H (¹H NMR δ 2.49 ppm and ¹³C NMR δ 39.43 ppm) were used as the internal standards. Assignments of ¹H and ¹³C signals were made with the aid of 2D COSY, TOCSY, NOESY, HSQC and HMBC NMR spectra where necessary. Mass spectra were taken either on a chromatography-mass (electron ionization, 70 eV, ion source temperature 200 °C, gas chromatographic inlet with an 5ms column) or a mass (electron ionization, 70 eV, ion source temperature 200 °C, direct inlet probe) spectrometer. Highresolution mass spectra (HRMS) were taken on a massspectrometer using direct analysis in real time (DART) ionization method (helium was used as DART gas, gas flow rate was 1 L/min, flow T 300 °C, discharge electrode was set to +4000 V, the mass scale was calibrated using PEG 600) or using an MALDI-TOF mass spectrometer, operated in positive reflectron mode (2,5-dihydroxybenzoic acid was used as matrix). These methods were used when no molecular ions were observed in the electron ionization mass spectra. The purity of the substances obtained and the composition of the reaction mixtures were monitored by TLC (SiO₂ plates) or, when possible, by GCMS. The purification of the final products was carried out by column chromatography on Al₂O₃ (activated, neutral, 50-200 mesh) or SiO₂ (40–100 mesh) or by fractional crystallization. Microanalyses were performed for C, H, N on a C,H,N,O,Sanalyzer and were within $\pm 0.4\%$ of theoretical values. The detailed X-ray description of structures 17a, 37a, 39a is given in the Supporting Information.

Multiscale synthesis of starting 5-arylfurfurals

5-Arylfurfurals (**f-h**) required for the synthesis of **1** (**2**) were obtained by the following procedures. Literature methods^{41,42} turned out to be either difficult to scale to multi-gram quantities^{41b,41d,42a-f} or hardly reproducible.^{41c,41g,42c,42i}

5-(2-Nitrophenyl)furan-2-carbaldehyde (**h**) and 5-(3nitrophenyl)furan-2-carbaldehyde (g). General experimental procedure. A mixture of nitroaniline (28.0 g, 0.20 mol), water (80 mL) and concentrated hydrochloric acid (45 mL) was cooled (-5 °C) and a solution of NaNO₂ (14.8 g, 0.20 mol) in water (50 mL) was added dropwise to the stirred mixture keeping the temperature below +5 °C. After the addition of NaNO₂ was complete, a solution of furfural (24.8 mL, 0.30 mol) in Me₂CO (100 mL) was quickly added to the mixture. Powdered Cu₂Cl₂ was added in small portions at 20-30 °C until nitrogen evolution had ceased (in general it took 15-25 min for the reaction to complete and required 3-5 g of the catalyst). The mixture was stirred at room temperature for 10 min and cold water (400 mL) was then added. The mixture was kept overnight at +4 °C and on the next day the greenish water layer was decanted from the semi-crystalline precipitate. The precipitate was then washed with water (2 \times 200 mL) and suspended in Et₂O (100 mL). The crystals formed were separated by filtration, washed with water $(2 \times 100 \text{ mL})$ and Et₂O $(2 \times 30 \text{ mL})$. The crude products were further purified if necessary by recrystallization from i-PrOH/DMF mixtures affording 5.2-15.5 g of 5-arylfuran-2carbaldehydes h, g as yellow crystals (12-35 % yield), m.p. 95–96 °C (**h**), 155–156 °C (**g**).

5-Phenylfuran-2-carbaldehyde (f). A solution of aniline (45.5 mL, 0.50 mol) in a mixture of concentrated HCl (100 mL) and water (100 mL) was diazotized with a solution of NaNO₂ (34.5 g, 0.50 mol) in water (100 mL) at 0 °C. A solution of furfural (41.4 mL, 0.50 mol) in Me₂CO (150 mL) was then added. The reaction mixture was brought to 20 °C and then a mixture consisting of 10 mol % CuCl₂·H₂O (7.60 g, 0.05 mol) and 10 mol % Cu₂Cl₂ (9.90 g, 0.05 mol) was added in small portions with intensive stirring during an ~ 1 h period and keeping the temperature within 25–35 °C (in general, no cooling was required). The mixture was stirred for another ~ 2 h until nitrogen evolution had ceased. Na₂CO₃ was added until the solution turned basic and the mixture was extracted with diethyl ether (1 \times 100 and 1 \times 180 mL). The combined organic layers were washed with a saturated NaHCO₃ solution (2 \times 150 mL), dried (MgSO₄), filtered and the solvent was removed in vacuo. The brownish liquid residue was distilled in vacuo and dark-yellow oil (b.p. 120-160/7 mm Hg, 20-25 g) was initially collected, then redistilled yielding 5-phenylfuran-2carbaldehyde (f) (14.2-18.8 g, 16-21 % yield) as a yellow liquid (b.p. 147–153 °C/ 7 mm Hg or 130–134 °C/ 1 mm Hg).

Physical and spectroscopic data for 5-arylfur furals $f\!-\!h$ match the literature data. 41,42

General procedure for preparation of tautomeric mixtures of imines (1a–h) and 2-furyl-1,3-oxazinanes (2a–h) and their adducts with maleic anhydride (3a–h).

Anhydrous powdered MgSO₄ (12.1 g, 0.10 mol) was added to a solution of 3-amino-1-propanol (7.80 mL, 0.10 mol) and corresponding furaldehyde (0.10 mol) in CH₂Cl₂ (70 mL). The mixture was stirred at room temperature for 4–24 h (in case of 5-nitrofurfural the mixture was stirred at 0 °C for 10 min). MgSO₄ was filtered off and washed with CH₂Cl₂ (40 mL). The organic phases were combined and used in the next step or concentrated under reduced pressure to give a mixture of tautomers **1a–h/2a–h** as yellow or reddish yellow oils. The tautomers **1a–h/2a–h** (or their solutions in CH₂Cl₂) were used for the next step without further purification, assuming quantitative yield. Tautomeric equilibrium of the condensation products **1a** (**2a**) and **1f** (**2f**) was investigated by NMR.

Mixture of tautomers **1a/2a** in ratio ~ 77/23, yellow viscous oil; v_{max} (liquid film) 3390, 2939, 2857, 1647 cm⁻¹; GC-MS (EI, 70 eV) *m*/z 153 (1, M⁺), 152 (2), 124 (2), 123 (11), 109 (100), 108 (52), 96 (12), 95 (32), 94 (39), 93 (6), 81 (62), 80 (30), 67 (10), 53 (20), 51 (14), 41 (14), 39 (36%).

3-[(2-Furylmethylene)amino]propan-1-ol (**1a**). $\delta_{\rm H}$ (400 MHz, CDCl₃) 8.07 (1 H, s, CH=N), 7.49 (1 H, dd, ${}^{3}J_{5',4'}$ 1.8, ${}^{4}J_{5',3'}$ 0.8 Hz, H-5'), 6.72 (1 H, br.d, ${}^{3}J_{4',3'}$ 3.6 Hz, H-3'), 6.46 (1 H, dd, ${}^{3}J_{4',3'}$ 3.6, ${}^{3}J_{4',5'}$ 1.8 Hz, H-4'), 3.81 (2 H, t, ${}^{3}J_{1,2}$ 5.8 Hz, H-1), 3.72 (2 H, dt, ${}^{3}J_{2,3}$ 5.8, *J* 1.2 Hz, H-3), 1.91–1.95 (2 H, m, H-2); $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 151.5 (C-2'), 150.0 (C=N), 144.9 (C-5'), 114.0 and 111.7 (C-3' and C-4'), 62.2 (C-1), 59.8 (C-3), 33.6 (C-2).

2-(Furan-2-yl)-1,3-oxazinane (2a). $\delta_{\rm H}$ (400 MHz, CDCl₃) 7.35 (1 H, dd, ${}^{3}J_{5',4'}$ 1.8, ${}^{4}J_{5',5'}$ 0.8 Hz, H-5'), 6.33–6.30 (2 H, m, H-3' and H-4'), 5.21 (1 H, s, H-2), 4.18–4.22 (1 H, m, H-6^{eq}), 3.92 (1 H, dt, ${}^{2}J_{6,6} \sim {}^{3}J_{6a,5a}$ 11.9, ${}^{3}J_{5e,6a}$ 2.3 Hz, H-6^{ax}), 3.22–3.25 (1 H, m, H-4^{eq}), 3.06 (1 H, ddd, ${}^{2}J_{4,4}$ 15.6, ${}^{3}J_{4a,5a}$ 12.4, ${}^{3}J_{4a,5a}$ 3.7 Hz, H-4^{ax}), 1.85–1.88 (1 H, m, H-5^{ax}), 1.41–1.45 (1 H, m, H-5^{eq}); $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 148.1 (C-2'), 142.2 (C-5'), 112.7 and 110.2 (C-3' and C-4'), 83.8 (C-2), 67.9 (C-6), 44.1 (C-4), 27.1 (C-5).

Mixture of tautomers 1f/2f in ratio ~ 86/14, yellow viscous oil.

3-{[(5-Phenyl-2-furyl)methylene]amino}propan-1-ol (1f). $\delta_{\rm H}$ (400 MHz, CDCl₃) 8.10 (1 H, s, CH=N), 7.75 (2 H, dd, ³J 7.8, ⁴J 1.3 Hz, H-Ph), 7.39 (2 H, dt, ³J 7.8, ⁴J 1.3 Hz, H-Ph), 7.30 (1 H,

br.t, ${}^{3}J$ 7.8 Hz, H-Ph), 6.85 (1 H, d, ${}^{3}J_{4',3'}$ 3.5 Hz, H-4'), 6.73 (1 H, d, ${}^{3}J_{3',4'}$ 3.5 Hz, H-3'), 3.90 (2 H, t, ${}^{3}J_{1,2}$ 5.5 Hz, H-1), 3.80 (2 H, br.t, ${}^{3}J_{2,3}$ 5.5 Hz, H-3), 1.97 (2 H, p, ${}^{3}J_{2,3} \sim {}^{3}J_{1,2}$ 5.5 Hz, H-2); $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 156.3 (C-5'), 150.7 (C-2'), 150.1 (C=N), 128.6 (C-1''), 128.8 (C-3'' and C-5''), 128.5 (C-4''), 124.6 (C-2'' and C-6''), 116.4 (C-3'), 107.1 (C-4'), 61.9 (C-1), 59.8 (C-3), 33.5 (C-2).

2-(5-Phenyl-2-furyl)-1,3-oxazinane (**2f**). $\delta_{\rm H}$ (400 MHz, CDCl₃) 7.67 (2 H, dd, ${}^{3}J$ 7.7, ${}^{4}J$ 1.2 Hz, H-Ph), 7.39–7.24 (3 H, m, H-Ph), 6.60 (1 H, d, ${}^{3}J_{4',3'}$ 3.5 Hz, H-4'), 6.42 (1 H, d, ${}^{3}J_{3',4'}$ 3.5 Hz, H-3'), 5.28 (1 H, s, H-2), 4.23–4.26 (1 H, m, H-6^{eq}), 3.96 (1 H, dt, ${}^{2}J_{6,6} \sim {}^{3}J_{6a,5a}$ 11.9, ${}^{3}J_{5e,6a}$ 2.3 Hz, H-6^{ax}), 3.29–3.32 (1 H, m, H-4^{eq}), 3.11 (1 H, br.dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,5a}$ 12.4, ${}^{3}J_{4a,5a}$ 3.2 Hz, H-4^{ax}), 1.75–1.79 (1 H, m, H-5^{ax}), 1.45–1.48 (1 H, m, H-5^{eq}); $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 149.5 (C-2'), 144.7 (C-5'), 129.5 and 129.9 (C-1'' and C-4''), 127.5 (C-3'' and C-5''), 124.0 (C-2'' and C-6''), 108.7 (C-3'), 105.6 (C-4'), 83.8 (C-2), 67.9 (C-6), 44.1 (C-4), 27.0 (C-5).

Maleic anhydride (9.80 g, 0.10 mol) was added to a solution of the tautomeric mixture 1 ± 2 (0.10 mol) in Me₂CO (100 mL) or CH₂Cl₂ (~ 100 mL). The mixture was stirred at 24 °C for 4–46 h. The precipitate formed was collected by filtration, washed with Me₂CO (2 × 30 mL) and Et₂O (2 × 30 mL) and dried in air until constant weight to give a diastereomeric pair of the title acids **3a–e,g,h** as white or slightly colored powders. Compound **3f** does not crystallize from the reaction mixture and thus was isolated by column chromatography on SiO₂. The yields were in general 5–7 % higher when the reactions were carried out in CH₂Cl₂. However, the products crystallized from Me₂CO were less tinted and did not require any purification after filtration and washing with Me₂CO. The yields of the mixtures of diastereomeric adducts **3** given below are for reactions in Me₂CO.

(6aRS,7SR,8RS,10aSR,10bRS)-6-Oxo-3,4,6,6a,7,8-hexahydro-2H-8,10a-epoxy[1,3]oxazino[2,3-*a*]isoindole-7-carboxylic acid (3Aa) and (6aRS,7SR,8RS,10aSR,10bSR)-6-oxo-3,4,6,6a,7,8hexahydro-2H-8,10a-epoxy[1,3]oxazino[2,3-*a*]isoindole-7-

carboxylic acid (3Ba). Ratio of isomers 3Aa/3Ba ~ 94/6, white powder (15.06 g, 60 %), m.p. 181-182 °C; [Found: C, 57.26; H, 5.34; N, 5.63. C₁₂H₁₃NO₅ requires C, 57.33; H, 5.22; N, 5.58%]; R_f (50% EtOH/DMF) 0.45; v_{max} (KBr) 3109, 3006, 2971, 1738, 1664, 1165, 1056, 917 cm⁻¹; $\delta_{\rm H}$ (400 MHz, DMSO- d_6) (3Aa) 12.22 (1 H, br.s, CO₂H), 6.60 (1 H, d, ${}^{3}J_{10,9}$ 5.8 Hz, H-10), 6.46 (1 H, dd, ${}^{3}J_{9,10}$ 5.8, ${}^{3}J_{9,8}$ 1.7 Hz, H-9), 5.12 (1 H, s, H-10b), 5.08 (1 H, d, ${}^{3}J_{8,9}$ 1.7 Hz, H-8), 4.14–4.10 (1 H, m, ${}^{2}J_{2,2}$ 11.6, ${}^{3}J_{2e,3a}$ 4.6, ${}^{3}J_{2e,3e} \sim {}^{4}J_{2e,4e}$ 1.6 Hz, H-2^{eq}), 3.87–3.91 (1 H, m, H-4^{eq}), 3.86–3.91 (1 H, m, H-2^{ax}), 3.09 (1 H, ddd, ${}^{2}J_{4,4}$ 13.2, ${}^{3}J_{4a,3a}$ 12.3, ${}^{3}J_{4a,3e}$ 3.7 Hz, H-4^{ax}), 2.73 (1 H, dd, ${}^{3}J_{6a,7}$ 9.2, ${}^{5}J_{6a,4}$ 0.5 Hz, H-6a), 2.52 (1 H, d, ${}^{3}J_{7,6a}$ 9.2 Hz, H-7), 1.68–1.57 (1 H, m, H-3^{ax}), 1.53-1.47 (1 H, m, H-3^{eq}); (**3Ba**) 12.22 (1 H, br.s, CO₂H), 6.60 (1 H, d, ${}^{3}J_{10,9}$ 5.7 Hz, H-10), 6.44 (1 H, dd, ${}^{3}J_{9,10}$ 5.7, ${}^{3}J_{9,8}$ 1.7 Hz, H-9), 5.40 (1 H, s, H-10b), 5.01 (1 H, d, ${}^{3}J_{8,9}$ 1.7 Hz, H-8), 4.09-4.04 (1 H, m, H-2^{eq}), 3.88-3.84 (1 H, m, H-4^{eq}), 3.67 (1 H, dt, ${}^{2}J_{2,2} \sim {}^{3}J_{2a,3a}$ 11.6, ${}^{3}J_{2a,3e}$ 3.0 Hz, H-2^{ax}), 3.01–2.93 (1 H, m, H- 4^{ax}), 2.76 (1 H, dd, ${}^{3}J_{6a,7}$ 9.2, ${}^{5}J_{6a,4a}$ 1.4 Hz, H-6a), 2.45 (1 H, d, ${}^{3}J_{7,6a}$ 9.2 Hz, H-7), 1.61–1.57 (2 H, m, H-3^{ax} and H-3^{eq}); δ_{C} (100.6 MHz, DMSO-d₆) (3Aa) 172.6 (s, CO₂H), 170.7 (s, C-6), 136.8 (d, J 177.5 Hz, C-9), 133.8 (d, J 179.5 Hz, C-10), 89.5 (s, C-10a), 85.1 (d, J 165.0 Hz, C-10b), 82.3 (d, J 169.0 Hz, C-8), 66.8 (t, J 144.0 Hz, C-2), 49.3 (d, J 137.5 Hz, C-6a), 44.0 (d, J 139.0 Hz, C-7), 38.3 (t, J 139.0 Hz, C-4), 25.1 (t, J 129.5 Hz, C-3); (**3Ba**) 172.9 (s, CO₂H), 167.9 (s, C-6), 137.2 (d, J 179.0 Hz, C-9), 134.6 (d, J 179.0 Hz, C-10), 89.5 (s, C-10a), 82.8 (d, J 160.0 Hz, C-

10b), 81.5 (d, *J* 169.0 Hz, C-8), 66.2 (t, *J* 144.5 Hz, C-2), 50.2 (d, *J* 138.5 Hz, C-6a), 44.4 (d, *J* 138.5 Hz, C-7), 37.4 (t, *J* 139.0 Hz, C-4), 23.4 (t, *J* 130.0 Hz, C-3); MS (EI, 70 eV) m/z 251 (2, M⁺), 207 (22), 206 (20), 178 (13), 153 (10), 152 (100), 150 (11), 138 (18), 124 (26), 122 (18), 121 (27), 110 (11), 99 (20), 96 (19), 95 (57), 94 (18), 86 (69), 85 (13), 65 (15), 56 (18), 41 (12), 39 (19), 28 (12), 27 (11%).

Similar procedure carried out in CH_2Cl_2 (150 mL) leads to a diastereomeric mixture **3Aa/3Ba** in ratio of ~ 95/5 (44 % yield). When PhMe was used (150 mL, 10 min reflux), only the *major* isomer **3Aa** (m.p. 182 °C from an *i*-PrOH/DMF mixture) was obtained in 27 % yield.

(6aRS,7SR,8RS,10aSR,10bRS)-8-Methyl-6-oxo-3,4,6,6a,7,8-

hexahydro-2*H*-8,10a-epoxy[1,3]oxazino[2,3-*a*]isoindole-7carboxylic acid (3Ab) and (6*aRS*,7*SR*,8*RS*,10*aSR*,10*bSR*)-8methyl-6-oxo-3,4,6,6a,7,8-hexahydro-2*H*-8,10a-

epoxy[1,3]oxazino[2,3-a]isoindole-7-carboxylic acid (3Bb). Ratio of isomers 3Ab/3Bb ~ 95/5, pale-yellow powder (15.3 g, 58 %), m.p. 148-155 °C (decomp.); [Found: C, 58.94; H, 5.77; N, 5.17. C₁₃H₁₅NO₅ requires C, 58.86; H, 5.70; N, 5.28%]; R_f (50% EtOH/DMF) 0.90; ν_{max} (KBr) 3083, 2985, 1741, 1661, 1450, 1170, 1075, 872 cm⁻¹; $\delta_{\rm H}$ (400 MHz, DMSO- d_6) (**3Ab**) 12.22 (1 H, br.s, CO₂H), 6.62 (1 H, br.d, ³J_{9,10} 5.7 Hz, H-10), 6.30 $(1 \text{ H}, d, {}^{3}J_{10,9} 5.7 \text{ Hz}, \text{H-9}), 5.11 (1 \text{ H}, \text{s}, \text{H-10b}), 4.11 (1 \text{ H}, \text{br.dd})$ ${}^{2}J_{2,2}$ 12.1, ${}^{3}J_{2e,3e}$ 4.0 Hz, H-2^{eq}), 3.92 (1 H, br.dd, ${}^{2}J_{4,4}$ 13.0, ${}^{3}J_{4e,3e}$ 5.0 Hz, H-4^{eq}), 3.88 (1 H, dt, ${}^{2}J_{2,2} \sim {}^{3}J_{2a,3a}$ 12.1, ${}^{3}J_{2a,3e}$ 2.0 Hz, H-2^{ax}), 3.08 (1 H, br.dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 13.0, ${}^{3}J_{4a,3e}$ 4.1 Hz, H-4^{ax}), 2.76 (1 H, d, ${}^{3}J_{7,6a}$ 8.9 Hz, H-7), 2.55 (1 H, d, ${}^{3}J_{6a,7}$ 8.9 Hz, H-6a), 1.68-1.55 (1 H, m, H-3^{ax}), 1.51 (3 H, s, Me-8), 1.50-1.45 (1 H, m, H-3^{eq}); (**3Bb**) 12.22 (1 H, br.s, CO₂H), 6.62 (1 H, br.d, ${}^{3}J_{9,10}$ 5.7 Hz, H-10), 6.25 (1 H, d, ${}^{3}J_{10,9}$ 5.7 Hz, H-9), 5.32 (1 H, s, H-10b), 4.07 (1 H, br.dd, ${}^{2}J_{2,2}$ 12.1, ${}^{3}J_{2e,3e}$ 3.8 Hz, H-2^{eq}), 3.94–3.88 (1 H, m, H-4^{eq}), 3.66 (1 H, dt, ${}^{2}J_{2,2} \sim {}^{3}J_{2a,3a}$ 12.1, ${}^{3}J_{2a,3e}$ 2.5 Hz, H-2^{ax}), 2.95 (1 H, br.dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 12.5, ${}^{3}J_{4a,3e}$ 4.5 Hz, H-4^{ax}), 2.74 (1 H, d, ${}^{3}J_{7,6a}$ 8.9 Hz, H-7), 2.47 (1 H, d, ${}^{3}J_{6a,7}$ 8.9 Hz, H-6a), 1.68-1.55 (1 H, m, H-3ax), 1.52 (3 H, s, Me-8), 1.50-1.45 (1 H, m, H-3^{eq}); δ_C (100.6 MHz, D₂O/NaOD (5 mol %)) (**3Ab**) 175.2 (CO₂H), 173.0 (C-6), 138.3 (C-9), 131.4 (C-10), 88.7 and 86.9 (C-10a and C-8), 84.4 (C-10b), 65.7 (C-2), 50.9 (C-7), 48.0 (C-6a), 36.9 (C-4), 22.7 (C-3), 13.4 (Me-8); δ_C (100.6 MHz, DMSO*d*₆) (**3Ab**) 171.4 (CO₂H), 170.8 (C-6), 139.6 (C-9), 134.5 (C-10), 89.6 (C-8), 89.0 (C-10a), 85.2 (C-10b), 66.7 (C-2), 52.4 (C-7), 47.4 (C-6a), 38.1 (C-4), 25.0 (C-3), 15.6 (Me-8); (3Bb) 171.5 (CO₂H), 170.8 (C-6), 139.9 (C-9), 135.7 (C-10), 88.79 (C-8), 88.76 (C-10a), 83.1 (C-10b), 66.2 (C-2), 53.5 (C-7), 47.6 (C-6a), 37.5 (C-4), 23.3 (C-3), 15.6 (Me-8); MS (EI, 70 eV) m/z 265 (3, M⁺), 221 (18), 220 (10), 219 (11), 204 (10), 167 (11), 166 (94), 152 (31), 138 (14), 137 (13), 136 (15), 135 (33), 123 (23), 122 (18), 111 (15), 110 (38), 109 (100), 108 (17), 99 (15), 95 (21), 86 (91), 79 (11), 58 (10), 56 (16), 55 (13), 54 (15), 53 (17), 43 (24), 41 (11), 28 (19), 27 (17), 26 (16%).

(6aRS,7RS,8SR,10aSR,10bRS)-8-Bromo-6-oxo-3,4,6,6a,7,8hexahydro-2*H*-8,10a-epoxy[1,3]oxazino[2,3-*a*]isoindole-7carboxylic acid (3Ac) and (6aRS,7RS,8SR,10aSR,10bSR)-8bromo-6-oxo-3,4,6,6a,7,8-hexahydro-2*H*-8,10a-

epoxy[1,3]oxazino[2,3-*a***]isoindole-7-carboxylic acid (3Bc).** Ratio of isomers **3Ac/3Bc** ~ 83/17, (13.86 g, 42 %), yellow powder, m.p. 205–208 °C; [Found: C, 43.59; H, 3.55; N, 4.16; Hal, 23.94. C₁₂H₁₂BrNO₅ requires C, 43.66; H, 3.66; N, 4.24; Br, 24.20%]; R_f (50% EtOH/DMF) 0.73; ν_{max} (KBr) 3136, 1751, 1670, 1449, 1162, 1056 cm⁻¹; δ_H (600 MHz, DMSO-*d*₆) (**3Ac**) 12.60 (1 H, br.s, CO₂H), 6.73 and 6.55 (1 H and 1 H, two d, ³*J*_{9,10} 5.5 Hz, H-9 and H-10), 5.18 (1 H, s, H-10b), 4.07 (1 H, br.dd,

 ${}^{2}J_{2,2}$ 11.7, ${}^{3}J_{2e,3a}$ 4.1 Hz, H-2^{eq}), 3.90–3.86 (2 H, m, H-4^{eq} and H- 2^{ax}), 3.09 (1 H, dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 12.4, ${}^{3}J_{4a,3e}$ 3.4 Hz, H-4^{ax}), 2.98 and 2.96 (1 H and 1 H, two d, ${}^{3}J_{6a,7}$ 8.9 Hz, H-7 and H-6a), 1.62– 1.55 (1 H, m, H-3^{ax}), 1.46–1.48 (1 H, m, H-3^{eq}); (3Bc) 12.60 (1 H, br.s, CO₂H), 6.76 and 6.48 (1 H and 1 H, two d, ${}^{3}J_{9,10}$ 5.5 Hz, H-9 and H-10), 5.37 (1 H, s, H-10b), 4.07 (1 H, br.dd, ²J_{2,2} 11.7, ${}^{3}J_{2e,3a}$ 4.1 Hz, H-2^{eq}), 3.85–3.82 (2 H, m, H-4^{eq} and H-2^{ax}), 3.66 (1 H, dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 11.7, ${}^{3}J_{4a,3e}$ 2.0 Hz, H-4^{ax}), 2.98 and 2.96 (1 H and 1 H, two d, ${}^{3}J_{6a,7}$ 8.9 Hz, H-7 and H-6a), 1.62–1.55 (1 H, m, H-3^{ax}), 1.55–1.51 (1 H, m, H-3^{eq}); δ_{C} (100.6 MHz, DMSO- d_{6}) (3Ac) 170.2 and 170.1 (CO₂H and C-6), 140.7 (C-9), 136.1 (C-10), 91.3 and 88.6 (C-10a and C-8), 84.8 (C-10b), 67.4 (C-2), 52.3 (br.s, C-7), 51.3 (C-6a), 38.8 (C-4), 25.5 (C-3); (3Bc) 170.13 and 170.07 (CO₂H and C-6), 140.8 (C-9), 137.5 (C-10), 91.4 and 88.1 (C-10a and C-8), 83.2 (C-10b), 67.0 (C-2), 53.4 (C-6a), 51.4 (br.s, C-7), 38.2 (C-4), 23.9 (C-3); δ_C (100.6 MHz, D₂O/NaOD (5 mol %)) (3Ac) 173.1 and 171.6 (CO₂H and C-6), 138.6 (C-10), 132.4 (C-9), 86.2 and 81.2 (C-10a and C-8), 83.7 (C-10b), 66.0 (C-2), 51.4 (br.s, C-7), 51.1 (C-6a), 37.0 (C-4), 22.7 (C-3); MS (EI, 70 eV) m/z 331 (1, M⁺, for Br⁸¹), 329 (1), 303 (3), 301 (5), 286 (7), 285 (7), 284 (7), 250 (10), 232 (25), 230 (25), 201 (11), 199 (11), 175 (13), 173 (11), 137 (18), 99 (12), 86 (100), 85 (18), 65 (15), 56 (24), 41 (18), 39 (19%).

(6aRS,7RS,8SR,10aSR,10bRS)-8-Iodo-6-oxo-3,4,6,6a,7,8hexahydro-2H-8,10a-epoxy[1,3]oxazino[2,3-*a*]isoindole-7carboxylic acid (3Ad) and (6aRS,7RS,8SR,10aSR,10bSR)-8iodo-6-oxo-3,4,6,6a,7,8-hexahydro-2H-8,10a-

epoxy[1,3]oxazino[2,3-a]isoindole-7-carboxylic acid (3Bd). Ratio of isomers **3Ad/3Bd** ~ 80/20, pale brown powder (20.75 g, 55 %), m.p. 178–182 °C (decomp.); [Found: C, 38.39; H, 3.29; N, 3.60. C₁₂H₁₂INO₅ requires C, 38.22; H, 3.21; N, 3.71%]; R_f (50% EtOH/DMF) 0.72; v_{max} (KBr) 3153, 1747, 1678 cm⁻¹; δ_{H} (400 MHz, DMSO-d₆) (3Ad) 12.54 (1 H, br.s, CO₂H), 6.62 and 6.57 (1 H and 1 H, two br.d, ${}^{3}J_{9,10}$ 5.7 Hz, H-9 and H-10), 5.18 (1 H, s, H-10b), 4.12 (1 H, br.dd, ${}^{2}J_{2,2}$ 11.4, ${}^{3}J_{3a,2e}$ 4.4 Hz, H-2^{eq}), 3.94–3.88 (2 H, m, H-2^{ax} and H-4^{eq}), 3.11 (1 H, br.dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 12.7, ³J_{4a,3e} 3.8 Hz, H-4^{ax}), 2.91 (2 H, s, H-7 and H-6a), 1.68–1.58 (1 H, m, H-3^{ax}), 1.53–1.47 (1 H, m, H-3^{eq}); (**3Bd**) 12.54 (1 H, br.s, CO₂H), 6.77 and 6.58 (1 H and 1 H, two br.d, ³J_{9.10} 5.7 Hz, H-9 and H-10), 5.21 (1 H, s, H-10b), 4.12 (1 H, br.dd, ${}^{2}J_{2,2}$ 11.4, ${}^{3}J_{3a,2e}$ 4.4 Hz, H-2^{eq}), 3.94–3.88 (2 H, m, H-2^{ax} and H-4^{eq}), 3.11 (1 H, br.dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 12.7, ${}^{3}J_{4a,3e}$ 3.8 Hz, H-4^{ax}), 3.02 and 3.00 (1 H and 1 H, two d, H-7 and H-6a), 1.68-1.58 (1 H, m, H-3^{ax}), 1.53–1.47 (1 H, m, H-3^{eq}); δ_{C} (100.6 MHz, DMSO- d_{6}) (3Ad) 170.3 and 169.7 (CO₂H and C-6), 143.3 (C-10), 134.7 (C-9), 88.9 (C-10a), 84.0 (C-10b), 66.8 (C-2), 65.5 (C-8), 52.5 (C-6a), 50.9 (C-7), 38.2 (C-4), 24.9 (C-3); (**3Bd**) 169.7 and 169.4 (CO₂H and C-6), 140.1 (C-10), 135.5 (C-9), 90.7 (C-10a), 84.2 (C-10b), 66.8 (C-2), 65.5 (C-8), 51.7 (C-6a), 50.7 (C-7), 38.2 (C-4), 24.9 (C-3); δ_C (100.6 MHz, D₂O/NaOD (5 mol %)) (3Ad) 173.9 and 171.7 (CO₂H and C-6), 141.9 (C-10), 131.6 (C-9), 87.0 and 80.8 (C-10a and C-8), 83.4 (C-10b), 66.0 (C-2), 53.5 (br.s, C-7), 53.3 (C-6a), 37.0 (C-4), 22.7 (C-3); MS (EI, 70 eV) *m/z* 377 (1, M⁺), 332 (12), 278 (100), 253 (11), 250 (34), 249 (47), 235 (65), 222 (50), 221 (68), 207 (21), 206 (31), 194 (17), 180 (28), 179 (42), 178 (62), 151 (29), 149 (36), 138 (24), 137 (47), 128 (46), 123 (49), 107 (59), 99 (50), 86 (77), 79 (38), 68 (32), 66 (35), 59 (54), 56 (79), 45 (19), 44 (28), 43 (35), 42 (37%).

(6aRS,7SR,8RS,10aSR,10bRS)-8-Nitro-6-oxo-3,4,6,6a,7,8hexahydro-2*H*-8,10a-epoxy[1,3]oxazino[2,3-*a*]isoindole-7carboxylic acid (3Ae) and

(6aRS,7SR,8RS,10aSR,10bSR)-8-nitro-6-oxo-3,4,6,6a,7,8hexahydro-2H-8,10a-epoxy[1,3]oxazino[2,3-*a*]isoindole-7-

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and

carboxylic acid (3Be). The reaction was carried out following the general procedure above, but the condensation step was performed at -5 - 0 °C, 10 min. Ratio of isomers 3Ae/3Be ~ 89/11, maize yellow powder (10.36 g, 35 %), m.p. > 229 °C (decomp.); [Found: C, 48.29; H, 4.30; N, 9.61. C₁₂H₁₂N₂O₇ requires C, 48.65; H, 4.08; N, 9.46%]; R_f (35% EtOAc/ hexane) 0.35; v_{max} (KBr) 3149, 1745, 1695, 1579, 1433, 1390 cm⁻¹; δ_{H} (600 MHz, DMSO-d₆) (**3Ae**) 13.02 (1 H, s, CO₂H), 7.00 and 6.89 (1 H and 1H, two d, ${}^{3}J_{9,10}$ 5.5 Hz, H-9 and H-10), 5.32 (1 H, s, H-10b), 4.11 (1 H, br.dd, ${}^{2}J_{2,2}$ 12.2, ${}^{3}J_{2e,3a}$ 4.8 Hz, H-2^{eq}), 3.91 (1 H, br.dt, ${}^{2}J_{2,2} \sim {}^{3}J_{2a,3a}$ 12.2, ${}^{3}J_{2a,3e}$ 2.1 Hz, H-2^{ax}), 3.88 (1 H, br.dd, ${}^{2}J_{4,4}$ 13.7, ${}^{3}J_{4e,3a}$ 5.5 Hz, H-4^{eq}), 3.34 and 3.24 (1 H and 1 H, two d, ${}^{3}J_{6a,7}$ 8.9 Hz, H-7 and H-6a), 3.14 (1 H, dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 13.7, ³J_{4a,3e} 4.1 Hz, H-4^{ax}), 1.65–1.57 (1 H, m, H-3^{ax}), 1.51–1.49 (1 H, m, H-3^{eq}); (**3Be**) 13.02 (1 H, br.s, CO₂H), 7.03 and 6.81 (1 H and 1 H, two d, ³J_{9,10} 5.5 Hz, H-9 and H-10), 5.47 (1 H, s, H-10b), 4.09 (1 H, br.dd, ${}^{2}J_{2,2}$ 11.7, ${}^{3}J_{2e,3a}$ 4.8 Hz, H-2^{eq}), 3.89–3.84 (2 H, m, H-4^{eq} and H-2^{ax}), 3.34 and 3.29 (1 H and 1 H, two d, ${}^{3}J_{6a,7}$ 8.9 Hz, H-7 and H-6a), 2.98 (1 H, dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 13.7, ${}^{3}J_{4a,3e}$ 4.1 Hz, H-4^{ax}), 1.65–1.57 (1 H, m, H-3^{ax}), 1.51–1.49 (1 H, m, H-3^{eq}); δ_C (150.9 MHz, DMSO-d₆) (3Ae) 169.6 and 169.2 (CO₂H and C-6), 137.1 and 135.8 (C-10 and C-9), 113.1 (C-8), 87.8 (C-10a), 84.4 (C-10b), 67.4 (C-2), 50.9 and 47.5 (C-6a and C-7), 38.8 (C-4), 25.5 (C-3); (3Be) 169.7 (CO₂H), 166.6 (C-6), 138.3 and 136.1 (C-10 and C-9), 113.0 (C-8), 88.3 (C-10a), 82.7 (C-10b), 66.8 (C-2), 51.9 and 48.0 (C-6a and C-7), 37.9 (C-4), 23.7 (C-3); MS (EI, 70 eV) *m/z* 296 (14, M⁺), 286 (4), 270 (3), 268 (5), 259 (3), 257 (6), 254 (9), 250 (68), 233 (16), 221 (15), 220 (24), 205 (22), 204 (38), 197 (78), 181 (25), 177 (33), 176 (56), 169 (38), 164 (10), 153 (11), 149 (17), 138 (41), 130 (15), 123 (26), 121 (41), 111 (20), 110 (38), 101 (16), 99 (100), 95 (56), 85 (57), 84 (67), 83 (70), 78 (55), 65 (69), 63 (74), 59 (25), 57 (50), 56 (80), 45 (30), 43 (35%).

The individual isomer **3Ae** was isolated by crystallization from Me₂CO/EtOH as a fine-crystalline yellow powder, m.p. 230-231 °C (decomp.).

(6aRS,7SR,8RS,10aSR,10bRS)-6-Oxo-8-phenyl-3,4,6,6a,7,8hexahydro-2H-8,10a-epoxy[1,3]oxazino[2,3-a]isoindole-

7(10bH)-carboxylic acid (3Af). Acids 3f (R Ph) could not be obtained by the general procedure due to their low crystallization propensity (after the reaction with maleic anhydride a brown oil soluble in Me₂CO or CH₂Cl₂ is formed). In this case, the solvent was evaporated, and the residue was purified by column chromatography (Al₂O₃, 3×14 cm) using hexane/EtOAc (5/1 \rightarrow 1/1) as eluent. Compound **3Af** was obtained in 16 % yield (5.23 g) as white needles, m.p. 124.8-127.1 °C (decomp., from EtOAc/EtOH); [Found: C, 66.19; H, 5.14; N, 4.40. C₁₈H₁₇NO₅ requires C, 66.05; H, 5.23; N, 4.28%]; v_{max} (KBr) 3195, 1753, 1682 cm⁻¹; $\delta_{\rm H}$ (400 MHz, DMSO- d_6) 11.84 (1 H, br.s, CO₂H), 7.41-7.27 (5 H, m, H-Ph), 6.77 (1 H, br.d, ³J_{9,10} 5.7 Hz, H-9), 6.55 (1 H, d, ³J_{10,9} 5.7 Hz, H-10), 5.31 (1 H, s, H-10b), 4.15 (1 H, br.dd, ²J_{2,2} 11.4, ³J_{2e,3e} 4.4 Hz, H-2^{eq}), 3.98–3.92 (2 H, m, H-2^{ax} and H-4^{eq}), 3.16 (1 H, dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 13.4, ${}^{3}J_{4a,3e}$ 3.8 Hz, H-4^{ax}), 3.03 and 2.96 (1 H and 1 H, two d, ${}^{3}J_{7,6a}$ 8.9 Hz, H-7 and H-6a), 1.71–1.62 (1 H, m, H-3^{ax}), 1.55–1.51 (1 H, m, H-3^{eq}); δ_{C} (100.6 MHz, DMSO-d₆) 170.7 and 170.4 (CO₂H and C-6), 140.0 (C-9), 136.4 (C-1'), 134.3 (C-10), 127.9 (2 C, C-2' and C-6'), 127.4 (C-4'), 125.3 (2 C, C-3' and C-5'), 93.6 and 89.2 (C-10a and C-8), 85.2 (C-10b), 66.8 (C-2), 52.3 (C-7), 48.6 (C-6a), 38.2 (C-4), 25.0 (C-3); MALDI-TOF HR: MH⁺, found 328.1192. C₁₈H₁₈NO₅ requires 328.1179.

(6aRS,7SR,8RS,10aSR,10bRS)-8-(3-Nitrophenyl)-6-oxo-3,4,6,6a,7,8-hexahydro-2H-8,10a-epoxy[1,3]oxazino[2,3*a*]isoindole-7-carboxylic acid (3Ag)(6aRS,7SR,8RS,10aSR,10bSR)-8-(3-nitrophenvl)-6-oxo-3,4,6,6a,7,8-hexahydro-2H-8,10a-epoxy[1,3]oxazino[2,3-

alisoindole-7-carboxylic acid (3Bg). The synthesis was carried out following the general procedure above, but CHCl3 was used as the reaction solvent because the starting 5-arylfurfural has a low solubility in CH₂Cl₂ and Me₂CO. Ratio of isomers 3Ag/3Bg ~ 91/9, pale yellow powder (17.1 g, 46 %), m.p. > 169 °C (decomp.); [Found: C, 58.23; H, 4.67; N, 7.41. C₁₈H₁₆N₂O₇ requires C, 58.06; H, 4.33; N, 7.52%]; v_{max} (KBr) 3413, 1730, 1692, 1531, 1348, 1195, 1056 cm⁻¹; $\delta_{\rm H}$ (600 MHz, DMSO- d_6) (3Ag) 12.05 (1 H, br.s, CO₂H), 8.17 (1 H, br.s, 1 H, H-2'), 8.16 $(1 \text{ H}, \text{ d}, {}^{3}J_{4',5'} 8.3 \text{ Hz}, \text{H-4'}), 7.90 (1 \text{ H}, \text{ d}, {}^{3}J_{6',5'} 7.7 \text{ Hz}, \text{H-6'}),$ 7.68 (1 H, ddd, ${}^{3}J_{5',4'}$ 8.3, ${}^{3}J_{5',6'}$ 7.7, ${}^{5}J_{5',2'}$ 1.3 Hz, H-5'), 6.79 and 6.64 (1 H and 1 H, two d, ³J_{9.10} 5.5 Hz, H-9 and H-10), 5.34 (1 H, s, H-10b), 4.13 (1 H, br.dd, ${}^{2}J_{2,2}$ 11.7, ${}^{3}J_{2e,3a}$ 4.1 Hz, H-2^{eq}), 3.94–3.90 (2 H, m, H-2^{ax} and H-4^{eq}), 3.15 (1 H, dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 13.1, ${}^{3}J_{4a,3e}$ 3.4 Hz, H-4^{ax}), 3.11 and 3.00 (1 H and 1 H, two d, ${}^{3}J_{7.6a}$ 8.9 Hz, H-7 and H-6a), 1.65–1.61 (1 H, m, H-3^{ax}), 1.51-1.49 (1 H, m, 3-H^{eq}); (**3Bg**) 12.05 (1 H, br.s, CO₂H), 8.28 (1 H, s, H-2'), 8.16 (1 H, d, ${}^{3}J_{4',5'}$ 8.3 Hz, H-4'), 7.90 (1 H, d, ${}^{3}J_{6',5'}$ 1, 5, 11-2), 5.16 (1 11, d, $J_{4,5}$), 8.5 (1 11, d, $J_{6,5}$, 7.7 Hz, H-6'), 7.68 (1 H, dd, ${}^{3}J_{5',4'}$, 8.3, ${}^{3}J_{5',6'}$, 7.7, ${}^{5}J_{5',2'}$, 1.3 Hz, H-5'), 6.81 and 6.54 (1 H and 1 H, two d, ${}^{3}J_{9,10}$, 5.5 Hz, H-9 and H-10), 5.45 (1 H, s, H-10b), 4.13 (1 H, dd, ${}^{2}J_{2,2}$, 11.7, ${}^{3}J_{2a,3e}$, 4.1 Hz, H-2^{eq}), 3.94-3.90 (2 H, m, H-2^{ax} and H-4^{eq}), 3.15 (1 H, dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 13.1, ${}^{3}J_{4a,3e}$ 3.4 Hz, 1 H, H-4^{ax}), 3.11 and 3.06 (1 H and 1 H, two d, ³J_{7,6a} 8.9 Hz, H-7 and H-6a), 1.65–1.61 (1 H, m, H-3^{ax}), 1.51–1.49 (1 H, m, H-3^{eq}); δ_{C} (150.9 MHz, DMSO- d_{6}) (3Ag) 171.0 and 170.9 (CO₂H and C-6), 147.9 (C-3'), 139.6 and 138.9 (C-10 and C-9), 135.3 (C-1'), 132.7 (C-6'), 130.4 (C-5'), 123.2 (C-2'), 120.7 (C-4'), 93.2 (C-8), 90.0 (C-10a), 85.5 (C-10b), 67.3 (C-2), 52.6 and 49.1 (C-6a and C-7), 38.7 (C-4), 25.5 (C-3); (**3Bg**) 171.1 and 168.1 (CO₂H and C-6), 147.9 (C-3'), 140.0 and 139.8 (C-10 and C-9), 135.3 (C-1'), 132.7 (C-6'), 130.3 (C-5'), 123.2 (C-2'), 120.7 (C-4'), 92.6 (C-8), 89.9 (C-10a), 83.6 (C-10b), 66.9 (C-2), 53.7 and 49.1 (C-6a and C-7), 38.1 (C-4), 25.3 (C-3); MS (EI, 70 eV) m/z 354 (5, M⁺-18), 340 (1), 311 (2), 293 (1), 274 (10), 272 (19), 257 (70), 244 (68), 230 (17), 229 (43), 216 (18), 213 (52), 202 (21), 188 (23), 173 (24), 172 (39), 161 (21), 158 (50), 146 (43), 145 (87), 144 (67), 130 (30), 128 (65), 120 (20), 119 (41), 116 (83), 104 (47), 102 (54), 98 (43), 92 (20), 89 (27), 88 (47), 85 (55), 76 (43), 59 (80), 54 (100), 53 (45), 48 (21), 47 (25), 45 (29), 43 (47%). (6aRS,7SR,8RS,10aSR,10bRS)-8-(2-Nitrophenyl)-6-oxo-

3,4,6,6a,7,8-hexahydro-2H-8,10a-epoxy[1,3]oxazino[2,3a]isoindole-7-carboxylic acid (6aRS,7SR,8RS,10aSR,10bSR)-8-(2-nitrophenyl)-6-oxo-

and

(3Ah)

3,4,6,6a,7,8-hexahydro-2H-8,10a-epoxy[1,3]oxazino[2,3alisoindole-7-carboxylic acid (3Bh). The synthesis was carried out in accordance with the general procedure described above, but CHCl₃ was used as the reaction solvent because the starting 5-arylfurfural has a low solubility in CH₂Cl₂ and Me₂CO. Ratio of isomers 3Ah/3Bh ~ 92/8, yellow powder (14.55 g, 39 %), m.p. 173-174 °C (decomp.); [Found: C, 58.29; H, 4.19; N, 7.28. C₁₈H₁₆N₂O₇ requires C, 58.06; H, 4.33; N, 7.52%]; v_{max} (KBr) 3509, 1722, 1649, 1531, 1348, 1050 cm⁻¹; $\delta_{\rm H}$ (400 MHz, DMSO d_6) (**3Ah**) 12.04 (1 H, br.s, CO₂H), 8.10 (1 H, d, ${}^{3}J_{3',4'}$ 7.6 Hz, H-3'), 7.76-7.72 (2 H, m, H-5' and H-6'), 7.66-7.59 (1 H, m, H-4'), 6.85 and 6.67 (1 H and 1 H, two d, ${}^{3}J_{9,10}$ 5.3 Hz, H-9 and H-10), 5.31 (1 H, s, H-10b), 4.17 (1 H, br.dd, ²J_{2,2} 11.4, ³J_{2e,3a} 4.4 Hz, H-2^{eq}), 4.00-3.93 (2 H, m, H-2^{ax} and H-4^{eq}), 3.26 and 3.07 (1 H and 1 H, two d, ${}^{3}J_{7.6a}$ 9.3 Hz, H-7 and H-6a), 3.16 (1 H, dt, ${}^{2}J_{4.4}$ ~ ${}^{3}J_{4a,3a}$ 13.4, ${}^{3}J_{4a,3e}$ 3.8 Hz, H-4^{ax}), 1.73–1.61 (1 H, m, H-3^{ax}),

1.55–1.52 (1 H, m, H-3^{eq}); (**3Bh**) 12.04 (1 H, br.s, CO₂H), 8.07 (1 H, dd, ${}^{4}J_{3',5'}$ 1.2, ${}^{3}J_{3',4'}$ 7.6 Hz, H-3'), 7.78–7.72 (2 H, m, H-5' and H-6'), 7.66-7.60 (1 H, m, H-4'), 6.87 and 6.61 (1 H and 1 H, two d, ${}^{3}J_{9,10}$ 5.3 Hz, H-9 and H-10), 5.49 (1 H, s, H-10b), 4.19-4.15 (1 H, m, H-2^{eq}), 4.00-3.93 (2 H, m, H-2^{ax} and H-4^{eq}), 3.26 and 3.07 (1 H and 1 H, two d, ${}^{3}J_{7.6a}$ 9.3 Hz, H-7 and H-6a), 3.18-3.14 (1 H, m, H-4^{ax}), 1.73-1.61 (1 H, m, H-3^{ax}), 1.55-1.52 (1 H, m, H-3^{eq}); δ_{C} (100.6 MHz, DMSO- d_{6}) (3Ah) 170.5 and 170.3 (CO₂H and C-6), 146.7 (C-2'), 138.8 and 134.9 (C-9 and C-10), 133.6, 129.6, 129.5 (C-4', C-5', C-6'), 130.9 (C-1'), 124.9 (C-3'), 92.7 and 88.1 (C-8 and C-10a), 85.1 (C-10b), 66.8 (C-2), 52.0 and 47.8 (C-6a and C-7), 38.1 (C-4), 24.9 (C-3); (3Bh) 170.5 and 167.5 (CO₂H and C-6), 146.7 (C-2'), 139.0 and 136.2 (C-9 and C-10), 135.5, 131.7, 130.0, 129.2 (C-4', C-5', C-6', C-1'), 124.8 (C-3'), 87.9, 83.2, 79.1 (C-8, C-10a, C-10b), 66.4 (C-2), 53.2 and 48.1 (C-6a and C-7), 37.6 (C-4), 23.2 (C-3); MS (EI, 70 eV) m/z 372 (6, M⁺), 328 (3), 284 (5), 276 (3), 274 (27), 244 (58), 231 (28), 230 (100), 217 (64), 216 (59), 202 (83), 183 (20), 172 (14), 156 (15), 128 (25), 115 (69), 89 (16), 76 (23), 63 (14), 59 (34), 54 (78), 43 (34%).

General procedure for preparation of epoxy[1,3]oxazino[2,3*a*]isoindoles (4a–c). Acryloyl chloride (7.21 mL, 0.09 mol) was added dropwise to a stirred and water cooled (+5 °C) solution of the tautomeric mixture 1 \leftrightarrows 2 obtained above (0.064 mol) and NEt₃ (16.6 mL, 0.12 mol) in PhH (80 mL). The reaction mixture was heated under reflux for 2 h (TLC control), then cooled and poured into cold water (250 mL). The organic layer was separated and the aqueous layer was extracted with EtOAc (2 × 100 mL). The combined organic phases were dried over MgSO₄. Solvents were removed under reduced pressure to give brown oil, which was purified by silica-gel column chromatography (3 × 10 cm) with Et₂O and then EtOAc/hexane, 1/1 as eluent. Adducts **4a–c** were obtained as colorless crystals.

(6aRS,8SR,10aSR,10bRS)-3,4,7,8-Tetrahydro-2H-8,10a-

epoxv[1,3]oxazino[2,3-a]isoindol-6(6aH,10bH)-one (4a). Fine colorless prisms (2.37 g, 18 %), m.p. 136-138 °C; [Found: C, 63.56; H, 6.42; N, 6.55. C₁₁H₁₃NO₃ requires C, 63.76; H, 6.32; N, 6.76%]; R_f (25% EtOAc/hexane) 0.35; v_{max} (KBr) 2854, 1679 (br), 1433, 1266, 1045 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 6.45 (1 H, d, ${}^{3}J_{10,9}$ 6.0 Hz, H-10), 6.39 (1 H, dd, ${}^{3}J_{9,10}$ 6.0, ${}^{3}J_{9,8}$ 1.8 Hz, H-9), 5.40 (1 H, s, H-10b), 5.12 (1 H, dd, ${}^{3}J_{8,7exo}$ 4.6, ${}^{3}J_{8,9}$ 1.8 Hz, H-8), 4.23–4.16 (2 H, m, H-2^{eq} and H-4^{eq}), 3.73 (1 H, dt, ${}^{3}J_{2a,3a} \sim {}^{2}J_{2,2}$ 12.0, ${}^{3}J_{2a,3e}$ 1.8 Hz, H-2^{ax}), 3.00 (1 H, dddd, ${}^{2}J_{4,4}$ 13.6, ${}^{3}J_{4a,3a}$ 11.4, ${}^{3}J_{4a,3e}$ 3.6, ${}^{5}J_{4a,6}$ 1.6 Hz, H-4^{ax}), 2.39 (1 H, ddd, ${}^{5}J_{4,6a}$ 1.6, ${}^{3}J_{6a,7exo}$ 4.0, ${}^{3}J_{6a,7exo}$ 8.7 Hz, H-6a), 2.23 (1 H, dt, ${}^{2}J_{7,7}$ 11.7, ${}^{3}J_{7exo,8}$ ~ ${}^{3}J_{7\text{exo},6a}$ 4.0 Hz, H-7^{*exo*}), 1.96–1.84 (1 H, m, H-3^{*ax*}), 1.60–1.54 (1 H, m, H-3^{eq}), 1.55 (1 H, dd, ${}^{2}J_{7,7}$ 11.7, ${}^{3}J_{7\text{endo},6a}$ 8.7 Hz, H-7^{endo}); δ_{C} (100.6 MHz, CDCl₃) 173.9 (C-6), 135.9 (C-10), 132.0 (C-9), 90.1 (C-10a), 86.7 (C-10b), 79.8 (C-8), 67.6 (C-2), 46.3 (C-6a), 38.8 (C-4), 27.6 (C-7), 25.3 (C-3). GC-MS (EI, 70 eV) m/z 207 (6, M⁺), 179 (57), 178 (19), 152 (17), 151 (11), 150 (11), 138 (11), 137 (22), 124 (16), 123 (12), 122 (19), 96 (31), 95 (65), 94 (53), 86 (100), 84 (28), 81 (12), 77 (11), 66 (27), 65 (13), 56 (31), 55 (67), 41 (23), 39 (18), 29 (15), 28 (27), 27 (29%).

(6aRS,8SR,10aSR,10bRS)-8-Methyl-3,4,7,8-tetrahydro-2H-

8,10a-epoxy[1,3]oxazino[2,3-*a***]isoindol-6(6***aH***,10***bH***)-one (4***b***). Fine white prisms (2.68 g, 19 %), m.p. 132–133 °C; [Found: C, 65.19; H, 6.88; N, 6.00. C_{12}H_{15}NO_3 requires C, 65.14; H, 6.83; N, 6.33%]; R_f (25% EtOAc/hexane) 0.36; v_{max} (KBr) 1698 cm⁻¹; \delta_H (400 MHz, CDCl₃) 6.57 and 6.20 (1 H and 1 H, two d, {}^{3}J_{9,10} 5.6 Hz, H-10 and H-9), 5.00 (1 H, s, H-10b), 4.24–4.19 (2 H, m, H-2^{eq} and H-4^{eq}), 3.87 (1 H, dt, {}^{3}J_{2a,3a} \sim {}^{2}J_{2,2} 12.5, {}^{3}J_{2a,3e} 2.5 Hz, H-2^{ax}), 3.09 (1 H, dt, {}^{3}J_{4,3a} \sim {}^{2}J_{4,4} 12.5, {}^{3}J_{4a,3e} 3.6 Hz, H-4^{ax}), 2.60 (1**

H, dd, ${}^{3}J_{6aendo,7endo}$ 8.7, ${}^{3}J_{6a,7exo}$ 3.7 Hz, H-6a^{endo}), 1.88 (1 H, dd, ${}^{2}J_{7,7}$ 11.8, ${}^{3}J_{7exo,6a}$ 3.7 Hz, H-7^{exo}), 1.96–1.84 (1 H, m, H-3^{ax}), 1.71 (1 H, dd, ${}^{2}J_{7,7}$ 11.8, ${}^{3}J_{7endo,6a}$ 8.7 Hz, H-7^{endo}), 1.64 (3 H, s, Me-8), 1.48–1.52 (1 H, m, 3-H^{eq}); $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 174.1 (C-6), 139.3 and 132.5 (C-9 and C-10), 89.8 and 88.2 (C-8 and C-10a), 86.9 (C-10b), 67.7 (C-2), 49.6 (C-6a), 38.9 (C-4), 33.9 (C-7), 25.4 (C-3), 18.7 (Me-8). GC-MS (EI, 70 eV) *m*/*z* 221 (26, M⁺), 193 (47), 178 (60), 166 (25), 152 (16), 151 (49), 150 (22), 139 (12), 138 (20), 137 (14), 136 (34), 124 (25), 123 (12), 122 (23), 111 (16), 110 (39), 109 (92), 108 (75), 107 (12), 98 (11), 95 (15), 94 (11), 86 (100), 80 (38), 79 (14), 56 (17), 55 (51), 53 (10), 43 (20), 41 (11), 27 (14%).

(6aRS,8RS,10aSR,10bRS)-8-Bromo-3,4,7,8-tetrahydro-2*H*-8,10a-epoxy[1,3]oxazino[2,3-*a*]isoindol-6(6*aH*,10b*H*)-one (4Ac) and (6aRS,8RS,10aSR,10bSR)-8-bromo-3,4,7,8-

tetrahydro-2H-8,10a-epoxy[1,3]oxazino[2,3-a]isoindol-

6(6aH,10bH)-one (4Bc). Ratio of isomers 4Ac/4Bc ~ 64/36, fine white crystals (3.86 g, 21 %), m.p. 113-114 °C; [Found: C, 46.21; H, 4.31; N, 5.12; Hal, 28.38. C₁₁H₁₂BrNO₃ requires C, 46.18; H, 4.23; N, 4.90; Br, 27.93%]; R_f (25% EtOAc/hexane) 0.40; v_{max} (KBr) 1690, 1045 cm⁻¹; δ_{H} (600 MHz, CDCl₃) (4Ac) 6.60 and 6.41 (1 H and 1 H, two d, ${}^{3}J_{9,10}$ 5.8 Hz, H-10 and H-9), 5.04 (1 H, s, H-10b), 4.23-4.18 (2 H, m, H-2^{eq} and H-4^{eq}), 3.88 $^{3}J_{6a,7ex0}$ 4.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd, $^{2}J_{7,7}$ 11.9, $^{3}J_{7ex0,64}$.1 Hz, H-6a^{endo}), 2.47 (1 H, dd), 2.47 (H-7^{exo}), 2.25 (1 H, dd, ²J_{7,7} 11.9, ³J_{7endo,6a} 8.9 Hz, H-7^{endo}), 1.93-1.86 (1 H, m, H-3^{ax}), 1.55-1.53 (1 H, m, H-3^{eq}); (4Bc) 6.68 and 6.37 (1 H and 1H, two d, ³J_{9,10} 5.8 Hz, H-10 and H-9), 5.04 (1 H, s, H-10b), 4.23–4.18 (2 H, m, H-2^{eq} and H-4^{eq}), 3.88 (1 H, dt, ${}^{2}J_{2,2} \sim {}^{3}J_{2a,3a}$ 12.7, ${}^{3}J_{2a,3e}$ 2.4 Hz, H-2^{ax}), 3.10 (1 H, dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 12.7, ${}^{3}J_{4a,3e}$ 3.4 Hz, H-4^{ax}), 2.69 (1 H, dd, ${}^{3}J_{6aendo,7endo}$ 8.9, ${}^{3}J_{6a,7exo}$ 4.1 Hz, H-6a^{endo}), 2.37 (1 H, dd, ${}^{2}J_{7,7}$ 11.9, ${}^{3}J_{7exo,6a}$ 4.1 Hz, H-7^{exo}), 2.19 (1 H, dd, ${}^{2}J_{7,7}$ 11.9, ${}^{3}J_{7endo,6a}$ 8.9 Hz, H-7^{endo}), 1.93–1.86 (1 H, m, H-3^{ax}), 1.55–1.53 (1 H, m, H-3^{ax}); δ_{C} (100.6 MHz, CDCl₃) (4Ac) 172.1 (C-6), 139.9 (C-10), 133.0 (C-9), 99.4 (C-8), 89.1 (C-10a), 85.8 (C-10b), 67.7 (C-2), 49.1 (C-6a), 38.9 (C-7), 37.1 (C-4), 25.2 (C-3); (4Bc) 172.2 (C-6), 138.5 (C-10), 133.3 (C-9), 99.4 (C-8), 88.1 (C-10a), 86.0 (C-10b), 67.7 (C-2), 49.2 (C-6a), 38.8 (C-7), 37.1 (C-4), 25.2 (C-3). GC-MS (EI, 70 eV) m/z 287 (2, M⁺, for Br⁸¹), 286 (1), 285 (2), 284 (1), 260 (2), 259 (15), 258 (3), 257 (15), 218 (2), 217 (6), 216 (2), 215 (6), 207 (10), 206 (66), 178 (12), 175 (15), 174 (20), 173 (14), 172 (17), 152 (37), 146 (16), 144 (16), 121 (10), 93 (10), 86 (100), 85 (10), 65 (28), 56 (20), 55 (44), 41 (10), 39 (16), 27 (10%).

Multigram synthesis of 4-amino-4-methylpentan-2-ol.

A modified procedure²⁴ was used for the preparation of 4-amino-4-methylpentan-2-ol. A moderate stream of ammonia gas was bubbled until saturation (4-6 h) through a stirred solution of 4methylpent-3-en-2-one (117 g, 1.20 mol) in MeOH (350 mL). The mixture was stirred at 26 °C for another 2 h. Then NaBH₄ (44.4 g, 1.80 mol) was added to this solution in small portions (during a ~ 1.5 h period). After addition was complete, the mixture was heated under reflux for another 2 h. The mixture was then cooled, poured into water (1400 mL) and extracted with Et_2O (4 × 250 mL). The organic phases were collected and dried over MgSO₄. Ether was removed under reduced pressure to give a pale-yellow oil. The residue was purified by distillation under reduced pressure to afford aminoalcohol as a colorless oil in 67% yield (86.4 g), b.p. 62-64 °C/21 mm Hg; [Found: C, 61.32; H, 12.81; N, 12.05. C₆H₁₅NO requires C, 61.49; H, 12.90; N, 11.95%]; n_D^{22} 1.3450; v_{max} (liquid film) 3359 (br.) cm⁻¹; δ_H (400 MHz, CDCl₃) 6.70 (1 H, br.s, OH), 3.97 (1 H, ddq, ³J_{2.3B} 8.0,

³ $J_{Me,2}$ 6.1, ³ $J_{2,3A}$ 5.5 Hz, H-2), 1.31–1.26 (2 H, m, H-3), 1.09 (3 H, s, Me-4), 1.08 (3 H, s, Me-5), 1.02 (3 H, d, ³ $J_{Me,2}$ 6.1 Hz, Me-1); δ_C (100.6 MHz, CDCl₃) 65.1 (C-2), 50.3 (C-4), 49.2 (C-3), 35.8 (C-1), 26.8 and 23.9 (C-5 and Me-4).

2e-(Furan-2-yl)-4e,4a,6e-trimethyl-1,3-oxazinane (5). А mixture of 4-amino-4-methylpentan-2-ol (7.50 g, 65.0 mmol) and furfural (5.37 g, 65.0 mmol) was heated under reflux (3 h) in PhH (60 mL) with azeotropic removal of water using a Dean-Stark apparatus. After the theoretical amount of water (~ 1.2 mL) was collected, the mixture was cooled and PhH was removed under reduced pressure to afford a brown residue. The residue was purified by distillation under reduced pressure to give 5 as a viscous yellow oil (8.43 g, 66 %), b.p. 156-159 °C/ 7 mm Hg; [Found: C, 67.46; H, 8.59; N, 7.50. C₁₁H₁₇NO₂ requires C, 67.66; H, 8.78; N, 7.17%]; v_{max} (liquid film) 3330 cm⁻¹; δ_{H} (400 MHz, CDCl₃) 7.55 (1 H, dd, ⁴J_{5',3'} 0.9, ³J_{5',4'} 1.8 Hz, H-5'), 6.39 (1 H, dd, ${}^{3}J_{4',5'}$ 1.8, ${}^{3}J_{3',4'}$ 3.2 Hz, H-4), 6.36 (1 H, d, ${}^{4}J_{5',3'}$ 0.9, ${}^{3}J_{3',4'}$ 3.2 Hz, H-3'), 5.25 (1 H, d, ${}^{3}J_{2,\text{NH}}$ 12.0 Hz, H-2), 3.87 (1 H, ddq, ${}^{3}J_{6,5a}$ 13.0, ³J_{6,Me} 6.1, ³J_{6,5e} 2.2 Hz, H-6), 2.16 (1 H, d, ³J_{NH,2} 12.2 Hz, NH), 1.41 (1 H, dd, ²J_{5.5} 13.0, ³J_{5e,6} 2.2 Hz, H-5^{eq}), 1.10 (1 H, t, ${}^{2}J_{5.5} \sim {}^{3}J_{6.5a}$ 13.0 Hz, H-5^{ax}), 1.14 (3 H, s, Me-4A), 1.09 (3 H, s, Me-4B), 1.08 (3 H, d, ${}^{3}J_{\text{Me,6}}$ 6.1 Hz, Me-6); δ_{C} (100.6 MHz, CDCl₃) 152.8 (C-2'), 141.7 (C-5'), 109.8 and 106.2 (C-3' and C-4'), 78.7 (C-2), 68.9 (C-6), 48.9 (C-4), 45.1 (C-5), 32.4 (Me-6), 23.4 and 22.1 (Me-4 \times 2). GC-MS (EI, 70 eV) m/z 195 (13, M⁺), 194 (47), 181 (3), 180 (14), 180 (24), 167 (12), 166 (11), 153 (3), 152 (27), 151 (67), 142 (3), 138 (5), 137 (24), 136 (53), 121 (5), 112 (11), 97 (28), 96 (100), 95 (95), 94 (38), 84 (44), 81 (11), 68 (8), 58 (8), 45 (7), 43 (13), 42 (35), 41 (32), 39 (24%).

(2RS,6aSR,7RS,8SR,10aRS,10bSR)-2,4,4-Trimethyl-6-oxo-3,4,6,6a,7,8-hexahydro-2H-8,10a-epoxy[1,3]oxazino[2,3*a*]isoindole-7(10bH)-carboxylic acid (6A) and

a]isoindole-7(10b*H*)-carboxylic acid (6A) (2*RS*,6a*RS*,7*SR*,8*RS*,10a*SR*,10b*SR*)-2,4,4-trimethyl-6-oxo-3,4,6,6a,7,8-hexahydro-2*H*-8,10a-epoxy[1,3]oxazino[2,3-

alisoindole-7(10bH)-carboxvlic acid (6B). A solution of 2furyloxazine 5 (2.35 g, 12.0 mmol) in Et₂O (20 mL) was added to a solution of maleic anhydride (1.27 g, 13.0 mmol) in Et₂O (20 mL). The mixture was stirred at room temperature for 8 h. The solvent was then decanted from a yellow oil. Precipitate formed upon trituration of the residue with Me₂CO (20 mL) was separated by filtration, washed with Me₂CO (2×15 mL) and dried in air to give product 6 (1.0 g, 28 %) as a pale-yellow powder, ratio of isomers 6A/6B ~ 75/25. M.p. 165-166 °C (for the mixture of isomers); [Found: C, 61.37; H, 6.94; N, 4.28. $C_{15}H_{19}NO_5$ requires C, 61.42; H, 6.53; N, 4.78%]; R_f (25%) EtOAc/hexane) 0.35; v_{max} (KBr) 3430, 1726, 1641 cm⁻¹; δ_{H} (400 MHz, DMSO-d₆) (6A) 12.06 (1 H, br.s, CO₂H), 6.54 (1 H, d, ${}^{3}J_{10,9}$ 5.6 Hz, H-10), 6.43 (1 H, dd, ${}^{3}J_{9,10}$ 5.6, ${}^{3}J_{9,8}$ 1.0 Hz, H-9), 5.17 (1 H, s, H-10b), 4.97 (1 H, d, ³J_{8,9} 1.0 Hz, H-8), 4.11 (1 H, ddq, ${}^{3}J_{2a,3a}$ 12.4, ${}^{3}J_{2,Me}$ 6.2, ${}^{3}J_{2a,3e}$ 2.4 Hz, H-2), 2.71 (1 H, d, ${}^{3}J_{7,6a}$ 9.1 Hz, H-7), 2.40 (1 H, br.d, ${}^{3}J_{6a,7}$ 9.1 Hz, H-6a), 1.55 (3 H, s, Me-4A), 1.53 (1 H, dd, ${}^{2}J_{3,3}$ 13.3, ${}^{3}J_{3e,2a}$ 2.4 Hz, H-3^{eq}), 1.49 (1 H, dd, ${}^{2}J_{3,3}$ 13.3, ${}^{3}J_{2a,3a}$ 12.4 Hz, H-3^{ax}), 1.29 (3 H, s, Me-4B), 1.17 (3 H, d, ${}^{3}J_{Me,2}$ 6.2 Hz, Me-2); (6B) 12.06 (1 H, br.s, CO₂H), 6.56 (1 H, d, ${}^{3}J_{10,9}$ 5.6 Hz, H-10), 6.40 (1 H, dd, ${}^{3}J_{9,10}$ 5.6, ${}^{3}J_{9,8}$ 1.0 Hz, H-9), 5.44 (1 H, s, H-10b), 4.96 (1 H, d, ${}^{3}J_{8,9}$ 1.0 Hz, H-8), 3.95–3.91 (1 H, m, H-2), 2.62 (1 H, d, ³J_{7.6a} 9.1 Hz, H-7), 2.41 (1 H, d, ${}^{3}J_{6a,7}$ 9.1 Hz, H-6a), 1.51 (3 H, s, Me-4A), 1.47–1.33 (2 H, m, H-3), 1.26 (3 H, s, Me-4B), 1.15 (3 H, d, ³J_{Me,2} 6.1 Hz, Me-2); $\delta_{\rm C}$ (100.6 MHz, DMSO- d_6) (6A) 172.9 and 169.5 (CO₂H and C-6), 136.8 and 133.3 (C-9 and C-10), 88.3 (C-10a), 83.0 and 82.0 (C-8 and C-10b), 69.9 (C-2), 54.0 (C-4), 46.2 (C-3), 49.7 and 44.7 (C-6a and C-7), 28.7 and 24.5 (Me-4 × 2), 21.3 (Me-2). MS

(EI, 70 eV) m/z 293 (23, M⁺), 278 (74), 275 (2), 249 (8), 220 (17), 204 (3), 194 (30), 182 (51), 180 (30), 165 (8), 162 (15), 152 (17), 138 (29), 136 (47), 121 (42), 110 (14), 99 (69), 95 (49), 84 (100), 70 (13), 69 (20), 65 (43), 58 (43), 55 (31), 43 (35), 42 (65), 41 (59), 40 (60), 29 (21), 27 (33%).

The individual isomer **6A** was obtained by crystallization of the isomer mixture from hexane/EtOAc as a colorless powder, m.p. 149-153 °C (decomp.).

Synthesis of azomethines 7.

3-{[1-(2-Furyl)ethylidene]amino}propan-1-ol (7a). A mixture of 2-acetylfurane (5.49 g, 0.05 mol) and 3-aminopropan-1-ol (3.80 mL, 0.05 mol) in PhH (60 mL) was heated under reflux for 2 h with azeotropic removal of water using a Dean-Stark apparatus until the theoretical amount of water (~ 0.9 mL) was collected. The mixture was cooled and activated charcoal (3.0 g) was added to it. The mixture was then heated under reflux for 5 min and filtered. Benzene was removed under reduced pressure. A yellow oil was obtained (7.38 g, 78 %), which contained more than 80% of the target compound 7a (according to ¹H NMR data, the compound was mainly contaminated with aminopropanol). The spectral data of the crude product are provided below. v_{max} (KBr) 3398, 1647 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 7.45 (1 H, br.d, ³*J*_{5',4'} 1.8 Hz, H-5'), 6.73 (1 H, br.d, ³*J*_{3',4'} 3.7 Hz, H-3'), 6.41 (1 H, dd, ${}^{3}J_{3',4'}$ 3.7, ${}^{3}J_{5',4'}$ 1.8 Hz, H-4'), 3.86–3.90 (2 H, m, H-1), 3.63 (2 H, br.t, ³J_{2,3} 6.0 Hz, H-3), 2.15 (3 H, s, Me), 1.94–1.97 (2 H, m, H-2); δ_C (100.6 MHz, CDCl₃) 156.7 and 153.6 (C-1 and C-2'), 143.9 (C-5'), 111.2 and 111.0 (C-3' and C-4'), 62.3 (C-1), 50.0 (C-3), 32.4 (C-2), 14.3 (Me). GC-MS (EI, 70 eV) m/z 167 (1, M⁺), 166 (2), 152 (12), 137 (17), 123 (79), 122 (45), 109 (10), 108 (10), 95 (26), 94 (100), 93 (21), 81 (84), 66 (26), 65 (31), 53 (15), 42 (27), 39 (57%).

3-{[1-(5-Methyl-2-furyl)ethylidene]amino}propan-1-ol (7b) was obtained in a similar way.

Attempted synthesis of 10b-methyl-6-oxo-3,4,6,6a,7,8hexahydro-2*H*-8,10a-epoxy[1,3]oxazino[2,3-*a*]isoindole-

7(10b*H*)- and 8,10b-dimethyl-6-oxo-3,4,6,6a,7,8-hexahydro-2*H*-8,10a-epoxy[1,3]oxazino[2,3-*a*]isoindole-7(10b*H*)carboxylic acids (see Scheme 3).

Maleic anhydride (4.31 g, 44.0 mmol) was added with stirring

and cooling (+10 °C) to a solution of the obtained above azomethine **7a** (7.38 g, 44.0 mmol) in CH₂Cl₂ (50 mL). The solution grew turbid and viscous brown oil formed almost right away. The reaction mixture was stirred at room temperature for 24 h. CH₂Cl₂ was decanted off and the obtained oil was washed with CH₂Cl₂ (2 × 15 mL). Our attempts to induce crystallization of the obtained mixture of products were unsuccessful. Use of PhH (reflux, 2 or 10 h) and Me₂CO as the reaction solvent lead to similar results.

Crystalline products of reaction of 7b with maleic anhydride in the same conditions could not be isolated either.

2-{[1-(5-Methyl-2-furyl)ethylidene]amino}ethanol (**8Ba**). A solution of 5-methylacetylfuran (11.7 mL, 0.10 mol) in MeOH (30 mL) was added to a solution of ethanolamine (6.0 mL, 0.10 mol) in MeOH (30 mL) and the cherry red mixture was stirred at room temperature for 24 h. MeOH was evaporated under reduced pressure, the residue was dissolved in CH₂Cl₂ (30 mL) and purified by column chromatography (3×5 cm) on Al₂O₃ using CH₂Cl₂. After evaporation of the eluent, the viscous brown oil was obtained, which slowly crystallizes at +4 °C. Sticky crystals (7.89 g) containing more than 90% of the target compound (according to ¹H NMR) were obtained by crystallization from a petroleum ether/EtOAc/EtOH mixture. Repeated crystallization from the petroleum ether/EtOAc gives rise to azomethine **8Ba** (4.51)

g, 27 %) as light-brown amorphous crystals, m.p. 61.8–66.4 °C; [Found: C, 64.44; H, 7.94; N, 8.60. C₉H₁₃NO₂ requires C, 64.65; H, 7.84; N, 8.38%]; ν_{max} (KBr) 3137, 1618, 1530 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 6.66 (1 H, d, ${}^{3}J_{3',4'}$ 3.2 Hz, H-3'), 6.03 (1 H, dq, ${}^{3}J_{3',4'}$ 3.2, ${}^{4}J_{\rm Me,4'}$ 0.9 Hz, H-4'), 3.92 (2 H, t, ${}^{3}J_{1,2}$ 5.5 Hz, H-1), 3.56 (2 H, t, ${}^{3}J_{1,2}$ 5.5 Hz, H-2), 2.78 (1 H, br.s, OH), 2.34 (3 H, br.s, Me-5'), 2.13 (3 H, s, Me-C=N); $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 158.1 (C=N), 154.8 and 152.4 (C-2' and C-5'), 113.2 and 107.9 (C-3' and C-4'), 62.5 (O-CH₂), 53.6 (N-CH₂), 15.2 and 14.0 (Me-5' and C(*Me*)=N).

2-[(3aRS,6RS,7aSR)-6-Methyl-3-methylene-1-oxo-1,6,7,7a-

tetrahydro-3a,6-epoxyisoindol-2(3H)-yl]ethyl acrylate (11). A solution of imine (8Ba) (0.50 g, 3.0 mmol), acryloyl chloride (0.36 mL, 4.50 mmol) and NEt₃ (0.83 mL, 6.0 mmol) in PhMe (50 mL) was heated under reflux for 4 h. The reaction mixture was cooled and poured into water (100 mL). The organic layer was separated and the water layer was extracted with EtOAc (3 \times 20 mL). The organic phases were combined and dried (MgSO₄). Pale brown oil was obtained after evaporation of the solvent. ¹H NMR revealed at least four products in the mixture with about 20 % of the target compound 11. The mixture was separated by column chromatography (1.8 \times 12 cm) on Al₂O₃ using hexane and then EtOAc/hexane $(1/20 \rightarrow 1/10)$ mixtures as eluent. First the product of crotonic condensation of 5-methyl-2-acetylfuran and 1,3-bis(5-methyl-2-furyl)but-2-en-1-one was separated (colourless oil, 50 mg, 7%), then epoxyisoindolone 11 (0.12 g, 15 %) was isolated as a vitreous pale oil. [Found: C, 65.37; H, 6.16; N, 5.31. C₁₅H₁₇NO₄ requires C, 65.44; H, 6.22; N, 5.09%]; v_{max} (liquid film) 2974, 2943, 1720, 1656, 1364, 1182, 1063, 990 cm ¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 6.53 (1 H, d, ³J_{5.4} 6.0 Hz, H-5), 6.41 (1 H, dd, ${}^{3}J_{3''trans,2''}$ 17.1, ${}^{2}J_{3'',3''}$ 1.4 Hz, H-3'', trans), 6.31 (1 H, d, ${}^{3}J_{5,4}$ 6.0 Hz, H-4), 6.10 (1 H, d, ${}^{3}J_{3"trans,2"}$ 17.1, ${}^{3}J_{2",3"cis}$ 10.3 Hz, H-2''), 5.85 (1 H, dd, ${}^{3}J_{2'',3''cis}$ 10.3, ${}^{2}J_{3'',3''}$ 1.4 Hz, H-3''^{cis}), 4.75 (1 H, d, ²J 2.3 Hz, C=CH₂A), 4.72 (1 H, d, ²J 2.3 Hz, C=CH₂B), 4.38–4.29 (2 H, m, N-CH₂-CH₂-O), 3.95 (1 H, dt, ${}^{2}J$ 14.7, ${}^{3}J \sim {}^{3}J$ 5.0 Hz, N-CH₂A-CH₂-O), 3.71 (1 H, dt, ${}^{2}J$ 14.7, ${}^{3}J \sim {}^{3}J$ 5.0 Hz, N-CH₂B-CH₂-O), 2.64 (1 H, dd, ³J_{7a,7endo} 9.0, ³J_{7exo,7a} 3.9 Hz, H-7a^{endo}), 1.98 (1 H, dd, ²J_{7,7} 11.7, ³J_{7exo,7a} 3.9 Hz, H-7^{exo}), 1.72 (1 H, dd, ${}^{2}J_{7,7}$ 11.7, ${}^{3}J_{7a,7endo}$ 9.0 Hz, H-7^{endo}), 1.64 (3 H, s, Me-6); δ_{C} (100.6 MHz, CDCl₃) 174.3 (C-1), 165.8 (O-CO-CH=CH₂), 143.2 (C-3), 141.3 (C-4), 132.6 (C-5), 131.2 (O-CO-CH=CH₂), 128.0 (O-CO-CH=CH₂), 90.0 (C₃=CH₂), 89.8 and 88.5 (C-3a and C-6), 60.6 (O-CH₂), 50.7 (C-7a), 39.2 (N-CH₂), 34.1 (C-7), 18.8 (Me-6). GC-MS (EI, 70 eV) m/z 220 (2, M⁺-55), 202 (2), 188 (4), 162 (7), 148 (4), 107 (18), 99 (19), 95 (7), 77 (11), 65 (5), 55 (100), 43 (14%).

Synthesis of compounds 12a, 12Ab/12Bb, 12c, some of their spectroscopic data and description of attempted synthesis of the isoindoles 13a–c.

2-[(2-Furylmethylene)amino]ethanol (12a) was obtained following the general synthetic procedure for compounds **1a–h** (**2a–h**). After MgSO₄ and CH₂Cl₂ were removed from a solution of furfural (0.10 mol) and 2-aminoethanol (0.10 mol), a pale yellow oil (~13 g), which contained ~ 90 % of the target compound, was isolated. The spectral data of the crude product are provided below. v_{max} (liquid film) 3390, 2889, 1647 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 8.07 (1 H, s, CH=N), 7.48 (1 H, br.d, $^{3}J_{5',4'}$ 1.8 Hz, H-5'), 6.72 (1 H, br.d, $^{3}J_{3',4'}$ 3.2 Hz, H-3'), 6.44 (1 H, dd, $^{3}J_{3',4'}$ 3.2, $^{3}J_{5',4'}$ 1.8 Hz, H-4'), 3.88 (2 H, t, $^{3}J_{1,2}$ 5.3 Hz, H-1), 3.68 (2 H, dt, $^{3}J_{1,2}$ 5.3, J 0.9 Hz, H-2), 2.79 (br.s, 1 H, OH); $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 151.6 (C=N), 151.3 (C-2'), 144.9 (C-5'), 114.4 (C-3'), 111.7 (C-4'), 63.6 (O-CH₂), 62.1 (N-CH₂); GC-MS (EI, 70)

eV) *m*/z 139 (8, M⁺), 108 (100), 94 (8), 81 (71), 53 (18), 39 (11%).

2-(2-Furyl)-4,4-dimethyl-1,3-oxazolidine (12Ab) and 2-[(2furylmethylene)amino]-2-methylpropan-1-ol (12Bb). solution of furfural (4.63 mL, 56.0 mmol) and 2-amino-2methylpropan-1-ol (5.40 mL, 56.0 mmol) in PhMe (60 mL) was heated under reflux for 2 h. After PhMe removing a brown quickly crystallizing oil was obtained. Recrystallization from hexane affords a pale brown crystalline compound (6.39 g, 69 %). Mixture of tautomers 12Ab/12Bb in ratio ~ 38/62. [Found: C, 64.64; H, 8.03; N, 8.42 C₉H₁₃NO₂ requires C, 64.65; H, 7.84; N, 8.38%]; $\delta_{\rm H}$ (600 MHz, CDCl₃) (**12Ab**) 7.39 (1 H, br.d, {}^{3}J_{5',4'} 1.4 Hz, H-5'), 6.39 (1 H, br.d, ${}^{3}J_{3',4'}$ 3.4 Hz, H-3'), 6.33 (1 H, dd, ³*J*_{3',4'} 3.4, ³*J*_{5',4'} 1.4 Hz, H-4'), 5.53 (1 H, s, H-2), 3.63 (1 H, d, ²*J*_{5,5} 6.9 Hz, H-5A), 3.50 (1 H, d, ²*J*_{5,5} 6.9 Hz, H-5B), 1.33 (3 H, s, Me-4A), 1.25 (3 H, s, Me-4B); (12Bb) 8.10 (1 H, s, CH=N), 7.50 (1 H, br.d, ${}^{3}J_{5',4'}$ 1.4 Hz, H-5'), 6.73 (1 H, dd, ${}^{3}J_{3',4'}$ 3.4, ${}^{4}J_{3',5'}$ 0.7 Hz, H-3'), 6.46 (1 H, dd, ³J_{3',4'} 3.4, ³J_{5',4'} 1.4 Hz, H-4'), 3.54 (2 H, s, CH₂-OH), 2.36 (1 H, br.s, OH), 1.23 (6 H, s, C(Me)₂); δ_C (100.6 MHz, CDCl₃) (12Ab) 152.2 (C-2'), 147.0 (C-5'), 111.6 (C-3'), 108.2 (C-4'), 85.9 (C-2), 77.2 (C-5), 59.7 (C-4), 26.2 (Me-4A), 25.8 (Me-4B); (12Bb) 151.8 (C-2'), 144.6 (C-5'), 142.7 (C=N), 114.1 (C-3'), 110.3 (C-4'), 71.4 (CH₂-OH), 61.2 (CMe₂), 23.8 (2 C, CMe₂).

2-[(2-Furylmethylene)amino]phenol (12c). A solution of 2aminophenol (10.0 g, 92.0 mmol) and furfural (7.61 mL, 92.0 mmol) in PhH (50 mL) was heated under reflux for 1.5 h with azeotropic removal of water using a Dean-Stark apparatus till the theoretical amount of water (~ 1.8 mL) was isolated. The solvent was evaporated under reduced pressure and the residue - a viscous brown oil - was used in the next step without further purification. The crude product contains ~ 92 % of the target compound according to ¹H NMR. The spectral data are given for this mixture. v_{max} (liquid film) 3394, 1628 cm⁻¹; δ_{H} (400 MHz, CDCl₃) 8.45 (1 H, s, CH=N), 7.60 (1 H, s, OH), 7.25-7.15 (3 H, m, H-3, H-5, H-6), 7.00-6.98 (2 H, m, H-3' and H-5'), 6.87 (1 H, br.t, ${}^{3}J_{3,4} \sim {}^{3}J_{5,4}$ 7.3 Hz, H-4), 6.56 (1 H, dd, ${}^{3}J_{3',4'}$ 3.7, ${}^{3}J_{5',4'}$ 1.8 Hz, H-4'); δ_C (100.6 MHz, CDCl₃) 152.4 (C=N), 146.0 (2C) and 144.8 (C-1, C-2', C-5'), 135.7 (C-2), 129.0 (C-5), 120.2 (C-4), 116.3, 115.8, 115.3 (C-3, C-3', C-6), 112.6 (C-4').

Attempted synthesis of **3,3-dimethyl-5-oxo-2,3,5,5a,6,7**hexahydro-7,9a-epoxy[1,3]oxazolo[2,3-*a*]isoindole-6-

carboxylic acid (13b). Treatment of 12b obtained above with an equimolar amount of maleic anhydride at room temperature in Me_2CO or CH_2Cl_2 (45 mL), or heating under reflux (2 h) in PhMe (50 mL) gives rise to (2*Z*)-4-[(2-hydroxy-1,1-dimethylethyl)amino]-4-oxobut-2-enoic acid (yields 17–38 %). According to ¹H NMR spectra, the reaction mixtures contain the target adduct 13b, but it could not be isolated.

Attempted synthesis of **11-oxo-1,2,11,11a-tetrahydro-2,4a-epoxyisoindolo**[**1,2-b**][**1,3]benzoxazole-1-carboxylic acid** (**13c**). Treatment of the imine **12c** obtained in a similar way with an equimolar amount of maleic anhydride at room temperature in Me₂CO or CH₂Cl₂ affords (2*Z*)-4-[(2-hydroxyphenyl)amino]-4-oxobut-2-enoic acid (**14**) as the main product (yield 52–63 %).

2,9b-Epoxy[1,3]oxazino[2,3-a]oxireno[g]isoindole-3-

carboxylic acids (15). General experimental procedure. Formic acid (4.12 mL, 100 mmol) followed by 30% H_2O_2 (4.30 mL, 150 mmol) were added to a solution of acid **3Aa** or **3Ab** (~ 3.0 g, 10 mmol) in CHCl₃ (40 mL). The clear two-phase mixture was heated under reflux with intensive stirring for 12 h. The crystals formed upon cooling (~ +4 °C) were collected by filtration, washed with H_2O (2 × 30 mL), Et₂O (3 × 7 mL) and dried in air. Diepoxides 15a,b were obtained as well-formed transparent crystals. In some cases, the diepoxide 15 samples contained 2–3 % of the starting acids 3. The latter can be easily removed by crystallization from an EtOH/DMF mixture.

(1aRS,2RS,3RS,3aSR,9bSR,9cRS)-4-Oxooctahydro-6H-2,9bepoxy[1,3]oxazino[2,3-a]oxireno[g]isoindole-3-carboxylic acid (15a). Colourless prisms or white powder (1.25 g, 47 %), m.p. 261.0-262.7 °C (decomp.); [Found: C, 54.08; H, 4.72; N, 5.15. C₁₂H₁₃NO₆ requires C, 53.93; H, 4.90; N, 5.24%]; R_f (50% EtOH/DMF) 0.57; v_{max} (KBr) 1745, 1672 cm⁻¹; δ_{H} (600 MHz, DMSO-d₆) 5.08 (1 H, s, H-9a), 4.63 (1 H, s, H-2), 4.09 (1 H, br.dd, ²J_{8.8} 11.7, ³J_{8e,7a} 4.0 Hz, H-8^{eq}), 3.90–3.84 (2 H, m, H-8^{ax} and H-6^{eq}), 3.62 and 3.56 (1 H and 1 H, two d, ${}^{3}J_{9c,1a}$ 3.5 Hz, H-9c and H-1a), 3.09 (1 H, d, ³J_{3,3a} 9.5 Hz, H-3), 3.05 (1 H, ddd, ²J_{6,6} 13.4, ³J_{6a,7a} 12.1, ³J_{6a,7e} 3.8 Hz, H-6^{ax}), 2.81 (1 H, d, ³J_{3a,3} 9.5 Hz, H-3a), 1.66–1.55 (1 H, m, H-7^{ax}), 1.51–1.47 (1 H, m, H-7^{eq}); δ_{C} (100.6 MHz, DMSO- d_{6}) 171.1 and 169.6 (CO₂H and C-4), 85.9 (C-9b), 84.1 and 78.6 (C-2 and C-9a), 66.5 (C-8), 50.7, 48.5, 47.0, 46.7 (C-1a, C-3, C-3a, C-9c), 38.1 (C-6), 24.8 (C-7). MS (EI, 70 eV) *m*/*z* 267 (50, M⁺), 260 (1), 252 (2), 250 (3), 239 (3), 238 (12), 224 (30), 223 (46), 208 (18), 194 (76), 180 (36), 167 (21), 166 (34), 164 (41), 152 (55), 138 (48), 137 (56), 125 (30), 123 (48), 122 (56), 111 (46), 110 (80), 99 (73), 87 (26), 86 (58), 85 (77), 84 (87), 71 (26), 69 (41), 66 (45), 57 (37), 56 (94), 55 (100), 45 (26), 43 (76%).

(1aRS,2SR,3SR,3aRS,9bRS,9cSR)-2-Methyl-4-oxooctahydro-6H-2,9b-epoxy[1,3]oxazino[2,3-*a*]oxireno[*g*]isoindole-3-

carboxylic acid (15b). Thick colourless plates (1.31 g, 47 %), m.p. 252.1-253.6 °C (decomp); [Found: C, 55.44; H, 5.29; N, 4.91. C₁₃H₁₅NO₆ requires C, 55.51; H, 5.38; N, 4.98%]; R_f (50% EtOH/DMF) 0.73; v_{max} (KBr) 3533, 1732, 1683 cm⁻¹; δ_{H} (400 MHz, DMSO-d₆) 12.33 (1 H, br.s, CO₂H), 5.05 (1 H, s, H-9a), 4.09 (1 H, br.dd, ${}^{2}J_{8,8}$ 11.4, ${}^{3}J_{8e,7a}$ 4.4 Hz, H-8^{eq}), 3.91–3.85 (2 H, m, H-8^{ax} and H-6^{eq}), 3.67 and 3.51 (1 H and 1 H, two d, ${}^{3}J_{9c,1a}$ 3.5 Hz, H-9c and H-1a), 3.05 and 2.80 (1 H and 1 H, two d, ${}^{3}J_{3,3a}$ 9.5 Hz, H-3 and H-3a), 3.07 (1 H, br.dt, ${}^{2}J_{6,6} \sim {}^{3}J_{6a,7a} \sim 13.4$, ${}^{3}J_{6a,7e} 3.8$ Hz, H-6^{ax}), 1.60–1.49 (1 H, m, H-7^{ax}), 1.43–1.41 (1 H, m, ${}^{2}J_{7,7}$ 13.2 Hz, H-7^{eq}), 1.32 (3 H, br.s, Me-2); ¹³C NMR (D₂O/NaOD (5%), 100.6 MHz) δ 173.6 and 171.7 (CO₂H and C-4), 83.3 (C-9a), 84.7 and 83.7 (C-2 and C-9b), 65.7 (C-8), 51.6, 50.9, 50.0, 47.7 (C-1a, C-3, C-3a, C-9c), 36.9 (C-6), 22.5 (C-7), 11.2 (Me-2). δ_C (100.6 MHz, DMSO-*d*₆) 170.2 and 169.9 (CO₂H and C-4), 85.7 and 85.6 (C-2 and C-9b), 84.4 (C-9a), 66.6 (C-8), 52.1, 51.7, 50.4, 48.8 (C-1a, C-3, C-3a, C-9c), 38.1 (C-6), 24.9 (C-7), 13.6 (Me-2). MS (EI, 70 eV) m/z 281 (12, M⁺), 280 (9), 265 (1), 263 (6), 252 (9), 246 (34), 238 (41), 235 (11), 234 (14), 221 (24), 220 (56), 210 (22), 208 (52), 207 (17), 206 (19), 195 (29), 194 (41), 192 (44), 182 (13), 181 (54), 180 (70), 168 (37), 151 (67), 150 (59), 140 (57), 138 (36), 136 (35), 135 (17), 125 (44), 124 (66), 123 (76), 113 (10), 111 (23), 110 (22), 109 (41), 108 (49), 101 (13), 99 (46), 95 (54), 87 (13), 86 (77), 79 (41), 71 (13), 70 (18), 69 (41), 59 (54), 58 (100), 56 (91), 55 (86), 53 (62), 45 (34), 44 (51), 43 (54), 41 (56%).

6-Oxo-3,4,6,10b-tetrahydro-2H-[1,3]oxazino[2,3-a]isoindole-

7-carboxylic acid (16). A solution of oxazino[2,3-*a*]isoindole carboxylic acid **3Aa** (1.01 g, 3.98 mmol) in 10% NaOH (15 mL) was heated under reflux for 2 h. After cooling, HCl (conc.) was added to the solution until it was slightly acidic. Crystals formed were collected by filtration, washed with water (2×20 mL) and Et₂O (2×10 mL) and dried in air until constant weight. The title acid **16** was obtained as a beige powder (0.12 g, 18 %), m.p. 169.2–172.2 °C; [Found: C, 61.68; H, 4.55; N, 5.97. C₁₂H₁₁NO₄ requires C, 61.80; H, 4.75; N, 6.01%]; v_{max} (KBr) 3518, 1716,

1622 cm⁻¹; $\delta_{\rm H}$ (600 MHz, DMSO- d_6) 8.09 (1 H, d, ${}^3J_{8,9}$ 7.6 Hz, H-8), 7.84 (1 H, d, ${}^3J_{10,9}$ 7.6 Hz, H-10), 7.79 (1 H, br.t, ${}^3J_{8,9} \sim {}^3J_{9,10}$ 7.6 Hz, H-9), 5.82 (1 H, s, H-10b), 4.24 (1 H, br.dd, ${}^2J_{2,2}$ 12.7, ${}^3J_{2e,3a}$ 4.2 Hz, H-2^{eq}), 4.13 (1 H, br.dd, ${}^2J_{4,4}$ 11.7, ${}^3J_{4e,3a}$ 2.8 Hz, H-4^{eq}), 4.00 (1 H, dt, ${}^2J_{4,4} \sim {}^3J_{4a,3a}$ 11.7, ${}^3J_{4a,3e}$ 2.8 Hz, H-4^{ax}), 3.44 (1 H, dt, ${}^2J_{2,2} \sim {}^3J_{2a,3a}$ 12.7, ${}^3J_{2a,3e}$ 4.8 Hz, H-2^{ax}), 1.72–1.68 (2 H, m, H-3); $\delta_{\rm C}$ (100.6 MHz, DMSO- d_6) 165.7 and 164.9 (CO₂H and C-6), 142.3 (C-10a), 132.8, 132.6, 127.4 (C-8, C-9, C-10), 129.1 (C-6a), 121.0 (C-7), 84.6 (C-10b), 66.4 (C-2), 38.1 (C-4), 24.0 (C-3). MS (EI, 70 eV) *m*/*z* 233 (8, M⁺), 232 (41), 216 (2), 214 (11), 204 (32), 203 (43), 190 (48), 189 (100), 175 (76), 160 (52), 157 (56), 149 (18), 130 (46), 105 (57), 104 (96), 103 (74), 77 (63), 76 (69), 75 (71), 63 (31), 59 (94), 53 (30), 43 (71%).

Esters of 8,10a-epoxy[1,3]oxazino[2,3-*a*]isoindole-7carboxylic acids (17a–e). General experimental procedure. A solution of the appropriate acid 3Aa–d (0.05 mol) and a catalytic amount of sulfuric acid (ca. 0.5 mL) in MeOH or abs. EtOH (150 mL) was heated under reflux for 5–25 h (the acid dissolves completely). The solvent was then evaporated under reduced pressure and the residue was purified by aluminum oxide column chromatography (with Et₂O and then EtOAc as eluent). Esters 17a–e were obtained as colorless or pale yellow crystals after crystallization from MeOH or EtOH.

Methyl (6aRS,7SR,8RS,10aSR,10bRS)-6-oxo-3,4,6,6a,7,8hexahydro-2H-8,10a-epoxy[1,3]oxazino[2,3-a]isoindole-7-

carboxylate (17a). Colorless rhombic crystals (6.89 g, 52 %), m.p. 169-170 °C (MeOH); [Found: C, 58.63; H, 5.57; N, 5.16. C₁₃H₁₅NO₅ requires C, 58.86; H, 5.70; N, 5.28%]; R_f (EtOAc) 0.62; v_{max} (KBr) 1742, 1699 cm⁻¹; δ_{H} (400 MHz, DMSO- d_6) 6.62 $(1 \text{ H}, \text{ d}, {}^{3}J_{10,9} 5.8 \text{ Hz}, \text{H-10}), 6.48 (1 \text{ H}, \text{ dd}, {}^{3}J_{9,10} 5.8, {}^{3}J_{9,8} 1.7 \text{ Hz},$ H-9), 5.14 (1 H, s, H-10b), 5.12 (1 H, d, ³J_{8.9} 1.7 Hz, H-8), 4.12 (1 H, br.dd, ${}^{2}J_{2,2}$ 11.4, ${}^{3}J_{2e,3e}$ 4.4 Hz, H-2^{eq}), 3.94–3.87 (2 H, m, H-2^{ax} and H-4^{eq}), 3.57 (3 H, s, OMe), 3.10 (1 H, dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 12.7, ³J_{4a,3e} 3.6 Hz, H-4^{ax}), 2.82 (1 H, br.d, ³J_{7,6a} 9.1 Hz, H-7), 2.67 (1 H, d, ${}^{3}J_{6a,7}$ 9.1 Hz, H-6a), 1.69–1.58 (1 H, m, H-3^{ax}), 1.52–1.48 (1 H, m, H-3^{eq}); δ_{C} (100.6 MHz, DMSO- d_{6}) 171.6 (CO₂Me), 170.4 (C-6), 136.6 (C-10), 133.6 (C-9), 89.4 (C-10a), 84.9 (C-10b), 81.8 (C-8), 66.7 (C-2), 51.3 (C-6a), 49.3 (CO₂Me), 43.7 (C-7), 38.1 (C-4), 25.0 (C-3). MS (EI, 70 eV) m/z 265 (12, M^+), 250 (1), 237 (3), 234 (6), 206 (23), 153 (9), 152 (100), 138 (8), 124 (19), 121 (27), 113 (19), 95 (22), 86 (30), 85 (9), 65 (6), 59 (5), 56 (5%).

Methyl (6aRS,7SR,8RS,10aSR,10bRS)-8-methyl-6-oxo-3,4,6,6a,7,8-hexahydro-2*H*-8,10a-epoxy[1,3]oxazino[2,3-

alisoindole-7-carboxylate (17b). Colorless prisms (6.42 g, 46 %), m.p. 177-179 °C (MeOH); [Found: C, 60.31; H, 6.07; N, 5.28. C14H17NO5 requires C, 60.21; H, 6.14; N, 5.02%]; Rf (EtOAc) 0.67; v_{max} (KBr) 1744, 1697 cm⁻¹; δ_{H} (400 MHz, CDCl₃) 6.70 (1 H, br.d, ³J_{9,10} 5.4 Hz, H-9), 6.22 (1 H, d, ³J_{10,9} 5.4 Hz, H-10), 5.15 (1 H, s, H-10b), 4.24-4.21 (2 H, m, H-2^{eq} and H-4^{eq}), 3.87 (1 H, br.t, ${}^{2}J_{2,2} \sim {}^{3}J_{2a,3a}$ 12.1 Hz, H-2^{ax}), 3.75 (3 H, s, OMe), 3.11 (1 H, dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 12.1, ${}^{3}J_{4a,3e}$ 2.8 Hz, H-4^{ax}), 2.85 (1 H, d, ³*J*_{7.6a} 8.9 Hz, H-7), 2.78 (1 H, d, ³*J*_{6a,7} 8.9 Hz, H-6a), 1.95–1.85 (1 H, m, H-3^{ax}), 1.62 (3 H, s, Me-8), 1.51–1.49 (1 H, m, H-3^{eq}); δ_{C} (100.6 MHz, DMSO-d₆) 170.5 and 170.4 (CO₂Me and C-6), 139.5 and 134.5 (C-9 and C-10), 89.6 and 89.0 (C-8 and C-10a), 85.1 (C-10b), 66.7 (C-2), 52.5, 51.1, 47.2 (CO₂Me, C-6a, C-7), 38.1 (C-4), 24.9 (C-3), 15.4 (Me-8). MS (EI, 70 eV) m/z 279 (54, M⁺), 261 (8), 251 (7), 249 (8), 248 (40), 247 (16), 236 (9), 220 (35), 219 (38), 204 (26), 194 (8), 190 (10), 176 (21), 167 (29), 166 (79), 150 (10), 139 (24), 138 (84), 137 (13), 136 (36), 135 (77), 124 (24), 123 (15), 122 (30), 114 (42), 113 (42), 110 (45), 109 (38), 108 (100), 107 (35), 101 (12), 99 (13), 95 (26), 94 (16),

87 (25), 86 (85), 85 (62), 81 (23), 80 (34), 67 (13), 59 (77), 57 (29), 56 (76), 55 (41), 54 (23), 53 (48), 51 (17), 44 (21), 43 (35), 42 (33), 41 (22%).

Methyl (6aRS,7RS,8SR,10aSR,10bRS)-8-bromo-6-oxo-3,4,6,6a,7,8-hexahydro-2*H*-8,10a-epoxy[1,3]oxazino[2,3-

a]isoindole-7(10bH)-carboxylate (17c). Fine yellow powder (7.88 g, 46 %), m.p. 201-202 °C (decomp., from MeOH); [Found: C, 45.39; H, 4.13; N, 4.04. C₁₃H₁₄BrNO₅ requires C, 45.37; H, 4.10; N, 4.07%]; R_f (65% EtOAc/hexane) 0.52; v_{max} (KBr) 1754, 1706 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 6.75 (1 H, d, ${}^{3}J_{9,10}$ 5.5 Hz, H-9), 6.44 (1 H, d, ${}^{3}J_{10,9}$ 5.5 Hz, H-10), 5.17 (1 H, s, H-10b), 4.22 (1 H, ddd, ${}^{2}J_{2,2}$ 13.1, ${}^{3}J_{2e,3a}$ 5.0, ${}^{3}J_{2e,3e}$ 1.2 Hz, H-2^{eq}), 4.22 (1 H, ddd, ${}^{2}J_{4,4}$ 13.0, ${}^{3}J_{4e,3a}$ 5.0, ${}^{3}J_{4e,3e}$ 1.2 Hz, H-4^{eq}), 3.88 (1 H, dt, ${}^{2}J_{2,2} \sim {}^{3}J_{2a,3a}$ 13.1, ${}^{3}J_{2a,3e}$ 2.7 Hz, H-2^{ax}), 3.80 (3 H, s, OMe), 3.19 (1 H, d, ${}^{3}J_{7,6a}$ 8.7 Hz, H-7), 3.13 (1 H, ddd, ${}^{3}J_{4a,3a}$ 14.3, ${}^{2}J_{4,4}$ 13.0, ³J_{4a,3e} 4.1 Hz, H-4^{ax}), 2.92 (1 H, d, ³J_{6a,7} 8.7 Hz, H-6a), 1.93–1.85 (1 H, m, H- 3^{ax}), 1.54–1.52 (1 H, m, H- 3^{eq}); δ_C (150.9 MHz, CDCl₃) 170.0 and 168.9 (CO₂Me and C-6), 140.0 and 135.8 (C-9 and C-10), 90.1 and 88.5 (C-8 and C-10a), 85.4 (C-10b), 67.9 (C-2), 52.6, 52.5, 51.5 (CO₂Me, C-6a, C-7), 39.1 (C-4), 25.2 (C-3). GC-MS (EI, 70 eV) *m/z* 345 (3, M⁺, for Br⁸¹), 343 (4), 317 (4), 312 (5), 284 (19), 264 (24), 232 (16), 230 (36), 202 (29), 198 (34), 199 (35), 176 (22), 175 (28), 173 (21), 151 (71), 145 (23), 119 (20), 113 (66), 86 (100), 85 (56), 65 (44), 63 (49), 59 (52), 56 (68), 51 (16), 39 (43%).

Methyl (6aRS,7RS,8SR,10aSR,10bRS)-8-iodo-6-oxo-3,4,6,6a,7,8-hexahydro-2H-8,10a-epoxy[1,3]oxazino[2,3-

a]isoindole-7(10bH)-carboxylate (17d). Yellow powder (9.80 g, 50 %), m.p. 179-181 °C (decomp., from MeOH); [Found: C, 39.82; H, 3.57; N, 3.83. C₁₃H₁₄INO₅ requires C, 39.92; H, 3.61; N, 3.58%]; R_f (65% EtOAc/hexane) 0.67; v_{max} (KBr) 1749, 1684 cm^{-1} ; δ_{H} (400 MHz, DMSO- d_{6}) 6.57 and 6.51 (1 H and 1 H, two d, ${}^{3}J_{910}$ 5.5 Hz, H-9 and H-10), 5.14 (1 H, s, H-10b), 4.23–4.16 (2 H, m, H-2^{eq} and H-4^{eq}), 3.86 (1 H, dt, ${}^{2}J_{2,2} \sim {}^{3}J_{2a,3a}$ 12.4, ${}^{3}J_{2a,3e}$ 2.3 Hz, H- 2^{ax}), 3.79 (3 H, s, OMe), 3.12 (1 H, dd, J 0.9, ${}^{3}J_{7,6a}$ 8.7 Hz, H-7), 3.10 (1 H, br.dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 12.8, ${}^{3}J_{2a,3e}$ 3.7 Hz, H-4^{ax}), 2.86 (1 H, d, ³J_{6a,7} 8.7 Hz, H-6a), 1.92–1.80 (1 H, m, H-3^{ax}), 1.51 (1 H, br.d, ${}^{3}J_{3,3}$ 14.7 Hz, H-3^{eq}); δ_{C} (100.6 MHz, DMSO- d_{6}) 169.8 and 169.3 (CO2Me and C-6), 143.0 and 135.0 (C-9 and C-10), 89.3 (C-10a), 85.0 (C-10b), 67.7 (C-2), 64.6 (C-8), 53.3, 52.2, 51.6 (C-6a, C-7, CO₂Me), 38.9 (C-4), 25.1 (C-3); MS (EI, 70 eV) m/z 391 (14, M⁺), 390 (2), 359 (3), 333 (75), 332 (20), 331 (6), 278 (32), 277 (51), 250 (16), 247 (37), 220 (17), 151 (24), 127 (27), 113 (30), 92 (19), 86 (59), 85 (50), 63 (46), 59 (68), 56 (100), 55 (22), 54 (15%).

Ethyl (6aRS,7SR,8RS,10aSR,10bRS)-6-oxo-3,4,6,6a,7,8hexahydro-2H-8,10a-epoxy[1,3]oxazino[2,3-*a*]isoindole-7-

carboxylate (17e). Pale yellow powder (14.1 g, 72 %), m.p. 129–130 °C (from EtOH); [Found: C, 60.14; H, 6.32; N, 5.27. C₁₄H₁₇NO₅ requires C, 60.21; H, 6.14; N, 5.02%]; R_f (EtOAc) 0.72; v_{max} (KBr) 1733, 1680 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 6.70 (1 H, d, ${}^{3}J_{0.9}$ 5.8 Hz, H-10), 6.43 (1 H, dd, ${}^{3}J_{9.10}$ 5.8, ${}^{3}J_{9.8}$ 1.6 Hz, H-9), 5.20 (1 H, d, ${}^{3}J_{8.9}$ 1.6 Hz, H-8), 5.15 (1 H, s, H-10b), 4.26–4.16 (2 H, m, H-2^{eq} and H-4^{eq}), 4.22 (2 H, q, ${}^{3}J_{CH2,Me}$ 7.1 Hz, CH₂CH₃), 3.88 (1 H, dt, ${}^{2}J_{2.2} \sim {}^{3}J_{2a,3a}$ 12.6, ${}^{3}J_{2a,3e}$ 2.1 Hz, H-2^{ax}), 3.10 (1 H, dt, ${}^{2}J_{4.4} \sim {}^{3}J_{4a,3e}$ 12.6, ${}^{3}J_{4a,3e}$ 3.5 Hz, H-4^{ax}), 2.82 and 2.75 (1 H and 1 H, two d, ${}^{3}J_{7,6a}$ 9.1 Hz, H-6a and H-7), 1.95–1.87 (1 H, m, H-3^{ax}), 1.55–1.51 (1 H, m, H-3^{eq}), 1.31 (3 H, t, ${}^{3}J_{CH2,Me}$ 7.1 Hz, CH₂CH₃); $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 171.2 and 170.8 (CO₂Me and C-6), 135.6 and 134.2 (C-10 and C-9), 89.6 (C-10a), 85.8 (C-10b), 82.4 (C-8), 67.5 (C-2), 60.9 (OCH₂CH₃), 49.8 (C-6a), 44.2 (C-7), 38.8 (C-4), 25.1 (C-3), 14.0 (OCH₂CH₃); MS (EI, 70 eV) *m*/z 279 (9, M⁺), 234 (7), 206 (26), 202 (3), 166 (13), 153

(6), 152 (100), 138 (27), 124 (15), 121 (35), 110 (6), 100 (5), 99 (34), 96 (11), 95 (36), 94 (9), 86 (40), 85 (5), 65 (8), 56 (8), 44 (13), 39 (11), 29 (17), 27 (16), 26 (14%).

Methvl (6aRS,7SR,8SR,9RS,10RS,10aSR,10bRS)-9,10dibromo-6-oxooctahydro-2H-8,10a-epoxy[1,3]oxazino[2,3alisoindole-7-carboxylate (18). A solution of methyl ester 17a (0.51 g, 2.00 mmol) and $Me_2N^+HCOMe \cdot Br_3 \cdot Me_2NCOMe$ (0.80 g, 2.00 mmol) in CHCl₃ (10 mL) was heated under reflux for 12 h. The residue obtained after evaporation of the solvent was purified by silica gel column chromatography $(1.2 \times 10 \text{ cm})$ eluent: EtOAc/hexane, 1/2). The target dibromide 18 was isolated as a white powder (0.45 g, 56 %). Analytical sample was obtained by crystallization from a hexane/EtOAc mixture as fine transparent needles (elongated prisms), m.p. 181.1-182.0 °C; [Found: C, 36.52; H, 3.28; N, 3.45; Hal, 37.91. C₁₃H₁₅Br₂NO₅ requires C, 36.73; H, 3.56; N, 3.30; Br 37.60%]; R_f (25% EtOAc/hexane) 0.73; v_{max} (KBr) 1733 (br.) cm⁻¹; δ_{H} (600 MHz, CDCl₃) 5.00 (1 H, s, H-10b), 4.81 (1 H, d, ³J_{10,9} 5.4 Hz, H-10), 4.44 (1 H, dd, ³J_{9,10} 5.4, ³J_{9,8} 2.2 Hz, H-9), 4.14 (1 H, d, ³J_{8,9} 2.2 Hz, H-8), 4.14–4.10 (2 H, m, H-2^{eq} and H-4^{eq}), 3.82 (1 H, d, ${}^{3}J_{7,6a}$ 9.6 Hz, H-6a), 3.77 (1 H, ddd, ²J_{2,2} 13.1, ³J_{2a,3a} 12.5, ³J_{2a,3e} 2.3 Hz, H-2^{ax}), 3.67 (3 H, s, CO₂Me), 3.09 (1 H, d, ${}^{3}J_{6a,7}$ 9.6 Hz, H-7), 3.04 (1 H, ddd, ${}^{2}J_{4,4}$ 13.3, ${}^{3}J_{4a,3a}$ 12.5, ${}^{3}J_{4a,3e}$ 3.8 Hz, H-4^{ax}), 1.81–1.73 (1 H, m, H-3^{ax}), 1.43–1.41 (1 H, m, H-3^{eq}); δ_{C} (100.6 MHz, CDCl₃) 170.4 and 170.2 (CO₂Me and C-6), 90.2 (C-10a), 86.0 and 83.4 (C-8 and C-10b), 67.6 (C-2), 53.0 and 52.4 (C-9 and C-10), 52.7 (CO₂Me), 50.6 (C-7), 46.1 (C-6a), 39.3 (C-4), 25.0 (C-3); GC-MS (EI, 70 eV) *m/z* 427 (1, M⁺ for Br⁸¹), 425 (2), 423 (1), 396 (1), 394 (2), 392 (1), 346 (18), 344 (18), 244 (2), 242 (3), 121 (3), 113 (2), 95 (2), 86 (3), 85 (5), 84 (4), 65 (6), 59 (100), 56 (14), 55 (6), 54 (3), 42 (4), 41 (9), 39 (5%). Methyl (3RS,3aSR,6RS,7SR,7aRS)-3-(acetyloxy)-2-[3-

Methyl (3KS,3aSK,6KS,7SK,7aKS)-3-(acetyloxy)-2-[3-(acetyloxy)propyl]-1-oxo-1,2,3,6,7,7a-hexahydro-3a,6-

epoxvisoindole-7-carboxvlate (19). BF₃·OEt₂ (1.42 mL, 11.3 mmol) was added to a cooled (-5 °C) solution of methyl ester 17a (1.00 g, 3.80 mmol) in Ac₂O (10 mL). The reaction mixture was stirred at room temperature for 24 h (TLC monitoring), poured into water (100 mL). A saturated sodium carbonate solution was added to the mixture until it was slightly basic. The mixture was extracted with CHCl₃ (3 \times 50 mL) and the combined organic phases were dried (MgSO₄). The residue obtained after evaporation of the solvent was purified by silica gel column chromatography (3 \times 10 cm, eluent: EtOAc/hexane, 1/2). The target product 19 was isolated as colorless rhombus-shaped crystals (0.17 g, 12 %), m.p. 74-76 °C; [Found: C, 55.35; H, 5.82; N, 3.77. C₁₇H₂₁NO₈ requires C, 55.58; H, 5.76; N, 3.81%]; R_{f} (EtOAc) 0.85; v_{max} (KBr) 1724, 1707 cm⁻¹; δ_{H} (600 MHz, CDCl₃) 6.48 (1 H, d, ${}^{3}J_{4,5}$ 5.9 Hz, H-4), 6.43 (1 H, dd, ${}^{3}J_{5,4}$ 5.9, ³*J*_{5,6} 1.7 Hz, H-5), 6.39 (1 H, s, H-3), 5.19 (1 H, d, ³*J*_{6,5} 1.7 Hz, H-6), 4.04 (2 H, t, ³J_{2',3'} 6.4 Hz, H-3'), 3.73 (3 H, s, CO₂Me), 3.54 (1 H, dt, ${}^{2}J_{1',1'}$ 14.2, ${}^{3}J_{1A',2'}$ 7.2 Hz, H-1'A), 3.29 (1 H, dt, ${}^{2}J_{1',1'}$ 14.2, ³*J*_{1B',2'} 7.2 Hz, H-1'B), 2.89 (1 H, d, ³*J*_{7,7a} 9.1 Hz, H-7), 2.73 $(1 \text{ H}, d, {}^{3}J_{7a,7} 9.1 \text{ Hz}, \text{H-7a}), 2.17 (3 \text{ H}, \text{s}, \text{OCOMe}), 2.03 (3 \text{ H}, \text{s}, \text{s})$ OCOMe), 1.94–1.81 (2 H, m, H-2'); δ_C (100.6 MHz, CDCl₃) 172.3, 171.6, 170.9, 169.9 (OCOMe × 2, CO₂Me, C-1), 136.5 and 133.4 (C-4 and C-5), 90.1 (C-3a), 82.4 and 81.6 (C-3 and C-6), 61.4 (C-3'), 52.1 (CO₂Me), 48.6 and 44.9 (C-7a and C-7), 39.0 (C-1'), 26.8 (C-2'), 20.8 (OCOMe × 2); MS (EI, 70 eV) m/z 367 (8, M⁺), 350 (1), 325 (3), 309 (6), 308 (14), 307 (19), 281 (5), 265 (15), 248 (14), 232 (7), 230 (12), 212 (38), 211 (46), 198 (15), 197 (17), 196 (99), 170 (61), 169 (70), 154 (10), 152 (44), 139 (44), 135 (57), 134 (73), 124 (27), 122 (38), 114 (43), 113

(82), 101 (94), 97 (100), 86 (14), 85 (19), 82 (29), 81 (37), 73 (25), 66 (17), 65 (26), 59 (40), 56 (54), 45 (43), 43 (85%).

6b,9-Epoxyisoindolo[2,1-a][3,1]benzoxazine-10-carboxylic

acids (20a–c). General experimental procedure. Method A (thermal). A mixture of (2-aminophenyl)methanol (10.0 g, 80.0 mmol) and a suitable furfural (80.0 mmol) was heated under reflux in PhH (60 mL) with azeotropic removal of water using a Dean-Stark apparatus (1–2 h). After the theoretical amount of water (~ 1.4 mL) was collected, the mixture was cooled and crude 2-furanyl-1,4-dihydro-2*H*-3,1-benzoxazine was used in the next step without further purification, assuming quantitative yield. According to ¹H NMR, in the case of 2-(furan-2-yl)-2,4-dihydro-1*H*-benzo[*d*][1,3]oxazine, the ratio of the ring/chain tautomeric forms (formed as viscous brown oil after solvent evaporation) in the mixture was ~ 78/22.

A solution of maleic anhydride (8.82 g, 0.09 mol) in PhH (50 mL) was added to a solution of the freshly prepared benzoxazine (0.08 mol) in PhH (~ 50 mL). The mixture was heated under reflux for 2 h and then cooled. Benzene was decanted from the viscous brown oil. The precipitate formed upon trituration of the residue with Me₂CO (40 mL) was collected by filtration, washed with acetone (2 × 30 mL), dried in air and further purified if necessary by recrystallization from *i*-PrOH/DMF mixtures. A white powder (6.45 g, 27 %) was obtained in the case of the synthesis of the acid **20a**. The **20Aa/20Ba** isomeric ratio was ~ 96/4.

Method B (ambient temperature) presented for the synthesis of the acid 20a, as an example (gives slightly lower yields of adducts 20, but higher diastereoselectivity). Anhydrous powdered MgSO₄ (12.0 g, 100 mmol) was added to a solution of (2-aminophenyl)methanol (5.0 g, 41 mmol) and furfural (3.4 mL, 41 mmol) in CH₂Cl₂ (80 mL). The mixture was stirred at room temperature for 24 h. MgSO₄ was then filtered off and washed with CH₂Cl₂ (2 × 40 mL). The organic phases were combined and concentrated under reduced pressure to give 2-(furan-2-yl)-2,4-dihydro-1H-benzo[d][1,3]oxazine as a yellow oil. The oxazinane was used in the next step without further purification, assuming quantitative yield.

Maleic anhydride (4.47 g, 45.0 mmol) was added to a solution of the 2-furyloxazine (41 mmol) in Me₂CO (60 mL). The mixture was stirred at 24 °C for 2 d. The yellowish precipitate formed was collected by filtration, washed with Me₂CO (2×15 mL), Et₂O (20 mL) and dried in air to give the acid **20Aa** as a light yellow powder (2.8 g, 23 %).

Other acids (20b,c) were obtained following *Method A*.

(6aRS,6bSR,9RS,10SR,10aRS)-11-Oxo-9,10,10a,11-

tetrahydro-5*H*-6b,9-epoxyisoindolo[2,1-*a*][3,1]benzoxazine-10(6*aH*)-carboxylic acid (20Aa) and

(6aRS,6bRS,9SR,10RS,10aSR)-11-oxo-9,10,10a,11-

tetrahydro-5H-6b,9-epoxyisoindolo[2,1-a][3,1]benzoxazine-

10(6*aH***)-carboxylic acid (20Ba)**. The adduct yields and the ratio of the isomers in the samples obtained following methods A and B are given above. The single isomer **20Aa** was isolated by crystallization of the isomer mixture from *i*-PrOH/DMF, m.p. 197–202 °C (decomp.); [Found: C, 64.49; H, 4.29; N, 4.31. C₁₆H₁₃NO₅ requires C, 64.21; H, 4.38; N, 4.68%]; R_f (50% EtOH/DMF) 0.46; v_{max} (KBr) 1710 cm⁻¹; δ_{H} (400 MHz, DMSO*d*₆) (**20Aa**) 8.36 (1 H, dd, ${}^{3}J_{1,2}$ 7.5, ${}^{4}J_{1,3}$ 1.0 Hz, H-1), 7.25 (1 H, dt, ${}^{3}J_{1,2} \sim {}^{3}J_{2,3}$ 7.5, ${}^{4}J_{2,4}$ 1.0 Hz, H-2), 7.17 (1 H, br.d, ${}^{3}J_{4,3}$ 7.5 Hz, H-4), 7.08 (1 H, dt, ${}^{3}J_{3,2} \sim {}^{3}J_{3,4}$ 7.5, ${}^{4}J_{3,1}$ 1.0 Hz, H-3), 6.64 (1 H, d, ${}^{3}J_{7,8}$ 5.7 Hz, H-7), 6.51 (1 H, dd, ${}^{3}J_{8,7}$ 5.7, ${}^{3}J_{8,9}$ 1.7 Hz, H-8), 5.93 (1 H, s, H-6a), 5.11 (1 H, d, ${}^{2}J_{5,5}$ 15.2 Hz, H-5A), 5.08 (1 H, d, ${}^{3}J_{9,8}$ 1.7 Hz, H-9), 4.96 (1 H, d, ${}^{2}J_{5,5}$ 15.2 Hz, H-5B), 3.13 (1 H, d, ${}^{3}J_{10a,10}$ 9.1 Hz, H-10a), 2.56 (1 H, d, ${}^{3}J_{10,10a}$ 9.1 Hz, H-10); (20Ba) 7.68 (1 H, dd, ${}^{3}J_{1,2}$ 7.5, ${}^{4}J_{1,3}$ 1.0 Hz, H-1), 7.25 (1 H, dt, ${}^{3}J_{1,2} \sim {}^{3}J_{2,3}$ 7.5, ${}^{4}J_{2,4}$ 1.0 Hz, H-2), 7.17 (1 H, br.d, ${}^{3}J_{4,3}$ 7.5 Hz, H-4), 7.08 (1 H, dt, ${}^{3}J_{3,2} \sim {}^{3}J_{3,4}$ 7.5, ${}^{4}J_{3,1}$ 1.0 Hz, H-3), 6.74 (1 H, d, ³*J*_{7,8} 5.7 Hz, H-7), 6.55 (1 H, dd, ³*J*_{8,7} 5.7, ³*J*_{8,9} 1.7 Hz, H-8), 5.47 (1 H, s, H-6a), 5.23 (1 H, d, ${}^{2}J_{5,5}$ 15.1 Hz, H-5A), 5.19 (1 H, d, ³*J*_{9,8} 1.7 Hz, H-9), 5.06 (1 H, d, ²*J*_{5,5} 15.1 Hz, H-5B), 3.05 (1 H, d, ${}^{3}J_{10a,10}$ 9.1 Hz, H-10a), 2.64 (1 H, d, ${}^{3}J_{10,10a}$ 9.1 Hz, H-10); δ_{C} (150.9 MHz, CDCl₃) (**20Aa**) 172.6 and 168.4 (CO₂H and C-11), 138.0 (C-7), 134.7 (C-12a), 134.3 (C-8), 128.0, 125.4, 123.9 (C-2, C-3, C-4), 123.5 (C-4a), 117.9 (C-1), 89.2 (C-6b), 83.6 (C-6a), 81.7 (C-9), 68.0 (C-5), 52.0 (C-10a), 45.3 (C-10); MS (EI, 70 eV) m/z 299 (38, M⁺), 281 (2), 255 (3), 237 (6), 201 (47), 200 (100), 172 (16), 159 (30), 158 (14), 131 (15), 132 (50), 130 (16), 117 (10), 106 (14), 105 (34), 104 (40), 95 (18), 91 (23), 78 (30), 77 (32), 54 (13), 51 (14), 44 (19), 39 (16), 28 (26), 26 (15%). (6aRS,6bSR,9RS,10SR,10aRS)-9-Methyl-11-oxo-9,10,10a,11tetrahydro-5H-6b,9-epoxyisoindolo[2,1-a][3,1]benzoxazine-10(6aH)-carboxylic acid (20Ab) (6aRS,6bRS,9SR,10RS,10aSR)-9-methyl-11-oxo-9,10,10a,11tetrahydro-5H-6b,9-epoxyisoindolo[2,1-a][3,1]benzoxazine-10(6aH)-carboxylic acid (20Bb). Ratio of isomers 20Ab/20Bb ~ 57/43, yellowish powder (5.76 g, 23 %), m.p. 167-168 °C (decomp.); [Found: C, 65.42; H, 4.57; N, 4.72. C₁₇H₁₅NO₅ requires C, 65.17; H, 4.83; N, 4.47%]; R_f (50% EtOH/DMF) 0.68; v_{max} (KBr) 3534, 1725, 1679 cm⁻¹; δ_{H} (400 MHz, DMSO d_6) (**20Ab**) 12.40 (1 H, br.s, CO₂H), 8.42 (1 H, d, ${}^3J_{1,2}$ 8.2 Hz, H-1), 7.31–7.22 (3 H, m, H-2, H-3, H-4), 6.67 (1 H, d, ³J_{8,7} 5.6 Hz, H-8), 6.33 (1 H, d, ³J_{7,8} 5.6 Hz, H-7), 5.85 (1 H, s, H-6a), 5.10 (1 H, d, ²J_{5.5} 15.3 Hz, H-5A), 4.95 (1 H, d, ²J_{5.5} 15.3 Hz, H-5B), 3.14 $(1 \text{ H}, \text{ d}, {}^{3}J_{10a,10} 8.9 \text{ Hz}, \text{H-10a}), 2.59 (1 \text{ H}, \text{ d}, {}^{3}J_{10,10a} 8.9 \text{ Hz}, \text{H-10}),$ 1.54 (3 H, s, Me-9); (20Bb) 12.40 (1 H, br.s, CO₂H), 7.66 (1 H, d, ³J_{1,2} 8.2 Hz, H-1), 7.31–7.22 (3 H, m, H-2, H-3, H-4), 6.77 (1 H, d, ³*J*_{8,7} 5.6 Hz, H-8), 6.39 (1 H, d, ³*J*_{7,8} 5.6 Hz, H-7), 5.45 (1 H, s, H-6a), 5.21 (1 H, d, ²J_{5,5} 15.3 Hz, H-5A), 5.06 (1 H, d, ²J_{5,5} 15.3 Hz, H-5B), 3.04 (1 H, d, ³J_{10a,10} 8.9 Hz, H-10a), 2.67 (1 H, d, ${}^{3}J_{10,10a}$ 8.9 Hz, H-10), 1.56 (3 H, s, Me-9); $\delta_{\rm C}$ (100.6 MHz, DMSO-d₆) (20Ab) 172.0 and 168.6 (CO₂H and C-11), 140.7 (C-8), 135.6 (C-7), 135.1 (C-12a), 127.9, 125.4, 123.8 (C-2, C-3, C-4), 123.4 (C-4a), 117.6 (C-1), 89.4 and 88.2 (C-6b and C-9), 83.9 (C-6a), 68.1 (C-5), 54.7 (C-10a), 48.6 (C-10b), 16.1 (Me-9); (20Bb) 171.8 (CO₂H), 170.9 (C-11), 140.3 (C-8), 134.9 (C-7), 134.0 (C-12a), 127.4, 125.4, 125.1 (C-2, C-3, C-4), 125.7 (C-4a), 121.4 (C-1), 90.4 and 88.9 (C-6b and C-9), 84.9 (C-6a), 67.7 (C-5), 53.2 (C-10a), 48.5 (C-10), 16.1 (Me-9); MS (EI, 70 eV) m/z 313 (36, M⁺), 295 (5), 268 (2), 251 (17), 215 (35), 214 (100), 213 (17), 186 (17), 183 (13), 172 (22), 159 (30), 158 (24), 152 (45), 135 (34), 134 (87), 133 (26), 132 (72), 130 (30), 110 (35), 109 (43), 106 (31), 105 (36), 104 (53), 99 (30), 95 (63), 93 (22), 91 (22), 82 (12), 79 (19), 78 (58), 77 (82), 65 (20), 54 (41), 53 (48), 52 (24), 51 (48), 50 (18), 45 (15), 43 (51), 39 (35), 38 (16%). (6aRS,6bSR,9SR,10RS,10aRS)-9-Bromo-11-oxo-9,10,10a,11tetrahydro-5H-6b,9-epoxyisoindolo[2,1-a][3,1]benzoxazine-

10(6aH)-carboxylic acid (20Ac) and (6aRS,6bRS,9RS,10SR,10aSR)-9-bromo-11-oxo-9,10,10a,11-

tetrahydro-5*H*-6b,9-epoxyisoindolo[2,1-*a*][3,1]benzoxazine-10(6*aH*)-carboxylic acid (20Bc). Ratio of isomers 20Ac/20Bc ~ 93/7, yellowish powder (5.15 g, 17 %), m.p. > 250 °C (decomp.); [Found: C, 50.80; H, 3.34; N, 3.58; Hal, 21.48. C₁₆H₁₂BrNO₅ requires C, 50.82; H, 3.20; N, 3.70; Br, 21.13%]; v_{max} (KBr) 3098, 1751, 1665, 525 cm⁻¹; $\delta_{\rm H}$ (400 MHz, DMSO-*d*₆) (20Ac) 12.33 (1 H, br.s, CO₂H), 8.41 (1 H, d, ³*J*_{1,2} 8.1 Hz, H-1), 7.26 (1 H, ddd, ³*J*_{2,1} 8.1, ³*J*_{2,3} 6.9, ⁴*J*_{2,4} 0.7 Hz, H-2), 7.20 (1 H, dd, ³*J*_{4,3}

6.9, ${}^{4}J_{4,2}$ 0.7 Hz, H-4), 7.12 (1 H, dt, ${}^{3}J_{3,4} \sim {}^{3}J_{3,2}$ 6.9, ${}^{4}J_{3,1}$ 0.7 Hz, H-3), 6.85 (1 H, d, ${}^{3}J_{8,7}$ 5.5 Hz, H-8), 6.60 (1 H, d, ${}^{3}J_{7,8}$ 5.5 Hz, H-7), 5.93 (1 H, s, H-6a), 5.13 (1 H, d, ${}^{2}J_{5,5}$ 15.4 Hz, H-5^A), 5.00 $(1 \text{ H}, \text{ d}, {}^{2}J_{5,5} 15.4 \text{ Hz}, \text{H-5B}), 3.32 (1 \text{ H}, \text{ d}, {}^{3}J_{10a,10} 8.8 \text{ Hz}, \text{H-10a}),$ 3.09 (1 H, d, ${}^{3}J_{10,10a}$ 8.8 Hz, H-10); (**20Bc**) 12.33 (1 H, br.s, CO₂H), 7.68 (1 H, d, ${}^{3}J_{1,2}$ 8.1 Hz, H-1), 7.44 (1 H, dd, ${}^{3}J_{4,3}$ 7.0, ⁴J_{2,4} 1.2 Hz, H-4), 7.39 (1 H, br.dd, ³J_{2,3} 7.0, ³J_{1,2} 8.1 Hz, H-2), 7.31–7.29 (1 H, m, H-3), 6.93 (1 H, d, ³J_{8.7} 5.7 Hz, H-7), 6.67 (1 H, d, ${}^{3}J_{7,8}$ 5.7 Hz, H-8), 5.57 (1 H, s, H-6a), 5.25 (1 H, d, ${}^{2}J_{5,5}$ 15.3 Hz, H-5A), 5.10 (1 H, d, ${}^{2}J_{5.5}$ 15.3 Hz, H-5B), ~ 3.30 (1 H, d, ${}^{3}J_{10a,10}$ 8.3 Hz, H-10a), 3.14 (1 H, d, ${}^{3}J_{10,10a}$ 8.3 Hz, H-10); δ_{C} (100.6 MHz, DMSO-d₆) (20Ac) 169.5 (CO₂H), 166.8 (C-11), 140.6 (C-8), 136.2 (C-7), 134.3 (C-12a), 127.4 (C-2), 124.9 (C-4), 123.6 (C-3), 122.9 (C-4a), 117.2 (C-1), 90.7 (C-9), 87.1 (C-6b), 82.9 (C-6a), 67.6 (C-5), 53.6 (C-10a), 51.4 (C-10); (20Bc) 169.1 (CO₂H), 166.7 (C-11), 140.3 (C-8), 135.4 (C-7), 133.1 (C-12a), 129.9, 128.6, 127.0 (C-2, C-3, C-4), 125.1 (C-4a), 120.9 (C-1), 90.7 (C-9), 87.5 (C-6b), 83.5 (C-6a), 67.3 (C-5), 52.1 (C-10a), 51.2 (C-10). MS (EI, 70 eV) m/z 379 (5, M⁺ for Br⁸¹), 377 (5), 333 (2), 315 (2), 313 (3), 298 (2), 280 (26), 278 (31), 270 (2), 252 (4), 224 (5), 214 (14), 198 (12), 185 (10), 174 (11), 173 (10), 170 (22), 159 (47), 158 (24), 134 (71), 132 (84), 121 (18), 104 (67), 99 (40), 91 (21), 77 (100), 65 (31), 51 (67), 44 (49), 39 (62%).

$\label{eq:methyl} Methyl \qquad (6aRS,6bSR,9RS,10SR,10aRS)-11-oxo-9,10,10a,11-tetrahydro-5H-6b,9-epoxyisoindolo[2,1-a][3,1]benzoxazine-$

10(6aH)-carboxylate (21). A solution of the acid 20Aa (15.65 g, 0.05 mol) and a catalytic amount of sulfuric acid (ca. 0.5 mL) in MeOH (160 mL) was heated under reflux for 8 h. The acid dissolved completely by the end of the reaction. The solvent was then evaporated under reduced pressure and the residue, a yellow oil, was purified by aluminum oxide column chromatography (2 \times 10 cm, eluent: EtOAc). Ester 21 was obtained as colorless prisms (12.67 g, 82 %), m.p. 183-184 °C; [Found: C, 65.14; H, 4.74; N, 4.58. C₁₇H₁₅NO₅ requires C, 65.17; H, 4.83; N, 4.47%]; R_{f} (65% EtOAc/hexane) 0.34; ν_{max} (KBr) 1703, 1600 cm $^{1};$ δ_{H} $(400 \text{ MHz}, \text{CDCl}_3) 8.37 (1 \text{ H}, \text{ dd}, {}^{3}J_{1,2} 8.3, {}^{4}J_{1,3} 1.1 \text{ Hz}, \text{H-1}), 7.20$ (1 H, ddd, ${}^{3}J_{1,2}$ 8.3, ${}^{3}J_{3,2}$ 7.6, ${}^{4}J_{2,4}$ 1.7 Hz, H-2), 7.03 (1 H, dt, ${}^{3}J_{2,3}$ ~ ${}^{3}J_{4,3}$ 7.6, ${}^{4}J_{3,1}$ 1.1 Hz, H-3), 6.98 (1 H, dd, ${}^{3}J_{3,4}$ 7.6, ${}^{4}J_{2,4}$ 1.7 Hz, H-4), 6.58 (1 H, d, ³J_{7,8} 5.8 Hz, H-7), 6.49 (1 H, dd, ³J_{7,8} 5.8, ³J_{9,8} 1.8 Hz, H-8), 5.70 (1 H, s, H-6a), 5.23 (1 H, d, ³J_{8.9} 1.8 Hz, H-9), 5.10 (1 H, d, ²J_{5.5} 15.0 Hz, H-5A), 4.98 (1 H, d, ²J_{5.5} 15.0 Hz, H-5B), 3.72 (3 H, s, CO₂Me), 2.97 (1 H, d, ³J_{10,10a} 9.0 Hz, H-10a), 2.74 (1 H, d, ${}^{3}J_{10,10a}$ 9.0 Hz, H-10); δ_{C} (100.6 MHz, CDCl₃) 171.5 and 167.1 (two s, CO₂Me and C-11), 137.7 (d, J 178.2 Hz, C-7), 134.0 (s, C-12a), 133.3 (d, J 179.5 Hz, C-8), 127.7 (d, J 162.0 Hz, C-2), 124.1 (d, J 158.0 Hz, C-4), 123.8 (d, J 163.0 Hz, C-3), 122.4 (s, C-4a), 118.5 (d, J 167.3 Hz, C-1), 88.6 (s, C-6b), 83.1 (d, J 161.2 Hz, C-6a), 81.6 (d, J 168.5 Hz, C-9), 68.3 (t, J 147.0 Hz, C-5), 52.1 (q, J 147.3 Hz, CO₂Me), 51.1 (d, J 140.0 Hz, C-10a), 45.3 (d, J 139.0 Hz, C-10). 1D NOE ¹H NMR, %: η_{H-1} {H-6a} 2.4 %; η_{H-6a} {H_A-5} 8.7 %; η_{H-6a} {H-10a} 3.1 %; η_{H-7} {H-6a} 5.1 %; η_{H-7} {H-10a} 2.4 %; η_{H-8} {H-10} 2.9 %; η_{H-9} {H-10} 5.6 %; η_{H-10} {H-10a} 10.0 %. MS (EI, 70 eV) m/z 313 (35, M⁺), 295 (1), 282 (2), 254 (2), 236 (2), 217 (4), 202 (19), 201 (23), 200 (100), 186 (22), 185 (11), 174 (19), 157 (9), 134 (10), 132 (36), 113 (45), 104 (12), 95 (10), 77 (17), 65 (7), 59 (10), 51 (7), 39 (7), 28 (4%)

2,4a-Epoxyisoindolo[1,2-b][1,3]benzoxazine-1(4bH)-

carboxylic acids (22). General experimental procedure. A mixture of 2-(aminomethyl)phenol^[27] (1.23 g, 10.0 mmol) and furfural (or 5-methylfurfural) (~1 mL, 10.0 mmol) in PhMe (50 mL) was heated under reflux with azeotropic removal of water

using a Dean-Stark apparatus for 0.5–1 h. About 0.2 mL of water was collected. The solvent was removed under reduced pressure and a yellow-brown oil was obtained. The ratio of the ring/chain tautomeric forms (according to ¹H NMR) in the case of 2-(furan-2-yl)-3,4-dihydro-2*H*-1,3-benzoxazine was found ~ 39/61. The oil was then dissolved in CH₂Cl₂ (30 mL) and maleic anhydride (0.98 g, 10.0 mmol) was added. The solution was stored at room temperature for 24 h. The solvent was removed and the residue, a viscous brown oil, was triturated with Et₂O (5 × 15 mL). A pale brown powder formed was collected by filtration, air-dried until constant weight and analyzed by NMR. Analytical samples of adducts **22A** were obtained by crystallization from an EtOAc/MeOH mixture.

(1RS,2SR,4aRS,4bSR,12aSR)-12-Oxo-1,2,12,12a-tetrahydro-10H-2,4a-epoxyisoindolo[1,2-b][1,3]benzoxazine-1(4bH)-

carboxylic acid (**22Aa**). Cream-colored powder (1.02 g, 34 %), m.p. 144.8–145.3 °C (decomp. from EtOAc/ MeOH); [Found: C, 64.17; H, 4.33; N, 4.53. $C_{16}H_{13}NO_5$ requires C, 64.21; H, 4.38; N, 4.68%]; v_{max} (KBr) 3468, 1739, 1676 cm⁻¹; δ_{H} (400 MHz, CDCl₃) 9.45 (1 H, br.s, CO₂H), 7.17 (1 H, dd, ${}^{3}J_{6,7}$ 8.2, ${}^{3}J_{8,7}$ 7.5 Hz, H-7), 7.08 (1 H, br.d, ${}^{3}J_{8,9}$ 7.5 Hz, H-9), 7.00 (1 H, br.t, ${}^{3}J_{8,9} \sim {}^{3}J_{8,7}$ 7.5 Hz, H-8), 6.95 (1 H, br.d, ${}^{3}J_{6,7}$ 8.2 Hz, H-6), 6.87 (1 H, d, ${}^{3}J_{3,4}$ 5.8 Hz, H-4), 6.49 (1 H, br.d, ${}^{3}J_{3,4}$ 5.8 Hz, H-3), 5.55 (1 H, s, H-4b), 5.30 (1 H, br.s, H-2), 4.96 (1 H, d, ${}^{2}J_{10,10}$ 16.8 Hz, H-10A), 4.36 (1 H, d, ${}^{2}J_{10,10}$ 16.8 Hz, H-10B), 2.93 (1 H, d, ${}^{3}J_{12a,1}$ 9.0 Hz, H-12a), 2.84 (1 H, d, ${}^{3}J_{1,12a}$ 9.0 Hz, H-1); δ_{C} (100.6 MHz, CDCl₃) 174.2 (CO₂H), 172.1 (C₁₂), 152.5 (C_{5a}), 136.1 (C₃), 134.1 (C₄), 128.3 (C₇), 127.2 (C₉), 122.7 (C₈), 119.7 (C_{9a}), 117.9 (C₆), 89.9 (C_{4a}), 83.3 (C₂), 82.9 (C_{4b}), 49.9 (C_{12a}), 44.6 (C₁), 40.0 (C₁₀). MS (EI, 70 eV) *m*/z 299 (39, M⁺), 281 (37), 202 (9), 201 (66), 200 (100), 184 (14), 172 (17), 108 (9), 107 (73), 99 (17), 81 (37), 78 (48), 77 (45), 26 (9%).

(1RS,2SR,4aRS,4bSR,12aSR)-2-Methyl-12-oxo-1,2,12,12atetrahydro-10H-2,4a-epoxyisoindolo[1,2-b][1,3]benzoxazine-1(4bH)-carboxylic acid (22Ab) and (1RS,2SR,4aRS,4bRS,12aSR)-2-methyl-12-oxo-1,2,12,12atetrahydro-10H-2,4a-epoxyisoindolo[1,2-b][1,3]benzoxazine-1(4bH)-carboxylic acid (22Bb). A pale-brown powder (1.11 g, 35 %) was obtained by trituration with ether, ratio of isomers **22Ab/22Bb** ~ 86/14. $\delta_{\rm H}$ (400 MHz, DMSO- d_6 , **22Bb** in mixture **22Ab/22Bb** ~ 86/14) 7.31 (1 H, dd, ${}^{4}J_{9,7}$ 1.3, ${}^{3}J_{8,9}$ 7.7 Hz, H-9), 7.23-6.70 (4 H, these signals are overlapping with the signals of *major* isomer **22Ab**, H-9, H-8, H-6 and H-4), 6.31 (1 H, d, ${}^{3}J_{3,4}$ 5.5 Hz, H-3), 5.84 (1 H, s, H-4b), 5.55 (1 H, d, ²J_{10,10} 16.9 Hz, H-10A), 4.31 (1 H, d, ²J_{10,10} 16.9 Hz, H-10B), 2.93 (1 H, d, ³J_{12a,1} 8.7 Hz, H-12a), 2.54 (1 H, d, ³J_{1,12a} 8.7 Hz, H-1), 1.52 (3 H, s, Me-2). Single isomer 22Ab was obtained by crystallization from EtOAc/MeOH as a white powder, m.p. 176.3-177.1 °C (decomp.); [Found: C, 64.91; H, 4.75; N, 4.49. C₁₇H₁₅NO₅ requires C, 65.17; H, 4.83; N, 4.47%]; v_{max} (KBr) 3441, 3182, 1743, 1696, 1439, 1189 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) (**22Ab**) 7.19 (1 H, br.dt, ${}^{3}J_{6,7} \sim {}^{3}J_{8,7}$ 7.7, ${}^{4}J_{9,7}$ 1.4 Hz, H-7), 7.10 (1 H, dd, ${}^{3}J_{8,9}$ 7.7, ${}^{4}J_{9,7}$ 1.4 Hz, H-9), 7.02 (1 H, dt, ${}^{3}J_{8,9} \sim {}^{3}J_{8,7}$ 7.7, ${}^{4}J_{8,6}$ 1.4 Hz, H-8), 6.96 (1 H, dd, ${}^{3}J_{6,7}$ 7.7, ${}^{4}J_{6,8}$ 1.4 Hz, H-6), 6.92 (1 H, d, ${}^{3}J_{3,4}$ 5.5 Hz, H-4), 6.31 (1 H, d, ${}^{3}J_{3,4}$ 5.5 Hz, H-3), 5.59 (1 H, s, H-4b), 5.04 (1 H, d, ${}^{2}J_{10,10}$ 16.7 Hz, H-10A), 4.41 (1 H, d, ${}^{2}J_{10,10}$ 16.7 Hz, H-10B), 3.00 (1 H, d, ${}^{3}J_{12a,1}$ 8.7 Hz, H-12a), 2.88 (1 H, d, ${}^{3}J_{1,12a}$ 8.7 Hz, H-1), 1.74 (3 H, s, Me-2); δ_C (100.6 MHz, CDCl₃) 173.2 (CO₂H), 172.7 (C-12), 152.6 (C-5a), 139.4 (C-3), 135.0 (C-4), 128.3 (C-7), 127.2 (C-9), 122.9 (C-8), 119.7 (C-9a), 118.0 (C-6), 91.1 (C-4a), 89.5 (C-2), 83.1 (C-4b), 53.2 and 48.2 (C-12a and C-1), 40.0 (C-10), 16.0 (Me-2). MALDI-TOF HR: MNa⁺, found 336.0853. C₁₇H₁₅NNaO₅ requires 336.0842.

(2RS,4aRS,4bSR,12aSR)-1,12a-Dihydro-10H-2,4a-

epoxyisoindolo[1,2-b][1,3]benzoxazin-12(2H,4bH)-one (23).The condensation adduct (20 mmol), obtained as described above from 2-hydroxybenzylamine and furfural, was heated under reflux with NEt₃ (4.16 mL, 30.0 mmol) and acryloyl chloride (1.92 mL, 24.0 mmol) in PhMe (50 mL) for 4 h. The mixture was cooled, poured into water (250 mL), acidified with hydrochloric acid and extracted with EtOAc (4 \times 60 mL). The combined organic phases were washed with a 2% HCl solution (100 mL), dried (MgSO₄), and concentrated. The residue was purified by silica gel column chromatography (2 \times 25 cm, eluent: EtOAc/hexane, 1/3). The isolated vitreous pale yellow oil (1.28 g, 25 %) slowly crystallized on standing to give elongated thin colorless needles, m.p. 106.1-110.5 °C; [Found: C, 70.36; H, 5.15; N, 5.54. C₁₅H₁₃NO₃ requires C, 70.58; H, 5.13; N, 5.49%]; v_{max} (KBr) 1708, 1432, 1215 cm⁻¹; δ_{H} (400 MHz, CDCl₃) 7.19 (1 H, ddd, ${}^{3}J_{6,7}$ 8.3, ${}^{3}J_{8,7}$ 7.5, ${}^{4}J_{9,7}$ 1.2 Hz, H-7), 7.09 (1 H, br.d, ${}^{3}J_{8,9}$ 7.5 Hz, H-9), 7.00 (1 H, dt, ${}^{3}J_{8,7} \sim {}^{3}J_{8,9}$ 7.5, ${}^{4}J_{8,6}$ 1.2 Hz, H-8), 6.97 (1 H, dd, ³*J*_{6,7} 8.3, ³*J*_{8,6} 1.2 Hz, H-6), 6.78 (1 H, d, ³*J*_{3,4} 5.6 Hz, H-dd, ${}^{3}J_{12a,1exo}$ 3.7, ${}^{3}J_{12a,1exo}$ 9.1 Hz, H-12a^{endo}), 2.22 (1 H, dd, ${}^{2}J_{1,1}$ 11.8, ${}^{3}J_{2,1exo}$ 4.4, ${}^{3}J_{12a,1exo}$ 3.7 Hz, H-1^{exo}), 1.68 (1 H, dd, ${}^{2}J_{1,1}$ 11.8, ${}^{3}J_{12a,1exo}$ 9.1 Hz, H-1^{endo}); $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 174.0 (C-12), 152.7 (C-5a), 136.1 (C-3), 131.8 (C-4), 128.1 (C-7), 127.0 (C-9), 122.3 (C-8), 119.8 (C-9a), 117.8 (C-6), 89.9 (C-4a), 83.3 (C-2), 80.0 (C-4b), 45.8 (C-12a), 39.7 (C-10), 27.5 (C-1). GC-MS (EI, 70 eV) m/z 255 (28, M⁺), 226 (4), 201 (31), 200 (63), 187 (7), 172 (5), 149 (7), 132 (11), 107 (28), 94 (8), 81 (17), 78 (42), 77 (31), 65 (10), 55 (100), 39 (24%).

7,9a-Epoxy[1,3]thiazolo[2,3-a]isoindole-6(9bH)-carboxylic

acids (24). Method A (two-step). Freshly distilled furfural (10.9 mL, 0.13 mol) was added in one portion to a stirred solution of 2aminoethanethiol hydrochloride (15.0 g, 0.13 mol) and anhydrous Na₂CO₃ powder (27.6 g, 0.26 mol) in CHCl₃ (250 mL) at room temperature. The mixture was stirred at room temperature for 24 h. The precipitate of inorganic salts was filtered off, washed with CHCl₃ (2×50 mL) and the solvent was then evaporated under reduced pressure. The residue was purified by column chromatography (silica gel, hexane/EtOAc, 1/1) to provide 2-(2-furyl)-1,3-thiazolidine as a yellow oil, which crystallized upon cooling to give a yellow powder (17.8 g, 87 %); [Found: C, 54.20; H, 5.82; N, 9.04. C7H9NOS requires C, 54.17; H, 5.84; N, 9.02%]; v_{max} (KBr) 3188, 1484, 930, 733 cm⁻¹; δ_{H} (400 MHz, CDCl₃) 7.38 (1 H, dd, ${}^{3}J_{4',5'}$ 1.6, ${}^{4}J_{3',5'}$ 0.6 Hz, H-5'), 6.34 (1 H, dd, ${}^{3}J_{3',4'}$ 3.1, ${}^{4}J_{3',5'}$ 0.6 Hz, H-3'), 6.31 (1 H, dd, ${}^{3}J_{3',4'}$ 3.1, ³*J*_{4',5'} 1.6 Hz, H-4'), 5.59 (1 H, s, H-2), 3.54–3.52 (1 H, m, H-5A), 3.19–3.04 (3 H, m, H-4, H-5B), 2.16 (1 H, br.s, NH); δ_C (100.6 MHz, CDCl₃) 152.7 (C-2'), 142.5 (C-5'), 110.2 and 107.5 (C-3' and C-4'), 65.4 (C-2), 52.4 (C-4), 36.0 (C-5). GC-MS (EI, 70 eV) m/z 155 (82, M⁺), 154 (9), 122 (11), 109 (78), 108 (26), 96 (51), 95 (51), 94 (100), 81 (73), 80 (74), 67 (22), 53 (23), 52 (37), 45 (36), 39 (70%).

2-[2-(5-Methylfuryl)]-1,3-thiazolidine was obtained in a similar way.

A solution of maleic anhydride (5.59 g, 57.0 mmol) in PhH (100 mL) was added to a solution of (2-furyl)-1,3-thiazolidine (8.81 g, 57.0 mmol) in PhH (100 mL) and the mixture was stirred overnight at room temperature. The formed precipitate was collected by filtration, washed first with PhH (50 mL) and then with Et_2O (2 × 25 mL), and dried in air affording acids **24a** as

white powders (7.71 g, 56 %) with the 24Aa/24Ba ratio of ~ 17/83.

This reaction can be carried out in a *one-pot* procedure (the yields of **24a** and the isomers ratio are comparable).

Method B (one pot). Freshly distilled furfural (1.78 mL, 21.7 mmol) was added in one portion at 0 °C to a stirred solution of 2-aminoethanethiol hydrochloride (2.47 g, 21.7 mmol) and NEt₃ (3.01 mL, 21.7 mmol) in CHCl₃ (15 mL). The mixture was stirred for 30 min at room temperature, then cooled to 0 °C and a solution of maleic anhydride (2.13 g, 21.7 mmol) in CHCl₃ (10 mL) was added at this temperature. The mixture was warmed to room temperature and stirred overnight. The formed precipitate was collected by filtration and dried affording **24a** (2.70 g, 49 %) as a white powder (ratio **24Aa/24Ba** is 15/85), m.p. 157–158 °C (EtOH/DMF, decomp.).

Generally, the title compound obtained by *Method B* was contaminated with 5-10 % NEt₃·HCl.

The individual isomer **24Ba** was obtained after recrystallization from EtOH/DMF as transparent tiny rhombus-shaped crystals. (5*aRS*,6*SR*,7*RS*,9*aSR*,9*bSR*)-5-Oxo-2,3,5,5*a*,6,7-hexahydro-

7,9a-epoxy[1,3]thiazolo[2,3-a]isoindole-6(9bH)-carboxylic

acid (24Ba). M.p. 155–156 °C (decomp.); [Found: C, 52.13; H, 4.57; N, 5.42. C₁₁H₁₁NO₄S requires C, 52.16; H, 4.38; N, 5.53%]; v_{max} (KBr) 3184, 1743, 1679, 1402, 1168 cm⁻¹; $\delta_{\rm H}$ (400 MHz, DMSO- d_6) 12.06 (1 H, br.s, CO₂H), 6.57 (1 H, d, ${}^3J_{8,9}$ 5.6 Hz, H-9), 6.48 (1 H, dd, ${}^3J_{8,9}$ 5.6, ${}^3J_{7,8}$ 1.8 Hz, H-8), 5.66 (1 H, s, H-9b), 4.99 (1 H, d, ${}^3J_{7,8}$ 1.8 Hz, H-7), 4.07 (1 H, ddd, ${}^2J_{3,3}$ 11.2, ${}^3J_{3A,2B}$ 5.6, ${}^3J_{3A,2A}$ 2.5 Hz, H-3A), 3.18 (1 H, d, ${}^3J_{5a,6}$ 8.7 Hz, H-6), 3.09–2.92 (2 H, m, H-2A and H-3B), 2.75–2.68 (1 H, m, H-2B), 2.49 (1 H, d, ${}^3J_{5a,6}$ 8.7 Hz, H-5a); $\delta_{\rm C}$ (100.6 MHz, DMSO- d_6) 172.6 and 170.3 (C-5 and CO₂H), 137.7 (C-9), 133.9 (C-8), 92.1 (C-9a), 80.5 (C-7), 63.7 (C-9b), 52.6 and 45.8 (C-5a and C-6), 44.9 (C-3), 31.9 (C-2). MS (EI, 70 eV) *m*/z 253 (10, M⁺), 209 (6), 156 (9), 155 (51), 154 (62), 127 (13), 122 (14), 111 (23), 108 (94), 99 (24), 96 (41), 88 (10), 85 (44), 80 (35), 71 (11), 68 (17), 61 (17), 60 (46), 56 (20), 55 (100), 45 (48), 43 (67%).

(5*aRS*,6*SR*,7*RS*,9*aSR*,9*bRS*)-5-Oxo-2,3,5,5*a*,6,7-hexahydro-7,9*a*-epoxy[1,3]thiazolo[2,3*-a*]isoindole-6(9*bH*)-carboxylic

acid (24Aa) (in the mixture of 24Aa/24Ba in ratio ~ 15/85). $\delta_{\rm H}$ (400 MHz, DMSO- d_6) 12.18 (1 H, br.s, CO₂H), 6.61 (1 H, d, ${}^3J_{8,9}$ 5.6 Hz, H-9), 6.52 (1 H, dd, ${}^3J_{8,9}$ 5.6, ${}^3J_{7,8}$ 1.4 Hz, H-8), 5.18 (1 H, s, H-9b), 5.16 (1 H, d, ${}^3J_{7,8}$ 1.4 Hz, H-7), 4.24–4.18 (1 H, m, H-3A), 3.06–3.02 and 2.98–2.92 (1 H and 1 H, two m, H-2A and H-3B), 2.70 (1 H, br.dt, ${}^3J_{2B,3}$ 6.4, ${}^2J_{2,2} \sim {}^3J_{2B,3}$ 9.6 Hz, H-2B), 2.76 and 2.63 (1 H and 1 H, two d, ${}^3J_{5a,6}$ 9.2 Hz, H-6 and H-5a); $\delta_{\rm C}$ (100.6 MHz, DMSO- d_6) 174.8 and 172.1 (C-5 and CO₂H), 137.4 (C-9), 135.6 (C-8), 90.0 (C-9a), 82.5 (C-7), 65.6 (C-9b), 49.7 and 44.4 (C-5a and C-6), 45.6 (C-3), 33.0 (C-2).

Acids **24b** were obtained according to *Method A*, ratio of **24Ab/24Bb** \sim 67/33, yield 48 %. The individual isomer **24Ab** was obtained after three-fold recrystallization from EtOH as crystalline aggregates of thick colorless needles.

(5aRS,6SR,7RS,9aSR,9bSR)-7-Methyl-5-oxo-2,3,5,5a,6,7hexahydro-7,9a-epoxy[1,3]thiazolo[2,3-*a*]isoindole-6(9bH)-

carboxylic acid (**24Ab**). M.p. > 160 °C (decomp.); [Found: C, 53.82; H, 5.14; N, 5.58. $C_{12}H_{13}NO_4S$ requires C, 53.92; H, 4.90; N, 5.24%]; v_{max} (KBr) 1717, 1697, 1666, 1220 cm⁻¹; δ_H (400 MHz, DMSO- d_6) 12.34 (1 H, br.s, CO₂H), 6.61 (1 H, d, ${}^{3}J_{8,9}$ 5.5 Hz, H-9), 6.36 (1 H, d, ${}^{3}J_{8,9}$ 5.5 Hz, H-8), 5.16 (1 H, s, H-9b), 4.24–4.16 (1 H, m, H-3A), 3.22–3.12 (3 H, m, H-2 and H-3B), 2.81 (1 H, d, ${}^{3}J_{5a,6}$ 8.9 Hz, H-5a), 2.65 (1 H, ${}^{3}J_{5a,6}$ 8.9 Hz, H-6), 1.53 (3 H, s, Me-7); δ_C (100.6 MHz, DMSO- d_6) 174.9 and 171.0 (C-5 and CO₂H), 140.3 and 136.3 (C-8 and C-9), 89.6 and 89.2

(C-7 and C-9a), 65.8 (C-9b), 52.7 and 48.0 (C-5a and C-6), 45.4 (C-3), 32.7 (C-2), 15.7 (Me-7). MS (EI, 70 eV) m/z 267 (M⁺, 17), 249 (20), 222 (4), 207 (2), 194 (5), 180 (5), 175 (4), 170 (27), 169 (100), 168 (87), 154 (36), 136 (45), 123 (50), 122 (96), 113 (13), 110 (42), 109 (73), 108 (42), 98 (72), 95 (47), 94 (38), 85 (68), 80 (37), 59 (40), 54 (84), 53 (95), 45 (37), 43 (47%).

(5aRS,6SR,7RS,9aSR,9bRS)-7-Methyl-5-oxo-2,3,5,5a,6,7hexahydro-7,9a-epoxy[1,3]thiazolo[2,3-a]isoindole-6(9bH)-

carboxylic acid (**24Bb**) (in the mixture of **24Ab/24Bb** in ratio ~ 10/90). White powder. $\delta_{\rm H}$ (400 MHz, DMSO- d_6) 12.18 (1 H, br.s, CO₂H), 6.62 (1 H, d, ${}^3J_{8,9}$ 5.7 Hz, H-9), 6.27 (1 H, d, ${}^3J_{8,9}$ 5.7 Hz, H-8), 5.60 (1 H, s, H-9b), 4.03 (1 H, ddd, ${}^2J_{3,3}$ 11.4, ${}^3J_{3,2}$ 6.4, ${}^3J_{3,2}$ 3.2 Hz, H-3A), 3.20 (1 H, dd, J 1.3, ${}^3J_{5a,6}$ 8.7 Hz, H-5a), 3.16–2.94 and 2.83–2.77 (2 H and 1 H, m and m, H-2 and H-3B), 2.52 (1 H, d, ${}^3J_{5a,6}$ 8.7 Hz, H-6), 1.55 (3 H, s, Me-7); $\delta_{\rm C}$ (100.6 MHz, DMSO- d_6) 171.3 and 169.8 (C-5 and CO₂H), 140.2 and 135.4 (C-8 and C-9), 90.8 and 87.9 (C-7 and C-9a), 64.0 (C-9b), 56.3 and 48.3 (C-5a and C-6), 44.6 (C-3), 32.3 (C-2), 15.7 (Me-7).

7,9a-Epoxy[1,3]thiazolo[2,3-a]isoindol-5(5aH,9bH)-ones (25). General experimental procedure. Freshly distilled furfural (8.0 mL, 96 mmol) was added in one portion at room temperature to a stirred solution of 2-aminoethanethiol hydrochloride (11.0 g, 96.0 mmol) and NEt₃ (13.3 mL, 96.0 mmol) in CHCl₃ (100 mL). The mixture was stirred for 30 min then cooled to 0 °C and a solution of acryloyl chloride (7.69 mL, 96.0 mmol) in CHCl₃ (100 mL) was added drop-wise at this temperature. The mixture was then warmed to room temperature and stirred overnight. The mixture was washed with water (100 mL) and brine (100 mL), dried over Na₂SO₄. The brown oil obtained after solvent evaporation was purified by silica gel column chromatography (hexane/EtOAc, 1/1). 3-Acryloyl-2-(2-furyl)-1,3-thiazolidine obtained this way was used for thermal IMDAF reaction (see below). Yellow powder (19.0 g, 95 %); [Found: C, 57.50; H, 5.22; N, 6.74. C₁₀H₁₁NO₂S requires C, 57.39; H, 5.30; N, 6.69%]; v_{max} (KBr) 1644, 1601 cm⁻¹. NMR spectra of this compound are difficult for interpretation due to strong widening of the signals even under heating up to 90 °C (DMSO- d_6). δ_H (600 MHz, CDCl₃) 7.37 (1 H, br.d, ${}^{3}J_{4',5'}$ 1.6 Hz, H-5'), 6.45 (1 H, dd, ${}^{3}J_{2'',3''}$ is 10.0, ${}^{2}J_{3'',3''}$ 1.2 Hz, H-3^{', cis}), 6.38 (1 H, dd, ³J_{2^{'',3''}trans} 15.6, ²J_{3^{'',3''}} 1.2 Hz, H-3''^{trans}), 6.12–6.30 (3 H, m, H-3', H-4', H-2"), 5.71 (1 H, br.s, H-2), 4.23 (1 H, m, H-5A), 3.95 (1 H, m, H-4A), 3.17-3.09 (2 H, m, H-4B, H-5B); δ_C (100.6 MHz, CDCl₃)164.4, 153.1, 142.9, 142.3, 129.4, 128.9, 128.2, 110.4, 107.3, 106.9, 58.29, 57.64, 49.13, 31.04, 29.10 some signals are double due to amidic rotation. GC-MS (EI, 70 eV) m/z 209 (40, M⁺), 182 (14), 181 (26), 155 (13), 154 (20), 148 (21), 126 (10), 122 (28), 109 (16), 108 (71), 96 (21), 95 (26), 88 (20), 80 (26), 55 (100), 45 (19), 43 (51%).

Intermediate 1-[2-(5-methylfuran-2-yl)thiazolidin-3-yl]prop-2en-1-one (pale yellow oil, yield 73 %) was obtained in a similar way.

(5aRS,7SR,9aSR,9bSR)-2,3,6,7-Tetrahydro-7,9a-

epoxy[1,3]thiazolo[2,3-*a***]isoindol-5(5a***H***,9b***H***)-one (25a). 3-Acryloyl-2-(2-furyl)-1,3-thiazolidine (10.6 g, 51.0 mmol) obtained in the previous step was heated under reflux in PhMe (100 mL) for 40 h. The precipitate formed upon cooling was collected by filtration and dried in air. Compound 25a** was obtained as a white powder (7.13 g, 67 %); m.p. 127–129 °C; [Found: C, 57.52; H, 5.24; N, 6.60. C₁₀H₁₁NO₂S requires C, 57.39; H, 5.30; N, 6.69%]; R_f (50% EtOAc/hexane) 0.45; v_{max} (KBr) 1689 cm⁻¹; δ_H (600 MHz, CDCl₃) 6.42 (1 H, dd, ${}^{3}J_{8,9}$ 5.5, ${}^{3}J_{7,8}$ 1.8 Hz, H-8), 6.37 (1 H, d, ${}^{3}J_{8,9}$ 5.5 Hz, H-9), 5.56 (1 H, s, H-9b), 5.06 (1 H, dd, ${}^{3}J_{6exo,7}$ 3.7, ${}^{3}J_{7,8}$ 1.8 Hz, H-7), 4.38 (1 H, ddd, ² $J_{3,3}$ 11.9, ³ $J_{3A,2B}$ 6.4, ³ $J_{3A,2A}$ 1.8 Hz, H-3A), 2.99 (1 H, dt, ² $J_{3,3}$ 11.9, ³ $J_{3B,2A} \sim {}^{3}J_{3B,2B}$ 6.4 Hz, H-3B), 2.93 (1 H, ddd, ² $J_{2,2}$ 11.0, ³ $J_{3B,2A}$ 6.4, ³ $J_{3A,2A}$ 1.8 Hz, H-2A), 2.85 (1 H, dt, ² $J_{2,2}$ 11.0, ³ $J_{2B,3A} \sim {}^{3}J_{2B,3B}$ 6.4 Hz, H-2B), 2.74 (1 H, dd, ³ $J_{5a,6endo}$ 8.3, ³ $J_{5a,6exo}$ 3.7 Hz, H-5a^{endo}), 2.26 (1 H, br.dt, ² $J_{6,6}$ 11.9, ³ $J_{6exo,7} \sim {}^{3}J_{5a,6exo}$ 3.7 Hz, H-6^{exo}), 1.52 (1 H, dd, ² $J_{6,6}$ 11.9, ³ $J_{5a,6endo}$ 8.3 Hz, H-6^{endo}); δ_{C} (100.6 MHz, CDCl₃) 174.2 (C-5), 138.0 and 131.7 (C-8 and C-9), 92.9 (C-9a), 78.7 (C-7), 65.3 (C-9b), 50.5 (C-5a) 45.5 (C-3), 32.3 (C-2), 28.6 (C-6). GC-MS (EI, 70 eV) *m*/*z* 209 (63, M⁺), 181 (36), 164 (5), 155 (19), 154 (37), 148 (24), 126 (12), 109 (24), 108 (40), 96 (32), 94 (78), 88 (78), 80 (32) 77 (15), 70 (21), 66 (19), 65 (50), 61 (63), 56 (24), 55 (100), 45 (32%).

(5aRS,7SR,9aSR,9bSR)-7-Methyl-2,3,6,7-tetrahydro-7,9a-

epoxy[1,3]thiazolo[2,3-a]isoindol-5(5aH,9bH)-one (25b). 1-[2-(5-Methyl-2-furyl)-1,3-thiazolidin-3-yl]prop-2-en-1-one (5.0 g, 22.4 mmol) obtained following the procedure above was heated under reflux in PhMe (100 mL) for 25 h. Evaporation of toluene under reduced pressure afforded a brown oil which solidified on cooling. The solid substance was purified by recrystallization (with activated carbon) from a hexane/EtOAc mixture. Compound 25b was obtained as colorless, long needles (hedgehog-like crystalline aggregates) (2.85 g, 57 %); m.p. 161-162 °C; [Found: C, 59.06; H, 5.91; N, 6.53. C₁₁H₁₃NO₂S requires C, 59.17; H, 5.87; N, 6.27%]; R_f (50% EtOAc/hexane) 0.42; v_{max} (KBr) 2942, 1682, 1396, 705 cm⁻¹; δ_{H} (400 MHz, CDCl₃) 6.40 (1 H, d, ³J_{8,9} 5.9 Hz, H-8), 6.27 (1 H, d, ³J_{8,9} 5.9 Hz, H-9), 5.53 (1 H, s, H-9b), 4.40 (1 H, ddd, ${}^{2}J_{3,3}$ 11.5, ${}^{3}J_{3A,2B}$ 5.9, ³*J*_{3A,2A} 2.2 Hz, H-3A), 3.07–2.89 (3 H, m, H-3B, H-2), 2.85 (1 H, ddd, ³J_{5a,6endo} 8.7, ³J_{5a,6exo} 3.3, ⁵J 1.2 Hz, H-5a^{endo}), 2.03 (1 H, dd, ${}^{2}J_{6,6}$ 11.8, ${}^{3}J_{6exo,5a}$ 3.3 Hz, H-6^{exo}), 1.66 (3 H, s, Me-7), 1.64 (1 H, dd, ${}^{2}J_{6,6}$ 11.8, ${}^{3}J_{6endo,5a}$ 8.7 Hz, H-6^{endo}); δ_{C} (100.6 MHz, CDCl₃) 174.2 (C-5), 141.0 (C-8), 132.3 (C-9), 92.5 (C-9a), 86.8 (C-7), 65.4 (C-9b), 53.6 (C-5a), 45.2 (C-3), 34.6 and 32.3 (C-6 and C-2), 18.7 (Me-7). GC-MS (EI, 70 eV) m/z 223 (18, M⁺), 208 (8), 195 (9), 180 (39), 168 (9), 162 (15), 152 (17), 136 (10), 122 (31), 113 (7), 109 (14), 108 (28), 95 (26), 88 (8), 79 (9), 55 (100), 53 (12), 43 (22%).

Methyl esters of 7,9a-epoxy[1,3]thiazolo[2,3-*a*]isoindole-6(9b*H*)carboxylic acids (26).

Methyl (5a*RS*,6S*R*,7*RS*,9a*SR*,9b*RS*)-5-oxo-2,3,5,5a,6,7hexahydro-7,9a-epoxy[1,3]thiazolo[2,3-*a*]isoindole-6(9b*H*)carboxylate (26Aa) and methyl (5a*RS*,6*SR*,7*RS*,9a*SR*,9b*SR*)-5-oxo-2,3,5,5a,6,7-hexahydro-7,9a-epoxy[1,3]thiazolo[2,3-

a]isoindole-6(9bH)-carboxylate (26Ba). The acid 24Ba (1.0 g, 3.91 mmol) was dissolved in MeOH (50 mL) and one drop of H₂SO₄ (conc.) was added. The mixture was refluxed for 4 h. EtOAc (200 mL) was then added. The mixture was washed with saturated sodium bicarbonate solution (50 mL), then with water (50 mL) and dried over Na2SO4. Evaporation of the solvent under reduced pressure yielded the desired product 26a (0.74 g, 73 %), as a mixture of isomers in ratio 26Aa/26Ba ~ 29/71, white powder, m.p. 122.0–122.8 °C (EtOAc). $\delta_{\rm H}$ (600 MHz, CDCl₃) (**26Aa**) 6.61 (1 H, dd, ${}^{3}J_{8,9}$ 6.0, ${}^{3}J_{7,8}$ 1.9 Hz, H-8), 6.43 (1 H, d, $^{(20\text{A}a)}_{J_{8,9}}$ 6.0 Hz, H-9), 5.22 (1 H, d, $^{3}_{J_{7,8}}$ 1.9 Hz, H-7), 5.20 (1 H, s, H-9b), 4.45 (1 H, ddd, ${}^{2}J_{3,3}$ 11.7, ${}^{3}J_{3A,2B}$ 6.7, ${}^{3}J_{3A,2A}$ 2.1 Hz, H-3A), 3.73 (3 H, s, CO₂Me), 3.16-3.13 (1 H, m, H-3B), 3.05-2.93 (2 H, m, H-2), 2.81 and 2.77 (1 H and 1 H, two d, ${}^{3}J_{5a,6}$ 8.9 Hz, H-6 and H-5a); δ_{C} (100.6 MHz, CDCl₃) 174.7 and 171.2 (C-2 and CO₂Me), 136.4 and 136.1 (C-9 and C-8), 90.4 (C-9a), 82.6 (C-7), 65.9 (C-9b), 52.2, 50.2 and 44.9 (C-5a, C-6 and CO₂Me), 45.9 (C-3), 33.4 (C-2). After recrystallization of the isomer mixture from EtOAc/heptane the isomer **26Ba** was obtained (0.40 g, 40 %) as white needles, m.p. 131.8-133.0 °C; [Found: C, 53.81; H,

4.83; N, 5.36. $C_{12}H_{13}NO_4S$ requires C, 53.92; H, 4.90; N, 5.24%]; R_f (EtOAc) 0.43; v_{max} (KBr) 2948, 1727, 1688, 1387, 1270 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 6.53 (1 H, dd, ${}^3J_{8,9}$ 5.8, ${}^3J_{7,8}$ 1.2 Hz, H-8), 6.49 (1 H, d, ${}^3J_{8,9}$ 5.8 Hz, H-9), 5.54 (1 H, s, H-9b), 5.22 (1 H, d, ${}^3J_{7,8}$ 1.2 Hz, H-7), 4.43 (1 H, ddd, ${}^2J_{3,3}$ 11.2, ${}^3J_{3A,2B}$ 4.4, ${}^3J_{3A,2A}$ 2.5 Hz, H-3A), 3.77 (3 H, s, CO₂Me), 3.11 (1 H, br.d, ${}^3J_{5a,6}$ 9.1 Hz, H-6), 3.04–2.92 (3 H, m, H-2 and H-3B), 2.72 (1 H, d, ${}^3J_{5a,6}$ 9.1 Hz, H-5a); $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 172.0 and 170.8 (C-2 and CO₂Me), 138.1 (C-9), 133.6 (C-8), 92.7 (C-9a), 80.8 (C-7), 64.2 (C-9b), 53.2, 52.1 and 46.1 (C-5a, C-6 and CO₂Me), 45.9 (C-3), 32.3 (C-2). GC-MS (EI, 70 eV) m/z 267 (8, M⁺), 235 (9), 208 (2), 180 (4), 156 (7), 155 (11), 154 (100), 153 (16), 152 (5), 122 (8), 113 (33), 108 (24), 96 (7), 94 (8), 85 (26), 81 (19), 80 (8), 60 (5), 59 (23), 53 (6), 39 (7%).

Isomeric mixture of methyl (5aRS,6SR,7RS,9aSR,9bRS)-7methyl-5-oxo-2,3,5,5a,6,7-hexahydro-7,9a-

epoxy[1,3]thiazolo[2,3-*a*]isoindole-6(9b*H*)-carboxylate (26Ab) and methyl (5*aRS*,6*SR*,7*RS*,9*aSR*,9*bSR*)-7-methyl-5-oxo-2,3,5,5*a*,6,7-hexahydro-7,9*a*-epoxy[1,3]thiazolo[2,3-

a]isoindole-6(9bH)-carboxylate (26Bb) (0.63 g, 63 %) is obtained in a similar way from acid 24Ab, 26Ab/26Bb ~ 67/33, white powder, m.p. 104.8-105.8 °C; [Found: C, 55.31; H, 5.38; N, 5.20. C₁₃H₁₅NO₄S requires C, 55.50; H, 5.37; N, 4.98%]; R_f (EtOAc) 0.74; v_{max} (KBr) 1720, 1687 (br.), 1030 cm⁻¹; δ_{H} (600 MHz, CDCl₃) (**26Bb**) 6.52 and 6.23 (1 H and 1 H, two d, ${}^{3}J_{8,9}$ 5.5 Hz, H-9 and H-8), 5.47 (1 H, s, H-9b), 4.38 (1 H, ddd, ²J_{3,3} 11.2, ${}^{3}J_{3A,2B}$ 4.1, ${}^{3}J_{3A,2A}$ 3.2 Hz, H-3A), 3.72 (3 H, s, CO₂Me), 3.16-2.98 (3 H, m, H-3B and H-2), 3.14 and 2.74 (1 H and 1 H, br.d, ³J_{5a,6} 9.0 Hz, H-5a and H-6), 1.68 (3 H, s, Me-7); (26Ab) 6.62 and 6.25 (1 H and 1 H, two d, ${}^{3}J_{8,9}$ 5.5 Hz, H-9 and H-8), 5.23 (1 H, s, H-9b), 4.50 (1 H, ddd, ${}^{2}J_{3,3}$ 11.7, ${}^{3}J_{3A,2B}$ 4.1, ${}^{3}J_{3A,2A}$ 2.2 Hz, H-3A), 3.75 (3H, s, CO₂Me), 3.16-2.98 (3 H, m, H-3B, H-2), 2.85 and 2.82 (1 H and 1 H, two d, ${}^{3}J_{5a6}$ 9.0 Hz, H-5a and H-6), 1.62 (3 H, s, Me-7); δ_{C} (100.6 MHz, DMSO- d_{6}) (mixture of **26Ab** and **26Bb**) 174.7, 170.6, 170.2, 170.0 (CO₂Me and C-5), 140.3, 140.2, 136.3, 135.4 (C-8 and C-9), 91.1, 89.7, 89.3, 87.7 (C-7 and C-9a), 65.7, 64.0 (C-9b), 56.5, 52.9, 51.3, 51.0, 48.0, 47.9 (C-5a, C-6, CO₂Me), 45.4, 44.7 (C-3), 32.8, 32.3 (C-2), 15.59, 15.56 (Me-1); GC-MS (EI, 70 eV) m/z 281 (5, M⁺), 250 (4), 249 (5), 248 (6), 222 (3), 194 (5), 169 (12), 168 (100), 136 (7), 135 (14), 122 (15), 114 (10), 113 (70), 108 (17), 95 (20), 85 (18), 59 (20), 53 (11), 43 (16%). All of the presented data was obtained for the mixture of isomers.

(3RS,3aSR,6SR,7aRS)-2-[2-(Acetylthio)ethyl]-1-oxo-

1,2,3,6,7,7a-hexahydro-3a,6-epoxyisoindol-3-yl acetate (27). A catalytic amount of BF3·Et2O (~ 0.2 mL) was added to a stirred solution of thiazolo[2,3-a]isoindol-5-one 25a (1.0 g, 4.8 mmol) in Ac₂O (10 mL). The reaction mixture was stirred at room temperature for 24 h, then poured into water (100 mL) and 1 M KOH solution was added to the cooled mixture till pH ~ 8. The mixture was extracted with $CHCl_3$ (4 × 60 mL) and the combined organic phases were dried over Na₂SO₄. A viscous brown residue obtained after solvent evaporation was purified by column chromatography (silica gel, hexane/EtOAc, 1/1) affording the compound 27 (0.39 g, 25 %) as a white powder. M.p. 102.7-104.3 °C; [Found: C, 54.04; H, 5.47; N, 4.72. C14H17NO5S requires C, 54.01; H, 5.50; N, 4.50%]; R_f (65% EtOAc/hexane) 0.43; v_{max} (KBr) 2946, 1758, 1712, 1705, 1225, 956, 623 cm⁻¹; δ_{H} (400 MHz, CDCl₃) 6.44 (1 H, s, H-3), 6.40 (1 H, dd, ³J_{5,4} 6.2, ³J_{5,6} 1.3 Hz, H-5), 6.38 (1 H, d, ${}^{3}J_{5,4}$ 6.2 Hz, H-4), 5.11 (1 H, dd, ${}^{3}J_{5,6}$ 1.3, ³J_{6.7exo} 4.6 Hz, H-6), 3.64–3.57 (1 H, m, NCH₂A), 3.48–3.41 (1 H, m, NCH₂B), 3.11-3.07 (2 H, m, SCH₂), 2.56 (1 H, dd, ³J_{7endo.7a} 8.7, ³J_{7exo.7a} 3.1 Hz, H-7a), 2.33 (3 H, s, OAc), 2.21 (3 H, s, SAc), 2.17 (1 H, ddd, ${}^{2}J_{7,7}$ 11.8, ${}^{3}J_{6,7exo}$ 4.6, ${}^{3}J_{7exo,7a}$ 3.1 Hz, H-7^{exo}), 1.61 (1 H, dd, ${}^{2}J_{7,7}$ 11.8, ${}^{3}J_{7endo,7a}$ 8.7 Hz, H-7^{endo}); $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 194.9 (SCOMe), 175.8 (C-1), 170.1 (OCOMe), 136.7 (C-5), 131.4 (C-4), 90.6 (C-3a), 82.7 and 80.2 (C-3 and C-6), 45.0 (C-7a), 41.6 (NCH₂), 30.6 (SCOMe), 28.1 (C-7), 27.2 (SCH₂), 21.0 (OCOMe); MS (EI, 70 eV) m/z 311 (2, M⁺), 252 (13), 236 (86), 222 (15), 210 (36), 209 (88), 180 (5), 154 (10), 150 (16), 139 (24), 126 (9), 122 (70), 108 (88), 98 (12), 97 (75), 81 (16), 66 (16), 55 (45), 43 (100%).

8,10a-Epoxy[1,3]thiazino[2,3-a]isoindole-7(10bH)-carboxylic

acids (31). General experimental procedure. A suspension of 3-aminopropane-1-thiol (0.91 g, 10.0 mmol), corresponding furfural (~ 0.9 mL, 10.0 mmol) and anhydrous powdered MgSO₄ (2.42 g, 20 mmol) in CH₂Cl₂ (30 mL) was stirred at room temperature for 2 h. MgSO₄ was filtered off and washed with CH₂Cl₂ (2 × 10 mL). To the obtained solution of 2-furanyl-1,3-thiazinane, maleic anhydride (1.08 g, 11.0 mmol) was added (some blurring and warming of the reaction mixture was observed). The mixture was stirred at room temperature for 24 h. Viscous yellow oil was formed. The solvent was removed under reduced pressure and the residue, a slowly crystallizing yellow oil was purified by crystallization from MeOH. The title acids **31** were obtained as colorless needle-shaped crystals.

Intermediate **2-(furan-2-yl)-1,3-thiazinane** was isolated and characterized during preparation of acid **31a**. $\delta_{\rm H}$ (400 MHz, CDCl₃) 7.35 (1 H, dd, ${}^{3}J_{5',4'}$ 1.9, ${}^{4}J_{5',3'}$ 0.8 Hz, H-5'), 6.31 (1 H, dd, ${}^{3}J_{4',3'}$ 3.2, ${}^{3}J_{5',4'}$ 1.9, Hz H-4'), 6.29 (1 H, br.d, ${}^{3}J_{4',3'}$ 3.2 Hz, H-3'), 5.29 (1 H, s, H-2), 3.33 (1H, ddd, ${}^{2}J_{4,4}$ 14.5, ${}^{3}J_{4e,5a}$ 3.5, ${}^{3}J_{4e,5e}$ 1.2 Hz, H-4^{eq}), 3.14 (1H, ddd, ${}^{2}J_{4,4}$ 14.5, ${}^{3}J_{4a,5e}$ 3.5 Hz, H-6^{ax}), 2.93 (1H, ddd, ${}^{2}J_{4,4}$ 14.5, ${}^{3}J_{5a,4a}$ 11.8, ${}^{3}J_{4a,5e}$ 3.5 Hz, H-4^{ax}), 2.90–2.85 (1 H, m, H-6^{eq}), 1.93 (1 H, br.s, NH), 1.79–1.63 (2 H, m, H-5); $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 153.1 (C-2'), 141.7 (C-5'), 110.0 and 106.0 (C-3' and C-4'), 58.5 (C-2), 46.7 (C-6), 29.0 (C-4), 25.6 (C-5). GC-MS (EI, 70 eV) *m*/*z* 169 (61, M⁺), 136 (12), 122 (100), 109 (59), 108 (35), 95 (82), 94 (74), 81 (61), 80 (54), 67 (24), 52 (25), 45 (24), 41 (33), 39 (57%).

(6aRS,7SR,8RS,10aSR,10bRS)-6-Oxo-3,4,6,6a,7,8-hexahydro-2H-8,10a-epoxy[1,3]thiazino[2,3-*a*]isoindole-7(10bH)-

carboxylic acid (31a). Thick colorless needles (1.29 g, 48 %), m.p. 183.5-189 °C (decomp., from MeOH); [Found: C, 53.98; H, 5.01; N, 5.41. C₁₂H₁₃NO₄S requires C, 53.92; H, 4.90; N, 5.24%]; v_{max} (KBr) 3006, 1735, 1658, 1444, 1172 cm⁻¹; δ_{H} (400 MHz, DMSO- d_6) 12.20 (1 H, br.s, CO₂H), 6.51 (1 H, dd, ${}^{3}J_{9,10}$ 5.7, ${}^{3}J_{9,8}$ 1.3 Hz, H-9), 6.49 (1 H, d, ³J_{10,9} 5.7 Hz, H-10), 5.26 (1 H, s, H-10b), 5.07 (1 H, d, ${}^{3}J_{8,9}$ 1.3 Hz, H-8), 3.99 (1 H, ddd, ${}^{2}J_{4,4}$ 13.4, ${}^{3}J_{4e,3a}$ 3.6, ${}^{3}J_{4e,3e}$ 1.9 Hz, H-4^{eq}), 3.17 (1 H, dt, ${}^{3}J_{2a,3a} \sim {}^{2}J_{2,2}$ 13.4, ${}^{3}J_{2e,2a}$ 2.5 Hz, H-2^{ax}), 2.92 (1 H, dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 13.4, ${}^{3}J_{4a,3e}$ 3.1 Hz, H-4^{ax}), 2.92–2.87 (1 H, m, H-2^{eq}), 2.75 (1 H, d, ³J_{6a,7} 9.1 Hz, H-6a), 2.56 (1 H, d, ³J_{7.6a}9.1 Hz, H-7), 1.87–1.80 (1 H, m, H-3^{eq}), 1.53-1.40 (1 H, m, H-3^{ax}); δ_C (100.6 MHz, DMSO-d₆) 172.4 (CO₂H), 168.9 (C-6), 137.6 (C-9), 133.0 (C-10), 89.8 (C-10a), 81.8 (C-8), 59.0 (C-10b), 49.3 (C-6a), 44.1 (C-7), 39.7 (C-4), 27.5 (C-2), 24.9 (C-3). MS (EI, 70 eV) m/z 267 (13, M⁺), 221 (7), 169 (12), 168 (100), 167 (13), 166 (8), 122 (11), 121 (8), 102 (11), 99 (30), 81 (12), 46 (11), 45 (45), 44 (7), 42 (6), 41 (24), 39 (16), 29 (14), 28 (16), 27 (17%).

(6aRS,7SR,8RS,10aSR,10bRS)-8-Methyl-6-oxo-3,4,6,6a,7,8hexahydro-2H-8,10a-epoxy[1,3]thiazino[2,3-a]isoindole-

7(10bH)-carboxylic acid (31b). Colorless needles (1.29 g, 46 %), m.p. 184.2–185.2 °C (MeOH); [Found: C, 55.54; H, 5.09; N, 5.34. $C_{13}H_{15}NO_4S$ requires C, 55.50; H, 5.37; N, 4.98%]; v_{max} (KBr) 1732, 1653, 1430, 1170, 1131, 871 cm⁻¹; δ_H (400 MHz, DMSO- d_6) 12.22 (1 H, br.s, CO₂H), 6.53 (1 H, d, ${}^3J_{9,10}$ 5.7 Hz, H-

9), 6.35 (1 H, d, ${}^{3}J_{10,9}$ 5.7 Hz, H-10), 5.25 (1 H, s, H-10b), 4.01–3.98 (1 H, m, ${}^{2}J_{4,4} \sim 14.0$ Hz, H-4^{eq}), 3.16 (1 H, dt, ${}^{3}J_{2a,3a} \sim {}^{2}J_{2,2}$ 13.5, ${}^{3}J_{2e,2a}$ 1.9 Hz, H-2^{ax}), 2.95–2.87 (2 H, m, H-4^{ax} and H-2^{eq}), 2.79 (1 H, d, ${}^{3}J_{6a,7}$ 8.9 Hz, H-6a), 2.58 (1 H, d, ${}^{3}J_{7,6a}$ 8.9 Hz, H-7), 1.85–1.80 (1 H, m, H-3^{eq}), 1.52 (3 H, s, Me-8), 1.55–1.42 (1 H, m, H-3^{ax}); $\delta_{\rm C}$ (100.6 MHz, DMSO- $d_{\rm 6}$) 171.3 (CO₂H), 169.2 (C-6), 140.4 (C-9), 134.0 (C-10), 89.2 (2C, C-10a and C-8), 59.3 (C-10b), 52.5 (C-6a), 47.5 (C-7), 39.7 (C-4), 27.4 (C-2), 24.8 (C-3), 15.5 (Me-8). MS (EI, 70 eV) *m/z* 281 (16, M⁺), 236 (7), 235 (19), 220 (13), 183 (16), 182 (100), 162 (7), 152 (8), 136 (19), 135 (11), 125 (10), 110 (9), 109 (8), 102 (18), 99 (8), 95 (10), 73 (10), 45 (8), 43 (16), 41 (11%).

(6aRS,8SR,10aSR,10bRS)-3,4,7,8-Tetrahydro-2H-8,10a-

epoxy[1,3]thiazino[2,3-a]isoindol-6(6aH,10bH)-one (32). Furfural (0.65 mL, 7.90 mmol) and anhydrous powdered MgSO₄ (1.89 g, 16.0 mmol) was added to a solution of 3aminopropanethiol (0.72 g, 7.90 mmol) in CH₂Cl₂ (30 mL). The reaction mixture was stirred at room temperature for 2 h. MgSO₄ was filtered off, washed with CH_2Cl_2 (2 × 10 mL), and the solvent was evaporated under reduced pressure. Obtained 2-(2furyl)-1,3-thiazinane (~ 7.9 mmol) was dissolved in PhMe (50 mL) and then acryloyl chloride (0.96 mL, 12.0 mmol) and NEt₃ (2.22 mL, 16.0 mmol) were added to the solution. The reaction mixture was heated under reflux for 3 h, then cooled and poured into water (50 mL). The organic layer was separated and the water layer was extracted with EtOAc (3×20 mL). The organic phases were combined and dried over MgSO₄. The brown oil obtained after solvent evaporation was crystallized in Et₂O (10 mL). After recrystallization of the precipitate from an EtOAc/hexane mixture, thiazino[2,3-a]isoindol 32 was isolated (0.46 g, 26 %) as fine colorless needles, m.p. 140–141 °C (EtOAc/hexane); [Found: C, 59.04; H, 5.60; N, 6.53, $C_{11}H_{13}NO_2S$ requires C, 59.17; H, 5.87; N, 6.27%]; v_{max} (KBr) 1676, 1419, 1253, 956 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 6.43 (1 H, d, ${}^{3}J_{10,9}$ 5.8 Hz, H-10), 6.37 (1 H, dd, ${}^{3}J_{9,10}$ 5.8, ${}^{3}J_{9,8}$ 1.5 Hz, H-9), 5.07 (1 H, dd, ${}^{3}J_{8,7exo}$ 4.4, ${}^{3}J_{8,9}$ 1.5 Hz, H-8), 5.04 (1 H, s, H-10b), 4.29 (1 H, ddd, ${}^{2}J_{4,4}$ 13.7, ${}^{3}J_{4e,3a}$ 2.5, ${}^{3}J_{4e,3e}$ 1.9 Hz, H-4^{eq}), 3.09 (1 H, ddd, ${}^{2}J_{2,2}$ 13.7, ${}^{3}J_{2a,3a}$ 12.5, ${}^{3}J_{2a,3e}$ 3.1 Hz, H-2^{ax}), 2.93–2.81 (2 H, m, H-2^{eq} and H-4^{ax}), 2.52 (1 H, dd, ³J_{6a,7endo} 8.7, ³J_{6a,7exo} 3.7 Hz, H-6a), 2.15 (1 H, ddd, ²J_{7,7} 11.5, ³J_{7exo.8} 4.4, ³J_{7exo.6a} 3.7 Hz, H-7^{exo}), 1.89–1.82 (1 H, m, H-3^{eq}), 1.80–1.72 (1 H, m, H-3^{ax}), 1.60 (1 H, dd, ${}^{2}J_{7,7}$ 11.5, ${}^{3}J_{7\text{endo},6a}$ 8.7 Hz, H-7^{endo}); δ_{C} (100.6 MHz, CDCl₃) 172.4 (C-6), 136.7 (C-9), 131.6 (C-10), 90.6 (C-10a), 79.5 (C-8), 61.0 (C-10b), 46.4 (C-6a), 40.5 (C-4), 28.6 and 27.8 (C-2 and C-7), 25.1 (C-3). GC-MS (EI, 70 eV) m/z 223 (50, M⁺), 196 (4), 195 (22), 168 (100), 148 (11), 134 (10), 122 (18), 111 (20), 108 (27), 102 (28), 94 (35), 81 (34), 65 (36), 56 (31), 55 (85), 45 (37), 41 (50), 39 (62%).

2,4a-Epoxyisoindolo[1,2-b][1,3]benzothiazole-1(4bH)-

carboxylic acids (33). General experimental procedure. Anhydrous powdered MgSO₄ (7.21 g, 60 mmol) and then 2aminothiophenol (3.30 mL, 30 mmol) were added to a solution of the corresponding furaldehyde (30 mmol) in CH₂Cl₂ (50 mL). The reaction mixture was stirred at room temperature for 3–4 h. MgSO₄ was filtered off and washed with CH₂Cl₂ (2×25 mL). The organic phases were combined and concentrated under reduced pressure. The residue, a yellow-brown oil, was dissolved in Me₂CO (30 mL) and a solution of maleic anhydride (3.09 g, 31.5 mmol) in Me₂CO (20 mL) was added in one portion with stirring. The reaction mixture was then stirred at room temperature for 12 h. The precipitate formed was collected by filtration, washed with Me₂CO (3×20 mL) and air-dried till constant weight. Isoindolo[1,2-b][1,3]benzothriazole acids **33a–c** were obtained as white easy-electrifiable powders.

(1RS,2SR,4aRS,4bRS,11aSR)-11-Oxo-1,2,11,11a-tetrahydro-2,4a-epoxyisoindolo[1,2-b][1,3]benzothiazole-1(4bH)-

carboxylic acid (33a). White powder (5.38 g, 60 %), m.p. 176-176.7 °C (decomp.); [Found: C, 59.63; H, 3.52; N, 4.78. C₁₅H₁₁NO₄S requires C, 59.79; H, 3.68; N, 4.65%]; v_{max} (KBr) 3540, 1719, 1707, 1470, 1362, 759 cm⁻¹; $\delta_{\rm H}$ (600 MHz, DMSO d_6) 12.32 (1 H, br.s, CO₂H), 7.42 (1 H, dd, ${}^{3}J_{8,9}$ 8.0, ${}^{4}J_{7,9}$ 1.4 Hz, H-9), 7.28 (1 H, br.d, ${}^{3}J_{6,7}$ 7.6 Hz, H-6), 7.08 (1 H, dt, ${}^{3}J_{6,7} \sim {}^{3}J_{7,8}$ 7.6, ${}^{4}J_{7,9}$ 1.4 Hz, H-7), 7.03 (1 H, ddd, ${}^{3}J_{8,9}$ 8.0, ${}^{3}J_{8,7}$ 7.6, ${}^{4}J_{6,8}$ 1.4 Hz, H-8), 6.68 (1 H, s, H-4b), 6.60 (1 H, d, ³J₃₄ 5.7 Hz, H-4), 6.52 (1 H, dd, ${}^{3}J_{3,4}$ 5.7, ${}^{3}J_{3,2}$ 1.6 Hz, H-3), 5.08 (1 H, d, ${}^{3}J_{2,3}$ 1.6 Hz, H-2), 3.40 (1 H, d, ³J_{1,11a} 8.9 Hz, H-1), 2.59 (1 H, d, ³J_{1,11a} 8.9 Hz, H-11a); δ_C (150.9 MHz, DMSO-d₆) 173.2 and 169.9 (CO₂H and C-11), 138.7, 135.4, 133.8, 132.4 (C-3, C-4, C-5a, C-9a), 125.8, 125.7, 123.2 (C-6, C-7, C-8), 116.4 (C-9), 91.7 (C-4a), 82.0 (C-2), 67.3 (C-4b), 54.8 and 46.5 (C-1 and C-11a). MS (EI, 70 eV) m/z 301 (66, M⁺), 285 (2), 258 (4), 228 (10), 205 (25), 204 (47), 203 (72), 202 (100), 186 (20), 175 (13), 174 (71), 173 (60), 172 (16), 170 (43), 162 (10), 149 (26), 136 (86), 135 (30), 117 (24), 110 (33), 109 (35), 101 (14), 99 (45), 83 (18), 77 (15), 69 (35), 65 (55), 59 (27), 55 (26), 54 (80), 51 (25), 45 (38), 43 (55), 41 (18%).

(1RS,2SR,4aRS,4bRS,11aSR)-2-Methyl-11-oxo-1,2,11,11a-tetrahydro-2,4a-epoxyisoindolo[1,2-b][1,3]benzothiazole-

1(4bH)-carboxylic acid (33b). White powder (5.10 g, 54 %) (33 % after recrystallization from i-PrOH/DMF), m.p. 129.1-129.6 °C (decomp.); [Found: C, 60.99; H, 4.39; N, 4.25. C₁₆H₁₃NO₄S requires C, 60.94; H, 4.16; N, 4.44%]; v_{max} (KBr) 3437, 1746, 1691, 1469, 1367, 1367, 1183, 754 cm⁻¹; δ_H (600 MHz, DMSOd₆) 12.34 (1 H, br.s, CO₂H), 7.46 (1 H, br.d, ³J_{8,9} 7.7 Hz, H-9), 7.28 (1 H, br.d, ${}^{3}J_{6,7}$ 7.7 Hz, H-6), 7.08 (1 H, br.t, ${}^{3}J_{6,7} \sim {}^{3}J_{7,8}$ 7.7 Hz, H-7), 7.03 (1 H, br.t, ${}^{3}J_{8,9} \sim {}^{3}J_{8,7}$ 7.7 Hz, H-8), 6.64 (1 H, d, ³J_{3,4} 5.8 Hz, H-4), 6.63 (1 H, s, H-4b), 6.34 (1 H, d, ³J_{3,4} 5.8 Hz, H-3), 3.41 (1 H, d, ³*J*_{1,11a} 9.1 Hz, H-1), 2.63 (1 H, d, ³*J*_{1,11a} 9.1 Hz, H-11a), 1.55 (3 H, s, Me-2); δ_C (100.6 MHz, DMSO-d₆) 171.2 and 169.0 (CO₂H and C-11), 140.7 and 134.6 (C-3 and C-4), 135.0 and 132.0 (C-5a and C-9a), 125.2 (2 C) and 122.7 (C-6, C-7, C-8), 115.5 (C-9), 90.0 and 88.9 (C-2 and C-4a), 67.1 (C-4b), 57.9 and 48.9 (C-1 and C-11a), 15.6 (Me-2). MS (EI, 70 eV) m/z (%): 315 (13 M⁺), 271 (1), 254 (3), 242 (6), 223 (9), 218 (20), 217 (85), 216 (100), 215 (30), 210 (8), 202 (40), 187 (8), 186 (10), 177 (26), 174 (89), 170 (10), 157 (9), 136 (78), 125 (12), 110 (20), 109 (38), 98 (33), 95 (47), 81 (22), 76 (14), 69 (24), 65 (30), 60 (14), 55 (14), 54 (39), 53 (35), 51 (10), 45 (32), 43 (25), 42 (10%).

(1RS,2SR,4aSR,4bSR,11aRS)-2-Bromo-11-oxo-1,2,11,11atetrahydro-2,4a-epoxyisoindolo[1,2-b][1,3]benzothiazole-

1(4bH)-carboxylic acid (33c) was obtained following the procedure above. After addition of maleic anhydride, a dark purple reaction mixture was formed with a little precipitate present. The mixture was stirred at room temperature for 4 h. The precipitate was collected by filtration, washed with boiling Me₂CO (4 × 15 mL), and finally recrystallized from an *i*-PrOH/DMF mixture. A pale pink powder was obtained (0.68 g, 6%), m.p. 145.4–147.5 °C (decomp.); [Found: C, 47.16; H, 2.68; N, 3.84; Hal, 21.32. C₁₅H₁₀BrNO₄S requires C, 47.38; H, 2.65; N, 3.68; Br, 21.02%]; v_{max} (KBr) 3413, 1752, 1691, 1554, 1469, 1370, 1237, 1178 cm⁻¹; $\delta_{\rm H}$ (400 MHz, DMSO-*d*₆) 12.62 (1 H, br.s, CO₂H), 7.50 (1 H, dd, ³*J*_{8,9} 7.6, ⁴*J*_{7,9} 1.2 Hz, H-9), 7.34 (1 H, dd, ³*J*_{6,7} 7.6, ⁴*J*_{6,8} 1.2 Hz, H-6), 7.14 (1 H, dt, ³*J*_{6,7} 7.6, ⁴*J*_{6,8} 1.2 Hz, H-8),

6.84 (1 H, d, ${}^{3}J_{3,4}$ 5.5 Hz, H-4), 6.71 (1 H, s, H-4b), 6.62 (1 H, d, ${}^{3}J_{3,4}$ 5.5 Hz, H-3), 3.62 (1 H, d, ${}^{3}J_{1,11a}$ 8.9 Hz, H-11a), 3.14 (1 H, d, ${}^{3}J_{1,11a}$ 8.9 Hz, H-1); $\delta_{\rm C}$ (100.6 MHz, DMSO-*d*₆) 169.3 and 167.9 (CO₂H and C-11), 140.7 and 136.0 (C-3 and C-4), 134.7 and 131.7 (C-5a and C-9a), 125.40, 125.34, 122.8 (C-6, C-7, C-8), 115.4 (C-9), 90.5 and 89.5 (C-2 and C-4a), 66.5 (C-4b), 57.3 and 52.1 (C-1 and C-11a). HRMS (DART): MH⁺, found 379.9569 (for Br⁷⁹) and 381.9585 (for Br⁸¹). C₁₅H₁₁BrNO₄S requires 379.9592 (for Br⁷⁹).

Synthesis of isoindolo[1,2-b][1,3]benzothiazol-11-one (34) and N-acryloyl derivative (34*). A suspension of 2aminobenzenethiol (3.30 mL, 30 mmol), furfural (2.52 mL, 30 mmol) and anhydrous powdered MgSO₄ (3.60 g, 30 mmol) in CH₂Cl₂ (50 mL) was stirred at room temperature for 3 h. MgSO₄ was filtered off, the solvent was evaporated and the residue, a yellow oil, was dissolved in PhMe (50 mL). NEt₃ (6.24 mL, 45 mmol) was added and the mixture was cooled to 0 °C. Acryloyl chloride (2.40 mL, 30.0 mmol) was then added with intensive stirring. The red-brown reaction mixture was heated under reflux for 4 h, then cooled and poured into a saturated solution of Na₂CO₃ (100 mL). The organic layer was separated and the water layer was extracted with EtOAc (2×70 mL). The organic phases were combined and dried over MgSO4. After filtration and solvent evaporation, the residue, a brown oil, was separated by silica gel column chromatography (2.5 \times 20 cm, eluent: EtOAc/hexane, 1/5) affording intermediate 3-acryloyl-2-(2furyl)-2,3-dihydro-1,3-benzothiazole (34*) 1.15 g (15 %). Then 2,4a-epoxyisoindolo[1,2-b][1,3]benzothiazol-11-one (34) 1.54 g (20 %) was isolated using a more polar eluent mixture (EtOAc/hexane, 1/1). Increase of the reaction time to 22 h raises the yield of the target adduct 34 to 2.15 g (28 %, after column chromatography).

3-Acryloyl-2-(2-furyl)-2,3-dihydro-1,3-benzothiazole (34*). Beige plates, m.p. 87.8-88.7 °C (from EtOAc/hexane); [Found: C, 65.64; H, 4.52; N, 5.08. C₁₄H₁₁NO₂S requires C, 65.35; H, 4.31; N, 5.44%]; R_f (65% EtOAc/hexane) 0.70; v_{max} (KBr) 1650, 1611, 1463, 1407, 1229, 750 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 7.65 (1 H, br.s, H-4), 7.34–7.32 (1 H, m, H-5'), 7.21 (1 H, dd, ⁴J_{7.5} 1.0, ${}^{3}J_{7,6}$ 7.5 Hz, H-7), 7.13 (1 H, dt, ${}^{3}J_{6,7} \sim {}^{3}J_{6,5}$ 7.5, ${}^{4}J_{6,4}$ 1.0 Hz, H-6), 7.07 (1 H, dt, ${}^{3}J_{5,6} \sim {}^{3}J_{5,4}$ 7.5, ${}^{4}J_{5,7}$ 1.0 Hz, H-5), 6.86 (1 H, br.s, H-2), 6.67 (1 H, dd, ${}^{3}J_{2'',3''trans}$ 16.8, ${}^{3}J_{2'',3''cis}$ 10.3 Hz, H-2''), 6.52 (1 H, dd, ${}^{3}J_{3''trans,2''}$ 16.8, ${}^{2}J_{3'',3''}$ 1.6 Hz, H-3'''^{trans}), 6.26–6.24 (2 H, m, H-3' and H-4'), 5.85 (1 H, dd, ${}^{3}J_{3'',2''cis}$ 10.3, ${}^{2}J_{3'',3''}$ 1.6 Hz, H-3""cis); δ_C (100.6 MHz, CDCl₃) 164.1 (C-1"), 152.0 (C-2'), 142.9 (C-5'), 137.1 (C-3a), 130.3 (C-3"), 128.1, 125.4 and 125.1 (C-5, C-6 and C-7), 122.9 and 119.0 (br.s, 2 C) (C-4, C-2" and C-7a), 110.4 (C-3'), 107.4 (C-4'), 61.0 (C-2). GC-MS (EI, 70 eV) m/z 259 (5, M^++2 for S^{34}), 257 (44, M^+), 255 (2), 228 (5), 204 (8), 203 (38), 202 (43), 201 (10), 174 (40), 173 (58), 172 (12), 147 (13), 136 (14), 135 (15), 109 (38), 108 (18), 94 (30), 82 (12), 69 (39), 65 (48), 55 (100), 51 (16), 45 (15), 39 (35%).

(2RS,4aRS,4bRS,11aSR)-1,11a-Dihydro-2,4a-

epoxyisoindolo[1,2-*b*][1,3]benzothiazol-11(2*H*,4b*H*)-one (34). White powder, m.p. 167.8–169.0 °C (from EtOAc/hexane); [Found: C, 65.35; H, 4.41; N, 5.14. C₁₄H₁₁NO₂S requires C, 65.35; H, 4.31; N, 5.44%]; R_f (50% EtOAc/hexane) 0.78; v_{max} (KBr) 1719, 1699, 1465, 1356, 1179, 753 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 7.61 (1 H, d, ${}^{3}J_{8,9}$ 7.5 Hz, H-9), 7.14 (1 H, d, ${}^{3}J_{6,7}$ 7.5 Hz, H-6), 7.08 (1 H, br.t, ${}^{3}J_{6,7} \sim {}^{3}J_{8,7}$ 7.5 Hz, H-7), 7.00 (1 H, br.t, ${}^{3}J_{8,7} \sim {}^{3}J_{8,9}$ 7.5 Hz, H-8), 6.52 (1 H, s, H-4b), 6.49 (1 H, dd, ${}^{3}J_{4,3}$ 5.6, ${}^{3}J_{2,3}$ 1.2 Hz, H-3), 6.44 (1 H, d, ${}^{3}J_{4,3}$ 5.6 Hz, H-4), 5.18 (1 H, dd, ${}^{3}J_{2,3}$ 1.2, ${}^{3}J_{2,\text{lexo}}$ 4.9 Hz, H-2), 2.89 (1 H, dd, ${}^{3}J_{1\text{endo,11a}}$ 8.7, ${}^{3}J_{1\text{exo,11a}}$ 3.7

Hz, H-1^{exo}), 1.65 (1 H, dd, ${}^{2}J_{1,1}$ 11.8, ${}^{3}J_{1\text{endo},11a}$ 8.7 Hz, H-1^{endo}); δ_C (100.6 MHz, CDCl₃) 172.3 (C-11), 138.3 (C-4), 135.1 and 131.8 (C-5a and C-9a), 131.3, 125.4 (2 C), 122.5, 116.8 (C-3, C-6, C-7, C-8, C-9), 91.6 (C-4a), 79.5 (C-2), 68.4 (C-4b), 52.2 (C-11a), 29.0 (C-1). GC-MS (EI, 70 eV) *m*/*z* 259 (4, M⁺+2 for S³⁴), 257 (26, M⁺), 256 (18), 255 (2), 228 (4), 203 (20), 202 (40), 201 (39), 174 (27), 173 (39), 172 (36), 147 (12), 135 (14), 130 (12), 109 (37), 94 (30), 82 (10), 69 (35), 65 (47), 55 (100), 51 (17), 45 (11), 39 (36%).

Methyl (1RS,2SR,4aRS,4bRS,11aSR)-11-oxo-1,2,11,11atetrahydro-2,4a-epoxyisoindolo[1,2-b][1,3]benzothiazole-

1(4bH)-carboxylate (35). Carbonic acid 33a (1.0 g, 3.30 mmol) was heated under reflux in MeOH (40 mL) with conc. H₂SO₄ (0.25 mL) for 2 h. White needle-shaped crystals of the target ester are formed on cooling of the yellow solution to +4 °C. The precipitate was collected by filtration, washed with Et_2O (2 × 20 mL), recrystallized from MeOH. The ester 35 was obtained as colorless needle-shaped crystals (0.75 g, 72 %), m.p. 180-180.6 °C; [Found: C, 60.86; H, 4.29; N, 4.51. C₁₆H₁₃NO₄S requires C, 60.94; H, 4.16; N, 4.44%]; R_f (EtOAc) 0.73; v_{max} (KBr) 1727, 1720, 1470, 1361, 1228, 1184, 761 cm⁻¹; δ_H (600 MHz, DMSO d_6) 7.40 (1 H, dd, ${}^{3}J_{8,9}$ 7.7, ${}^{4}J_{7,9}$ 1.3 Hz, H-9), 7.28 (1 H, dd, ${}^{3}J_{6,7}$ 7.7, ${}^{4}J_{6,8}$ 1.3 Hz, H-6), 7.08 (1 H, dt, ${}^{3}J_{6,7} \sim {}^{3}J_{8,7}$ 7.7, ${}^{4}J_{9,7}$ 1.3 Hz, H-7), 7.04 (1 H, dt, ${}^{3}J_{8,7} \sim {}^{3}J_{8,9}$ 7.7, ${}^{4}J_{6,8}$ 1.3 Hz, H-8), 6.69 (1 H, s, H-4b), 6.62 (1 H, d, ${}^{3}J_{4,3}$ 5.8 Hz, H-4), 6.53 (1 H, dd, ${}^{3}J_{4,3}$ 5.8, ³J_{2,3} 1.7 Hz, H-3), 5.13 (1 H, d, ³J_{2,3} 1.7 Hz, H-2), 3.56 (3 H, s, CO_2Me), 3.46 (1 H, d, ${}^{3}J_{1,11a}$ 9.1 Hz, H-1), 2.75 (1 H, d, ${}^{3}J_{1,11a}$ 9.1 Hz, H-11a); δ_C (150.9 MHz, DMSO-d₆) 172.4 and 170.0 (CO₂Me and C-11), 138.7 and 133.9 (C-3 and C-4), 135.3 and 132.3 (C-5a and C-9a), 125.93, 125.70, 123.2 (C-6, C-7, C-8), 116.6 (C-9), 91.9 (C-4a), 81.6 (C-2), 67.3 (C-4b), 55.0 and 46.3 (C-1 and C-11a), 52.0 (CO₂Me). GC-MS (EI, 70 eV) m/z 315 (11, M⁺), 272 (2), 228 (1), 203 (33), 202 (100), 174 (26), 172 (24), 147 (5), 121 (7), 114 (10), 113 (95), 109 (24), 85 (14), 69 (15), 65 (26), 59 (21), 45 (7), 39 (27%).

Synthesis of epoxides 36. General experimental procedure. Compound 4a, 17a or 17b (24 mmol) was added to a solution of 85% *m*-CPBA (9.74 g, 48.0 mmol) in CHCl₃ (90 mL) at 0 °C. The mixture was stirred at 0 °C for 30 min and then for 4–14 h at room temperature. The reaction mixture was then poured into a saturated Na₂CO₃ solution (150 mL). The organic layer was separated and the water layer was extracted with CHCl₃ (2 × 80 mL). Combined organic extracts were washed with a saturated sodium bicarbonate solution (3 × 80 mL) and dried over MgSO₄. Then MgSO₄ was then filtered off. Crystals obtained by solvent evaporation were recrystallized from a hexane/EtOAc mixture using activated carbon. Diepoxides **36a–c** were obtained as colorless crystalline compounds.

Methyl (1aRS,2RS,3RS,3aSR,9aSR,9bSR,9cRS)-4oxooctahydro-6H-2,9b-epoxy[1,3]oxazino[2,3-

a]oxireno[*g*]isoindole-3(9*aH*)-carboxylate (36*a*). Colorless prisms (5.33 g, 79 %), m.p. 231–233 °C; [Found: C, 55.63; H, 5.48; N, 4.93. C₁₃H₁₅NO₆ requires C, 55.51; H, 5.38; N, 4.98%]; R_f (EtOAc) 0.50; v_{max} (KBr) 1753, 1704, 1436, 1211, 1173, 1071, 1039 cm⁻¹; $\delta_{\rm H}$ (600 MHz, CDCl₃) 5.09 (1 H, s, H-9a), 4.75 (1 H, s, H-2), 4.17 (1 H, ddd, ²J_{8,8} 12.6, ³J_{8e,7a} 5.0, ³J_{8e,7e} 2.2 Hz, H-8^{*eq*}), 4.12 (1 H, ddd, ²J_{6,6} 13.2, ³J_{6e,7a} 5.0, ³J_{6e,7e} 1.7 Hz, H-6^{*eq*}), 3.83 (1 H, dt, ²J_{8,8} ~ ³J_{8a,7a} 12.6, ³J_{8a,7e} 2.2 Hz, H-8^{*ex*}), 3.69 (3 H, s, CO₂Me), 3.63 (1 H, dt, ³J_{1a,9c} 3.3 Hz, H-1a), 3.41 (1 H, dt, ³J_{9c,1a} 3.3 Hz, H-9c), 3.06 (1 H, ddd, ²J_{6,6} 13.2, ³J_{6a,7a} 12.6, ³J_{6a,7e} 3.8 Hz, H-6^{*ax*}), 2.98 and 2.93 (1 H and 1 H, two dt, ³J_{3,3a} 9.5, H-3 and H-3a), 1.86–1.78 (1 H, m, H-7^{*ax*}), 1.50–1.47 (1 H, m, H-7^{*eq*}); $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 170.3 and 169.6 (CO₂Me and C-4), 86.0 (C-

9b), 84.1 (C-9a), 78.2 (C-2), 66.6 (C-8), 51.4 (CO_2Me), 51.0, 48.5, 46.7, 46.6 (C-1a, C-3, C-3a, C-9c), 38.2 (C-6), 24.9 (C-7). MS (EI, 70 eV) *m*/2 281 (15, M⁺), 280 (8), 250 (14), 238 (2), 232 (1), 224 (2), 222 (4), 220 (4), 209 (3), 194 (9), 192 (13), 180 (10), 167 (11), 164 (11), 152 (13), 151 (17), 139 (30), 138 (80), 137 (18), 136 (10), 123 (11), 113 (10), 109 (16), 98 (7), 95 (10), 86 (46), 85 (26), 84 (25), 81 (100), 79 (17), 69 (4), 59 (21), 58 (11), 56 (38), 55 (10), 53 (15), 41 (21), 29 (19), 28 (22), 27 (12%).

Methyl (1aRS,2SR,3SR,3aRS,9aRS,9bRS,9cSR)-2-methyl-4oxooctahydro-6H-2,9b-epoxy[1,3]oxazino[2,3-

a]oxireno[g]isoindole-3(9aH)-carboxylate (36b). Colorless prisms (4.10 g, 58 %), m.p. 190-193 °C; [Found: C, 56.59; H, 5.35; N, 5.03. C₁₄H₁₇NO₆ requires C, 56.94; H, 5.80; N, 4.74%]; R_f (EtOAc) 0.47; v_{max} (KBr) 1747, 1691, 1436, 1203, 1064, 1029 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 4.97 (1 H, s, H-9a), 4.00–4.08 (2 H, m, H-8^{eq} and H-6^{eq}), 3.73 (1 H, br.dt, ${}^{2}J_{8,8} \sim {}^{3}J_{8a,7a}$ 12.5, ${}^{3}J_{8a,7e}$ 1.9 Hz, 8-H^{ax}), 3.58 (3 H, s, CO₂Me), 3.56 (1 H, d, ${}^{3}J_{1a,9c}$ 3.1 Hz, H-1a), 3.18 (1 H, d, ${}^{3}J_{9c,1a}$ 3.1 Hz, H-9c), 2.97 (1 H, br.dt, ${}^{2}J_{6,6}$ ~ ${}^{3}J_{6a,7a}$ 13.1, ${}^{3}J_{6a,7e}$ 3.7 Hz, 6-H^{ax}), 2.84 and 2.79 (1 H and 1 H, two d, ${}^{3}J_{3,3a}$ 9.4 Hz, H-3 and H-3a), 1.76–1.65 (1 H, m, H-7^{*ax*}), 1.39 (3 H, s, Me-2), 1.39–1.36 (1 H, m, H-7^{eq}); δ_C (100.6 MHz, CDCl₃) 170.0 and 169.1 (CO2Me and C-4), 86.4, 85.9, 85.1 (C-2, C-9a, C-9b), 67.5 (C-8), 52.8, 51.9, 51.8, 50.8, 49.1 (CO₂Me, C-1a, C-3, C-3a, C-9c), 38.9 (C-6), 25.0 (C-7), 13.7 (Me-2). MS (EI, 70 eV) m/z 295 (3, M⁺), 279 (2), 266 (30), 264 (43), 252 (53), 246 (38), 220 (40), 206 (24), 192 (33), 180 (100), 179 (32), 166 (24), 164 (28), 152 (50), 151 (69), 139 (26), 136 (23), 123 (40), 95 (37), 86 (33), 56 (39), 43 (39%).

(1aRS,2RS,3aSR,9aSR,9bSR,9cRS)-Hexahydro-6H-2,9bepoxy[1,3]oxazino[2,3-a]oxireno[g]isoindol-4(2H,9aH)-one

(36c). Colorless prisms (3.84 g, 71 %), m.p. 198–199.2 °C; [Found: C, 59.19; H, 5.87; N, 6.27. $C_{11}H_{13}NO_4$ requires C, 59.43; H, 5.76; N, 6.09%]; R_f (80% EtOAc/EtOH) 0.73; v_{max} (KBr) 1694, 1435, 1263, 1064, 744 cm⁻¹; δ_{H} (400 MHz, DMSO- d_6) 5.06 (1 H, s, H-9a), 4.58 (1 H, d, ${}^{3}J_{2,3exo}$ 4.5 Hz, H-2), 4.12 (1 H, br.dd, ${}^{3}J_{8e,7a}$ 4.5, ${}^{2}J_{8,8}$ 11.5 Hz, H-8^{eq}), 3.93–3.87 (2 H, m, H-8^{ax} and H-6^{eq}), 3.60 and 3.54 (1 H and 1 H, two d, ${}^{3}J_{1a,9c}$ 3.5 Hz, H-1a and H-9c), 3.09 (1 H, dt, ${}^{2}J_{6,6} \sim {}^{3}J_{6a,7a}$ 12.7, ${}^{3}J_{6a,7e}$ 3.8 Hz, H-6^{ax}), 2.73 (1 H, dd, ${}^{3}J_{3endo,3a}$ 8.9, ${}^{3}J_{3exo,3a}$ 5.1 Hz, H-3a), 1.84–1.74 (2 H, m, H-3), 1.68–1.56 (1 H, m, H-7^{ax}), 1.53–1.47 (1 H, m, H-7^{eq}); δ_{C} (100.6 MHz, DMSO- d_{6}) 172.4 (C-4), 86.2 (C-9b), 84.7 (C-9a), 75.7 (C-2), 66.7 (C-8), 49.1, 47.0, 46.4 (C-1a, C-3a, C-9c), 38.2 (C-6), 30.5 (C-3), 25.0 (C-7). GC-MS (EI, 70 eV) m/z 223 (20, M⁺), 222 (15), 194 (10), 180 (12), 166 (14), 152 (22), 151 (35), 139 (34), 123 (24), 109 (30), 94 (37), 86 (94), 81 (86), 80 (26), 66 (44), 56 (93), 55 (100), 53 (72), 41 (74), 39 (84%).

(1aRS,2RS,3aSR,8aRS,8bSR,8cRS)-Hexahydro-2,8b-

epoxyoxireno[g][1,3]thiazolo[2,3-a]isoindol-4(2H,8aH)-one

8,8-dioxide (38a). Formic acid (3.6 mL, 96.0 mmol) and then 35% H₂O₂ (8.2 mL, 96.0 mmol) were added to a solution of thiazoloisoindolone **25a** (2.0 g, 9.60 mmol) in CH₂Cl₂ (30 mL). The reaction mixture was intensively stirred at room temperature for 16 h (TLC monitoring) and then poured into water (150 mL). Aqueous ammonia was added to the mixture until pH ~ 8–9. The organic layer was separated and the water layer was extracted with CH₂Cl₂ (4 × 50 mL). The combined organic phases were dried over MgSO₄. After the solvent was evaporated, the residue was triturated with Et₂O (15 mL). The crystals formed were collected by filtration and recrystallized from a heptane/EtOAc mixture. The sulfone **38a** was obtained as a white powder (0.97 g, 40 %), m.p. 238–241 °C (decomp.); R_f (EtOAc) 0.80; v_{max} (KBr) 1707, 1387, 1314, 1119, 736 cm⁻¹; $\delta_{\rm H}$ (600 MHz, DMSO- d_6) 5.27 (1 H, s, H-8a), 4.60 (1 H, d, ³J_{2,3exo} 4.8 Hz, H-2), 4.16 (1

H, ddd, ${}^{2}J_{6,6}$ 12.6, ${}^{3}J_{6A,7B}$ 7.6, ${}^{3}J_{6A,7A}$ 2.8 Hz, H-6A), 3.77 (1 H, d, ${}^{3}J_{8c,1a}$ 3.4 Hz, H-8c), 3.53 (1 H, d, ${}^{3}J_{1a,8c}$ 3.4 Hz, H-1a), 3.47 (1 H, ddd, ${}^{2}J_{7,7}$ 12.6, ${}^{3}J_{7A,6B}$ 5.8, ${}^{3}J_{7A,6A}$ 2.8 Hz, H-7A), 3.34 (1 H, ddd, ${}^{2}J_{7,7}$ 12.6, ${}^{3}J_{6B,7B}$ 10.3, ${}^{3}J_{6B,7A}$ 5.8 Hz, H-6B), 3.14 (1 H, ddd, ${}^{2}J_{3,aendo,3endo}$ 9.2, ${}^{3}J_{3aendo,3exo}$ 3.4 Hz, H-3a^{endo}), 3.00 (1 H, ddd, ${}^{2}J_{7,7}$ 12.6, ${}^{3}J_{7B,6B}$ 10.3, ${}^{3}J_{7B,6A}$ 7.6 Hz, H-7B), 1.81 (1 H, ddd, ${}^{2}J_{3,3}$ 12.4, ${}^{3}J_{3exo,2}$ 4.8, ${}^{3}J_{3exo,3aendo}$ 3.4 Hz, H-3 endo), 1.67 (1 H, dd, ${}^{2}J_{3,3}$ 12.4, ${}^{3}J_{3endo,3eado}$ 9.2 Hz, H-3 endo); δ_{C} (100.6 MHz, DMSO- d_{6}) 173.8 (C-4), 86.2 (C-8b), 75.6 (C-2), 71.8 (C-8a), 52.6 (C-3a), 49.6 (C-7), 49.1 and 47.1 (C-1a and C-8c), 38.5 (C-6), 29.4 (C-3). GC-MS (EI, 70 eV) m/z 207 (3, M⁺-64), 194 (5), 193 127 (1), 121 (2), 97 (3), 93 (3), 77 (5), 73 (53), 72 (6), 45 (8), 44 (100), 43 (9), 42 (40%). HRMS (DART): MH⁺, found 258.0422. C₁₀H₁₂NO₅S requires 258.0436.

(1aRS,2SR,3aRS,8aSR,8bRS,8cSR)-2-Methylhexahydro-2,8b-epoxyoxireno[g][1,3]thiazolo[2,3-a]isoindol-4(2H,8aH)-one

8,8-dioxide (38b). A solution of m-CPBA (3.87 g, 22.4 mmol) and thiazoloisoindolone 25b (1.0 g, 4.50 mmol) in CH₂Cl₂ (25 mL) was stirred for 7 h. The reaction mixture was then poured into a saturated Na₂CO₃ solution (100 mL). The organic layer was separated and the water layer was extracted with CH_2Cl_2 (3 × 50 mL). The organic phases were combined, washed with saturated Na₂CO₃ solution (2×60 mL) and dried over MgSO₄. The residue obtained after the filtration and the solvent evaporation was triturated with Et₂O (20 mL). The crystalline precipitate formed was collected by filtration and crystallized from a hexane/EtOAc mixture. The target diepoxide 38b (3.94 g, 65 %) was obtained as a white powder, m.p. 239.4-241.2 °C; R_f (50% EtOAc/EtOH) 0.51; v_{max} (KBr) 2949, 1711, 1367, 1338, 1310, 1256, 1117, 902 cm⁻¹; $\delta_{\rm H}$ (600 MHz, DMSO- d_6) 5.23 (1 H, s, H-8a), 4.15 (1 H, ddd, ${}^{2}J_{6.6}$ 12.6, ${}^{3}J_{6A.7B}$ 7.7, ${}^{3}J_{6A.7A}$ 3.3 Hz, H-6A), 3.82 (1 H, d, ${}^{3}J_{8c,1a}$ 3.5 Hz, H-8c), 3.46 (1 H, ddd, ${}^{2}J_{7,7}$ 12.6, ³J_{7A,6B} 6.0, ³J_{7A,6A} 3.3 Hz, H-7A), 3.43 (1 H, d, ³J_{1a,8c} 3.5 Hz, H-1a), 3.34 (1 H, ddd, ${}^{2}J_{6,6}$ 12.6, ${}^{3}J_{6B,7B}$ 9.3, ${}^{3}J_{6B,7A}$ 6.0 Hz, H-6B), 3.16 (1 H, dd, ${}^{3}J_{3aendo,3endo}$ 9.1, ${}^{3}J_{3aendo,3exo}$ 3.5 Hz, H-3a^{endo}), 3.00 (1 H, ddd, ${}^{2}J_{7,7}$ 12.6, ${}^{3}J_{7B,6B}$ 9.3, ${}^{3}J_{7B,6A}$ 7.7 Hz, H-7B), 1.77 (1 H, dd, ${}^{2}J_{3,3}$ 12.2, ${}^{3}J_{3endo,3endo}$ 9.1 Hz, H-3^{endo}), 1.55 (1 H, dd, ${}^{2}J_{3,3}$ 12.2, ³J_{3exo,3aendo} 3.5 Hz, H-3^{exo}), 1.38 (3 H, s, Me-2); δ_C (100.6 MHz, DMSO-d₆) 174.0 (C-4), 86.2 (C-8b), 84.0 (C-2), 72.1 (C-8a), 54.1 (C-3a), 51.7 and 49.0 (C-1a and C-8c), 49.7 (C-7), 38.6 (C-6), 35.3 (C-3), 16.4 (Me-2). MS (EI, 70 eV) m/z 207 (63, M⁺-64), 206 (11), 192 (12), 178 (16), 165 (32), 164 (91), 153 (12), 138 (13), 137 (32), 136 (52), 122 (33), 111 (57), 108 (48), 95 (50), 83 (31), 82 (64), 67 (63), 59 (37), 56 (36), 55 (100), 54 (56), 53 (59), 43 (67%). HRMS (DART): MH⁺, found 272.0584. C₁₁H₁₄NO₅S requires 272.0593.

Methyl (6aRS,7SR,8SR,9RS,9aRS,10RS,10aRS)-9,10bis(acetyloxy)-6-oxodecahydro-2H-8,10-

epoxycyclopenta[4,5]**pyrido**[2,1-*b*][1,3]**oxazine-7-carboxylate** (**37a**). Boron trifluoride etherate (BF₃·OEt₂, 1.52 mL, 11.4 mmol) was added in one portion to an intensively stirred and cooled (0 °C) suspension of the ester **36a** (1.0 g, 3.79 mmol) in Ac₂O (15 mL). The pale yellow transparent reaction mixture was stirred at room temperature for 1 h, and then poured into water (150 mL).

room temperature for 1 h, and then poured into water (150 mL). Saturated Na₂CO₃ was added till the mixture was basic. The mixture was extracted with CHCl₃ (3×50 mL). The organic phases were combined, washed with saturated Na₂CO₃ solution (100 mL) and dried over MgSO₄. MgSO₄ was filtered off and the solvent was evaporated. The residue was recrystallized twice from a hexane/EtOAc mixture. The compound **37a** was obtained as colorless prism-shaped crystals (0.36 g, 25 %), m.p. 186–187 °C; [Found: C, 53.42; H, 5.37; N, 3.52. C₁₇H₂₁NO₉ requires C, 53.26; H, 5.52; N, 3.65%]; R_f (EtOAc) 0.45; v_{max} (KBr) 3475,

1743, 1648, 1367, 1250, 1226, 1063 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 5.34 (1 H, s, H-10a), 4.75 (1 H, br.d, ³J_{9,9a} 1.7 Hz, H-9), 4.73 (1 H, t, ${}^{3}J_{8,7} \sim {}^{3}J_{8,9}$ 1.0 Hz, H-8), 4.56 (1 H, ddt, ${}^{2}J_{4,4}$ 13.2, ${}^{3}J_{4e,3a}$ 4.9, ${}^{3}J_{4e,3e} \sim {}^{4}J_{4e,2e}$ 1.7 Hz, H-4^{eq}), 4.03 (1 H, ddt, ${}^{2}J_{2,2}$ 12.7, ${}^{3}J_{2e,3a}$ 5.1, ${}^{3}J_{2e,3e} \sim {}^{4}J_{2e,4e}$ 1.7 Hz, H-2^{eq}), 3.62 (1 H, ddd, ${}^{2}J_{2,2}$ 12.7, ${}^{3}J_{2a,3e}$ 11.4, ${}^{3}J_{2a,3e}$ 2.6 Hz, H-2^{*ax*}), 3.54 (3 H, s, CO₂Me), 3.17 (1 H, dd, ${}^{3}J_{6a,9a}$ 4.5, ${}^{3}J_{9,9a}$ 1.7 Hz, H-9a), 3.10 (1 H, dd, ${}^{3}J_{6a,7}$ 11.5, ${}^{3}J_{6a,9a}$ 4.5 Hz, H-6a), 3.09 (1 H, dd, ³J_{6a,7} 11.5, ³J_{7,8} 1.0 Hz, H-7), 2.70 (1 H, dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 13.2, ${}^{3}J_{4a,3e}$ 3.3 Hz, H-4^{*ax*}), 1.99 (3 H, s, OCOMe), 1.94 (3 H, s, OCOMe), 1.87–1.75 (1 H, m, H-3^{ax}), 1.45–1.41 (1 H, m, ${}^{2}J_{3,3}$ 13.6 Hz, H-3^{eq}). The ¹H NMR spectrum was recorded in deuterobenzene for unequivocal assignment of signals. In this case traces of pentadeuteriobenzene (C_6D_5H , δ 7.15 ppm) were used as the internal standard. $\delta_{H}~(400$ MHz, $C_{6}D_{6})$ 5.73 (1 H, s, H-10a), 4.69 (1 H, ddt, ${}^{2}J_{4,4}$ 13.2, ${}^{3}J_{4e,3a}$ 4.9, ${}^{3}J_{4e,3e} \sim {}^{4}J_{4e,2e}$ 1.7 Hz, H-4^{eq}), 4.54 (1 H, t, ${}^{3}J_{8,7} \sim {}^{3}J_{8,9}$ 1.0 Hz, H-8), 4.29 (1 H, br.d, ${}^{3}J_{9,9a}$ 1.7 Hz, H-9), 3.71 (1 H, ddt, ${}^{2}J_{2,2}$ 11.4, ${}^{3}J_{2e,3a}$ 5.1, ${}^{3}J_{2e,3e} \sim {}^{4}J_{2e,4e}$ 1.7 Hz, H-2^{eq}), 3.30 (1 H, ddd, ${}^{2}J_{2,2}$ 12.7, ${}^{3}J_{2a,3a}$ 11.4, ${}^{3}J_{2a,3e}$ 2.6 Hz, H-2^{ax}), 3.29 (3 H, s, CO₂Me), 3.21 (1 H, dd, ³J_{6a,9a} 4.5, ³J_{9,9a} 1.7 Hz, H-9a), 2.89 (1 H, dd, ³J_{6a,7} 11.5, ³J_{6a,9a} 4.5 Hz, H-6a), 2.57 (1 H, dd, ${}^{3}J_{6a,7}$ 11.5, ${}^{3}J_{7,8}$ 1.0 Hz, H-7), 2.43 (1 H, dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 13.2, ³J_{4a,3e} 3.3 Hz, H-4^{ax}), 1.70 (3 H, s, OCOMe), 1.63 (3 H, s, OCOMe), 1.63–1.56 (1 H, m, H-3^{ax}), 0.68–0.64 (1 H, m, H-3^{eq}); δ_{C} (100.6 MHz, CDCl₃) 169.7 (s, C(9)-OC=O), 168.0 (s, C(10)-OC=O), 167.4 (s, CO₂Me), 164.7 (s, C-6), 102.2 (s, C-10), 85.2 (d, J 165.7 Hz, C-10a), 81.6 (d, J 175.8 Hz, C-8), 76.2 (d, J 159.0 Hz, C-9), 68.4 (t, J 144.5 Hz, C-2), 51.9 (q, J 147.5 Hz, CO₂Me), 45.2 (d, J 135.0 Hz, C-7), 44.0 (d, J 157.2 Hz, C-9a), 41.2 (t, J 141.5 Hz, C-4), 37.9 (d, J 144.5 Hz, C-6a), 24.8 (t, J 129.5 Hz, C-3), 21.3 (q, J 129.8 Hz, OCOMe), 20.4 (q, J 130.0 Hz, OCOMe). 1D NOE ¹H NMR (CDCl₃), %: η_{H-10a} {2-H_a} 12.6 %; η_{H-10a} {4-H_a} 9.5 %; NOE 1D (C₆D₆), %: η_{H-9} {H-9_a} 5.9 %; η_{H-9} $_{6a} \{ H\text{-}9_a \} \ 6.8 \ \%; \ \eta_{Ha\text{-}2} \{ H\text{-}10_a \} \ 8.0 \ \%; \ \eta_{Ha\text{-}4} \{ H\text{-}10_a \} \ 6.3 \ \%; \ \eta_{H\text{-}7} \{ H\text{-}10_a \}$ 8} 7.3 %; η_{H-8} {H-9} 6.8 %; η_{H-6a} {H-9} 13.0 %; η_{H-7} {H-9} 11.3 %; η_{H-9a} {H-9} 3.9 %. GC-MS (EI, 70 eV) m/z 383 (4, M⁺), 352 (2), 341 (35), 340 (45), 324 (11), 310 (7), 299 (4), 281 (5), 264 (22), 255 (36), 236 (4), 213 (5), 166 (7), 151 (6), 124 (7), 113 (10), 86 (100), 81 (4), 58 (5), 43 (51%).

(6aRS,8SR,9RS,9aRS,10RS,10aRS)-6-Oxooctahydro-2H-8,10epoxycyclopenta[4,5]pyrido[2,1-b][1,3]oxazine-9,10(10aH)-

diyl diacetate (37b) was obtained similar to the preparation of the polycyclic compound 37a from diepoxide 36b (0.45 g, 2.02 mmol), BF3·OEt2 (0.89 mL, 7.06 mmol) and Ac2O (6 mL). About 0.5 g of dark brown oil was isolated, which was purified by Al_2O_3 column chromatography (3 × 20 cm, eluent: hexane/EtOAc, $4/1 \rightarrow 1/1$). After recrystallization from EtOAc with a little EtOH added, the compound 37b was isolated as a white powder (0.20 g, 30 %), m.p. 166 °C; [Found: C, 55.45; H, 5.81; N, 4.47. C₁₅H₁₉NO₇ requires C, 55.38; H, 5.89; N, 4.31%]; v_{max} (KBr) 1738, 1666, 1256, 1232, 1080, 1059, 904 cm⁻¹; δ_{H} (400 MHz, CDCl₃) 5.59 (1 H, s, H-10a), 4.74 (1 H, s, H-9), 4.64 (1 H, ddd, ${}^{2}J_{4,4}$ 13.1, ${}^{3}J_{4e,3a}$ 5.0, ${}^{3}J_{4e,3e}$ 1.9 Hz, H-4^{*eq*}), 4.59 (1 H, s, H-8), 4.13 (1 H, ddd, ${}^{2}J_{2,2}$ 12.5, ${}^{3}J_{2e,3a}$ 5.0, ${}^{3}J_{2e,3e} \sim 1.0$ Hz, H-2^{eq}), 3.75 (1 H, dt, ${}^{2}J_{2,2} \sim {}^{3}J_{2a,3a}$ 12.5, ${}^{3}J_{2a,3a}$ 2.5 Hz, H-2^{*ax*}), 3.20 (1 H, br.d, ${}^{3}J_{6a,9a}$ 3.1 Hz, H-9a), 2.89 (1 H, dt, ${}^{3}J_{6a,7exo}$ 12.1, ${}^{3}J_{6a,9a} \sim {}^{3}J_{6a,7endo}$ 3.1 Hz, H-6a), 2.76 (1 H, dt, ${}^{2}J_{4,4} \sim {}^{3}J_{4a,3a}$ 13.1, ${}^{3}J_{4a,3e}$ 3.1 Hz, H-4^{*ax*}), 2.15 (1 H, dd, ²J_{7,7} 13.7, ³J_{6a,7exo} 12.1 Hz, H-7^{*exo*}), 2.07 (3 H, s, OCOMe-9), 2.02 (3 H, s, OCOMe-10), 1.88 (1 H, m, H- 3^{ax}), 1.60 (1 H, dd, ${}^{2}J_{7,7}$ 13.7, ${}^{3}J_{6a,7endo}$ 3.1 Hz, H-7^{endo}), 1.48–1.44 (1 H, m, H-3^{eq}); δ_{C} (100.6 MHz, CDCl₃) 170.2 (C(9)-OC=O), 169.2 (C-6), 168.7 (C(10)-OC=O), 102.4 (C-10), 85.3 (C-10a), 80.7 (C-8), 78.0 (C-9), 69.0 (C-2), 43.5 (C-9a), 42.0 (C-4), 35.8 (C-6a), 33.4 (C-7), 25.7 (C-3), 21.9 (C(10)-OCOMe), 20.8 (C(9)- OCOMe). GC-MS (EI, 70 eV) m/z 325 (2, M^+), 284 (4), 282 (30), 281 (46), 266 (13), 265 (22), 264 (13), 224 (9), 223 (12), 222 (8), 206 (23), 205 (62), 166 (13), 138 (5), 128 (5), 97 (6), 93 (7), 86 (69), 83 (19), 66 (14), 55 (11), 43 (100%).

7,9-epoxycyclopenta[d][1,3]thiazolo[3,2-Synthesis of a)pyridines (39). General experimental procedure. $BF_3 \cdot OEt_2$ (1.33 mL, 10.5 mmol) was added to a cooled (0 °C) suspension of diepoxide 38a (38b) (~ 0.54 g, 2.1 mmol) in Ac₂O (6 mL) and the mixture was cooled at this temperature for 2 h (TLC monitoring). The reaction mixture was then poured into a saturated Na₂CO₃ solution (70 mL) and extracted with CHCl₃ (3 \times 50 mL). The combined organic phases were washed with saturated Na₂CO₃ solution (100 mL) and dried with MgSO₄, which was then removed by filtration. The solvent was removed under reduced pressure and the dark brown oil obtained was purified by column chromatography (Al₂O₃, 2×20 cm, eluent: hexane/EtOAc, $4/1 \rightarrow 1/1$). The polycycles 39 were obtained as white or pale yellow crystalline compounds.

(5aRS,7SR,8RS,8aRS,9RS,9aSR)-1,1-Dioxido-5-oxooctahydro-7,9-epoxycyclopenta[d][1,3]thiazolo[3,2-a]pyridine-8,9(9aH)-

diyl diacetate (39a). White powder (0.50 g, 46 %), m.p. 253.7-254.8 °C (decomp., from EtOAc); R_f (EtOAc) 0.46; v_{max} (KBr) 1746, 1677, 1256 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 5.07 (1 H, s, H-9a), 4.83 (1 H, br.s, H-7), 4.82 (1 H, d, ³J_{8,8a} 1.8 Hz, H-8), 4.59 (1 H, ddd, ${}^{2}J_{3,3}$ 12.6, ${}^{3}J_{3A,2A}$ 8.2, ${}^{3}J_{3A,2B}$ 3.7 Hz, H-3A), 3.99 (1 H, ddd, ${}^{3}J_{8a,5a}$ 4.6, ${}^{3}J_{8aa}$ 1.8, ${}^{4}J_{7,8a}$ 0.9 Hz, H-8a), 3.47 (1 H, ddd, ${}^{2}J_{3,3}$ 12.6, ³J_{3B,2A} 8.7, ³J_{3B,2B} 7.3 Hz, H-3B), 3.29–3.15 (2 H, m, H-2), 3.03 (1 H, ddd, ${}^{3}J_{5a,6endo}$ 11.5, ${}^{3}J_{8a,5a}$ 4.6, ${}^{3}J_{5a,6exo}$ 3.5 Hz, H-5a), 2.31 (1 H, ddd, ²J_{6,6} 13.3, ³J_{5a,6endo} 11.5, ³J_{7,6endo} 0.9 Hz, H-6^{endo}), 2.17 (3 H, s, OAc), 2.08 (3 H, s, OAc), 1.73 (1 H, ddd, ²J_{6.6} 13.3, ${}^{3}J_{5a,6exo}$ 3.5, ${}^{3}J_{7,6exo}$ 1.4 Hz, H-6^{exo}); δ_{C} (100.6 MHz, DMSO-d₆) 169.5, 169.0, 168.7 (2 × COMe and C-5), 103.6 (C-9), 80.1, 75.6, 70.0 (C-7, C-8, C-9a), 47.3 (C-2), 43.7 (C-8a), 38.7 (C-3), 36.0 (C-5a), 33.8 (C-6), 21.1 and 20.3 $(2 \times COMe)$. GC-MS (EI, 70 eV) m/z 341 (1, M⁺-18), 323 (1), 295 (6, M⁺-64), 281 (3), 266 (4), 253 (14), 235 (5), 207 (5), 193 (6), 164 (3), 138 (3), 111 (2), 96 (3), 82 (6), 81 (2), 66 (26), 65 (7), 44 (13), 43 (100%). HRMS (DART): MH⁺, found 360.3580. C₁₄H₁₈NO₈S requires 360.3596. (5aRS,7SR,8RS,8aRS,9RS,9aSR)-7-Methyl-1,1-dioxido-5oxooctahydro-7,9-epoxycyclopenta[d][1,3]thiazolo[3,2-

a]pyridine-8,9(9aH)-diyl diacetate (39b). Pale yellow powder

(0.52 g, 67 %), m.p. 225.8-226.4 °C (decomp., from EtOH/DMF); R_f (EtOAc) 0.61; v_{max} (KBr) 1742, 1666, 1327, 1261, 1248, 1225, 1140 cm⁻¹; $\delta_{\rm H}$ (600 MHz, CDCl₃) 5.03 (1 H, s, H-9a), 4.86 (1 H, d, ${}^{3}J_{8,8a}$ 1.4 Hz, H-8), 4.55 (1 H, ddd, ${}^{2}J_{3,3}$ 13.1, ${}^{3}J_{3A,2A}$ 8.9, ${}^{3}J_{3A,2B}$ 4.1 Hz, H-3A), 3.94 (1 H, dd, ${}^{3}J_{8a,5a}$ 3.8, ${}^{3}J_{8,8a}$ 1.4 Hz, H-8a), 3.47 (1 H, br.dt, ${}^{2}J_{3,3}$ 13.1, ${}^{3}J_{3B,2A} \sim {}^{3}J_{3B,2B}$ 8.3 Hz, H-3B), 3.27–3.16 (2 H, m, H-2), 3.00 (1 H, br.dt, ³J_{5a,6endo} 11.7, ${}^{3}J_{8a,5a} \sim {}^{3}J_{5a,6exo}$ 3.8 Hz, H-5a), 2.24 (1 H, dd, ${}^{2}J_{6,6}$ 13.7, ${}^{3}J_{5a,6endo}$ 11.7 Hz, H-6^{endo}), 2.15 (3 H, s, OAc), 2.09 (3 H, s, OAc), 1.69 (1 H, dd, ${}^{2}J_{6,6}$ 13.7, ${}^{3}J_{5a,6exo}$ 3.8 Hz, H-6^{exo}), 1.45 (3 H, s, Me-7); δ_{C} (100.6 MHz, DMSO-d₆) 169.5, 169.1, 168.8 (2 × COMe and C-5), 103.8 (C-9), 87.8 (C-7), 76.0 (C-8), 70.2 (C-9a), 47.3 (C-2), 45.0 (C-8a), 38.9 and 38.7 (C-3 and C-6), 36.0 (C-5a), 21.2 and 20.3 (2 \times COMe), 14.5 (Me-7). The C₃ and C₆ signals in a ¹³C NMR spectrum of **39b** in DMSO were overlapped by solvent peaks, therefore, a spectrum in CDCl₃ was recorded. $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 170.1, 169.7, 168.9 ($2 \times COMe$ and C-5), 103.9 (C-9), 88.7 (C-7), 76.6 (C-8), 71.3 (C-9a), 48.7 (C-2), 45.4 (C-8a), 39.9 and 38.9 (C-2 and C-6), 36.7 (C-5a), 21.7 and 20.7 (2 \times COMe), 15.0 (Me-7). GC-MS (EI, 70 eV) m/z 309 (3, M⁺-64), 272 (3), 271 (15), 270 (5), 250 (3), 243 (2), 207 (5), 202 (6), 179 (4), 150 (3), 138 (8), 124 (7), 109 (6), 96 (14), 80 (13), 69 (7), 55

(7), 44 (8), 43 (100%). HRMS (DART): MH^+ , found 374.0867. $C_{15}H_{20}NO_8S$ requires 374.0910.

Synthesis of 7b,10-epoxyisoindolo[2,1-*a*]perimidines (41). General experimental procedure. A solution of naphthalene-1,8-diamine (0.79 g, 5.0 mmol) and furfural (5-methylfurfural) (~ 0.5 mL, 5.0 mmol) in EtOH (10 mL) was stirred at room temperature for 10 min. The solvent was removed under reduced pressure affording intermediate 2-(2-furyl)-2,3-dihydro-1*H*-perimidines (40a,b) as brown, quickly crystallizing oils, which were used for the next step without further purification and assuming quantitative yield. No spectral data on perimidines (40) were found in the literature, therefore, we obtained analytically pure samples by recrystallization and characterized them by NMR spectra.

2-(2-Furyl)-2,3-dihydro-1H-perimidine colorless (40a), needles (yield 85 %), m.p. 101.2-102.0 °C (hexane-EtOAc), [Lit.:^[36a] m.p. 100–101 °C]; v_{max} (KBr) 3346, 3307, 1597, 1409, 749 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 7.42 (1 H, br.d, ${}^{3}J_{5',4'}$ 1.6 Hz, H-5'), 7.28–7.21 (4 H, m, H-Ar), 6.57 (2 H, dd, ${}^{3}J_{4(9),5(8)}$ 7.2, ${}^{4}J_{4(9),6(7)}$ 1.6 Hz, H-4 and H-9), 6.40 (1 H, br.d, ³J_{3',4'} 3.3 Hz, H-3'), 6.35 (1 H, dd, ³*J*_{3',4'} 3.3, ³*J*_{5',4'} 1.6 Hz, H-4'), 5.63 (1 H, s, H-2), 4.45 (2 H, br.s, NH); δ_C (100.6 MHz, CDCl₃) 153.5 (C-2'), 142.7 (2 C, C-3a and C-9a), 140.8 (C-5'), 134.8 (C-6a), 127.0 (2 C, C-5 and C-8), 118.3 (2 C, C-6 and C-7), 114.0 (C-9b), 110.6 and 107.8 (2 C, C-3' and C-4'), 106.7 (2 C, C-4 and C-9), 61.6 (C-2). GC-MS (EI, 70 eV) m/z 236 (100, M⁺), 235 (87), 219 (14), 207 (18), 206 (20), 205 (22), 182 (9), 169 (53), 168 (50), 140 (25), 127 (11), 115 (41), 103 (12), 39 (20%).

2-[2-(5-Methylfuryl)]-2,3-dihydro-1*H***-perimidine (40b), aggregates of tiny yellow needles (yield 57 %), m.p. 124.7–126.5 °C (hexane–EtOAc), [Lit:.^[36a] m.p. 123 °C]; v_{max} (KBr) 3332, 3272, 1600, 1411, 821, 758 cm⁻¹; \delta_{\rm H} (400 MHz, CDCl₃) 7.27–7.20 (4 H, m, H-Ar), 6.56 (2 H, dd, {}^{3}J_{4(9),5(8)} 6.9, {}^{4}J_{4(9),6(7)} 1.5 Hz, H-4 and H-9), 6.30 (1 H, d, {}^{3}J_{3',4'} 3.0 Hz, H-3'), 5.94 (1 H, dq, {}^{3}J_{3',4'} 3.0, {}^{4}J_{{\rm Me},5',4'} 1.1 Hz, H-4'), 5.55 (1 H, s, H-2), 4.65 (2 H, br.s, NH), 2.32 (3 H, br.s, Me-5'); \delta_{\rm C} (100.6 MHz, CDCl₃) 152.7 (C-2'), 151.3 (C-5'), 141.1 (2 C, C-3a and C-9a), 134.9 (C-6a), 126.9 (2 C, C-5 and C-8), 118.3 (2 C, C-6 and C-7), 114.0 (C-9b), 108.9 and 106.5 (2 C, C-3' and C-4'), 106.7 (2 C, C-4 and C-9), 61.8 (C-2), 13.7 (Me-5'). GC-MS (EI, 70 eV)** *m***/z 250 (2, M⁺), 248 (100), 219 (16), 207 (34), 205 (49), 166 (7), 140 (31), 113 (10), 53 (21), 44 (19%).**

Crude perimidines **40** (~ 5 mmol) were dissolved in the appropriate solvent (CH₂Cl₂, Me₂CO or PhMe, 10 mL) and maleic anhydride (0.49 g, 5.0 mmol) was added with intensive stirring. The orange-red color of the reaction mixture disappeared in 2–3 min, and a plentiful precipitation formed. The mixture was stored at room temperature for 1 d. The precipitate was collected by filtration, washed with CH₂Cl₂ (2 × 5 mL), dried in air till constant weight and its isomeric composition was analyzed by ¹H NMR.

(7aRS,7bRS,10SR,11RS,11aSR)-12-Oxo-7,7a,10,11,11a,12-

hexahydro-7b,10-epoxyisoindolo[2,1-a]perimidine-11carboxylic acid (41Aa) and (7aRS,7bSR,10RS,11SR,11aRS)-

12-oxo-7,7a,10,11,11a,12-hexahydro-7b,10-

epoxyisoindolo[2,1-*a*]perimidine-11-carboxylic acid (41Ba). Colorless powder. Reaction in CH₂Cl₂ gives rise to a mixture of isomers in ratio 41Aa/41Ba ~ 60/40 (1.52 g, 91 %). The ratio of 41Aa/41Ba ~ 89/11, (1.08 g, 65 %) was obtained in Me₂CO. A similar synthesis in toluene under reflux (Δ , 10 min) affords the following mixture of isomers: 41Aa/41Ba ~ 54/46 (1.58 g, 95 %). M.p. 202–206 °C (decomp.); [Found: C, 68.17; H, 4.08; N, 8.48. C₁₉H₁₄N₂O₄ requires C, 68.26; H, 4.22; N, 8.38%]; v_{max}

(KBr) 3298, 1725, 1688, 1610, 1421, 1210 cm⁻¹; $\delta_{\rm H}$ (400 MHz, DMSO- d_6) (41Aa) 12.38 (1 H, br.s, CO₂H), 7.62 (1 H, dd, ${}^{3}J_{3,2}$ 7.7, ${}^{4}J_{1,3}$ 1.6 Hz, H-3), 7.49 (1 H, dd, ${}^{3}J_{1,2}$ 7.1, ${}^{4}J_{1,3}$ 1.6 Hz, H-1), 7.46 (1 H, dd, ${}^{3}J_{2,3}$ 7.7, ${}^{3}J_{1,2}$ 7.1 Hz, H-2), 7.38 (1 H, dd, ${}^{3}J_{5,4}$ 7.7, ${}^{3}J_{5,6}$ 7.2 Hz, H-5), 7.35 (1 H, s, NH), 7.32 (1 H, dd, ${}^{3}J_{4,5}$ 7.7, ${}^{4}J_{4,6}$ 0.9 Hz, H-4), 7.01 (1 H, d, ${}^{3}J_{9.8}$ 5.8 Hz, H-8), 6.89 (1 H, dd, ${}^{3}J_{5.6}$ 7.2, ${}^{4}J_{6,4}$ 0.9 Hz, H-6), 6.61 (1 H, dd, ${}^{3}J_{9,8}$ 5.8, ${}^{3}J_{9,10}$ 1.6 Hz, H-9), 5.21 (1 H, d, ³J_{9.10} 1.6 Hz, H-10), 5.20 (1 H, s, H-7a), 3.09 (1 H, d, ${}^{3}J_{11,11a}$ 9.1 Hz, H-11a), 2.69 (1 H, d, ${}^{3}J_{11,11a}$ 9.1 Hz, H-11); (**41Ba**) 12.36 (1 H, br.s, CO₂H), 8.21 (1 H, dd, ${}^{3}J_{1,2}$ 7.6, ${}^{4}J_{1,3}$ 0.8 Hz, H-1), 7.53 (1 H, dd, ${}^{3}J_{3,2}$ 7.6, ${}^{4}J_{1,3}$ 0.8 Hz, H-3), 7.42 (1 H, t, ${}^{3}J_{1,2} \sim {}^{3}J_{2,3}$ 7.6 Hz, H-2), 7.32 (1 H, t, ${}^{3}J_{5,4} \sim {}^{3}J_{5,6}$ 7.5 Hz, H-5), 7.23 (1 H, dd, ${}^{3}J_{4,5}$ 7.5, ${}^{4}J_{4,6}$ 0.7 Hz, H-4), 7.09 (1 H, d, ${}^{3}J_{\rm NH,7a}$ 1.3 Hz, NH), 6.81 (1 H, dd, ³J_{5,6} 7.5, ⁴J_{6,4} 0.7 Hz, H-6), 6.73 (1 H, d, ${}^{3}J_{9,8}$ 5.7 Hz, H-8), 6.57 (1 H, dd, ${}^{3}J_{9,8}$ 5.7, ${}^{3}J_{9,10}$ 1.7 Hz, H-9), 5.64 (1 H, d, ³J_{NH,7a} 1.3 Hz, H-7a), 5.16 (1 H, d, ³J_{9,10} 1.7 Hz, H-10), 3.21 (1 H, d, ³J_{11,11a} 9.1 Hz, H-11a), 2.66 (1 H, d, ³J_{11,11a} 9.1 Hz, H-11); δ_C (100.6 MHz, DMSO-d₆) (41Aa) 172.5 (CO₂H), 169.4 (C-12), 141.5 (C-6a), 136.6 (C-9), 133.9 (2 C, C-3a and C-8), 131.2 (C-13a), 127.0 (C-5), 125.8 (C-2), 123.6 (C-3), 117.6 (C-4), 115.6 (C-13b), 114.7 (C-1), 108.5 (C-6), 89.2 (C-7b), 81.9 (C-10), 67.4 (C-7a), 50.0 (C-11a), 44.5 (C-11); (41Ba) 172.6 (CO₂H), 168.5 (C-12), 140.7 (C-6a), 137.2 (C-9), 134.5 (C-8), 133.9 (C-3a), 132.3 (C-13a), 127.0 (C-5), 125.9 (C-2), 122.5 (C-3), 116.8 (C-4), 113.5 (C-13b), 110.9 (C-1), 107.8 (C-6), 88.4 (C-7b), 81.3 (C-10), 65.3 (C-7a), 51.0 (C-11a), 45.0 (C-11). MS (EI, 70 eV) *m/z* for isomer **41Aa** 334 (19, M⁺), 316 (10), 300 (2), 290 (5), 272 (7), 262 (4), 243 (4), 237 (41), 236 (70), 235 (100), 234 (73), 219 (8), 205 (41), 200 (15), 184 (27), 183 (26), 169 (46), 168 (50), 154 (11), 143 (26), 140 (36), 127 (16), 118 (14), 115 (34), 106 (23), 98 (32), 91 (24), 81 (25), 73 (28), 65 (17), 54 (51), 43 (50%).

The single isomer **41Aa** was isolated by fractional crystallization of the isomer mixtures from *i*-PrOH/DMF as a grey powder, m.p. $205.5-207 \text{ }^{\circ}\text{C}$ (decomp.).

(7aRS,7bRS,10SR,11RS,11aSR)-10-Methyl-12-oxo-

7,7a,10,11,11a,12-hexahydro-7b,10-epoxyisoindolo[2,1 *a*]perimidine-11-carboxylic acid (41Ab) and (7a*RS*,7b*SR*,10*RS*,11*SR*,11a*RS*)-10-methyl-12-oxo-

7,7a,10,11,11a,12-hexahydro-7b,10-epoxyisoindolo[2,1-

a]perimidine-11-carboxylic acid (41Bb). The mixture of isomers 41Ab/41Bb ~ 54/46 was obtained when the reaction was carried out in CH₂Cl₂ (1.63 g, 94 %), beige powder, m.p. 168.3-169.4 °C (decomp.); [Found: C, 69.12; H, 4.60; N, 8.25. C₂₀H₁₆N₂O₄ requires C, 68.96; H, 4.63; N, 8.04%]; v_{max} (KBr) 3444, 1728, 1666 cm⁻¹; $\delta_{\rm H}$ (400 MHz, DMSO- d_6) (41Ab) 12.43 (1 H, br.s, CO₂H), 7.60 (1 H, dd, ${}^{3}J_{5,4}$ 7.5, ${}^{3}J_{5,6}$ 6.5 Hz, H-5), 7.48–7.45 (2 H, m, H-4 and H-6), 7.37 (1 H, dd, ³J_{2,3} 7.6, ³J_{2,1} 6.4 Hz, H-2), 7.32 (1 H, dd, ³J_{3,2} 7.6, ⁴J_{1,3} 0.8 Hz, H-3), 7.31 (1 H, br.s, NH), 7.02 (1 H, d, ³J_{8,9} 5.7 Hz, H-8), 6.87 (1 H, dd, ³J_{2,1} 6.4, ⁴*J*_{1,3} 0.8 Hz, H-1), 6.43 (1 H, d, ³*J*_{8,9} 5.7 Hz, H-9), 5.18 (1 H, s, H-7a), 3.10 (1 H, d, ³J_{11.11a} 8.9 Hz, H-11a), 2.71 (1 H, d, ³J_{11.11a} 8.9 Hz, H-11), 1.61 (3 H, s, Me-10); (41Bb) 12.38 (1 H, br.s, CO₂H), 8.29 (1 H, d, ³*J*_{2,1} 7.6 Hz, H-1), 7.53 (1 H, br.d, ³*J*_{3,2} 7.6 Hz, H-3), 7.42 (1 H, t, ${}^{3}J_{2,1} \sim {}^{3}J_{2,3}$ 7.6 Hz, H-2), 7.31 (1 H, t, ${}^{3}J_{5,6} \sim {}^{3}J_{5,4}$ 7.6 Hz, H-5), 7.23 (1 H, br.d, ³J_{5,4} 7.6 Hz, H-4), 7.08 (1 H, br.s, NH), 6.82 (1 H, dd, ${}^{3}J_{5,6}$ 7.6, ${}^{4}J_{4,6}$ 1.3 Hz, H-6), 6.74 (1 H, d, ${}^{3}J_{8,9}$ 5.1 Hz, H-8), 6.37 (1 H, d, ${}^{3}J_{9,8}$ 5.1 Hz, H-9), 5.55 (1 H, s, H-7a), 3.21 (1 H, d, ³J_{11,11a} 8.9 Hz, H-11), 2.70 (1 H, d, ³J_{11,11a} 8.9 Hz, H-11a), 1.62 (3 H, s, Me-10); δ_{C} (100.6 MHz, DMSO- d_{6}) (41Ab) 171.4 (CO₂H), 169.7 (C-12), 141.5 (C-6a), 139.5 (C-9), 134.9 (C-8), 133.9 (C-13a), 131.2 (C-3a), 127.0 (C-2), 125.9 (C-6), 123.6 (C-5), 117.6 (C-3), 115.7 (C-13b), 115.0 (C-4), 108.5 (C-

1), 89.3 (C-10), 88.8 (C-7b), 67.8 (C-7a), 53.3 (C-11a), 48.0 (C-11), 15.6 (Me-10); (**41Bb**) 171.4 (CO₂H), 168.9 (C-12), 140.8 (C-6a), 139.9 (C-9), 135.8 (C-8), 133.9 (C-13a), 132.6 (C-3a), 127.1 (C-2), 125.9 (C-6), 122.6 (C-5), 116.8 (C-3), 113.5 (C-13b), 110.8 (C-4), 107.8 (C-1), 88.8 (C-10), 87.8 (C-7b), 65.7 (C-7a), 54.3 (C-11a), 48.2 (C-11), 15.7 (Me-10). MS (EI, 70 eV) m/z 348 (4, M⁺), 330 (2), 273 (3), 252 (23), 251 (64), 250 (100), 249 (47), 236 (6), 219 (8), 206 (27), 195 (4), 183 (5), 169 (43), 168 (66), 140 (14), 127 (10), 115 (14), 106 (7), 97 (8), 54 (27), 43 (38%).

(7a*RS*,7b*RS*,10*RS*,11a*SR*)-7,7a,11,11a-Tetrahydro-7b,10epoxyisoindolo[2,1-*a*]perimidin-12(10*H*)-one (42A) and (7a*RS*,7b*SR*,10*SR*,11a*RS*)-7,7a,11,11a-tetrahydro-7b,10-

epoxyisoindolo[2,1-a]perimidin-12(10H)-one (42B). A mixture of 2-(2-furyl)-2,3-dihydro-1H-perimidine (40a) (0.46 g, 1.90 mmol), acryloyl chloride (0.16 mL, 2.0 mmol) and $\ensuremath{\text{NEt}}_3$ (0.32 mL, 2.3 mmol) in PhMe (30 mL) was heated under reflux for 2 h, then cooled and poured into water (50 mL). The organic layer was separated and the water layer was extracted with EtOAc (3 \times 20 mL). The organic phases were combined and dried over MgSO₄, which was then filtered off. After the solvents were evaporated under reduced pressure, dark-brown oil was obtained, which was triturated with EtOAc (4 mL). Brown crystals obtained were collected by filtration and recrystallized from a hexane/EtOAc mixture affording a mixture of 42A/42B in the ratio of ~ 48/52 (0.15 g, 27 %). The mother liquors were combined and purified by silica gel column chromatography (1.8 \times 11.5 cm, eluent: EtOAc/hexane 1/10 \rightarrow 1/1). Additional 70 mg (9%) of the individual isomer **42B** (70 mg, 9%) were isolated.

Compound 42B, thick colourless plates, m.p. 199–200 °C (hexane/EtOAc); [Found: C, 74.53; H, 4.87; N, 9.74. C₁₈H₁₄N₂O₂ requires C, 74.47; H, 4.86; N, 9.65%]; v_{max} (KBr) 3292, 1685, 1596, 1423, 817, 765 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) (42B) 8.33 (1 H, dd, ${}^{3}J_{1,2}$ 7.3, ${}^{4}J_{1,3}$ 0.9 Hz, H-1), 7.58 (1 H, dd, ${}^{3}J_{3,2}$ 7.3, ${}^{4}J_{1,3}$ 0.9 Hz, H-3), 7.47 (1 H, t, ${}^{3}J_{1,2} \sim {}^{3}J_{2,3}$ 7.3 Hz, H-2), 7.45 (1 H, dd, ${}^{3}J_{4,5}$ 8.0, ${}^{4}J_{4,6}$ 0.9 Hz, H-4), 7.36 (1 H, dd, ${}^{3}J_{5,4}$ 8.0, ${}^{3}J_{5,6}$ 7.3 Hz, H-5), 6.92 (1 H, dd, ³J_{5,6} 7.3, ⁴J_{6,4} 0.9 Hz, H-6), 6.59 (1 H, d, ³J_{9,8} 6.0 Hz, H-8), 6.53 (1 H, dd, ${}^{3}J_{9,8}$ 6.0, ${}^{3}J_{9,10}$ 1.6 Hz, H-9), 5.56 (1 H, s, H-7a), 5.21 (1 H, dd, ${}^{3}J_{11ex0,10}$ 4.6, ${}^{3}J_{9,10}$ 1.6 Hz, H-10), 2.77 (1 H, dd, ³J_{11a,11exo} 3.7, ³J_{11endo,11a} 8.7 Hz, H-11a), 2.39 (1 H, ddd, ${}^{3}J_{11\exp,10}$ 4.6, ${}^{2}J_{11,11}$ 11.9, ${}^{3}J_{11a,11\exp}$ 3.7 Hz, H-11^{*exo*}), 1.74 (1 H, dd, ${}^{2}J_{11,11}$ 11.9, ${}^{3}J_{11endo,11a}$ 8.7 Hz, H-11^{*endo*}); δ_{C} (100.6 MHz, CDCl₃) (42B) 171.2 (C-12), 138.1 and 132.2 (C-9 and C-8), 137.9 (C-6a), 134.4 (C-3a), 131.8 (C-13a), 126.7 and 126.6 (C-5 and C-2), 123.6 and 121.6 (C-3 and C-4), 116.6 (C-13b), 113.9 and 113.5 (C-1 and C-6), 89.2 (C-7b), 79.5 (C-10), 67.2 (C-7a), 48.5 (C-11a), 28.7 (C-11). MALDI-TOF HR: MNa⁺, found 313.1015. $C_{18}H_{14}N_2NaO_2$ requires 313.0953. $\delta_{\rm H}$ (400 MHz, CDCl_3) (42A in mixture **42A**/**42B** ~ 13/87) 7.76 (1 H, dd, ${}^{3}J_{1,2}$ 7.5, ${}^{4}J_{1,3}$ 1.2 Hz, H-1), 7.57 (1 H, br.d, ${}^{3}J_{3,2}$ 7.5 Hz, H-3), 7.47 (4 H, three m overlapping with signals of 42B, H-2, H-4, H-5), 6.77 (1 H, dd, ³*J*_{6.5} 7.5, ⁴*J*_{6.4} 1.2 Hz, H-6), 6.65 (1 H, d, ³*J*_{9.8} 6.2 Hz, H-8), 6.46 (1 H, dd, ³J_{9,8} 6.2, ³J_{9,10} 1.8 Hz, H-9), 5.24 (1 H, s, H-7a), 5.19 (1 H, dd, ³J_{11exo,10} 4.5, ³J_{9,10} 1.8 Hz, H-10), 2.70 (1 H, dd, ³J_{11endo,11a} 8.7, ${}^{3}J_{11a,11exo}$ 3.7 Hz, H-11a), 2.36 (1 H, ddd, ${}^{2}J_{11,11}$ 11.8, ${}^{3}J_{11exo,10}$ 4.5, ³*J*_{11a,11exo} 3.7 Hz, H-11^{*exo*}), 1.70 (1 H, dd, ²*J*_{11,11} 11.8, ³*J*_{11endo,11a} 8.7 Hz, H-11^{endo}); δ_C (100.6 MHz, CDCl₃) (**42A**) 171.1 (C-12), 140.4 (C-6a), 137.4 and 131.1 (C-9 and C-8), 134.4 (C-3a), 131.8 (C-13a), 126.6 and 126.5 (C-5 and C-2), 124.2 and 119.8 (C-3 and C-4), 115.8 (C-13b), 113.5 and 113.3 (C-1 and C-6), 90.0 (C-7b), 79.9 (C-10), 69.1 (C-7a), 47.3 (C-11a), 28.3 (C-11).

Synthesis of 2,4a-epoxyisoindolo[1,2-b]quinazolin-12-ones (44). General experimental procedure. A mixture of 2(aminomethyl)aniline (1.22 g, 10.0 mmol), furfural (0.89 mL, 10.5 mmol) and anhydrous powdered MgSO₄ (2.40 g, 20.0 mmol) in CH₂Cl₂ (20 mL) was stirred at room temperature for 2 h. MgSO₄ was filtered off and washed with CH₂Cl₂ (2×10 mL). The obtained solution was used in the next step with maleic anhydride assuming quantitative yield of quinazoline 43a. According to ¹H NMR, the mixture contains ~ 93% of the target 2-(furan-2-yl)-1,2,3,4-tetrahydroquinazoline (43a) (the chain tautomeric form was not observed). Some spectral data of 43a are presented. White needle-shaped crystals. $\delta_{\rm H}$ (400 MHz, CDCl₃) 7.40 (1 H, br.d, ${}^{3}J_{5',4'}$ 1.6 Hz, H-5'), 7.04 (1 H, br.dd, ${}^{3}J_{7,8}$ 8.1, ${}^{3}J_{6,7}$ 7.5 Hz, H-7), 6.91 (1 H, br.d, ${}^{3}J_{5,6}$ 7.5 Hz, H-5), 6.72 (1 H, dt, ${}^{3}J_{6,5} \sim {}^{3}J_{6,7}$ 7.5, ${}^{4}J_{6,8}$ 1.2 Hz, H-6), 6.59 (1 H, br.d, ${}^{3}J_{7,8}$ 8.1 Hz, H-8), 6.38 (1 H, br.d, ${}^{3}J_{3',4'}$ 3.1 Hz, H-3'), 6.35 (1 H, dd, ${}^{3}J_{3',4'}$ 3.1, ${}^{3}J_{5',4'}$ 1.6 Hz, H-4'), 5.35 (1 H, s, H-2), 4.17 (1 H, d, ${}^{2}J_{4,4}$ 16.8 Hz, H-4A), 3.96 (1 H, d, ${}^{2}J_{4,4}$ 16.8 Hz, H-4B); δ_{C} (100.6 MHz, CDCl₃) 154.0 (C-2'), 142.8 (C-8a), 142.4 (C-5'), 127.5 and 126.3 (C-5 and C-7), 121.4 (C-4a), 118.6 (C-6), 115.5 (C-8), 110.4 and 106.8 (C-3' and C-4'), 63.9 (C-2), 45.6 (C-4).

Maleic anhydride (1.03 g, 10.5 mmol) was added to a solution of quinazoline **43a** (~ 10 mmol) obtained above in CH₂Cl₂ (~ 35 mL). The brown-red transparent reaction mixture was stirred for 1 h and then stored at room temperature for 24 h. The crystals formed were collected by filtration, washed with Me₂CO (3 × 6 mL), Et₂O (2 × 10 mL) and air-dried till constant weight. The mixture of isomers **44a** was obtained as pale pink powder (2.34 g, 78 %), isomer ratio **44Aa/44Ba** ~ 82/18; m.p. 134.3–136 °C (decomp.).

A transparent solution is formed when this mixture (1.0 g) was brought to reflux in MeOH/DMF (10 mL/7 mL). The solution was cooled and Me₂CO (5 mL) was added to it. The solution was stored at -4 °C for 24 h. The precipitate formed was collected by filtration, washed with Me₂CO (2 × 3 mL) and air-dried till constant weight. The *minor* isomer **44Ba** (95 mg) was obtained as a white powder. The mother liquor was stored open at 26 °C for a week. The crystals formed were collected by filtration, washed with acetone (2 × 5 mL) and dried in air. The isomer **44Aa** (0.3 g) was obtained as elongated pale-yellow needles.

When Me₂CO (20 mL) was used as the solvent in the second step with the same load and the mixture was then stored for 2 d at room temperature, the title acids **44Aa**/**44Ba** (1.45 g, 48 %) were obtained in the ratio of 82/18 (small needles).

When PhMe (20 mL) was used as the solvent in the second step with the same load and heated under reflux for 10 min, the title acids **44Aa/44Ba** (1.0 g, 33 %) were obtained in the ratio of 50/50. In this case, some resinification occurred and the products were obtained by multiple trituration of the brown oil with Me₂CO.

(1RS,2SR,4aRS,4bRS,12aSR)-12-Oxo-1,2,5,10,12,12ahexahydro-4bH-2,4a-epoxyisoindolo[1,2-b]quinazoline-1-

carboxylic acid (44Aa). *Major* isomer, transparent needles, m.p. 123.1–125 °C (decomp., from MeOH/DMF); [Found: 64.45; H, 4.84; N, 9.17. C₁₆H₁₄N₂O₄ requires C, 64.42; H, 4.73; N, 9.39%]; v_{max} (KBr) 3611, 3293, 1728, 1674, 1636, 1492, 1060 cm⁻¹; δ_H (400 MHz, DMSO-*d*₆) 12.20 (1 H, br.s, CO₂H), 7.00 (1 H, br.t, ${}^{3}J_{7.8} \sim {}^{3}J_{6.7}$ 7.3 Hz, H-7), 6.99 (1 H, d, ${}^{3}J_{8.9}$ 7.3 Hz, H-9), 6.83 (1 H, d, ${}^{3}J_{4.3}$ 6.0 Hz, H-4), 6.71 (1 H, br.d, ${}^{3}J_{6.7}$ 7.3 Hz, H-6), 6.68 (1 H, br.t, ${}^{3}J_{3.4}$ 6.0, ${}^{3}J_{7.8}$ 7.3 Hz, H-8), 6.51 (1 H, br.s, NH), 6.48 (1 H, dd, ${}^{3}J_{3.4}$ 6.0, ${}^{3}J_{2.3}$ 0.9 Hz, H-3), 5.08 (1 H, d, ${}^{3}J_{2.3}$ 0.9 Hz, H-2), 4.99 (1 H, s, H-4b), 4.72 (1 H, d, ${}^{2}J_{10,10}$ 16.9 Hz, H-10A), 4.22 (1 H, d, ${}^{2}J_{10,10}$ 16.9 Hz, H-10B), 2.74 (1 H, d, ${}^{3}J_{1,12a}$ 9.2 Hz, H-12a), 2.52 (1 H, d, ${}^{3}J_{1,12a}$ 9.2 Hz, H-1); $\delta_{\rm C}$ (100.6 MHz, DMSO-*d*₆) 172.2 (CO₂H), 170.3 (C-12), 143.0 (C-5a), 136.2 (C-3), 134.0

(C-4), 127.0 and 126.5 (C-7 and C-9), 118.51 and 118.46 (C-8 and C-9a), 116.2 (C-6), 89.8 (C-4a), 81.8 (C-2), 65.7 (C-4b), 48.8 (C-12a), 44.0 (C-1), 40.0 (C-10). MS (EI, 70 eV) m/z 298 (4, M⁺), 235 (2), 220 (4), 201 (11), 200 (82), 199 (49), 184 (41), 183 (67), 170 (11), 154 (12), 133 (12), 131 (29), 107 (14), 106 (100), 104 (30), 91 (19), 81 (28), 78 (33), 77 (48), 73 (29), 54 (82), 43 (32%). (1RS,2SR,4aRS,4bSR,12aSR)-12-Oxo-1,2,5,10,12,12a-hexahydro-4bH-2,4a-epoxyisoindolo[1,2-b]quinazoline-1-

carboxylic acid (44Ba). Minor isomer, white powder, m.p. 166.5-167 °C (decomp., from MeOH/DMF); [Found: C, 64.38; H, 4.90; N, 9.24. C₁₆H₁₄N₂O₄ requires C, 64.42; H, 4.73; N, 9.39%]; v_{max} (KBr) 3345, 1729, 1670, 1503, 1181 cm⁻¹; δ_{H} (400 MHz, DMSO-*d*₆) 12.15 (1 H, br.s, CO₂H), 7.04 (1 H, dd, ³*J*_{8,9}7.5, ${}^{4}J_{7,9}$ 1.3 Hz, H-9), 7.00 (1 H, br.dt, ${}^{3}J_{7,8} \sim {}^{3}J_{6,7}$ 7.5, ${}^{4}J_{7,9}$ 1.3 Hz, H-7), 6.76 (1 H, dd, ${}^{3}J_{6,7}$ 7.5, ${}^{4}J_{6,8}$ 1.0 Hz, H-6), 6.67 (1 H, d, ${}^{3}J_{4,3}$ 5.7 Hz, H-4), 6.66 (1 H, br.dt, ${}^{3}J_{8,9} \sim {}^{3}J_{7,8}$ 7.5, ${}^{4}J_{8,6}$ 1.0 Hz, H-8), 6.50 (1 H, dd, ${}^{3}J_{3,4}$ 5.7, ${}^{3}J_{2,3}$ 1.7 Hz, H-3), 6.17 (1 H, d, ${}^{3}J_{NH,4b}$ 2.0 Hz, NH), 5.33 (1 H, d, ³*J*_{NH,4b} 2.0 Hz, H-4b), 5.06 (1 H, d, ³*J*_{2,3} 1.7 Hz, H-2), 4.53 (1 H, d, ²J_{10,10} 16.9 Hz, H-10A), 4.21 (1 H, d, ²J_{10,10} 16.9 Hz, H-10B), 2.90 (1 H, d, ³J_{1.12a} 8.9 Hz, H-12a), 2.53 (1 H, d, ${}^{3}J_{1,12a}$ 8.9 Hz, H-1); δ_{C} (100.6 MHz, DMSO- d_{6}) 172.7 (CO₂H), 168.6 (C₁₂), 142.8 (C_{5a}), 136.9 (C₃), 135.0 (C₄), 127.1 (C₉), 126.9 (C7), 117.8 (C8), 116.9 (C9a), 116.0 (C6), 89.7 (C4a), 81.1 (C2), 63.8 (C_{4b}), 50.6 (C_{12a}), 44.4 (C₁), 40.2 (C₁₀). MS (EI, 70 eV) m/z 298 (9, M⁺), 280 (7), 263 (3), 254 (4), 235 (4), 225 (4), 220 (4), 200 (99), 199 (100), 184 (39), 183 (97), 169 (27), 154 (12), 133 (18), 132 (19), 131 (39), 107 (40), 106 (94), 104 (61), 98 (20), 91 (19), 81 (22), 79 (25), 78 (50), 77 (77), 69 (10), 65 (18), 54 (93), 51 (33), 43 (39%).

(1RS,2SR,4aRS,4bRS,12aSR)-2-Methyl-12-oxo-

1,2,5,10,12,12a-hexahydro-4bH-2,4a-epoxyisoindolo[1,2-

b]quinazoline-1-carboxylic acid (44Ab). Similarly to the procedure for preparation of 44a, 2-(aminomethyl)aniline (1.22 g, 10.0 mmol), 5-methylfurfural (1.05 mL, 10.5 mmol), anhydrous powdered MgSO₄ (2.40 g, 20.0 mmol) and maleic anhydride (1.03 g, 10.5 mmol) in CH₂Cl₂ (30 mL) were used to afford 44Ab (1.21 g, 39 %) as white powder, m.p. 115.6-116.9 °C (decomp., from *i*-PrOH/DMF); [Found: C, 65.31; H, 5.04; N, 9.14. C₁₇H₁₆N₂O₄ requires C, 65.38; H, 5.16; N, 8.97%]; v_{max} (KBr) 3515, 3381, 1727, 1649, 741 cm⁻¹; δ_H (400 MHz, DMSOd₆) 12.24 (1 H, br.s, CO₂H), 7.05–7.02 (2 H, m, H-7 and H-9), 6.90 (1 H, d, ${}^{3}J_{3,4}$ 5.7 Hz, H-3), 6.75 (1 H, br.d, ${}^{3}J_{7,6}$ 7.7 Hz, H-6), 6.73 (1 H, t, ${}^{3}J_{7,8} \sim {}^{3}J_{9,8}$ 7.7 Hz, H-8), 6.48 (1 H, br.s, NH), 6.35 (1 H, d, ${}^{3}J_{3,4}$ 5.7 Hz, H-4), 5.01 (1 H, s, H-4b), 4.78 (1 H, d, ${}^{2}J_{10,10}$ 16.5 Hz, H-10A), 4.24 (1 H, d, ${}^{2}J_{10,10}$ 16.5 Hz, H-10B), 2.81 (1 H, d, ${}^{3}J_{1,12a}$ 8.9 Hz, H-12a), 2.60 (1 H, d, ${}^{3}J_{1,12a}$ 8.9 Hz, H-1), 1.55 (3 H, s, Me-2); δ_C (100.6 MHz, DMSO-d₆) 180.9 (CO₂H), 180.2 (C-12), 152.7 (C-5a), 148.8 (C-3), 144.5 (C-4), 136.6 (C-7), 136.1 (C-9), 127.97 (C-6), 127.93 (C-9a), 125.7 (C-8), 98.8 (C-2), 98.6 (C-4a), 75.2 (C-4b), 61.6 (C-1), 57.0 (C-12a), 49.4 (C-10), 25.0 (Me-2). MS (EI, 70 eV) m/z 294 (3, M⁺-18), 263 (3), 257 (4), 215 (59), 214 (78), 213 (67), 199 (46), 198 (69), 197 (90), 186 (18), 184 (27), 171 (38), 169 (31), 156 (28), 154 (31), 144 (13), 133 (30), 131 (36), 121 (39), 110 (42), 108 (86), 107 (81), 106 (63), 98 (73), 94 (60), 92 (62), 91 (100), 81 (32), 79 (42), 77 (71), 73 (23), 65 (53), 60 (29), 54 (68), 53 (90), 51 (64), 43 (56%).

(1RS,2SR,4aSR,4bSR,12aRS)-2-Bromo-12-oxo-

1,2,5,10,12,12a-hexahydro-4bH-2,4a-epoxyisoindolo[1,2-

b]quinazoline-1-carboxylic acid (44Ac) and (1*RS*,2*SR*,4*aSR*,4*bRS*,12*aRS*)-2-bromo-12-oxo-

1,2,5,10,12,12a-hexahydro-4bH-2,4a-epoxyisoindolo[1,2-

b]quinazoline-1-carboxylic acid (44Bc). Similarly to the preparation procedure presented for 44a, 2-(aminomethyl)aniline

(0.61 g, 5.0 mmol), 5-bromofurfural (0.88 g, 5.0 mmol), anhydrous powdered MgSO₄ (1.20 g, 10.0 mmol) and maleic anhydride (0.49 g, 5.0 mmol) in CH₂Cl₂ (30 mL) were used to afford 44c (0.46 g, 26 %) as a yellow powder, isomer ratio 44Ac/44Bc ~ 76/24. The minor isomer 44Bc (0.08 g) was obtained as a pale yellow powder by recrystallization of the mixture from EtOH/DMF, m.p. > 210.3 °C (decomp.); [Found: C, 50.79; H, 3.30; N, 7.46. C₁₆H₁₃BrN₂O₄ requires C, 50.95; H, 3.47; N, 7.43; Br, 21.18%]; v_{max} (KBr) 3514, 3338, 1740, 1667, 1185, 744 cm⁻¹; $\delta_{\rm H}$ (400 MHz, DMSO- d_6) (44Bc) 12.51 (1 H, br.s, CO₂H), 7.05 (1 H, br.d, ³J_{9.8} 7.4 Hz, H-9), 7.05 (1 H, dd, ³J_{7.6} 8.0, ³*J*_{7.8} 7.4 Hz, H-7), 6.86 (1 H, d, ³*J*_{3.4} 5.4 Hz, H-4), 6.76 (1 H, br.d, ${}^{3}J_{7,6}$ 8.0 Hz, H-6), 6.68 (1 H, br.t, ${}^{3}J_{7,8} \sim {}^{3}J_{9,8}$ 7.4 Hz, H-8), 6.57 (1 H, d, ³*J*_{3,4} 5.4 Hz, H-3), 6.40 (1 H, br.s, NH), 5.34 (1 H, s, H-4b), 4.53 (1 H, d, ${}^{2}J_{10,10}$ 16.5 Hz, H-10A), 4.24 (1 H, d, ${}^{2}J_{10,10}$ 16.5 Hz, H-10B), 3.10 (1 H, d, ³J_{1,12a} 8.3 Hz, H-12a), 3.01 (1 H, d, ${}^{3}J_{1,12a}$ 8.3 Hz, H-1); δ_{C} (100.6 MHz, DMSO- d_{6}) (44Bc) 169.5 (CO₂H), 167.6 (C-12), 142.6 (C-5a), 139.9 (C-3), 137.3 (C-4), 127.2 (C-7), 126.9 (C-9), 117.8 (C-8), 116.3 (C-9a), 115.6 (C-6), 90.7 (C-2), 88.3 (C-4a), 63.8 (C-4b), 53.3 (C-12a), 50.9 (C-1), 40.4 (C-10); $\delta_{\rm H}$ (400 MHz, DMSO- d_6) (44Ac in mixture with 44Bc, ratio 44Ac/44Bc ~ 76/24) 12.60 (1 H, br.s, CO₂H), 7.06–7.00 (2 H, m, H-9 and H-7), 6.74 (1 H, d, ${}^{3}J_{3,4}$ 5.7 Hz, H-4), 6.77–6.71 (2 H, m, H-6 and H-8), 6.62 (1 H, d, ${}^{3}J_{3,4}$ 5.7 Hz, H-3), 6.54 (1 H, br.s, NH), 5.12 (1 H, s, H-4b), 4.78 (1 H, d, ²J_{10.10} 16.7 Hz, H-10A), 4.28 (1 H, d, ${}^{2}J_{10,10}$ 16.7 Hz, H-10B), 3.05–3.01 (2 H, m, H-12a and H-1); δ_{C} (100.6 MHz, DMSO- d_{6}) (44Ac in mixture with 44Bc, ratio 44Ac/44Bc ~ 76/24) 169.6 and 169.5 (CO₂H and C-12), 142.9 (C-5a), 139.7 (C-3), 136.0 (C-4), 127.2 (C-7), 126.6 (C-9), 118.7 (C-8), 118.3 (C-9a), 116.3 (C-6), 90.5 (C-2), 88.4 (C-4a), 65.1 (C-4b), 51.5 (C-12a), 50.8 (C-1), 40.0 (C-10). MS (EI, 70 eV) m/z 378 (6, M⁺ for ⁸¹Br), 376 (5), 357 (9), 329 (1), 280 (91), 278 (100), 263 (56), 261 (28), 250 (8), 199 (70), 197 (17), 202 (14), 199 (70), 184 (33), 182 (35), 174 (18), 169 (32), 133 (37), 121 (42), 107 (73), 106 (76), 98 (89), 91 (71), 85 (78), 78 (62), 77 (77), 72 (38), 65 (32), 61 (34), 54 (100), 45 (76), 43 (78%).

Synthesis of 2,4a-epoxyisoindolo[1,2-b]quinazolin-12-ones (45, 46). Furfural (0.42 mL, 5.10 mmol) and anhydrous powdered $MgSO_4$ (1.20 g, 10.0 mmol) were added to a solution of 2-aminobenzylamine (0.61 g, 5.0 mmol) in CH₂Cl₂ (20 mL). The reaction mixture was stirred at room temperature for 1 h. MgSO₄ was filtered off and washed with CH_2Cl_2 (2 × 10 mL), the solvent was evaporated under reduced pressure. A solution of the obtained quinazoline 43a (5 mmol), acryloyl chloride (0.44 mL, 5.50 mmol) and NEt₃ (0.97 mL, 7.0 mmol) in PhMe (30 mL) was heated under reflux for 3 h. The mixture was cooled and poured into water (50 mL). The organic layer was separated and the water layer was extracted with EtOAc (3 \times 20 mL). The organic phases were combined and dried over MgSO4. The darkbrown oil obtained by evaporation of the solvent, was crystallized in EtOAc to afford a brown crystalline compound (0.23 g, 18 %) - the mixture of isomers 45A/45B in the ratio of ~ 70/30. The mother liquor was separated by silica gel column chromatography (1.7 \times 17 cm) using EtOAc/hexane (1/10 \rightarrow 1/1) mixtures. Isoindologuinazoline 45A (70 mg) is obtained first, followed by isoindoloquinazoline 45B (120 mg, ~ 92 % pure). The latter fraction was recrystallized from a hexane/EtOAc mixture to obtain an analytical sample of the isomer 45B. Then the compound 46 (60 mg) was eluated. The ratio of the isomers and the yields of the isolated products are the following: **45A/45B/46** ~ 50 (18 %)/ 39 (14 %)/ 11 (4 %).

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(2RS,4aRS,4bRS,12aSR)-1,5,10,12a-Tetrahydro-4bH-2,4a-

epoxyisoindolo[1,2-b]quinazolin-12(2H)-one (45A). Fine colorless needles (hedgehog-like crystalline aggregates), m.p. 212.5-214.5 °C (decomp., from EtOAc); [Found: C, 70.71; H, 5.63; N, 11.21. C15H14N2O2 requires C, 70.85; H, 5.55; N, 11.02%]; v_{max} (KBr) 3315, 1676, 1610, 1493, 1418, 1257, 1054, 757, 709 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 7.08 (1 H, ddd, ${}^{3}J_{7,6}$ 8.1, ${}^{3}J_{7,8}$ 7.5, ${}^{4}J_{7,9}$ 1.2 Hz, H-7), 7.03 (1 H, dd, ${}^{3}J_{9,8}$ 7.5, ${}^{4}J_{7,9}$ 1.2 Hz, H-9), 6.85 (1 H, dt, ${}^{3}J_{8,9} \sim {}^{3}J_{7,8}$ 7.5, ${}^{4}J_{6,8}$ 1.2 Hz, H-8), 6.73 (1 H, br.d, ³J₆₇ 8.1 Hz, H-6), 6.61 (1 H, d, ³J₄₃ 6.0 Hz, H-4), 6.44 (1 H, dd, ${}^{3}J_{3,4}$ 6.0, ${}^{3}J_{2,3}$ 1.8 Hz, H-3), 5.15 (1 H, dd, ${}^{3}J_{1exo,2}$ 4.4, ${}^{3}J_{2,3}$ 1.8 Hz, H-2), 5.06 (1 H, s, H-4b), 5.01 (1 H, d, ${}^{2}J_{10,10}$ 16.8 Hz, H-10A), 4.29 (1 H, d, ${}^{2}J_{10,10}$ 16.8 Hz, H-10B), 2.55 (1 H, dd, ${}^{3}J_{1\text{end},12a}$ 9.3, ${}^{3}J_{1\text{exo},12a}$ 4.4 Hz, H-12a), 2.20 (1 H, dt, ${}^{2}J_{1,1}$ 11.8, ${}^{3}J_{1\text{exo},12a} \sim {}^{3}J_{1\text{exo},2}$ 4.4 Hz, H-1^{exo}), 1.63 (1 H, dd, ${}^{2}J_{1,1}$ 11.8, ${}^{3}J_{1\text{endo},12a}$ 9.3 Hz, H-1^{endo}); δ_{C} (100.6 MHz, CDCl₃) 173.7 (C-12), 142.3 (C-5a), 137.2 (C-3), 131.3 (C-4), 127.7 and 127.2 (C-7 and C-9), 120.6 (C-8), 119.8 (C-9a), 117.1 (C-6), 90.7 (C-4a), 79.8 (C-2), 67.3 (C-4b), 46.2 (C-12a), 40.9 (C-10), 27.7 (C-1). MALDI-TOF HR: MNa⁺, found 277.0962. $C_{15}H_{14}N_2NaO_2$ requires 277.0947.

(2RS,4aRS,4bSR,12aSR)-1,5,10,12a-Tetrahydro-4bH-2,4a-

epoxyisoindolo[1,2-b]quinazolin-12(2H)-one (45B). Colorless plats, m.p. 192.2-192.5 °C (hexane/EtOAc); [Found: C, 70.75; H, 5.58; N, 11.17. C₁₅H₁₄N₂O₂ requires C, 70.85; H, 5.55; N, 11.02%]; v_{max} (KBr) 3298, 1660, 1495, 1260, 966, 780, 707 cm⁻¹; $δ_{\rm H}$ (400 MHz, CDCl₃) 7.11 (1 H, dd, ${}^{3}J_{7,6}$ 8.2, ${}^{3}J_{7,8}$ 7.5 Hz, H-7), 7.08 (1 H, br.d, ${}^{3}J_{9,8}$ 7.5 Hz, H-9), 6.92 (1 H, br.t, ${}^{3}J_{8,9} \sim {}^{3}J_{7,8}$ 7.5 Hz, H-8), 6.84 (1 H, br.d, ³J_{6.7} 8.2 Hz, H-6), 6.53 (1 H, d, ³J_{4.3} 5.7 Hz, H-4), 6.45 (1 H, dd, ³J_{3,4} 5.7, ³J_{2,3} 1.5 Hz, H-3), 5.28 (1 H, br.s, H-4b), 5.14 (1 H, dd, ${}^{3}J_{1exo,2}$ 4.4, ${}^{3}J_{2,3}$ 1.5 Hz, H-2), 4.84 (1 H, d, ${}^{2}J_{10,10}$ 17.0 Hz, H-10A), 4.37 (1 H, d, ${}^{2}J_{10,10}$ 17.0 Hz, H-10B), 4.24 (1 H, br.s, NH), 2.56 (1 H, dd, ${}^{3}J_{1\text{endo},12a}$ 9.0, ${}^{3}J_{1\text{exo},12a}$ 3.3 Hz, H-12a), 2.27 (1 H, ddd, ${}^{2}J_{1,1}$ 12.0, ${}^{3}J_{1exo,2}$ 4.4, ${}^{3}J_{1exo,12a}$ 3.3 Hz, H-1^{exo}), 1.63 (1 H, dd, ${}^{2}J_{1,1}$ 12.0, ${}^{3}J_{1\text{endo},12a}$ 9.0 Hz, H-1^{endo}); δ_{C} (100.6 MHz, CDCl₃) 172.0 (C-12), 141.1 (C-5a), 137.6 (C-3), 132.6 (C-4), 127.7 and 127.3 (C-7 and C-9), 121.8 (C-8), 120.9 (C-9a), 120.2 (C-6), 90.3 (C-4a), 79.2 (C-2), 65.6 (C-4b), 48.0 (C-12a), 40.9 (C-10), 28.2 (C-1). MS (EI, 70 eV) m/z 254 (72, M⁺), 237 (7), 225 (4), 207 (6), 199 (27), 183 (100), 181 (14), 132 (14), 131 (43), 106 (34), 104 (18), 78 (27), 77 (32), 59 (32), 55 (51), 43 (49%).

(2RS,4aRS,4bRS,12aSR)-5-Acryloyl-1,5,10,12a-tetrahydro-

4bH-2,4a-epoxyisoindolo[1,2-b]quinazolin-12(2H)-one (46). Colorless needles, m.p. 209.5-210 °C (decomp., from EtOH); [Found: C, 70.18; H, 5.39; N, 9.09. C₁₈H₁₆N₂O₃ requires C, 70.12; H, 5.23; N, 9.09%]; v_{max} (KBr) 1685, 1648, 1615, 1409, 1328, 1251 cm⁻¹; $\delta_{\rm H}$ (400 MHz, CDCl₃) 7.25–7.22 (2 H, m, H-7 and H-9), 7.17 (1 H, dt, ${}^{3}J_{8,9} \sim {}^{3}J_{7,8}$ 7.5, ${}^{4}J_{6,8}$ 1.2 Hz, H-8), 7.07 (1 H, br.d, ${}^{3}J_{7,6}$ 8.4 Hz, H-6), 6.75 (1 H, d, ${}^{3}J_{4,3}$ 6.2 Hz, H-4), 6.61 (1 H, s, H-4b), 6.50 (1 H, dd, ³J_{3'trans,2'} 16.8, ²J_{3',3'} 3.1 Hz, H-3'^{trans}), 6.44 (1 H, dd, ${}^{3}J_{3'\text{trans},2'}$ 16.8, ${}^{3}J_{3'\text{cis},2'}$ 9.3 Hz, H-2'), 6.30 (1 H, dd, ${}^{3}J_{3,4}$ 6.2, ${}^{3}J_{2,3}$ 1.9 Hz, H-3), 5.71 (1 H, dd, ${}^{3}J_{3'cis,2'}$ 9.3, ${}^{2}J_{3',3'}$ 3.1 Hz, H-3^{, cis}), 4.95 (1 H, d, ²J_{10,10} 14.7 Hz, H-10A), 4.85 (1 H, dd, ${}^{3}J_{1\text{exo},2}$ 4.6, ${}^{3}J_{2,3}$ 1.9 Hz, H-2), 3.86 (1 H, d, ${}^{2}J_{10,10}$ 14.7 Hz, H-10B), 2.64 (1 H, dd, ${}^{3}J_{1\text{endo},12a}$ 8.7, ${}^{3}J_{1\text{exo},12a}$ 3.1 Hz, H-12a), 2.07 (1 H, ddd, ${}^{2}J_{1,1}$ 11.8, ${}^{3}J_{1\text{exo},2}$ 4.6, ${}^{3}J_{1\text{exo},12a}$ 3.1 Hz, H-1^{exo}), 1.49 (1 H, dd, ${}^{2}J_{1,1}$ 11.8, ${}^{3}J_{1\text{end},12a}$ 8.7 Hz, H-1^{endo}); δ_{C} (100.6 MHz, CDCl₃) 172.9 (C-12), 165.6 (COCH=CH₂), 136.3 (C-5a), 135.9 (C-3), 133.7 (C-4), 131.3 (C-9a), 129.5 (COCH=CH₂), 128.9 (COCH=CH₂), 128.1 (C-7), 126.4 (C-9), 125.9 (C-8), 125.8 (C-6), 92.4 (C-4a), 78.8 (C-2), 69.4 (C-4b), 48.9 (C-12a), 40.3 (C-10), 28.2 (C-1). GC-MS (EI, 70 eV) m/z 308 (2, M⁺), 291 (1),

280 (5), 264 (3), 253 (9), 237 (11), 207 (4), 199 (23), 183 (10), 160 (4), 132 (20), 131 (14), 130 (9), 104 (6), 77 (13), 55 (100%). **Preparation of compounds 48–50 and attempted synthesis of 8,10a-epoxypyrimido[2,1-***a***]isoindole-7-carboxylic acid (51).**

A suspension of propane-1,3-diamine (8.33 mL, 0.10 mol), furfural (8.27 mL, 0.10 mol) and anhydrous powdered MgSO4 (24.0 g, 0.20 mol) in CH₂Cl₂ (100 mL) was stirred at room temperature for 1 h. MgSO₄ was filtered off and washed with CH_2Cl_2 (2 × 30 mL), the solvent was removed under reduced pressure. The residue, a brown mobile oil, was analyzed by NMR. The mixture contained three major products (~ 95 % overall): 2-(2-furyl)hexahydropyrimidine (48).N-(2furylmethylene)propane-1,3-diamine (49) and N,N'-bis(2furylmethylene)propane-1,3-diamine (50) in ratio 48/49/50 ~ 58/28/14. $\delta_{\rm H}$ (400 MHz, CDCl₃) (48) 7.31 (1 H, dd, ${}^{3}J_{5',4'}$ 1.8, ${}^{4}J_{5',3'}$ 0.8 Hz, H-5'), 6.28 (1 H, dd, ${}^{3}J_{3',4'}$ 3.1, ${}^{3}J_{5',4'}$ 1.8 Hz, H-4'), 6.25 (1 H, dd, ${}^{3}J_{3',4'}$ 3.1, ${}^{4}J_{5',3'}$ 0.8 Hz, H-3'), 4.67 (1 H, s, H-2), 3.23 (2 H, ddd, ${}^{2}J_{4(6),4(6)}$ 13.7, ${}^{3}J_{4e(6e),5a}$ 4.4, ${}^{3}J_{4e(6e),5e}$ 2.5 Hz, H-4^{eq} and H-6^{eq}), 2.95 (2 H, dddd, ${}^{2}J_{4(6),4(6)}$ 13.7, ${}^{3}J_{4a(6a),5a}$ 11.8, ${}^{3}J_{4a(6a),5e}$ 3.1, J 1.2 Hz, H-4^{ax} and H-6^{ax}), 1.66–1.46 (2 H, m, H-5); (49) 8.07 (1 H, s, N=CH), 7.47 (1 H, d, ${}^{3}J_{5',4'}$ 1.8 Hz, H-5'), 6.70 (1 H, d, ${}^{3}J_{3',4'}$ 3.3 Hz, H-3'), 6.44 (1 H, dd, ${}^{3}J_{3',4'}$ 3.3, ${}^{3}J_{5',4'}$ 1.8 Hz, H-4'), 3.61 (2 H, dt, J 1.2, J 6.9 Hz, CH=N-CH₂-CH₂-CH₂-NH₂), 2.76 (2 H, t, ³*J* 6.9 Hz, CH=N-CH₂-CH₂-CH₂-NH₂), 1.83 (2 H, p, ³*J* 6.9 Hz, CH=N-CH₂-CH₂-CH₂-NH₂); (**50**) 8.07 (2 H, s, N=CH), 7.47 (2 H, d, ³J_{5',4'} 1.8 Hz, H-5'), 6.70 (2 H, d, ³J_{3',4'} 3.3 Hz, H-3'), 6.44 (2 H, dd, ³J_{3',4'} 3.3, ³J_{5',4'} 1.8 Hz, H-4'), 3.64 (4 H, dt, J 1.2, J 6.9 Hz, CH=N-CH₂-CH₂-CH₂-N=CH), 2.11 (2 H, p, ³J 6.9 Hz, CH=N-CH₂-CH₂-CH₂-N=CH); δ_{C} (100.6 MHz, CDCl₃) (48) 151.7 (C-2'), 141.8 (C-5'), 110.1 (C-4'), 105.3 (C-3'), 68.7 (C-2), 45.7 (2 C, C-3 and C-5), 27.5 (C-4); (49) 155.0 (C-2'), 149.8 (CH=N), 144.7 (C-5'), 113.8 (C-3'), 111.6 (C-4'), 59.5 (CH=N- CH_2 -CH₂-CH₂-NH₂), 40.2 (CH=N-CH₂-CH₂-NH₂), 34.8 (CH=N-CH₂-CH₂-CH₂-NH₂); (50) 155.0 (2 C, C-2'), 150.1 (2 C, CH=N), 144.7 (2 C, C-5'), 113.8 (2 C, C-3'), 111.6 (2 C, C-4'), 59.4 (2 C, CH=N-CH2-CH2-CH2-N=CH), 31.9 (CH=N-CH2-CH2-CH2-N=CH).

The obtained mixture of products **48–50** was divided into 5 portions and introduced into a reaction with equimolar amount of maleic anhydride (3.92 g, 0.04 mol) in CH₂Cl₂ (50 mL), or Me₂CO (50 mL) at room temperature or at -10 °C, or stirred under reflux in PhMe (50 mL) for 1 h. In all of the five cases, a viscous brown oil was obtained after the solvent was removed. According to ¹H NMR, the mixtures contained mainly polymeric products.

A procedure for the synthesis of 8-aminonaphthalene-1-thiol from readily available potassium 8-aminonaphthalene-1-sulfonate is reported^[39] and is well-reproducible (Scheme 19), whereas the methods of preparation of 8-aminonaphtalen-1-ol from the same starting compound were described in relatively old and hard to access sources.^[40] We could not find any spectral data on this compound. Therefore, we describe its preparation here.

8-Aminonaphthalen-1-ol. A fine mixture of potassium 8aminonaphthalene-1-sulfonate (26.1 g, 0.10 mol) and KOH (84.2 g, 1.50 mol) was melted in a steel bowl with an untight cover and then heated at 220–225 °C for 1.5 h with periodical stirring. After cooling, the melt was dissolved in water (300 mL), filtered through coarse Al₂O₃ (3×6 cm) and Al₂O₃, washed with water (2 × 50 mL). HCl (18%) was added at 15–20 °C to the brown solution until it was strongly acidic (pH ~ 1–2). The mixture was filtered through Al₂O₃ (3×6 cm) again and washed with water (50 mL). NH₄OH (25 %) was carefully added to the obtained solution at stirring until the precipitation formation ceased (pH ~ 7). The grey solid was filtered off and dried in the air to a

constant weight. The obtained product (3.7–4.0 g, 23–25%) was rather pure for most purposes and stable in the air at +4 °C (unlike its analogue, 8-aminonaphthalene-1-thiol, which quickly turns orang and decomposes at this temperature). Attempted recrystallization from hexane/ethyl acetate mixtures lead to resinification of the product. Analytically pure sample may be obtained by slow vaporization of the ethereal solution. M.p. > 82 °C (decomp.); $\delta_{\rm H}$ (400 MHz, CDCl₃) 7.42 (1 H, d, $^3J_{5.6}$ 7.9 Hz, H-5), 7.29–7.20 (3 H, m, H-3, H-4, H-6), 6.79 (1 H, d, $^3J_{6.7}$ 7.5 Hz, H-7), 6.76 (1 H, d, $^3J_{2.3}$ 6.9 Hz, H-2), 6.30 (3 H, br.s, NH₂ and OH); $\delta_{\rm C}$ (100.6 MHz, CDCl₃) 155.2 (C-1), 139.9 (C-8), 136.8 (C-4a), 126.9 and 125.9 (C-3 and C-6), 123.0 (C-4), 119.5 (C-2), 118.3 (C-8a), 117.7 (C-5), 109.6 (C-7).

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Supplementary data

Copies of ¹H, ¹³C and DEPT-135 NMR spectra for all new compounds, X-ray description of compounds **17a**, **37a** and **39b** are available free of charge *via* the Internet at http://

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Supporting Information

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

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1

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X-Ray description of compounds 17a, 37a and 39b

X-ray structure determination. The crystal of 17a (C₁₃H₁₅NO₅, M = 265.26) is orthorhombic, space group $P2_12_12_1$, at T = 293 K: a = 9.327(2) Å, b = 10.719(2) Å, c =12.261(2) Å, V = 1225.8(4) Å³, Z = 4, $d_{calc} = 1.437$ g/cm³, F(000) = 560, $\mu = 0.111$ mm⁻¹. 2058 total reflections (2035 unique reflections, $R_{int} = 0.009$) were measured on a four-circle Enraf Nonius CAD-4 diffractometer (λ (MoK $_{\alpha}$)-radiation, graphite monochromator, $\omega/2\theta$ scan mode, $2\theta_{max} = 60^{\circ}$). The structure was determined by direct methods and refined by full-matrix least squares technique on F^2 with anisotropic displacement parameters for non-hydrogen atoms. The hydrogen atoms were placed in calculated positions and refined within the riding model with fixed isotropic displacement parameters [U_{iso} (H) = $1.5U_{eq}$ (C) for the CH₃-groups and U_{iso} (H) = $1.2U_{eq}$ (C) for the other groups]. The final divergence factors were $R_1 = 0.051$ for 1683 independent reflections with $I > 2\sigma(I)$ and $wR_2 = 0.143$ for all independent reflections, S = 0.998. All calculations were carried out using the SHELXTL program.¹

The crystal of **37a**•¹/4*C*₆*H*₁₄•¹/4*C*₄*H*₈*O*₂ (C_{19.5}H_{26.5}NO_{9.5}, *M* = 426.92) is monoclinic, space group *P*2₁/c, at *T* = 100 K: *a* = 9.7028(4) Å, *b* = 10.9965(5) Å, *c* = 19.1443(9) Å, β = 98.299(1)°, *V* = 2021.25(16) Å³, *Z* = 4, *d*_{calc} = 1.403 g/cm³, *F*(000) = 906, μ = 0.113 mm⁻¹. 29463 total reflections (4854 unique reflections, *R*_{int} = 0.029) were measured on a three-circle diffractometer (λ (MoK_{α})-radiation, graphite monochromator, φ and ω scan mode, $2\theta_{max}$ = 56°). The structure was determined by direct methods and refined by full-matrix least squares technique on *F*² with anisotropic displacement parameters for non-hydrogen atoms. There was a region in the asymmetric unit of **37a** that appeared to be occupied by solvent molecules. It looked like this region contained hexane and ethyl acetate molecules, which were strongly disordered around the inversion center. Attempts to model this region were unsatisfactory. The contribution to the scattering by the solvate molecules was removed by the use of the SQUEEZE utility in *PLATON98*.² The hydrogen atoms were placed in calculated positions and refined within the

riding model with fixed isotropic displacement parameters $[U_{iso}(H) = 1.5U_{eq}(C)$ for the CH₃groups and $U_{iso}(H) = 1.2U_{eq}(C)$ for the other groups]. The final divergence factors were $R_1 =$ 0.038 for 4246 independent reflections with $I > 2\sigma(I)$ and $wR_2 = 0.098$ for all independent reflections, S = 1.002. All calculations were carried out using the SHELXTL program.¹

The crystal of **39b** (C₁₅H₁₉NO₈S, M = 373.37) is orthorhombic, space group *P*bca, at T = 100 K: a = 9.3166(14) Å, b = 15.249(2) Å, c = 23.198(3) Å, V = 3295.7(8) Å³, Z = 8, $d_{calc} = 1.505$ g/cm³, F(000) = 1568, $\mu = 0.242$ mm⁻¹. 29586 total reflections (3261 unique reflections, $R_{int} = 0.049$) were measured on a three-circle diffractometer (λ (MoK_{α})-radiation, graphite monochromator, φ and ω scan mode, $2\theta_{max} = 52^{\circ}$). The structure was determined by direct methods and refined by full-matrix least squares technique on F^2 with anisotropic displacement parameters for non-hydrogen atoms. The hydrogen atoms were placed in calculated positions and refined within the riding model with fixed isotropic displacement parameters [U_{iso} (H) = $1.5U_{eq}$ (C) for the CH₃-groups and U_{iso} (H) = $1.2U_{eq}$ (C) for the other groups]. The final divergence factors were $R_1 = 0.039$ for 2342 independent reflections were carried out using the SHELXTL program.¹

Crystallographic data for the investigated compounds have been deposited with the Cambridge Crystallographic Data Center, CCDC 824681 (**17a**), CCDC 824682 (**37a**) and CCDC 824683 (**39b**). Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk or www.ccdc.cam.ac.uk).

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

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Figure S1Molecular structure of methyl $(6aR^*,7S^*,8R^*,10aS^*,10bR^*)$ -6-oxo-3,4,6,6a,7,8-hexahydro-2H-8,10a-epoxy[1,3]oxazino[2,3-a]isoindole-7-carboxylate17a.Displacement ellipsoids are depicted at the 40% probability level. Only hydrogen atoms at the asymmetric centers are presented.



Figure S2 Molecular structure of methyl (6a*RS*,7*SR*,8*SR*,9*RS*,9*aRS*,10*RS*,10*aRS*)-9,10bis(acetyloxy)-6-oxodecahydro-2*H*-8,10-epoxycyclopenta[4,5]pyrido[2,1-*b*][1,3]oxazine-7carboxylate **37a**. Displacement ellipsoids are depicted at the 50% probability level. Only hydrogen atoms at the asymmetric centers are presented.

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.



Figure S3 Molecular structure of (5aRS,7SR,8RS,8aRS,9RS,9aSR)-7-methyl-1,1-dioxido-5-oxooctahydro-7,9-epoxycyclopenta[d][1,3]thiazolo[3,2-a]pyridine-8,9-diyl diacetate **39b**. Displacement ellipsoids are depicted at the 50% probability level. Only hydrogen atoms at the asymmetric centers are presented.

According to the X-ray data, the molecules 17a, 37a and 39b are fused tetracyclic systems containing: 17a – three five-membered (two tetrahydrofurans and one pyrrolidinone) and one six-membered (oxazinane), 37a – two five-membered (cyclopentane and tetrahydrofuran) and two six-membered (tetrahydropyridinone and oxazinane), and **39b**: three five-membered (cyclopentane, tetrahydrofuran and thiazolidine) and one six-membered (tetrahydropyridinone) rings. All the five-membered rings have the regular envelope conformation, and the oxazole rings have the usual *chair* conformation. The conformation of the six-membered tetrahydropyridinone ring in 37a is a flattened *boat*, whereas, in 39b, this ring adopts a distorted *chair* conformation. The nitrogen atoms in all of the compounds have a slightly pyramidalized configuration. The two O-carboxylate substituents in 37a and 39b occupying the same positions in the 2-oxabicyclo[2.2.1]heptane fragment (C9 and C10 in **37a**, and C8 and C9 in **39b**) are in the sterically unfavorable *syn*-periplanar configuration relative to the tetrahydrofuran ring. Such a disposition is determined by the direction of the Wagner-Meerwein rearrangement.

5

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References

X-Ray

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Copies of ¹H, ¹³C and ¹³C DEPT-135 spectra of the compounds reported and

characterized in the main article

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

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			Comp	ounds 34	Aa/3Ba	6a N-4		N-4
					⁹ / ₁₀		и 10/104 Н	10b 3
					60	10ba 1	40	i de
			3.36					
								.55
								2 2
						2.74		2.5
						2.76		2.50
3.93 11 3 ⁻ 3.90						77 77 2.75		2.49
3.94 3.9 3.85 3.88 3.88 37	0 68 3.67			4 3.10 3.10	202	2		
S S	3.84 3.71 3.71 3.71 3.65 3.65 3.64			e. e	3.0 3.0 3.0 01 3.0 0 3.00 3.00 3.00 3.00	88		
MA	- MAX		\bigcirc	M	Hen a a a a			
2.68	0.65			1.	07 0.66	1.62		1.07
	06 E 88 E 16 E 2.68 3.9	06 88 10 <td< td=""><td>06 88 10 <td< td=""><td>Comp</td><td>Compounds 3.</td><td>Compounds 3Aa/3Ba</td><td>Compounds 3Aa/3Ba $\begin{array}{c} & &$</td><td>$Compounds 3Aa/3Ba = \begin{pmatrix} y_1 \\ y_2 \\ y_3 \\ y_6 \\$</td></td<></td></td<>	06 88 10 <td< td=""><td>Comp</td><td>Compounds 3.</td><td>Compounds 3Aa/3Ba</td><td>Compounds 3Aa/3Ba $\begin{array}{c} & &$</td><td>$Compounds 3Aa/3Ba = \begin{pmatrix} y_1 \\ y_2 \\ y_3 \\ y_6 \\$</td></td<>	Comp	Compounds 3.	Compounds 3Aa/3Ba	Compounds 3Aa/3Ba $\begin{array}{c} & & & & & & & & & & & & & & & & & & &$	$Compounds 3Aa/3Ba = \begin{pmatrix} y_1 \\ y_2 \\ y_3 \\ y_6 \\ $

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

21.05.2012 19:00:55

ormula C ₂₄ H ₂₆ N ₂ O ₁₀ ?		FW 502.4706+? (2	251.2353+251.2353+	?+?)				
cquisition Time (sec)	1.5000	Comment	ILTP8qq4absur	d ser.20070601		Date	Feb 19 2009	
ate Stamp	Feb 19 2009	File Name	D:\NMR\C_13\T	УРЧИН зима 2009\Nic 209 =	FZ 506 (оксазол,	Турчин)\FZNic209cpp_	190209	
requency (MHz)	100.58	Nucleus	13C	Number of Transients	54000	Original Points Cou	int 37500	
oints Count	65536	Pulse Sequence	s2pul	Receiver Gain	56.00	Solvent	DMSO-d6	
pectrum Offset (Hz)	9462.2764	Sweep Width (Hz)	25000.00	Temperature (degree C)	29.000			
				Compo	unds 3Aa	a/3Ba	$ \begin{array}{c} & & & \\ & & & & \\ & & & \\ & & & & \\ & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & $	35 34 36 0 35 34 36 0 33 36 0 114 10a 10b 3 10b 1 10b 1
Nic209cpp_190209								09.6
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								and the second sec
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0.71		77		0	9.48		49	
170		33.			89 9 89 88		43	
36		1 159			82.7	6/10	42	5.1
172		134			3	66.	44	42 2 2
						Ō		37
			4					
lings the production of the second	dentacio de la constanció		hind and the ball of the second	n in de la service de la s	n suite san suite san suite		e a contraction de la	ing "Witness de the anten de la service
ou the ellips was a dalle to a dall when	or complete difficult	to a color of the time of a share of the second	a data and a many car success with	A Real of the second	we allow a second second second	terrori e tradicio ante e colarito de cola	and a strain a strain strain the	and comparishing several second second
								and a second

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

21.05.2012 19:01:53

auisition Time (sec)	1.5000	Comment	ILTP8qq4absu	rd ser.20070601		Date	Feb 19 2009	
te Stamp	Feb 19 2009	File Name	D:\NMR\C 13	ТУРЧИН зима 2009\Nic 209 =	FZ 506 (оксазол,	Турчин)\FZNic209c	pp_190209	
equency (MHz)	100.58	Nucleus	13C	Number of Transients	54000	Original Points	Count 37500	
ints Count	65536	Pulse Sequence	s2pul	Receiver Gain	56.00	Solvent	DMSO-d6	
ectrum Offset (Hz)	9462.2764	Sweep Width (Hz)	25000.00	Temperature (degree C)	29.000			
				Compour	nds 3Aa/3	3Ba	$ \begin{array}{c} 16^{-18} & 0 \\ 115 \\ 0 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ $	$ \begin{array}{c} 35 \\ 35 \\ 36 \\ 9 \\ 9 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10$
Nic209cpp_190209						6	0.60	1
							ř	
							9.81	
							Ř	
							9.18	
							3	
48 5.05					49.26	95		
89.	2.30				10	43.6		~
2.79	8.1.48		0		0.2	57	.25	5.13
60	ò		.19		2	44.4	38	21
			- 99				37.4	
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					communication and a second second second	and the second se	LOUVER	and the opposite the second second second second second
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17 Formula C.,H.,NO,

FW

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 17:53:11

Acquisition Time (sec)	1.5729	Comment	5 mm QNP	1H/15N/13C/31P Z3379/0400		Date	21 Jun 2012 17:18:56
Date Stamp	21 Jun 2012 17	7:18:56					
File Name	C:\Users\Fedor	hDesktop/C13 Рома Для Стать	ы в ЈОС 25.0	05.12\rudn-250512-3Bb\rudn-2505	512-3Bb_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Temperature (degree C	32 000						



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 17:53:20

Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15	5N/13C/31P Z3379/0400		Date	21 Jun 2012 17:18:56
Date Stamp	21 Jun 2012 17:18	:56	*				
File Name	C:\Users\Fedor\De	sktop\C13 Рома Для Стат	ьи в JOC 25.05.12\г	udn-250512-3Bb\rudn-2505	12-3Bb_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Temperature (degree C	32.000						

H-10B

S

rudn-250512-3Bb_001000fid



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0



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 17:53:33

Formula C ₁₃ H ₁₅ NO ₅	<i>FW</i> 265.2619						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	21 Jun 2012 17:18:56
Date Stamp	21 Jun 2012 17:18	:56					
File Name	C:\Users\Fedor\De	sktop\C13 Рома Для Стати	ы в JOC 25.05.12\и	rudn-250512-3Bb\rudn-2505	512-3Bb_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Temperature (degree (C) 32.000						



Chemical Shift (ppm)

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 18:00:09

16-0H

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10

H₃C

Compounds 3Ab/3Bb

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15.56

Formula C ₁₃ H ₁₅ NO ₅	FW 265.2619						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N	I/13C/31P Z3379/0400		Date	21 Jun 2012 22:09:04
Date Stamp	21 Jun 2012 22:09:0	4					
File Name	C:\Users\Fedor\Desl	top\C13 Рома Для Статьи	в JOC 25.05.12\rudn	-250512-3Bb-c13dec\rudn-2	250512-3Bb-c13dec_0	01000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	31322	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.8018
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				





rudn-250512-3Bb-c13dec_001000fid

6

000H

171.36

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 18:00:19

10

134.52

OH

0

Formula C13H15NO5	<i>FW</i> 265.2619						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N	/13C/31P Z3379/0400		Date	21 Jun 2012 22:09:04
Date Stamp	21 Jun 2012 22:09:04	4					
File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Статьи	в JOC 25.05.12\rudn-	-250512-3Bb-c13dec\rudn-2	50512-3Bb-c13dec_00)1000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	31322	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.8018
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 18:00:36

Formula C ₁₃ H ₁₅ NO ₅	FW 265.2619						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	//15N/13C/31P Z3379/0400		Date	21 Jun 2012 22:09:04
Date Stamp	21 Jun 2012 22:09:0)4	,				
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Статьи	в ЈОС 25.05.12	rudn-250512-3Bb-c13dec\rudn-2	250512-3Bb-c13de	ec_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	31322	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.8018
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000			huit huit ha	

Compounds 3Ab/3Bb



rudn-250512-3Bb-c13dec_001000fid



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265 2619

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 18:00:51

Me-8

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114

H₃C

Compounds 3Ab/3Bb

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Formula C ₁₃ H ₁₅ NO ₅	FW 265.2619						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15	5N/13C/31P Z3379/0400		Date	21 Jun 2012 22:09:04
Date Stamp	21 Jun 2012 22:09:0	4					
File Name	C:\Users\Fedor\Dest	top\C13 Рома Для Статьи	B JOC 25.05.12\ruo	dn-250512-3Bb-c13dec\rudn-2	250512-3Bb-c13dec_	001000fid	1
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	31322	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.8018
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				





General Synthetic Approach towards Annelated 3a,6-Epoxylsoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 17:58:15

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Formula C ₁₃ H ₁₅ NO ₅	FW 265.2619						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1	H/15N/13C/31P Z3379/0400		Date	21 Jun 2012 17:21:04
Date Stamp	21 Jun 2012 17:21:0	4					
File Name	C:\Users\Fedor\Des	top\C13 Рома Для Статьи в	в ЈОС 25.05.12	2\rudn-250512-3Bb-dept135\rudn-2	50512-3Bb-dept1	35_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	10000	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9104.3936
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				



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of Furylazaheterocycles. Scope and Limitations.

22.06.2012 17:58:41

OH

0

H₃C

Compounds 3Ab/3Bb

O

Formula C13H15NO5	FW 265.2619						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	H/15N/13C/31P Z3379/0400		Date	21 Jun 2012 17:21:04
Date Stamp	21 Jun 2012 17:21:0)4					
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Статьи	в JOC 25.05.12	\rudn-250512-3Bb-dept135\rudn-2	250512-3Bb-dept1	35_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	10000	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9104.3936
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.04.2010 13:39:55



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.04.2010 13:40:47

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Acquisition Time (sec) 1.454	49 Comment	single_pulse	Date	09 Apr 2010	13:35:52		Date Stamp	09 Apr 2010 13:34:40
File Name D:\N	MR\6.04.10\FZ1138-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	8
Origin ECA	600 Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain 32.00	0 Solvent	DMSO-d6	Spectrum Offset (Hz)	3000.8616	Sweep Width (Hz)	11261.26	Temperature (degree C	21.200



Chemical Shint (ppm)

General Synthetic Approach towards Annelated 3a,6-Epoxylsoindoles by Tandem Acylation/IMDAF Reaction of Furvlazaheterocycles. Scope and Limitations.

Formula C., H., BrNO, FW 330,1314 Acquisition Time (sec) 1.4549 Comment single pulse Date 09 Apr 2010 13:35:52 File Name Date Stamp D:\NMR\6.04.10\FZ1138-1.idf 09 Apr 2010 13:34:40 Frequency (MHz) 600.17 Nucleus 1H Origin Number of Transients ECA 600 8 **Original Points Count** 16384 Owner delta Points Count 16384 **Receiver** Gain Pulse Sequence 32.00 Solvent single pulse.ex2 DMSO-d6 Spectrum Offset (Hz) 3000 8616 Sweep Width (Hz) 11261.26 Temperature (degree C) 21,200 0 .OH 19 0 H-2160 2,98, d (8.9) 2,96, d (8.9) Br Compounds 3Ac/3Bc 15 0 11. 0a H 10ba FZ1138-1.idf 2.50 pmp NO. Mesco minamas H.-2B) 4-4A 2.97 3 H-2A 0 05 M N tma/ B, MIN (F.11; 1,4) 4 her in V 12.4 2.91 3,4; N F.11 2.99 A 3.90 3.88 3.88 3.88 95 10 09 07 N -1 m N 2.53 2.48 2.47 40 07 3.68 3.68 3.68 3.68 3.66 3.66 3.66 3.64 3.64 62 .60 .59 05 85 84 3.30 46 2.02 26 44 2.07 (m m N 1.29 2.30 0.21 1.08 2.26 1.45 0.99 4.0 3.5 3.0 2.5 2.0 1.5

Chemical Shift (ppm)

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

13.04.2010 13:41:38

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.05.2012 15:50:37



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Acquisition Time (sec)	1.0433	Comment	single pulse d	lecoupled gated NOE		Date	26 Apr 201	2 13:23:04	
Date Stamp	26 Apr 2012 1	7:12:02		File Name	D:\NMR\20.04.	12\FZ2357-1.jdf		Frequency (MHz)	100.53
Nucleus	13C	Number of Transients	229	Origin	ECS 400	Original Points Count	32768	Owner	delta
Points Count	32768	Pulse Sequence	single_pulse_	dec		Receiver Gain	60.00	Solvent	DMSO-d6
Spectrum Offset (Hz)	10052.5303	Sweep Width (Hz)	31407.04	Temperature (degree C)	24.400				
					Compo	unds 3Ac/3B	В 1	$ \begin{array}{c} O_{17} > 0 \\ I_{17} > 10 \\ I_{19} \\ I_{19} \\ O_{/18} \\$	-4
2357-1.jdf			Q	10					.2
			5						84
									10
				0.11					40
				13(
COOPT,									100
/									100
6			0.71					0	
0			14(8	~
									8.58
.07									8
170								31	
22								6	
70.2									
5			82	7.51					თ
			140.	13.					33.1
			5					.43	4
								ō	60
									Ø

130 125 Chemical Shift (ppm)

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.05.2012 15:52:31

equisition Time (sec)	1.0433	Comment	single pulse de	coupled gated NOE		Date	26 Apr 2012	2 13:23:04	
ate Stamp	26 Apr 2012 1	7:12:02		File Name	D:\NMR\20.0	4.12\FZ2357-1.jdf		Frequency (MHz)	100.53
ucleus	13C	Number of Transients	229	Origin	ECS 400	Original Points Count	32768	Owner	delta
oints Count	32768	Pulse Sequence	single_pulse_d	ec		Receiver Gain	60.00	Solvent	DMSO-d6
pectrum Offset (Hz)	10052.5303	Sweep Width (Hz)	31407.04	Temperature (degre	ee C) 24.400				
					Comp	ounds 3Ac/3	Bc ^{Bi}		-1
								0	-0
2357-1.jdf			1	51.26					
			52.27	0.7		_			
			- fille	66,5		0.1			3
22						4			6
-						32 30 76			25.4
						39.6			
7.38									
9						1			
						4			
						0 0			
			44			39.6			
			53.						
90			4						35
66.9			4.1			.74			23.6
			4			38			1240
Lotra Maria	M. Los C.	a an ista aver-		in the state states		1. Miles Holes II		h that is a	all in
When the art the state of the	allow the state of	and a strain and the low on the Articles.	AMONTH AND AND	West of the Alternation of the Content	AN DE AND	A HULLET I THE AND A SHITTER MARK	人物的制造和	the should be a set of the set of the set	MAN BULLINES (DE MAN
It is dentered on the solution	the short of the short of the	the first of the other of the Lord Hilds of the bird	In all ad the starting	designed turburning a first statistic	and a subra addition of the	in the constant of the standard	· It is take the site	to also she is the line of the line in	Char I dil Harris da

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.06.2012 15:22:16

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0

2.91

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OH

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Formula C ₁₂ H ₁₂ INO ₅	FW 377.13	19					
Acquisition Time (sec,	1.5729	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	08 Jun 2012 15:28:00
Date Stamp	08 Jun 2012 15	:28:00					
File Name	C:\Users\Fedor	Desktop\C13 Рома Для Стат	ьи в ЈОС 25.05.1	2\rudn-040612-3d\rudn-04061	2-3d_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	64	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Temperature (degree	32.000						

Compounds 3Ad/3Bd



after recrystallization



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.06.2012 15:22:56

Formula C12H12INO5	<i>FW</i> 377.1319						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	08 Jun 2012 15:28:00
Date Stamp	08 Jun 2012 15:28	:00					
File Name	C:\Users\Fedor\De	sktop\C13 Рома Для Стат	ьи в JOC 25.05.12\ru	udn-040612-3d\rudn-04061	2-3d_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	64	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Taman anatura (danna (21 22 000						

Temperature (degree C) 32.000



0.72

6.7

6.8



12.53 13.47

6.6

6.5

6.4

6.3

6.2

6.1

6.0

Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

5.9

5.8

5.7

5.6

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A





MIN

5.3

5.21

13.37

5.2



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.06.2012 15:23:10

		5					
cquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	08 Jun 2012 15:28:00
ate Stamp	08 Jun 2012 15:2	28:00					
le Name	C:\Users\Fedor\D	Desktop\C13 Рома Для Стат	ы в JOC 25.05.12\г	udn-040612-3d\rudn-04061	2-3d_001000fid	Frequency (MHz)	400.14
ucleus	1H	Number of Transients	64	Origin	spect	Original Points Count	16384
wner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
W(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
				H-6a H-7			$\begin{array}{c} O_{17} & OH \\ I_{19} & O \\ I_{15} & I_{15} & I_{15} \\ I_{15} & I_{15} & I_{16} \\ I_{15} & I_{16} & I_{19} \\ I_{15} & I_{16} & I_{19} \\ I_{16} & I_{19} \\ I_{16} & I_{19} \\ I_{18} & I_{16} \\ I_{19} & I_{18} \\ I_{15} & I_{16} \\ I_{16} & I_{19} \\ I_{15} & I_{16} \\ I_{16} & I_{19} \\ I_{16} & I_{19} \\ I_{16} & I_{19} \\ I_{16} & I_{19} \\ I_{15} & I_{16} \\ I_{16} & I_{19} \\ I_{16} & I_{19$
				S, 2H maj	Compo	ounds 3Ad/3	$\operatorname{Bd}^{9 \longrightarrow 10}_{10ba} \operatorname{Bd}^{10a}_{10ba} \operatorname{Bd}^{10a}_{10ba}$
n-040612-3d_001000fic	d		H-4ax	-2.91			
2 eg	H-2 H-2	ax 1eq (62 dt 3,8;12,7)				H-3
Id (1.4)	m,	24	may +		(PM SC	0	min + n
+ maj	mint	mag H.	20 1 d	d (3)	*		
1 4 3.94 91	3.91	330	- 0 F	in			0 0
m	3.88	\wedge	3.15 3.14 3.14 3.15 3.14 3.13 3.09 3.03	3.01 2.99 2.93 2.89	2.54		1.68 1.67 1.67 1.66 1.66 1.66 1.66 1.66 1.66
4.14 4.14 4.14 4.14			JUM,	lit illi			- DAMAN A

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 21:47:02

-OH

0

0 18

0 16

Formula C12H12INO5	FW 377.1319						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N	/13C/31P Z3379/0400		Date	09 Jun 2012 12:05:20
Date Stamp	09 Jun 2012 12:05:2	0	×				
File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Статьи	в JOC 25.05.12\rudn	-040612-3d-c13dec\rudn-04	40612-3d-c13dec_001	000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	3328	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10552.1055
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000			19 - 19 - 19 - 19 - 19 - 19 - 19 - 19 -	

Compounds 3Ad/3Bd


General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 21:47:34

OH 19

0

Formula C ₁₂ H ₁₂ INO ₅	FW 377.1319						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	09 Jun 2012 12:05:20
Date Stamp	09 Jun 2012 12:05:20	0					
File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Статьи	в ЈОС 25.05.12\г	udn-040612-3d-c13dec\rudn-04	10612-3d-c13dec_001	000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	3328	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10552.1055
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				



36

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C ₁₂ H ₁₂ INO ₅	FW 377.1319						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	09 Jun 2012 12:05:20
Date Stamp	09 Jun 2012 12:05:	20				· · · · · · · · · · · · · · · · · · ·	
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Стат	гьи в JOC 25.05.12	rudn-040612-3d-c13dec\rudn-0	40612-3d-c13dec	001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	3328	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10552.1055
Sweep Width (Hz)	29409 97	Temperature (degree	C) 27 000				

09.06.2012 21:47:52



37

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 21:52:07

OH

•O

15

Compounds 3Ad/3Bd

Formula C ₁₂ H ₁₂ INO ₅	FW 377.1319							
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	09 Jun 2012 13:11:28	
Date Stamp	09 Jun 2012 13:11:2	8						
File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Статьи	в ЈОС 25.05.12\гис	dn-040612-3d-dept135\rudn-0	40612-3d-dept135_00	01000fid		
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	129	Origin	spect	
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135	
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9101.6973	
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000					



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

wixture 21.05.2012 20:16:38 FW Formula C24H24N4O14? 592,4658+? (296,2329+296,2329+?+?) Acquisition Time (sec) 1.4549 Comment single pulse Date 09 Apr 2010 12:57:25 Date Stamp 09 Apr 2010 12:56:14 1H File Name D:\NMR\06.04.10\FZ1136-1.idf Frequency (MHz) 600 17 Nucleus Number of Transients 8 Points Count 16384 Pulse Sequence single pulse.ex2 Origin ECA 600 **Original Points Count** 16384 Owner delta Temperature (degree C) 21.500 DMSO-d6 Sweep Width (Hz) 11261.26 Receiver Gain 42.00 Solvent Spectrum Offset (Hz) 3000.8616 OH 42 OH 0 18 21 0 20 0 0 20 An 0= 02 Compounds 3Ae/3Be -0 37 16 11. H 10ba 11 10bb 89 3Ae/3Be = 89/11S MBS + Me, CD 2.05 5.32 7.01 -7.00 -6.90 3.89 2.58 3.35 3.33 3.25 23 MOOH 2.47 3.16 3.16 3.14 3.13 5.47 91 89 89 7.04 82 82 81 ó 52 83 30 e e 00 0.90 0.12 0.99 0.12 0.13 1.080.14 1.02 11 11 1 1 비니니 186 6 13.0 10.5 9.5 9.0 7.0 6.5 5.5 2.0 1.5 12.5 12.0 11.5 11.0 10.0 8.5 8.0 5.0 3.5 3.0 2.5 1.0 7.5 6.0 4.5 4.0 Chemical Shift (ppm)

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

21.05.2012 20:16:46

Formula C ₂₄ H ₂₄ N ₄ O ₁₄ ?		FW 592.4658+?	(296.2329+296	.2329+?+?)					
Acquisition Time (sec)	1.4549	Comment	single_pulse	Date	09 Apr 2010	12:57:25		Date Stamp	09 Apr 2010 12:56:14
File Name	D:\NMR\06.0	4.10\FZ1136-1.idf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	8
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	42.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	3000.8616	Sweep Width (Hz)	11261.26	Temperature (degree C) 21.500



Compounds 3Ae/3Be

H-10B

5

FZ1136-1.jdf

H-10 d H-9 d (5,5)



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

21.05.2012 20:16:58



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

20.04.2012 12:02:58

	0.6921	Comment	single pulse	decoupled gated NOE		Date	15 Apr 2010	0 14:03:50	
te Stamp	15 Apr 2010 1	4:02:46		File Name	D:\NMR\14.04	4.10\FZ1148-1.jdf		Frequency (MHz)	150.91
icleus	13C	Number of Transients	200	Origin	ECA 600	Original Points Count	32768	Owner	delta
oints Count	32768	Pulse Sequence	single_pulse	_dec		Receiver Gain	50.00	Solvent	DMSO-d6
ectrum Offset (Hz)	15091.3428	Sweep Width (Hz)	47348.49	Temperature (deg	ree C) 22.000				
					Compou	unds 3Ae/3Be	0 16	$\begin{array}{c} 0 & 18 & 17 & 21 \\ 0 & 18 & 17 & 21 \\ 0 & 14 & 7 \\ 15 & 8 & 0 \\ 11 & 10 \\ 9 & 10 & 10 \\ H \end{array}$	N - 4 b 0 0 0 0 3 0 0 3 0 3 0 3 0 3 0 0 3 0 0 0 0 0 0 0 0
								10ba	1
148-1.jdf								0.02	
								14	
								39.8	
								40	
							21		
							50.9		
					ē		5		
					7.75		47.5		
-		.10			8				
9.6		137							
16		80			43			74	
		135.			84	.42		39.	5.46
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								.86	
12.5								38	
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66				13.				0.4	
		25		03	70	œ	.92	4	2
9.74				13	82	6.7	51	6.	9.0
169.74 166.56		138		÷	~	100	5 K.	100	

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

20.04.2012 12:01:32

Formula C ₁₂ H ₁₂ N ₂ O ₇	FW 296.2	329							
Acquisition Time (sec)	0.6921	Comment	single pulse d	lecoupled gated NOE		Date	15 Apr 201	0 14:03:50	
Date Stamp	15 Apr 2010 1	4:02:46		File Name	D:\NMR\14.0	04.10\FZ1148-1.jdf		Frequency (MHz)	150.91
Nucleus	13C	Number of Transients	200	Origin	ECA 600	Original Points Count	32768	Owner	delta
Points Count	32768	Pulse Sequence	single_pulse_	dec		Receiver Gain	50.00	Solvent	DMSO-d6
Spectrum Offset (Hz)	15091.3428	Sweep Width (Hz)	47348.49	Temperature (degree	e C) 22.000				





FZ1148-1.jdf



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

20.04.2012 12:02:13



Formula C₁₂H₁₂N₂O₇ FW 296.2329

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

04.05.2010 18:05:06

cquisition Time (sec)	2.9098	Comment	single pulse	Date	04 May 2010	10:35:03		Data Stamp	04 May 2010 10:22:40
ile Name	D:\NMR\22.	.04.10\FZ1178-1.jdf	0	Frequency (MHz)	600.17	Nucleus	111	Number of Transiente	04 May 2010 10:33:45
rigin	ECA 600	Original Points Count	32768	Owner	delta	Points Count	32768	Rulso Seguence	o ainele aules au?
eceiver Gain	26.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	3000 8616	Sween Width (Hz)	11261.26	Tomporature (degree (single_pulse.ex2
						,		$O_{18} = 17 - OH_{21}$	0 3Ae
						1 /	($N_{16} = \frac{15}{15} \frac{8}{8} - \frac{7}{0} \frac{6a}{16}$	/19 maj
					Comp	ounds 3Ae/	3Be	$\stackrel{ }{=} \stackrel{ }$	N - 4 10b 3 (+
178-1.jdf								H [*] 10ba	0_{1}^{-2}
								2.51	
						32			
						ů.			
Cooff				1 7.00 6.90	თ		• 8	34 25 35	
1				0.2	6.8			333	
03								3.24	
13									
						mΝ	11 3.91 3.89 37 7	13	51
						4.12	4.4. 3.8 3.8	3.12 3.11 2.41 1.63	1.45 1.45 1.45
									AL
1.76				0.9	В	1.00	2.00	1.01	1.00

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

04.05.2010 18:06:14



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

04.05.2010 18:06:25



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 18:49:44

Formula C ₁₈ H ₁₇ NO ₅	FW 327.3313						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	08 Jun 2012 15:21:36
Date Stamp	08 Jun 2012 15:21:	36					
File Name	C:\Users\Fedor\Des	sktop\C13 Рома Для Стать	ы в JOC 25.05.12\г.	udn-040612-3Af\rudn-0406	12-3Af_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Tanan anafuna (damas o	1 22 000						





Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 18:53:00

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0 114

OH 24

Formula C ₁₈ H ₁₇ NO ₅	FW 327.3313						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15N	/13C/31P Z3379/0400		Date	08 Jun 2012 15:21:36
Date Stamp	08 Jun 2012 15:21:	36	•				
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Стать	и в ЈОС 25.05.12\ruc	in-040612-3Af\rudn-04061	12-3Af_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Temperature (degree C) 32.000						



49

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

00 06 2012 18-55-07

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						The second second	
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/	15N/13C/31P Z3379/0400		Date	08 Jun 2012 15:21:36
Date Stamp	08 Jun 2012 15:21:	36					
File Name	C:\Users\Fedor\Des	sktop\C13 Рома Для Стат	ьи в ЈОС 25.05.12	2\rudn-040612-3Af\rudn-0406	12-3Af_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
			420 X	H- 6q		2	$\begin{array}{c c} & & & \\ 9 \\ & & \\ 10 \\ & & \\ 10 \\ & \\ 10 \\ & \\ 10 \\ & \\ 10 \\ & \\ 0 \\ & \\ 0 \\ & \\ 2 \end{array}$
udn-040612-3Af_001000f	īd		30	d d	BMSC	2	1
			с П	(8.8)	*	Comp	oound 3Af
1-70	H-Cax+				.2(



2.16

3.9

3.8

3.7

3.6

4.0

1.13

4.2

4.1





50

2.51



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 19:09:15

quisition Time (sec)	0.5571	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	08 Jun 2012 16:42:40
te Stamp	08 Jun 2012 16:42:4	40					
e Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Ста	тьи в JOC 25.05.12\ru	dn-040612-3Af-c13dec\rudn-	040612-3Af-c13dec	_001000fid	
equency (MHz)	100.62	Nucleus	13C	Number of Transients	386	Origin	spect
iginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
ceiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.8018
reep Width (Hz)	29409.97	Temperature (degree	e C) 27.000				
							$ \begin{array}{c} 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 $
						1 - + 6	H' O
					Cor	npound 3Af	10ba 1
n-040612-3Af-c13dec	001000fid		92		-	T	
	5.501.5 <i>5.55</i> .05		27.5				
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***********************************	- Design of the state of the st	obviolant and and and and and	transfer de la contraction de la construction de la construcción de la	the foot for the foot for the foot of the			
192 184 1	176 168 160	152 144 13	36 128 120	112 104 96	88 80 72	64 56 48	40 32 24 16 8

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 19:09:28

OH

Formula C ₁₈ H ₁₇ NO ₅	FW 327.3313						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	08 Jun 2012 16:42:40
Date Stamp	08 Jun 2012 16:42:4	0					
File Name	C:\Users\Fedor\Des	top\C13 Рома Для Статьи	в JOC 25.05.12\r	udn-040612-3Af-c13dec\rudn-0	040612-3Af-c13dec	_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	386	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.8018
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				



52

COOH

and

6

170.43

170.73

170

168

172

Formula O II NO

207 2212

EIA/

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 19:09:43

Formula C ₁₈ H ₁₇ NO ₅	- 327.3313						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	15N/13C/31P Z3379/0400		Date	08 Jun 2012 16:42:40
Date Stamp	08 Jun 2012 16:42:40	0					
File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Статьи	в ЈОС 25.05.12\	rudn-040612-3Af-c13dec\rudn-0	040612-3Af-c13dec	_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	386	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.8018
Sweep Width (Hz)	29409.97	Temperature (degree C	27.000				



rudn-040612-3Af-c13dec_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 19:20:38

Formula C ₁₈ H ₁₇ NO ₅	<i>FW</i> 327.3313						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/	5N/13C/31P Z3379/0400		Date	08 Jun 2012 16:51:12
Date Stamp	08 Jun 2012 16:51:	12					
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Статьи	в JOC 25.05.12\r	udn-040612-3Af-dept135\rudn-0	040612-3Af-dept1	35_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	166	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9104.3936
Sweep Width (Hz)	29409 97	Temperature (degree C)	27 000				



38.16

25.00



66.78

alimitinitini Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

C ₁₈ H ₁₆ N ₂ O ₇	FW 372.3	3288		100		MINNYKK	40.17		
Acquisition Time (sec)	1.4549	Comment	single_pulse	Date	09 Apr 2010	13:02:56		Date Stamp	09 Apr 2010 13:01:44
File Name	D:\NMR\6.04	.10\FZ1139-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	8
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	32.00	Solvent	DMSO-d6	Spectrum Offset (H	<i>lz</i>) 3000.8616	Sweep Width (Hz)	11261.26	Temperature (degree C	21.300
				Co	ompound	ls 3Ag/3Bg	O 25 N 24 0 27	$ \begin{array}{c} 4 \\ 3 \\ 3 \\ 3 \\ 2 \end{array} \right) \begin{array}{c} 0 \\ 22 \\ 1 \\ 3 \\ 9 \\ 10 \end{array} \right) \begin{array}{c} 0 \\ 22 \\ 1 \\ 1 \\ 9 \\ 10 \end{array} \right) $	OH 26 O 23 0 10 10a 10b 10b 3 10b 3 10b 3 10b 10b 3 10b 10b 10b 3 10b
g/3Bg = 91/	9							2.50	$10ba \frac{0}{1}$
								5	
								2.0	
						4			
			17			5.3			
CODH			œ	4	0				
200				6.8 6.6			2 23.10	5	
10			16 91	688				3.00	
0			28 8. 7.5	7.7 7 7.6 7 81 81	45	4722	3.94 3.92 17 15 15	0.0 4 0.0	52 1 1 49
3			8	6.5	9	4444	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	2.2 2.5 1.6 1.6 1.6 1.6	
12							10		and the second se
12					l	1		L	A

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.04.2010 14:24:42 372.3288 FW Formula C18H16N2O7 1.4549 Comment single pulse Date 09 Apr 2010 13:02:56 Date Stamp 09 Apr 2010 13:01:44 Acquisition Time (sec) File Name D:\NMR\6.04.10\FZ1139-1.jdf Frequency (MHz) 600.17 Nucleus 1H Number of Transients 8 **Original Points Count** Points Count 16384 single_pulse.ex2 Origin ECA 600 16384 Owner delta Pulse Sequence 32.00 Solvent DMSO-d6 Sweep Width (Hz) 11261.26 Temperature (degree C) 21.300 **Receiver Gain** Spectrum Offset (Hz) 3000.8616 OH min+mes, H-4' 0 8,16, d (8.3) -0 24 11 0 11. May 27 H-21 Η 10ba 8,17, 5 Brd 6,81, min of FZ1139-1.jdf Compounds 3Ag/3Bg 4-106 min + meg, H-6' d (7.7) maj Min H-2' 8,28,\$ (2.2) 5.34 8.17 H-A H-10 Min + mej; H-5' dld (t.3; 7.7; 8.3) 6.80 6.79 6.65 6.64 7.68 8.16 .91 min MIG 70 8.28 45 81 54 12 5 i 00 ŝ 2.17 1.13 1.13 1.08 1.00 0.09 0.09 1.00 8.0 7.5 7.0 6.5 6.0 5.5 Chemical Shift (ppm)

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.04.2010 14:24:56



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

19.04.2012 16:23:21

cquisition Time (sec)	0.6921	Comment	single pulse de	ecoupled gated NOE		Date	15 Apr 201	0 14:38:46	
ate Stamp	15 Apr 2010 14	4:37:42		File Name	D:\NMR	\14.04.10\FZ1147-1.jdf		Frequency (MH	z) 150.91
ucleus	13C	Number of Transients	200	Origin	ECA 60	Original Points Coun	t 32768	Owner	delta
oints Count	32768	Pulse Sequence	single_pulse_	dec		Receiver Gain	50.00	Solvent	DMSO-d6
bectrum Uπset (Hz)	15091.3428	Sweep Width (Hz)	4/348.49	Temperature (degr	Compo	unds 3Ag/3Bg	O 27 N 24 0 25	$ \begin{array}{c} & & & \\ & & & & \\ & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & $	$ \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\ \begin{array}{c} \end{array} \\ \end{array} \\$
147-1.jdf								40.16 40.03 39.88	1000 1
170.93		6 5	130.40 .20 120.73		-93.25 -90.05		.61 49.16	40.30 39.75	
-171.02		-138.97 139.5 135.28 132.71	123			67.32	22	- 40.94 - 38.72	70.07
	28.09	17.91 0.02 9.83	80.29		92.58 9.89	3.61	3.72	3.13	28

Date Stamp

Points Count

FZ1147-1.jdf

170.93

171.02

171.09

172

170

168.09

168

166

164

162

160

158

156

154

152

150

Nucleus

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

FW 372.3288 Formula C1.H.N.O7 0.6921 Comment single pulse decoupled gated NOE Date 15 Apr 2010 14:38:46 Acquisition Time (sec) 15 Apr 2010 14:37:42 D:\NMR\14.04.10\FZ1147-1.jdf Frequency (MHz) 150.91 File Name 13C Number of Transients 32768 Owner delta 200 Origin ECA 600 **Original Points Count** 32768 **Receiver Gain** 50.00 Solvent DMSO-d6 Pulse Sequence single_pulse_dec Spectrum Offset (Hz) 15091.3428 Sweep Width (Hz) 47348.49 Temperature (degree C) 21.300 0-22 OH. 26 0 24 0 114 25 Η 10ba Compounds 3Ag/3Bg 3 96 4 147. COOH, G 120.73 30.40 23.20 139.56 132.75 35.28 138.97

140.02

140

138

136

134

132.68

132

130.29

130

128

126

124

122

120

19.04.2012 16:23:54

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144

142

146 Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

147.91

148

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

19.04.2012 16:24:17



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.05.2012 20:08:37

Formula C ₃₆ H ₃₂ N ₄ O ₁₄ ?		FW 744.6577+? (3	372.3288+372.328	8+?+?)					
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H	I/15N/13C/31P Z3379/0400		Date	08 Jun 2010 05:58:24		
Date Stamp	08 Jun 2010 05:	58:24		File Name	D:\NMR\C_13\0)7.06.10 (Рома)\rudn_1_N	16\rudn_1_N6_001000fid		
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	32	Origin	spect		
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg		
Receiver Gain	512.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542		
Sweep Width (Hz)	10416.03	Temperature (degree 0	C) 27.000						
				Compour	nds 3Ah/	'3Bh	$\begin{array}{c} 0 \\ 22 \\ 22 \\ 22 \\ 21 \\ 26 \\ 22 \\ 22 \\ 22$		$\begin{array}{c} 48 & 53 \\ 7 & 6a \\ 0 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10$
$\mathbf{Ah/3Bh} = 92$	/8					5.31			
				7.73				2.50	
								3.25	
				g	.84 6.67		20	3.09	
				-7.74	6.66			3.06	
Bz.S				8.09			96 d	2	
COOFI				8. 61 7.62 60			16 3.93 3.93 3.93	3.16	55 8.6
12.04				7.78 7.79 7.59 7.7 7.59 7.59	6.60	5.49	4.19 4.18 4.18 3.90	-2.5	1.70 1.68 1.66
0.89		ene e al lisso a consilo		1.01 1.15 1	.050.09	1.00	1.16 0.13 0.10	1.16	2.35

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.05.2012 20:11:20

OH

0.

,OH

0.

-0

Formula C ₃₆ H ₃₂ N ₄ O ₁₄ ?		FW 744.6577+? (37	72.3288+372.32	288+?+?)			
Acquisition Time (sec)	1.5729	Comment	5 mm QNP	1H/15N/13C/31P Z3379/0400		Date	08 Jun 2010 05:58:24
Date Stamp	08 Jun 2010	05:58:24		File Name	D:\NMR\C_13	3\07.06.10 (Рома)\rudn_1_N6	6\rudn_1_N6_001000fid
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	32	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	512.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542
Sweep Width (Hz)	10416.03	Temperature (degree C	27.000				



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.05.2012 20:13:37

ormula C ₃₆ H ₃₂ N ₄ O ₁₄ ?		FW 744.6577+?	(372.3288+372.32	288+?+?)				
cquisition Time (sec)	1.5729	Comment	5 mm QNP	1H/15N/13C/31P Z3379/0400		Date	08 Jun 2010 05:58:24	
ate Stamp	08 Jun 2010 0	5:58:24		File Name	D:\NMR\C_13	\07.06.10 (Рома)\rudn_1_N	6\rudn_1_N6_001000fid	
requency (MHz)	400.14	Nucleus	1H	Number of Transients	32	Origin	spect	
riginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg	
eceiver Gain	512.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	
veep Width (Hz)	10416.03	Temperature (degre	e C) 27.000	2H, m			$\begin{array}{c} O \\ 22 \\ 22 \\ 22 \\ 22 \\ 22 \\ 22 \\ 22 \\$	$\begin{array}{c} \begin{array}{c} \begin{array}{c} \\ \\ \\ \\ \end{array} \end{array} \\ \begin{array}{c} \\ \\ \end{array} \\ \\ \\ \end{array} \\ \begin{array}{c} \\ \\ \\ \end{array} \\ \\ \\ \end{array} \\ \begin{array}{c} \\ \\ \\ \end{array} \\ \\ \end{array} \\ \begin{array}{c} \\ \\ \\ \\ \end{array} \\ \\ \end{array} \\ \begin{array}{c} \\ \\ \\ \\ \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \\ \\ \\ \end{array} \\ \begin{array}{c} \\ \\ \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \\ \\ \end{array} \\ \begin{array}{c} \\ \\ \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \\ \end{array} \\ \end{array} \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \\ \\ \end{array} $
				H-Zax H-Geg		0	Compounds	3Ah/3Bh
1_1_N6_001000fid				1. 1	11-7	4-69	O SMAR O	
4-106		ł	1-20g		Q (9,3	b)	2.50	
\$		(4	dd 4:11.4)	1	27 3.25	<u>3</u> .09		H-3ax H-3
				Hz	3 ×	H-4ax		m m
min			(3.96	3.29 16 M	(3.8; 13,	4)	1 /
5.49			4.19 4.18 4.15 4.15	BE Mih	-3:18 3.17 3.14 3.	July -	2.54	1.73 1.72 1.70 1.70 1.68 1.67 1.67 1.67 1.65 1.65 1.55 1.55
0.08 1.00			1.16	2.14 0.13 0.10	1.21	1.16		2.35

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

8 Jun 2010



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

8 Jun 2010



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

8 Jun 2010



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

8 Jun 2010



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

19.04.2012 17:26:30

Formula C ₁₁ H ₁₃ NO ₃	FW 207.22	258							
Acquisition Time (sec)	2.1837	Comment	single_pulse	Date	26 Apr 201	1 09:38:00		Date Stamp	26 Apr 2011 13:26:06
File Name	D:\NMR\19.04	.11\FZ1784-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	18
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	30.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	7503.00
Temperature (degree C) 21.400								







General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

19.04.2012 17:27:11

cquisition Time (sec)	2.1837	Comment	single_pulse	Date	26 Apr 2011 (09:38:00		Date Stamp	26 Apr 2011 13:26:06
le Name	D:\NMR\19.04	.11\FZ1784-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	18
rigin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
eceiver Gain	30.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	7503.00
					Com	pound 4a		$ \begin{array}{c} $	-4
1784-1.jdf								10ba 1	-2 X-7
4-29		t	1-Ya lddd			H-7(280		(8,d,
(+18; 119 d+		(1.	4;3.7;	11,7;13,7)	H-69 endo ddd	dt (4.0331	7)	H-39 M H-3 M	57 1.54 -1.55
3.73 3.73					(1,8;4,0;	(+;8) 525 223		3	1.52
3.76 3.76 3.70	2		3.04 3.04 3.03 3.03 3.00 2.98 3.00		2.41	2.40 2.39 2.39 2.39 2.38 2.38 2.38 2.38 2.38 2.38 2.28	00 10 10 10	1.93 1.92 1.92 1.87 1.87 1.85 1.86	
1.02			1.00			0.87 0.98		1.15	2.03

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

19.04.2012 17:26:55

uisition Time (sec)	2.1837	Comment	single_pulse	Date	26 Apr 20	011 09:38:00		Date Stamp	26 Apr 2011 13:26:0
Name	D:\NMR\19.04	.11\FZ1784-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	18
gin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
eiver Gain	30.00	Solvent	CHLOROFOR	M-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	7503.00
perature (degree C	/ 21.400								4
0 f	1-9			H-10	2,30	Compound	l 4a	9 10 10a 10b H 0- 10ba 1	
14-1.jdf	de			5.4					
e) (1	. 8', 50)					H - 8			H-2e
6.44						dd (1.8; 4.6)			H-9e M, 2t
6.40									
6.39 6.38						5.12 5.12 5.11 5.11			4.19
Ht									4.17
1.74				0.88	1	0.88			2.00

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

12 May 2009


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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

1 Apr 2010

Acquisition Time (sec)	1.6056	Comment	Imported fro	om UXNMR.		Date	30 Mar 20	10 14:24:00	
File Name	D:\NMR\23 a	and 25.03.10\N846\N846_0010	000fid	Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	32
Original Points Count	16384	Points Count	16384	Pulse Sequence	za	Solvent	CHLORO	FORM-D	
Sweep Width (Hz)	10204.08	Temperature (degree C)	27.000		-9	Contoint	Oneonto	OT WED	

Compound 4b







1 Apr 2010

Acquisition Time (sec)	1.6056	Comment	Imported fro	om UXNMR.		Date	30 Mar 20	10 14:24:00	
File Name	D:\NMR\23 a	and 25.03.10\N846\N846	001000fid	Frequency (MHz)	400 14	Muslaus	50 IVIAI 20	10 14.24.00	
Original Points Count	16384	Points Count	16384	Pulse Sequence	400.14	Nucleus	1H	Number of Transients 32	
Sweep Width (Hz)	10204.08	Temperature (degr	ee C) 27.000	i dise beguenee	29	Solvent	CHLORO	FORM-D	







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1 Apr 2010



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

1 Apr 2010

cquisition Time (sec)	0.6226	Comment	Imported from UX	NMR.		Date	30 Mar 2010 14:26:08	
ie Name	D:\NMR\23 and 25	5.03.10\N846c13dec\N846c	c13dec_001000fid			Frequency (MHz)	100.62	
ucieus	13C	Number of Transients	305	Original Points Count	16384	Points Count	16384	
lise Sequence	zgpg	Solvent	CHLOROFORM-I	D		Sweep Width (Hz)	26315.79	
					Compour	nd 4b	H ₃ C 9 0 11 10a	O // ¹⁶ N-4
						2 22:	H 10 4	Oba 0 - 2 3
		9				67	38.9	
		30						7 3
		10			106			33.89 25.42 (
		132.55			86.9		19.62 0	18.76 Za
				8,100	A			
				ŝ	77.44 ×	76.82		
Statefolisian and an	stanni an	andung Westerstrateringersmunisterspron W	ndetten understatet et derstatet anderstatet	the of the sectors and the sector and the sector and the sector and	aliteration of the second s	numinalitation or comprise animality of a setting and	hadananterskalanskalande (Madarspilalande standerskalande Versende	dynhan tamagasyddianiaisiaph dwinawweiriawaanaa
		З						

1 Apr 2010

Acquisition Time (sec)	0.6226	Comment	Imported from	IVNIND				1 Apr 2010
File Name	D:\NMR\23 an	d 25 03 10\N846dept135\N946	deputed nom	UXNMR.		Date	30 Mar 2010 14:32:32	
Nucleus	130	Number of Transient	ocep135_001000	Jfid		Frequency (MHz)	100.62	
Pulse Sequence	dent135	Solvert	185	Original Points Count	16384	Points Count	16384	
Temperature (degree C)	27.000	Solvent	CHLOROFOR	M-D		Sweep Width (Hz)	26315.79	



ormula C22H24Br2N2O6	?	FW 572.2438+?	(286.1219+286	5.1219+?+?)					
cquisition Time (sec)	1.8193	Comment	single pulse	Date	24 May 2010	11:59:32		Date Stamp	24 May 2010 11:58:1
ile Name	D:\NMR\11.05	.10\FZ1232-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	2
rigin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
ceiver Gain	38.00	Solvent	CHLOROFOR	RM-d		Spectrum Offset (Hz)	3601.0339	Sweep Width (Hz)	9005.76
mperature (degree C)	25.000								
						Br 15		baa 1	$ \begin{array}{c} & & & & & \\ & & & & & \\ & & & & \\ 9 \\ & & & &$
				5.02			33	Compounds 4	³⁴ Ac/4Bc
								I I I I	
							6	54/36	
CHC3									
7.25	6.59 6.58	6.39 6.39							
	-6.67 -6.66	6.35			.23 4.21 .22 4.21	88 88 3.86 84 3.86 34	3.09	2.66 2.65 2.64 2.64 2.64 2.47 2.45 2.25 9 2.22	- 2.21 .89 .1.86 .1.53
					44	3.5 3.5	3.11 3.10 3.06	2.66	1.92 2.15 1.90 2.15 1.86 1.84 1.66
	0.59	0.31		0.90	1,98	1.00	0.99	0.90 0.34 1.02	1.00 1.00

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

24.05.2012 16:49:11

Acquisition Time (sec)	1.8193	Comment	single_pulse	Date	24 May 20	10 11:59:32		Date Stamp	24 May 201	10 11:58:13
File Name	D:\NMR\11.0	5.10\FZ1232-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	2	
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_puls	e.ex2
Receiver Gain	38.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	3601.0339	Sweep Width (Hz)	9005.76	
Temperature (degree C,	25.000									
								0		0
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						Br	-8/1~6a-	Br 31	/ 60-	6
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Z1232-1.jdf	A A					6	6	1		
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0.29 0.59	0.31					0.90		1.98		1.00
6.5		60		5.5		5.0	1	5	4.0	

Chemical Shift (ppm)

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

24.05.2012 16:49:25

	4 9402	Commont	-timele	Data	24 May 24	10 11:50:22		Data Stamp	24 May 2010 11-58-12
Acquisition Time (sec)	1.8193	Comment	single_pulse	Date	24 Iviay 20	Nuclous	14	Number of Transients	24 May 2010 11.56.15
nie Name	D.(NIVIR(11.0	Original Points Coun	4 16204	Owner	dolta	Points Count	16384	Pulse Sequence	single nulse ex2
rigin	ECA 600	Criginal Points Coun		Owner	ueita	Fornts Count	3601 0230	Swoon Width (Hz)	0005 76
eceiver Gain	38.00	Solvent	CHLOROFOR	KIVI-Q		Spectrum Onset (Hz)	3001.0333	Sweep widdi (112)	3003.70
						Br 15		O 16 N 31 31 10b 3	3 - 0 - 0 - 0 - 0 - 0 - 0 - 0 - 0 - 0 -
			H-	6 endo	k	l - 7 endo	H 101 64 33	0-2	$H_{10bb} O_{1} - 2$
1232-1.jdf		4-	(-) M	,0, 22.0)	(8.9;12.0)	(Compounds 4	Ac/4Bc
H-4 ax		ex	0		dol	dd	H-	- Zax	H-31
min + mai	7	(4.0; 8	3.9)	dd			N	M	min + v
(3.4:13	.0)	Min	maj	maj	Mag	3	mil	n + moj	M
3.09		2.66	2.65 2.64 2.64	2.45	12	9 221 222 222 221 222 222 222 222 222 222	.89	Hz X	-1.53 -1.53 1.51 50
3.11 3.10 3.05		2.68	2.66	2.37	2.35	2.17 2.17 2.15	1.92	1.85 1.86 1.83 1.83	
0.00			0.90	0.65 0	34	1.02	1.00		1.00

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28.06.2010 21:32:34

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114

Br

15

Formula C11H12BrNO3 FW 286.1219

Acquisition Time (sec)	0.6921	Comment	single pulse de	coupled gated NOE		Date	23 Jun 2010	10:49:17	
Date Stamp	23 Jun 2010 1	0:47:50		File Name	D:\NMR\16.06	.10\FZ1279.jdf		Frequency (MHz)	150.91
Nucleus	13C	Number of Transients	200	Origin	ECA 600	Original Points Count	32768	Owner	delta
Points Count	32768	Pulse Sequence	single_pulse_d	ec		Receiver Gain	50.00		
Solvent	CHLOROFOR	RM-d		Spectrum Offset (Hz)	15091.3428	Sweep Width (Hz)	47348.49	Temperature (degree	C) 22.600

Compounds 4Ac/4Bc



28.06.2010 21:45:23

Formula C ₁₁ H ₁₂ BrNO ₃	FW 286.1	1219							
Acquisition Time (sec)	0.6921	Comment	single pulse dec	oupled gated NOE		Date	23 Jun 2010	10:49:17	
Date Stamp	23 Jun 2010 1	0:47:50		File Name	D:\NMR\16.06	.10\FZ1279.jdf		Frequency (MHz)	150.91
Nucleus	13C	Number of Transients	200	Origin	ECA 600	Original Points Count	32768	Owner	delta
Points Count	32768	Pulse Sequence	single_pulse_de	C		Receiver Gain	50.00		
Solvent	CHLOROFOR	RM-d		Spectrum Offset (Hz)	15091.3428	Sweep Width (Hz)	47348.49	Temperature (degree	C) 22.600



Compounds 4Ac/4Bc

FZ1279.jdf



Formula C₁₁H₁₂BrNO₃ FW 286.1219

28.06.2010 21:40:41

106

Acquisition Time (sec)	0.6921	Comment	single pulse	decoupled asted NOF						
Date Stamp	23 Jun 2010	10:47:50	origie puise (Date	23 Jun 2010	10:49:17		
Nuclous	120	10.47.50	1.000	File Name	D:\NMR\16.06	5.10\FZ1279.jdf		Frequency (MHz)	150.91	
Rucieus -	130	Number of Transients	200	200 Origin		Original Points Count	32768	Owner	dolta	
Points Count	32768	Pulse Sequence	single pulse	dec		Receiver Gain	50.00	owner	ueita	
Solvent	CHLOROFO	DRM-d		Spectrum Offeet (U=)	15001 0400		50.00			
				Spectrum Onset (Hz)	15091.3428	Sweep Width (Hz)	47348.49	Temperature (degree	C) 22.600	

0 /16 114 Н 10ba

Br

Compounds 4Ac/4Bc

FZ1279.jdf



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.06.2010 21:42:37

Acquisition Time (sec)	0.6921	Comment	single pulse	decoupled gated NOE		Date	23 Jun 2010	10:49:17	
Date Stamp	23 Jun 2010	10:47:50		File Name	D:\NMR\16.06	5.10\FZ1279.jdf		Frequency (MHz)	150.91
Nucleus	13C	Number of Transients	200	Origin	ECA 600	Original Points Count	32768	Owner	delta
Points Count	32768	Pulse Sequence	single_pulse	e_dec		Receiver Gain	50.00		
Solvent	CHLOROF	ORM-d		Spectrum Offset (Hz)	15091.3428	Sweep Width (Hz)	47348.49	Temperature (degree	C) 22.600



FZ1279.jdf

49.26

49.35



Compounds 4Ac/4Bc

Chemical Shift (ppm) Fedor 1. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

19.04.2012 20:16:1

Acquisition Time (sec)	1.4909	Comment	FZ.9	Date	22/06/2004	00:00:00		Date Stamp	22/06/2004 00:00	00:00
File Name	D:\NMR\24	4.06.04\FZ.013		Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	16	
Original Points Count	8192	Points Count	8192	Spectrum Offset (Hz)	1605.9999	Sweep Width (Hz)	5494.51	Temperature (degree	C) 24.000	
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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

19.04.2012 20:16:29





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

3 Jun 2011



3 Jun 2011

1.26

Acquisition Time (sec)	1.6056	Comment	Imported from UXNMF	۲.		Date	31 May 2011 14:11:12
File Name	C:\Users\Fedor\Deskto	ор\19.05.2011 С 13 Для Ир	оы Статья в Тетраэдро	н\rudn-300511-N4\rudn-30	0511-N4_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	8	Original Points Count	16384	Points Count	16384
Pulse Sequence	zg	Solvent	CHLOROFORM-D	Sweep Width (Hz)	10204.08	Temperature (degree (C) 27.000
						181 - W	CH

Compound 5



5



3 Jun 2011

Acquisition Time (sec)	1.6056	Comment	Imported from UXNM	R.		Date	31 May 2011 14:11:12
File Name	C:\Users\Fedor\Deskto	р\19.05.2011 С 13 Для Ир	ы Статья в Тетраэдро	on/rudn-300511-N4\rudn-30	0511-N4_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	8	Original Points Count	16384	Points Count	16384
Pulse Sequence	zg	Solvent	CHLOROFORM-D	Sweep Width (Hz)	10204.08	Temperature (degree	C) 27.000
							CH3

Compound 5

H₃C

H₃C

H 3



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

3 Jun 2011

Acquisition Time (sec)	1.6056	Comment	Imported from UXNM	R.		Date	31 May 2011 14:11:12	2
ile Name	C:\Users\Fedor\D)esktop\19.05.2011 С 13 Для И	ры Статья в Тетраэдр	он\rudn-300511-N4\rudn-30	0511-N4_001000fid	Frequency (MHz)	400.14	,
ucleus	1H	Number of Transients	8	Original Points Count	16384	Points Count	16384	Mark
Ilse Sequence	zg	Solvent	CHLOROFORM-D	Sweep Width (Hz)	10204.08	Temperature (degree	C) 27.000	Me
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				the second se		1 1 1 1 1 1 1		

Acquisition Time (sec) 0.5571 31 May 2011 14:58:08 Comment Imported from UXNMR. Date C:\Users\Fedor\Desktop\19.05.2011 C 13 Для Иры Статья в Тетраздрон\rudn-300511-N4-c13dec\rudn-300511-N4-c13dec 001000fid File Name Frequency (MHz) 100.62 Nucleus Number of Transients **Original Points Count** 16384 13C 258 Points Count 16384 29411.77 Pulse Sequence Solvent CHLOROFORM-D Sweep Width (Hz) zapa Temperature (degree C) 27.000 CH₃ H₃C Compound 5 H₃C H 3 23.41 22.07 Me-i 1 6 Me-6 109.82 45.09 2 68.87 06.23 32.44 78.72 V 141.72 48.93 152.76 77.44 77.11 76.79 160 152 144 32 24 16

152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladin**i (An Zaytsev), Vigtor** N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov 3 Jun 2011

 (\mathbf{x})

3 Jun 2011

cquisition Time (sec)	0.5571	Comment	Imported from UXNMR.	Date	31 May 2011 15:04:32		
ile Name	C:\Users\Fedor\Desktop\	19.05.2011 С 13 Для Ирь	Статья в Тетраздрон\rud	n-300511-N4-dept135\rudr	n-300511-N4-dept135_0010	DOOfid	
requency (MHz)	100.62	Nucleus	13C	Number of Transients	212	Original Points Count	16384
oints Count	16384	Pulse Sequence	dept135	Solvent	CHLOROFORM-D	Sweep Width (Hz)	29411.77
				Compoun	d 5	$H_{3C} \rightarrow H_{3C} \rightarrow H$	DO 1 2 2 3 4 5 5 5 5 5 5 5 5 5 5 5 5 5
141.72		109.82		78.72	68.87		32.44
	nd tra dit dag par salak dan menjabagan kenada sala pa					45	

150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimin En Zaytson V. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

06.06.2012 18:42:59



6.6

6.5

6.4

6.3

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

06.06.2012 18:44:26

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4.4

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equisition Time (sec)	2.1837	Comment	single_pulse	Date	06 Jun 2012	11:15:07		Date Stamp	06 Jun 2012 15	:04:23
le Name	D:\NMR\04.06	5.12\FZ2451-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	25	
rigin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex	2
eceiver Gain	38.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	2414.2141	Sweep Width (Hz)	7503.00	Temperature (degree C) 23.300	
					Со	mpound 6		$ \begin{array}{c} 8 \\ 9 \\ 9 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\$	CH ₃	
451-1.jdf (-10 h	1-9 dd				H-	106 H-	8			
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 Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

0.90

4.99

0.99

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

06.06.2012 18:55:32





24 Aug 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.03.2012 21:27:57



Date Stamp

Points Count

Nucleus

Solvent

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.03.2012 21:32:47 167.2050 Formula C₉H₁₃NO₂ FW Acquisition Time (sec) 0.5217 Comment single pulse decoupled gated NOE Date 06 Mar 2012 09:58:23 06 Mar 2012 14:33:11 File Name D:\NMR\05.03.12\13C_FZ2264-1.jdf Frequency (MHz) 100.53 13C Number of Transients 89 Origin ECS 400 **Original Points Count** 16384 Owner delta 16384 Pulse Sequence single_pulse_dec **Receiver Gain** 54.00 CHLOROFORM-d 10052.5303 Temperature (degree C) 24.400 Spectrum Offset (Hz) Sweep Width (Hz) 31407.04 Compound 8Ba cul 3 62.52 13C_FZ2264-1.jdf Mo-5 113.21 107.92 14.00 Me-C=N N-CUS 5.20 53.57 7



4

11

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.05.2012 15:22:55

Formula	C15H17NO4	FW	275.2998
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Acquisition Time (sec)	2.1837	Comment	single_pulse	Date	05 May 201	2 09:04:34		Date Stamp	05 May 2012 12:53:53
File Name	D:\NMR\03.0	5.12\FZ2367-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	4
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	40.00	Solvent	CHLOROFOR	RM-d		Spectrum Offset (Hz)	1992.4193	Sweep Width (Hz)	7503.00
Temperature (degree C) 23.200								

Compound 11







General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.05.2012 14:53:00



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.05.2012 14:58:41

Acquisition Time (sec)	2.1837	Comment	single_pulse	Date	05 May 2012 (09:04:34		Date Stamp	05 May 2	2012 12:53
File Name	D:\NMR\03.05	.12\FZ2367-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	; 4	
Drigin	ECS 400	Original Points Count	16384 *	Owner	delta	Points Count	16384	Pulse Sequence	single_p	ulse.ex2
Receiver Gain	40.00	Solvent	CHLOROFOR	RM-d		Spectrum Offset (Hz)	1992.4193	Sweep Width (Hz)	7503.00	
emperature (degree C)	23.200									
Z2367-1.jdf				0	CH ₂	Сс	ompoun	d 11	1.64	
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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

14.07.2012 14:10:31

Formula C15H17NO4	FW 275.2998						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	I/15N/13C/31P Z3379/0400		Date	28 May 2012 17:16:48
Date Stamp	28 May 2012 17:16:4	48					
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Статьи	B JOC 25.05.12	rudn-250512-11-c13dec\rudn-25	0512-11-c13dec_0010	00fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	445	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9627.5156
Sweep Width (Hz)	29409 97	Temperature (degree C)	27 000				





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

14.07.2012 14:14:30

Formula C ₁₅ H ₁₇ NO ₄	FW 275.2998						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	28 May 2012 17:16:48
Date Stamp	28 May 2012 17:16:4	18	'				
File Name	C:\Users\Fedor\Desk	ktop\C13 Рома Для Статы	в JOC 25.05.12\	rudn-250512-11-c13dec\rudn-25	0512-11-c13dec_0010	00fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	445	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9627.5156
Sweep Width (Hz)	29409.97	Temperature (degree C	27.000				
							0



Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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075 0000

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

14.07.2012 14:17:33

Formula C ₁₅ H ₁₇ NO ₄	-W 275.2998						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP	1H/15N/13C/31P Z3379/0400		Date	28 May 2012 17:16:48
Date Stamp	28 May 2012 17:16:4	8					
File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Статьи	в JOC 25.05.	12\rudn-250512-11-c13dec\rudn-25	0512-11-c13dec_0010	00fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	445	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9627.5156
Sween Width (Hz)	29409 97	Temperature (degree C)	27 000				





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

14.07.2012 14:21:58

0

H

Formula C ₁₅ H ₁₇ NO ₄	FW 275.2998						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400	/13C/31P Z3379/0400		28 May 2012 17:25:20
Date Stamp	28 May 2012 17:25:2	20	1				
File Name	С:\Users\Fedor\Desktop\C13 Рома Для Статьи в JOC 25.05.12\rudn-250512-11-dept135\rudn-250512-11-dept135_001000fid						
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	273	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9627.5049
Sween Width (Hz)	29409 97	Temperature (degree C	27 000				

Compound 11


Formula C₇H₉NO₂ FW 139.1519

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.07.2011 17:40:16

Acquisition Time (sec)	2.1837	Comment	single_pulse	Date	ate 13 Jul 2011 08:58:11			Data Stamp	12 1.1 0011 10 10 50
File Name	D:\NMR\01.0	07.11\FZ1919-1.idf	Corolline	Frequency (MHz)	200 79	Nucleur		Date Stamp	13 Jul 2011 12:46:56
Origin	ECS 400	Original Data to Control		riequency (miliz)	399.70	Nucleus	ıн	Number of Transients	8
Oligin	EC3 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulso Soquence	cingle pulse and
Receiver Gain	30.00	Solvent	CHLOROFOR	P MS			10004	r uise sequence	single_pulse.exz
Temperature (degree C)	24.300		on concorron of	(W-G		Spectrum Offset (Hz)	1998.9109	Sweep Width (Hz)	7503.00

Compound 12a reaction mixture





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.07.2011 17:41:11

OH

Acquisition Time (sec)	2.1837	Comment	single_pulse	Date	13 Jul 2011 (08:58:11		Date Stamp	13 Jul 2011 12:46:56
File Name	D:\NMR\01.07	.11\FZ1919-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	8
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	30.00	Solvent	CHLOROFOR	RM-d		Spectrum Offset (Hz)	1998.9109	Sweep Width (Hz)	7503.00
Temperature (degree C)	24.300								
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								1	
				Compo	und 120	E	11	2 - 11	-



Formula C₇H_oNO₂

FW

139.1519

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.07.2011 17:41:22

Acquisition Time (sec)	2.1837	Comment	single pulse	Date	13 Jul 201	1 08:58:11	Date Stamp	13 Jul 2011 12:46:56	
File Name	D:\NMR\01.0	07.11\FZ1919-1.jdf	5-1	Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	8
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single pulse.ex2
Receiver Gain	30.00	Solvent	CHLOROFOR	RM-d		Spectrum Offset (Hz)	1998.9109	Sweep Width (Hz)	7503.00
Temperature (degree C,	24.300								





Formula C ₇ H ₉ NO ₂	FW 139.1519	1					25.07.2011 16:37:31
Acquisition Time (sec	0.5898	Comment	5 mm ONP 1	H/15N/13C/31P 73370/0400			
Date Stamp	22 Jul 2011 11:54	:40		File Name	DUNING OF 11	Date	22 Jul 2011 11:54:40
Frequency (MHz)	100.62	Nucleus	130	Number of T	D:\NIVIR\19.07.11	(Рома)\rudn-190711-N9	-c13dec\rudn-190711-N9-c13dec_001000fid
Original Points Count	16384	Ownor	150	Number of Transients	636	Origin	spect
Receiver Gain	20769.00	Owner	root	Points Count	16384	Pulse Sequence	Zapa
Receiver Gam	32768.00	SW(cyclical) (Hz)	27777.78	Solvent	CHLOROFORM-C	1	-545
Spectrum Offset (Hz)	9634.7207	Sweep Width (Hz)	27776.08	Temperature (degree C	27 000		



50

25.07.2011 16:35:19

Conte Sterme	0.0071	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	22 Jul 2011 12:09:36
ate Stamp	22 Jul 2011 12:09	9:36		File Name	D:\NMR\19.07.11 (Рома)\rudn-190711-N9-	-dept135\rudn-190711-N9-dept135_001000fid
requency (MHz)	100.62	Nucleus	13C	Number of Transients	659	Origin	spect
riginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
eceiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d		
Dectrum Offset (Hz)	9634.8008	Sweep Width (Hz)	29409.97	Temperature (degree C	27.000		
n-190711-N9-dept135_	001000fid	elanteration and the contraction of the second s		anang nan mala ang sang sang sang sang sang sang sang	tablertysta förskrigt tig fall är efter sörigan	distan filosofia a forma a constructiva de constructiva de constructiva de constructiva de constructiva de cons	Compound 12a reaction mixture
						14	

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

27.10.2010 19:52:29

cquisition Time (sec)	1.4549	Comment	single_pulse	Date	26 Oct 201	0 12:01:47		Date Stamp	26 Oct 2010 11:14:25
le Name rigin	D:\NMR\22.10 ECA 600	0.10\FZ1406-1.jdf	16384	Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	8
eceiver Gain	32.00	Solvent	CHLOROFOF	RM-d	deita	Spectrum Offset	(Hz) 3000.8616	Pulse Sequence Sweep Width (Hz)	single_pulse.ex2 11261 26
emperature (degree C)	21.700								-
1406-1.jdf		2	2°C						\$ (+2AC
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8.0 7.5	5 7.0	6.5 6.0	5.5	5.0 4	4.5 4.0) 35	30 25	20 15	10

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

27.10.2010 19:53:01

1.4549	Comment	single_pulse	Date 26 Oct 2010 12:01:47				Date Stamp	26 Oct 2010 11:14:25
D:\NMR\22.1	0.10\FZ1406-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	8
ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
32.00	Solvent	CHLOROFOR	RM-d		Spectrum Offset (Hz)	3000.8616	Sweep Width (Hz)	11261.26
	1.4549 D:\NMR\22.1 ECA 600 32.00	1.4549 Comment D:\NMR\22.10.10\FZ1406-1.jdf ECA 600 Original Points Count 32.00	1.4549 Comment single_pulse D:\NMR\22.10.10\FZ1406-1.jdf ECA 600 Original Points Count 16384 32.00 Solvent CHLOROFOF	1.4549 Comment single_pulse Date D:\NMR\22.10.10\FZ1406-1.jdf Frequency (MHz) ECA 600 Original Points Count 16384 Owner 32.00 Solvent CHLOROFORM-d	1.4549 Comment single_pulse Date 26 Oct 201 D:\NMR\22.10.10\FZ1406-1.jdf Frequency (MHz) 600.17 ECA 600 Original Points Count 16384 Owner delta 32.00 Solvent CHLOROFORM-d CHLOROFORM-d	1.4549 Comment single_pulse Date 26 Oct 2010 12:01:47 D:\NMR\22.10.10\FZ1406-1.jdf Frequency (MHz) 600.17 Nucleus ECA 600 Original Points Count 16384 Owner delta Points Count 32.00 Solvent CHLOROFORM-d Spectrum Offset (Hz) Spectrum Offset (Hz)	1.4549 Comment single_pulse Date 26 Oct 2010 12:01:47 D:\NMR\22.10.10\FZ1406-1.jdf Frequency (MHz) 600.17 Nucleus 1H ECA 600 Original Points Count 16384 Owner delta Points Count 16384 32.00 Solvent CHLOROFORM-d Spectrum Offset (Hz) 3000.8616	1.4549 Comment single_pulse Date 26 Oct 2010 12:01:47 Date Stamp D:\NMR\22.10.10\FZ1406-1.jdf Frequency (MHz) 600.17 Nucleus 1H Number of Transients ECA 600 Original Points Count 16384 Owner delta Points Count 16384 Pulse Sequence 32.00 Solvent CHLOROFORM-d Spectrum Offset (Hz) 3000.8616 Sweep Width (Hz)

Temperature (degree C) 21.700

FZ1406-1.jdf

Compounds 12Ab/12Bb



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

27.10.2010 19:53:15

1.23

Acquisition Time (sec)	1.4549	Comment	single_pulse	Date	26 Oct 201	0 12:01:47	Date Stamp	26 Oct 2010 11:14:25	
File Name	D:\NMR\22.10).10\FZ1406-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	8
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single pulse ex2
Receiver Gain	32.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	3000 8616	Sween Width (Hz)	11261.26
Temperature (degree C)	21.700					opeen ann erneer (112)	0000.0010	encep main (112)	11201.20

FZ1406-1.jdf



01 Sep 2011 09:53:04 Acquisition Time (sec) 0.5898 Comment Imported from UXNMR. Date Frequency (MHz) File Name 100.62 C:\Users\Fedor\Desktop\26.08.11\rudn-260811-N5-c13dec\rudn-260811-N5-c13dec_001000fid **Original Points Count** 16384 16384 Nucleus 13C Number of Transients 564 Points Count Pulse Sequence Solvent CHLOROFORM-D Sweep Width (Hz) 27777.78 Temperature (degree C) 27.000 zgpg Η H₃C H₃(62% 38% B Compounds 12Ab/12Bb 5 2 0.30 A 77.21 85.94 2× Me-4 14.08 42.69 146.99 111.64 -26.22 -25.77 22 08 77.13 76.81 45 59.65 17 والمستحد والمرتبع والمراجع والمرجع والمرجع المرجع والمحاصر المتحاص المتحاص المحاص والمحاص والمحاص والمحاط المحاص والمحاص والمحا 25 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 50 45 40 35 30 55

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

2 Sep 2011

2 Sep 2011

quisition Time (sec)	0.5571	Comment	Imported from UXNN	IR.		Date	01 Sep 2011 10:10:08
e Name	C:\Users\Fedor\	Desktop\26.08.11\rudn-2608	311-N5-dept135\rudn-2608	11-N5-dept135_001000fid	1	Frequency (MHz)	100.62
cleus	13C	Number of Transien	ts 251	Original Points Count	16384	Points Count	16384
lse Sequence	dept135	Solvent	CHLOROFORM-D	Sweep Width (Hz)	29411.77	Temperature (degree	C) 27.000
						Сотрог	nds 12Ab/12Bb
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					77.21		
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Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.02.2012 19:16:58



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.03.2012 21:44:46

cquisition Time (sec)	0.5217	Comment	single pulse	decoupled gated NOE		Date	06 Mar 2012 09:53:18
ate Stamp	06 Mar 2012 14	4:28:06	54.V 21	File Name	D:\NMR\05.03	3.12\13C_FZ2243-1.jdf	
equency (MHz)	100.53	Nucleus	13C	Number of Transients	106	Origin	ECS 400
ginal Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse_dec
ceiver Gain	54.00	Solvent	CHLOROFO	DRM-d		Spectrum Offset (Hz)	10052.5303
eep Width (Hz)	31407.04	Temperature (deg	gree C) 24.400				
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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 19:54:53

OH

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Formula C ₁₂ H ₁₃ NO ₆	FW 267.2347						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	08 Jun 2012 15:15:12
Date Stamp	08 Jun 2012 15:15:	12					
File Name	C:\Users\Fedor\Des	sktop\C13 Рома Для Стат	ы в ЈОС 25.05.12	\rudn-040612-15a\rudn-0406	12-15a_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	20	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Temperature (degree (32.000						



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 19:56:54

Formula C ₁₂ H ₁₃ NO ₆	FW 267.2347							
Acquisition Time (sec) 1.5729	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	08 Jun 2012 15:15:12	
Date Stamp	08 Jun 2012 15:15	:12						
File Name	C:\Users\Fedor\De	sktop\C13 Рома Для Стат	ьи в ЈОС 25.05.12	rudn-040612-15a\rudn-0406	12-15a_001000fid	Frequency (MHz)	400.14	
Nucleus	1H	Number of Transients	20	Origin	spect	Original Points Count	16384	
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00	
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03	
H−39 £ rudn-040832-15a 00100	DOfid	H. S	- 2	Со	mpound	15a	$\begin{array}{c} 2 \\ 0 \\ a \\ 0 \\ 0 \\ 0 \\ 1 \\ 0 \\ 0 \\ 1 \\ 0 \\ 0 \\ 0$	H-19,0



09.06.2012 19:57:10



Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 20:09:21

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Formula C ₁₂ H ₁₃ NO ₆	FW 267.2347						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15M	V/13C/31P Z3379/0400		Date	08 Jun 2012 13:39:12
Date Stamp	08 Jun 2012 13:39:1	2	10				
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Статьи	в JOC 25.05.12\rudr	n-040612-15a-c13dec\rudn-(040612-15a-c13dec_0	01000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	427	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.7930
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				



09.06.2012 20:09:33

$\mathbf{brmula} \ \mathbf{C}_{12} \mathbf{H}_{13} \mathbf{NO}_{6} \mathbf{F}_{12} \mathbf{H}_{13} \mathbf{NO}_{6} \mathbf{F}_{13} $	W 267.2347						
cquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N/	13C/31P Z3379/0400		Date	08 Jun 2012 13:39:12
ate Stamp	08 Jun 2012 13:39:12	2	,				
e Name	C:\Users\Fedor\Desk	top\C13 Рома Для Статы	в JOC 25.05.12\rudn-	040612-15a-c13dec\rudn-0	040612-15a-c13dec_0	01000fid	
equency (MHz)	100.62	Nucleus	13C	Number of Transients	427	Origin	spect
riginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
eceiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.7930
veep Width (Hz)	29409.97	Temperature (degree C) 27.000				
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				Chemical Shift (ppm)			

Formula C12H13NO6	FW 267.2347						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	08 Jun 2012 13:45:36
Date Stamp	08 Jun 2012 13:45:3	36					
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Статьи	в JOC 25.05.12\ru	dn-040612-15a-dept135\rudn-	040612-15a-dept1	35 001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	322	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9104.3838
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				

Compound 15a



125

09.06.2012 20:07:06

OH

0 104

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 20:38:47

0.5

Formula C13H15NO6	FW 281.2613						
Acquisition Time (sec,) 1.5729	Comment	5 mm QNP 1H/15	5N/13C/31P Z3379/0400		Date	08 Jun 2012 15:17:20
Date Stamp	08 Jun 2012 15:17	:20					
File Name	C:\Users\Fedor\De	sktop\C13 Рома Для Стат	ы в JOC 25.05.12\r	udn-040612-15b\rudn-0406	12-15b_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	20	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Temperature (degree	C) 32 000						



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 20:36:29

Formula C ₁₃ H ₁₅ NO ₆	FW 281.2613						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/	15N/13C/31P Z3379/0400		Date	08 Jun 2012 15:17:20
Date Stamp	08 Jun 2012 15:17:	20					
File Name	C:\Users\Fedor\De	sktop\C13 Рома Для Стати	и в ЈОС 25.05.12	Vrudn-040612-15b\rudn-0406	12-15b_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	20	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Tama antina (damas (21 22 000						

Temperature (degree C) 32.000





rudn-040612-15b 001000fid



09.06.2012 20:36:45

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S, 3H

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H₃C

Formula C ₁₃ H ₁₅ NO ₆	<i>FW</i> 281.2613						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	08 Jun 2012 15:17:20
Date Stamp	08 Jun 2012 15:17:	:20	'				
File Name	C:\Users\Fedor\De	sktop\C13 Рома Для Стати	ы в JOC 25.05.12\	rudn-040612-15b\rudn-0406	12-15b_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	20	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03

Temperature (degree C) 32.000

Compound 15b





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.06.2012 20:44:44

Formula C ₁₃ H ₁₅ NO ₆	W 281.2613						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	08 Jun 2012 16:34:08
Date Stamp	08 Jun 2012 16:34:0	08					
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Стать	и в ЈОС 25.05.12	rudn-040612-15b-c13dec\rudn-	040612-15b-c13de	ec_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	227	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10556.1514
Sweep Width (Hz)	29409.97	Temperature (degree	C) 27.000				

Compound 15b



rudn-040612-15b-c13dec_001000fid



09.06.2012 20:45:01

$\frac{1}{16} \operatorname{Ramp} \qquad 0 \text{ Jun 2012 163.08} \\ \frac{1}{16} \operatorname{Ramp} \qquad C.UsersPiedor/Deektop(C)13 Powe Jun Cramue a JOC 25 05 12 rudn-040612-15b-c13dec/00100/rld $	equisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N/	13C/31P Z3379/0400		Date	08 Jun 2012 16:34:08	
e Name C.Ubers/Fedor/Desktop/C13 Powa J.nc Cramue e JOC 25.05.12/udn-040612-15b-c13dec/	te Stamp	08 Jun 2012 16:34:	08	*					
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	Name	C:\Users\Fedor\Des	sktop\C13 Рома Для Стать	и в JOC 25.05.12\rudn-	040612-15b-c13dec\rudn-	040612-15b-c13dec (001000fid		
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	quency (MHz)	100.62	Nucleus	13C	Number of Transients	227	Origin	spect	
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	ginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	ząpą	
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	eiver Gain	32768.00	SW(cvclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10556.1514	
Compound 15b $G_{0} = 10^{10}$ $G_{10} = 10^{10}$	eep Width (Hz)	29409.97	Temperature (degree (27.000					
$\begin{array}{cccccccccccccccccccccccccccccccccccc$					Com	pound 15b	H ₃ C.	$\begin{array}{c} O \\ 19 \\ 18 \\ 20 \\ 0 \\ 17 \\ 2 \\ 2 \\ 2 \\ 3a \\ 4 \\ 4 \\ 4 \\ 4 \\ 4 \\ 4 \\ 4 \\ 4 \\ 4 \\ $	
940612-15b-c13dec_001000fid 9 9 9 9 9 9 9 9 9 9 9 9 9				8	3,39			$\begin{array}{c} 0 \\ 1a \\ 9c \\ 9c \\ 9aa \\ 9aa \\ 0 \\ 9aa \\ 0 \\ 8 \end{array}$	Μ
	040612-15b-c13dec_	_001000fid		19,90	- ~			ġ.	12 67
	ga			\sim	\sim			Z	
56.58 ~~~~ 66.61			8				2		
36.58 			66.61		13 -50.41	5	>	-24.87	
35.58 	1				51.68	80 C			
	35.58					<u>.94/</u> 39.75 10 39.33			а ,
	al a construction of the	M. San and M. Stranger, Starley, and Stranger and Stranger	na benerated on a tark and	harditelebrachan saken kallena jaran kana k		30	oo ay Maaraa - Alia, bala ahaa ahaa ahaa ahaa ahaa ahaa ahaa	from allow mark both (Jacob) (Jacob) and a second second Mr	n. malanta (n

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

130

09.06.2012 20:43:25

Formula C ₁₃ H ₁₅ NO ₆	FW 281.2613						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	(15N/13C/31P Z3379/0400		Date	08 Jun 2012 16:40:32
Date Stamp	08 Jun 2012 16:40:3	2	,				
File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Стат	ы в ЈОС 25.05.12	rudn-040612-15b-dept135\rudn-	040612-15b-dept1	35_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	129	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9105.7412
Sweep Width (Hz)	29409.97	Temperature (degree	C) 27.000				



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

07.07.2011 18:45:45

OH

0

0

Formula C ₁₂ H ₁₁ NO ₄	FW 233.2200						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15N	1/13C/31P Z3379/0400		Date	06 Jul 2011 16:25:36
Date Stamp	06 Jul 2011 16:25:3	36					
File Name	C:\Users\Fedor\Des	sktop\19.05.2011 С 13 Для I	Иры Статья в Тетра	аздрон\rudn-0611-N38\rudi	n-0611-N38_001000f	id	
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	16	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	256.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542
Sweep Width (Hz)	10416.03	Temperature (degree C)	32.000				

Compound 16



Chemical Shift (ppm)

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C12H11NO4	FW 233.2200						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H	H/15N/13C/31P Z3379/0400		Date	06 Jul 2011 16:25:36
Date Stamp	06 Jul 2011 16:25:	36					
File Name	C:\Users\Fedor\De	sktop\19.05.2011 С 13 Для	я Иры Статья в	Тетраэдрон\rudn-0611-N38\rud	n-0611-N38_001	000fid	
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	16	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	256.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542
Sweep Width (Hz)	10416.03	Temperature (degree (32,000				





1.00

5.8

5.7

5.9

6.0

rudn-0611-N38 001000fid



Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

6.7

6.6

6.5

6.4

6.3

6.2

6.1

07.07.2011 18:46:15

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

07.07.2011 18:46:45

OH

0.

Formula C ₁₂ H ₁₁ NO ₄	FW 233.2200						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	06 Jul 2011 16:25:36
Date Stamp	06 Jul 2011 16:25:	36					
File Name	C:\Users\Fedor\De	sktop\19.05.2011 C 13 Для	я Иры Статья в Те	етраэдрон\rudn-0611-N38\rud	n-0611-N38_001	000fid	
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	16	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	256.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542
Sween Width (Hz)	10416.03	Temperature (degree (32 000				

Compound 16

H-2.eq ddd (1.8; 3.8; 11.4)







Formula C12H11NO4

FW

233.2200

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.04.2011 17:37:41

-OH 17

0

0

39.53

0

Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N/13C/31P Z3379/0400			Date	14 Apr 2011 06:24:00
Date Stamp	14 Apr 2011 0	6:24:00		File Name	D:\NMR\13.04.20	11 C-13\rudn-130411-N6-0	13dec\rudp_130411_N6_c13dec_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	671	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	Speci
Receiver Gain	32768.00	SW(cvclical) (Hz)	29411 77	Solvent	DMSO d6	Pulse Sequence	2gpg
Sweep Width (Hz)	29409 97	Temperature (degree C) 32 000		content	DIVISO-00	Spectrum Onset (H2)	10548.9658

Compound 16

rudn-130411-N6-c13dec_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.04.2011 17:36:39

Formula C ₁₂ H ₁₁ NO ₄	FW 233.2200						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	H/15N/13C/31P Z3379/0400		Date	14 Apr 2011 06:38:56
Date Stamp	14 Apr 2011 06:38	:56		File Name	D:\NMR\13.04.201	1 C-13\rudn-130411-N6-d	lept135\rudn-130411-N6-dept135_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	448	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9098.5479
Sween Width (Hz)	29409 97	Temperature (degree)	C1 32 000	a second a shaper.			







General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

29.05.2012 9:59:05

Formula C13H15NO5	<i>FW</i> 265.2619						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	28 May 2012 16:36:16
Date Stamp	28 May 2012 16:36	:16					
File Name	C:\Users\Fedor\De	sktop\C13 Рома Для Стать	ы в JOC 25.05.12\ru	udn-250212-17a\rudn-2502	12-17a_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	6	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Temperature (degree 0	32.000						



3.57



rudn-250212-17a_001000fid

17a



29.05.2012 9:59:32

Formula C ₁₃ H ₁₅ NO ₅	205.2019						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	28 May 2012 16:36:16
Date Stamp	28 May 2012 16:36	5:16					
File Name	C:\Users\Fedor\De	esktop\C13 Рома Для Стате	ы в ЈОС 25.05.12	rudn-250212-17a\rudn-2502	12-17a_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	6	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
T	00000						

Temperature (degree C) 32.000

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rudn-250212-17a_001000fid





General Synthetic Approach towards Annelated 3a,6-Epoxylsoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

29.05.2012 9:59:59

Formula C ₁₃ H ₁₅ NO ₅	<i>FW</i> 265.2619						1. ¹
Acquisition Time (sec,	1.5729	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	28 May 2012 16:36:16
Date Stamp	28 May 2012 16:36	:16					
File Name	C:\Users\Fedor\De	sktop\C13 Рома Для Стат	ы в ЈОС 25.05.12	rudn-250212-17a\rudn-2502	212-17a_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	6	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
				the second s			

Compound 17a

Temperature (degree C) 32.000

S, 3H









General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

29.05.2012 9:41:57

ate Stamp le Name	28 May 2012 16:36	10						
le Name		. 16						
oquoney (MHz)	C:\Users\Fedor\Des	sktop\C13 Рома Для Статі	ыя в JOC 25.05.12\гі	udn-250512-17a-c13dec\rudn-2	250512-17a-c13dec	_001000fid		
equency (MITZ)	100.62	Nucleus	13C	Number of Transients	222	Origin	spect	_
riginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg	
eceiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10548.9570	
veep Width (Hz)	29409.97	Temperature (degree	C) 27.000					
				Co	mpound	17a	17×16^{-18} O 17×16^{-18} O 10^{-10} N 100^{-15} N 100^{-10} N	
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in-250512-17a-c13dec_	001000fid	10				6.33		
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ana manananan mananananan mananan karakan	the mean of an antisenside day of spicing a	nandarharbarahahan kanan k	a haranazartari bezeren berezeta azilizita arabite	and the first of the second	ana molon, adolalak Prosperioant alperato)	une ministration addition and an addition and	an	North Section

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

29.05.2012 9:42:15

aquisitian Time (ass)	0 5571	Commont	5 mm OND 14/15N	120/210 72270/0400		Data	28 May 2012 16-36-16
to Stomp	0.0071	16	STITLONP TH/ ISN/	130/312 233/9/0400		Date	20 Way 2012 10.00.10
le Stamp	20 May 2012 10:36:	Iton/C12 Doug Dag Creer v	D IOC 25 05 12)	250512 17a a12dachada	250512 170 012400 (00100064	
	100 62	Мистон Рома для статьи	B JOC 25.05.12\ruan-	Aumber of Transients	200012-178-C1300C_0	Origin	spect
equency (WHZ)	100.62	Nucleus	13C	Number of Transients	16284	Origin Bulas Seguence	speci
Iginal Points Count	10304	Sil/(evolice)) (Hz)	20411 77	Points Count	DMSO de	Spectrum Officet (Uz)	10548 9570
eceiver Gain	32768.00	Sw(cyclical) (Hz)	29411.77	Solvent	DIVISO-06	Spectrum Onset (HZ)	10546.9570
veep width (Hz)	29409.97	Temperature (degree C)	27.000		Compound	17a	
	8				f, Come		$\begin{array}{c} 17 \\ 17 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\$
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1-250512-17a-c13dec	001000fid				33		
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and a substance of the second s	and a set in the start of a start of the set	Control American and Month and Million of Million of American Sciences and Ameri	and the second	an and have been been able to the sea of the main start offert	a leade of a second residence of the fight of	A Add Angelia that we the sound and the line of a sound of the sound o	ى مىڭ قۇلىرلىلىغۇ ئۇ مۇللىلىغۇلىيە تەتلىغۇ ھەرىپىغىغى قەرقىيە تەپ ئەسلىك تەرەپ ² مەت مەتمەت.
20 25	80	75 70	65	60 55	50	45 40	35 30 24
00	50	10	00	Chemical Shift (nnm)	00	40	55 55 25

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

29.05.2012 9:48:36

CH₃

Formula C ₁₃ H ₁₅ NO ₅	FW 265.2619						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	28 May 2012 16:40:32
Date Stamp	28 May 2012 16:40:	32					
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Статьи	в ЈОС 25.05.12\	rudn-250512-17a-dept135\rudn-2	250512-17a-dept1	135_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	203	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9098.5479
Sweep Width (Hz)	29409 97	Temperature (degree C	27 000				



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5 Dec 2007


General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

5 Dec 2007



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

5 Dec 2007



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

23 Jan 2009



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

23 Jan 2009



Formula C13H14BrNO5 FW

344.1580

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.04.2010 12:18:44

CH₃

Acquisition Time (sec)	1.4549	Comment	single_pulse	Date 09 Apr 2010 10:36:13		Date Stamp	09 Apr 2010 10:35:01		
File Name	D:\NMR\6.04	4.10\FZ1128-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	8
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single pulse ex2
Receiver Gain	26.00	Solvent	CHLOROFOR	HLOROFORM-d		Spectrum Offset (Hz)	3014 4822	Sween Width (Hz)	11261.26
Temperature (degree C	21,200					opeca and enset (n2)	0011.4022	Sincep main (112)	11201.20



Chemical Shift (ppm)

Formula C. H. BrNO EW

344 1580

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.04.2010 12:19:13

COME S

CH₂

requisition Time (Sec)	1.4549	Comment	single_pulse	Date 09 Apr 2010 10:36:13				Date Stamp	09 Apr 2010 10:35:01
File Name	D:\NMR\6.04	4.10\FZ1128-1.jdf		Frequency (MHz)	Frequency (MHz) 600.17 Nucleus 1H				
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single pulse.ex2
Receiver Gain	26.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	3014.4822	Sweep Width (Hz)	11261.26

Compound 17c



Formula C., H., BrNO, FW

344,1580

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.04.2010 12:19:25

CH3

Acquisition Time (sec)	1 4549	Comment	single pulse	Date	09 Apr 201	0 10:36:13		Date Stamp	09 Apr 2010 10:35:01
File Name	D:\NMR\6.0	4.10\FZ1128-1.jdf		Frequency (MHz)	600.17 <i>Nucleus</i> 1H		Number of Transients	8	
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	26.00	Solvent	CHLOROFOR	RM-d		Spectrum Offset (Hz)	3014.4822	Sweep Width (Hz)	11261.26
Temperature (degree C)	21.200								

Compound 17c





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

19.04.2010 13:02:22



17d

Office

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.06.2012 15:59:27

Formula C13H14INO5	FW	391.1584
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H-9,10 d d

(2, 2)

Acquisition Time (sec)	2.1837	Comment	single_pulse	Date	Date 06 Jun 2012 09:08:00			Date Stamp	06 Jun 2012 12:57:17
File Name	D:\NMR\04.06	5.12\FZ2443-1.jdf	,	Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	32
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	36.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	7503.00
Temperature (degree C) 22.900								





4.0

3.5

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.06.2012 16:01:56

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$\begin{array}{c c c c c c c c c c c c c c c c c c c $	equisition Time (sec)	2.1837	Comment	single_pulse	Date	06 Jun 2012	09:08:00		Date Stamp	06 Jun 2012 12:57:17
$\frac{1}{12} \underbrace{\text{ECS 400}}_{\text{perature (degree C) 22.900}} Original Points Count 1334 Points Count 134 Poin$	le Name	D:\NMR\04.06	5.12\FZ2443-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	32
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	rigin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
$\begin{array}{c} \begin{array}{c} & & & \\ & & $	ceiver Gain	36.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	7503.00
$\begin{array}{c} \begin{array}{c} \begin{array}{c} \begin{array}{c} G, G, Me, 3H \end{array}{)} \\ \begin{array}{c} H-4 ax \\ dt (3.7; 12.8) \end{array} \end{array} \end{array} \\ \begin{array}{c} \begin{array}{c} H-4 ax \\ dt (3.7; 12.8) \end{array} \end{array} \\ \begin{array}{c} \begin{array}{c} H-7 \\ H-6 \\ dd \end{array} \end{array} \\ \begin{array}{c} H-7 \\ H-6 \\ dd \end{array} \\ \begin{array}{c} H-6 \\ H-3 \\ H$	perature (degree C)	22.900	1						O 17 16 19 O	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		S,	OgMe, 3H		H-40 dt	xx (3,7;1	(8,8)		$ \begin{array}{c} I_{15} \\ 8 \\ 9 \\ 9 \\ 10 \\ H_{10} \\ H_{10} \\ 0 \\ H_{10} \\ H_{10} \\ 0 \\ H_{10} \\ M_{10} \\ 0 \\ H_{10} \\ M_{10} \\ M_{10} \\ M_{10} \\ M_{10} \\ M_{10} \\ $	-4
$\begin{array}{c} H - 4 \\ dd \\ H - 6a \\ dd \\ H - 3ax \\ H $	I3-1.jdf	3.79		Ţ.				Cor	npound 17d	
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		MM	Λ.		M'M	M				- <u>6</u>

Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

2.5

2.0

3.0

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.06.2012 15:39:55

are stamp 26 Jun 2012 17:14:40 ile Name C:\Users\Fedor\Deskto requency (MHz) 100.62 triginal Points Count 16384 eceiver Gain 32768.00 weep Width (Hz) 29409.97 Jn-140612-17d-c13dec_001000fid 87 G 6 S 6 S 6 S 6 S 6	ор\C13 Рома Для Стал Nucleus Owner SW(cyclical) (Hz) Temperature (degree	тьи в JOC 25.05.12\ru 13C root 29411.77 e C) 27.000	dn-140612-17d-c13dec\rudn-1 Number of Transients Points Count Solvent	40612-17d-c13dec_00 218 16384 CHLOROFORM-d	1000fid Origin Pulse Sequence Spectrum Offset (Hz)	spect zgpg
Image: C:\Users\FedorLesktoreguency (MHz) 100.62 riginal Points Count 16384 eceiver Gain 32768.00 weep Width (Hz) 29409.97 Im-140612-17d-c13dec_001000fid 87 Im-140612-17d-c13dec_001000fid	ор\С13 Рома Для Ста Nucleus Owner SW(cyclical) (Hz) Temperature (degree	ты в JOC 25.05.12\ru 13C root 29411.77 e C) 27.000	dn-140612-17d-c13dec\rudn-1 Number of Transients Points Count Solvent	40612-1/d-c13dec_00 218 16384 CHLOROFORM-d	Origin Pulse Sequence Spectrum Offset (Hz)	spect Zgpg
requency (MHz) 100.62 riginal Points Count 16384 eceiver Gain 32768.00 weep Width (Hz) 29409.97 Jon-140612-17d-c13dec_001000fid 000000000000000000000000000000000000	Nucleus Owner SW(cyclical) (Hz) Temperature (degree	13C root 29411.77 e C) 27.000	Number of Transients Points Count Solvent	218 16384 CHLOROFORM-d	Origin Pulse Sequence Spectrum Offset (Hz)	spect zgpg 11074 4607
In-140612-17d-c13dec_001000fid Image: Contemporal contempora contemporal	Owner SW(cyclical) (Hz) Temperature (degree	29411.77 e C) 27.000	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2gpg
Image: Weep Width (Hz) 29409.97 Image: Ima	SW(cyclical) (Hz) Temperature (degree	29411.77 e C) 27.000	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	7 7 () / A ALCO /
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Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.06.2012 15:34:20

autisition Time (see)	0 5571	Commont	5 mm OND 14/15	N/13C/31D 73370/0400		Data	26 Jun 2012 17:18:56
to Stamp	26 Jun 2012 17:18:6	Comment	STILL QINE 11/15	11/13C/31F 23579/0400		Date	20 301 2012 17:10:00
lo Namo	C:\Llsers\Fedor\Dos	kton)C13 Pous Ing Crath	P IOC 25 05 12\rude	140612 17d dopt135\rudp 1	40612 17d dopt135 0	01000fid	
	100 62	Миссис	1300 25.05.121100	Number of Transients	40012-170-dep(155_0	Origin	spect
riginal Doints Count	16394	Owner	root	Reinte Count	100	Dulas Seguence	dopt135
riginal Points Count	10304	Owner	100t	Points Count		Puise Sequence	0624 0222
	32768.00	Sw(cyclical) (Hz)	29411.77	Solvent	CHLOROFORINI-a	Spectrum Onset (Hz)	9024.0332
reep wiath (Hz)	29409.97	Temperature (degree 0) 27.000				
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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.05.2012 13:04:25

CH3

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Formula C14H17NO5 FW 279.2885

Acquisition Time (sec)	2.1837	Comment	single_pulse	Date 05 May 2012 09:16:46				Date Stamp	05 May 2012 13:06:05
File Name	D:\NMR\03.05	5.12\FZ2374-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	4
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	26.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	1998.9109	Sweep Width (Hz)	7503.00
Temperature (degree C)	23.300							and a second	



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.05.2012 13:05:03

Formula C14H17NO5	FW 279.	2885							
Acquisition Time (sec	2.1837	Comment	single_pulse	Date	05 May 201	12 09:16:46		Date Stamp	05 May 2012 13:06:05
File Name	D:\NMR\03.0	05.12\FZ2374-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	4
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	26.00	Solvent	CHLOROFO	RM-d		Spectrum Offset (Hz)	1998.9109	Sweep Width (Hz)	7503.00
Temperature (degree	C) 23.300								
					Со	mpound 17e		O 17 16 0 18 0 /15	



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.05.2012 13:04:48

CH3

Formula C ₁₄ H ₁₇ NO ₅	-77 279.	2003							
Acquisition Time (sec)	2.1837	Comment	single_pulse	Date	05 May 201	12 09:16:46		Date Stamp	05 May 2012 13:06:05
File Name	D:\NMR\03.0	05.12\FZ2374-1.jdf		Frequency (MHz) 399.78 Nucleus 1H				Number of Transients	4
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	26.00	Solvent	CHLOROFOR	RM-d		Spectrum Offset (Hz)	1998.9109	Sweep Width (Hz)	7503.00
Temperature (degree C	23.300								



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

23 Jan 2009



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

23 Jan 2009



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C ₁₃ H ₁₅ Br ₂ NO ₅		FW 425.0699					
Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H/15M	V/13C/31P Z3379/0400		Date	31 May 2012 15:49:20
Date Stamp	31 May 2012 15:49	9:20	'				
File Name	C:\Users\Fedor\De	sktop\C13 Рома Для Стать	и в ЈОС 25.05.12\ruc	in-250512-18\rudn-250512	-18_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	4	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2602.0486	Sweep Width (Hz)	10203.46
-							

Compound 18

3.69

Temperature (degree C) 27.000



01.06.2012 14:32:49

rudn-250512-18_001000fid

18



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C13H15Br2NO5		FW 425.0699					
Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H/15M	V/13C/31P Z3379/0400		Date	31 May 2012 15:49:20
Date Stamp	31 May 2012 1	5:49:20	· ·				
File Name	C:\Users\Fedor	NDesktop/C13 Рома Для Стать	и в ЈОС 25.05.12\гис	dn-250512-18\rudn-250512	2-18_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	4	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2602.0486	Sweep Width (Hz)	10203.46
Temperature (degree C	27 000						

01.06.2012 14:32:56

CH₃



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

01.06.2012 14:33:05

Formula C13H15Br2NO5		FW 425.0699					
Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H/15N	I/13C/31P Z3379/0400		Date	31 May 2012 15:49:20
Date Stamp	31 May 2012 15	:49:20	· ·				
File Name	C:\Users\Fedor\	Desktop\C13 Рома Для Стать	и в ЈОС 25.05.12\гис	in-250512-18\rudn-250512	-18_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	4	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2602.0486	Sweep Width (Hz)	10203.46
Temperature (degree C)	27.000						

Compound 18



rudn-250512-18_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

01.06.2012 14:47:56

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Formula $C_{13}H_{15}Br_2NO_5$		FW	425.0699					
Acquisition Time (sec)	0.5571	Comment	5 mm QN	P 1H/15N/1	3C/31P Z3379/0400		Date	31 May 2012 15:51:28
Date Stamp	31 May 2012 15:51:28	3	×.					
File Name	C:\Users\Fedor\Deskt	ор\С13 Рома Д	Іля Статьи в JOC 25.	05.12\rudn-2	50512-18-c13dec\rudn-25	0512-18-c13dec_0010	00fid	
Frequency (MHz)	100.62	Nucleus	13C		Number of Transients	337	Origin	spect
Original Points Count	16384	Owner	root		Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical)	(Hz) 29411.77		Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9631.5508
Sweep Width (Hz)	29409.97	Temperature	(degree C) 27.000					
								CH ₃

Compound 18



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C13H15Br2NO5		FW 425.06	699				
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/	/15N/13C/31P Z3379/0400		Date	31 May 2012 15:51:28
Date Stamp	31 May 2012 15:51:	28	,				
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Ст	гатьи в JOC 25.05.12\r	rudn-250512-18-c13dec\rudn-25	0512-18-c13dec_0010	000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	337	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9631.5508
Sweep Width (Hz)	29409 97	Temperature (deg	ree C) 27 000				

01.06.2012 14:48:15



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C13H15Br2NO5 FW 425.0699 Acquisition Time (sec) 0.5571 Comment 5 mm QNP 1H/15N/13C/31P Z3379/0400 Date 31 May 2012 15:57:52 31 May 2012 15:57:52 Date Stamp C:\Users\Fedor\Desktop\C13 Рома Для Статьи в JOC 25.05.12\rudn-250512-18-dept135\rudn-250512-18-dept135_001000fid File Name 100.62 Frequency (MHz) Nucleus 13C Number of Transients 252 Origin spect **Original Points Count** 16384 Owner Points Count **Pulse Sequence** dept135 root 16384 32768.00 **Receiver Gain** SW(cyclical) (Hz) 29411.77 Solvent CHLOROFORM-d Spectrum Offset (Hz) 9631.5508 29409.97 Sweep Width (Hz) Temperature (degree C) 27.000



Compound 18



Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

01.06.2012 14:46:46

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.05.2012 17:03:37

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Acquisition Time (sec)	1.8193	Comment	single_pulse	Date	27 Apr 2009	9 12:30:02		Date Stamp	27 Apr 2009 12:27:30
File Name	D:\NMR\27.04	1.09\fz647-2.jdf	-	Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	2
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	32.00	Solvent	CHLOROFO	RM-d		Spectrum Offset (Hz)	3000.8616	Sweep Width (Hz)	9005.76
Temperature (degree C) 22.400								
								CH ₃	





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.05.2012 17:04:04

Acquisition Time (sec)	1.8193	Comment	single_pulse	Date	27 Apr 200	9 12:30:02		Date Stamp	27 Apr 2009 12:27:30
File Name	D:\NMR\27.0	4.09\fz647-2.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	2
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	32.00	Solvent	CHLOROFOR	RM-d		Spectrum Offset (Hz)	3000.8616	Sweep Width (Hz)	9005.76
Temperature (degree C	22.400								
								CH3	
								0 0	
					6	1		0 22 21 25 0	
1	1. 0. 4				Com	oound 19		$O_{22} > 21 - O_{25} O_{120} $	



fz647-2.jdf











General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.05.2012 17:04:14

cquisition Time (sec)	1.8193	Comment	single_pulse	Date	27 Apr 2009	12:30:02		Date Stamp	27 Apr 2009 12:27:30
ile Name	D:\NMR\27.0	4.09\fz647-2.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	2
rigin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
ceiver Gain	32.00	Solvent	CHLOROFO	RM-d		Spectrum Offset (Hz)	3000.8616	Sweep Width (Hz)	9005.76
nperature (degree C)	22.400							CH ₃	
		CozMe, s, 3H			Com	pound 19		$\begin{array}{c} 0 \\ 22 \\ 22 \\ 21 \\ 22 \\ 21 \\ 20 \\ 20 \\ $	CH ₃ 7 \\ 0 19
7-2.jdf	3.73							OAC	OAC 34
4-3					H-4	H-Ja		3H, 5	2 , 51
+		H-S'A	H-	1B 1+	d (3.1)	d (9.1)			H
6.9)		~ at	~ 0	01/42					2
		(7,2;14,2)	(4	(2, 19, 2)					Ŷ
									1
06 4.05 04 4.05		-			2.88	2.74			
		3.56 3.55 3.57 3.51	3.32 3.30 3.29 3.28	3.27		J.			1.93 1.92 1.92 1.92 1.92 1.90 1.89
2.05	2.98	1.02	1.02		0.97	0.98		2.95	3.01 2.12

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

12 May 2009



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

12 May 2009



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.05.2012 13:21:43

Formula C16H13NO5	FW 299.278	31							
Acquisition Time (sec)	2.1845	Date	02 Feb 2009	06:56:00		Date Stamp	02 Feb 2009	06:56:00	
File Name	D:\NMR\C_13\	Кеня и Инга (ІОС коне	4 2008)\6nik (DM	SO)\6nik (DMSO)_002000fid		Frequency (MHz)	600.22	Nucleus	1H
Number of Transients	4	Origin	spect	Original Points Count	32768	Owner	root	Points Count	32768
Pulse Sequence	zg	Receiver Gain	128.00	SW(cyclical) (Hz)	15000.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	3144.6580
Sweep Width (Hz)	14999.54	Temperature (degree	e C) 22.500						

Compound 20Aa



3.39



20Aa



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.05.2012 13:22:11

Acquisition Time (sec)	2.1845	Date	02 Feb 2009 06	:56:00		Date Stamp	02 Feb 2009	06:56:00	
ile Name	D:\NMR\C_13	Женя и Инга (ІОС конец 2	008)\6nik (DMSO)\6nik (DMSO)_002000fid		Frequency (MHz)	600.22	Nucleus	1H
lumber of Transients	4	Origin	spect	Original Points Count	32768	Owner	root	Points Count	32768
Pulse Sequence	zg	Receiver Gain	128.00	SW(cyclical) (Hz)	15000.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	3144.6580
weep Width (Hz)	14999.54	Temperature (degree () 22.500						
ik (DMSO)_002000fid H-L Jd (7.5; 1.0)			(F,S	H-4 bzd (7, 1 H-2 dt ;1.0)	Co s) H	$\frac{1-3}{1+2}$ $\frac{1-3}{1+2}$ $\frac{1}{7}$ $\frac{1}{7}$ $\frac{1}{7}$ $\frac{1}{7}$ $\frac{1}{7}$ $\frac{1}{7}$)Aa	$\begin{array}{c} & 4a & 0 \\ & & 6aa \\ 1 & & 12a \\ & & 6ba \\ & & & & 6ba \\ & & & & 6ba \\ & & & & & 6ba \\ & & & & & & & & & & \\ & & & & & & & $	H-(\$
									5.95
8.34				7.28 7.19 7.19 7.11	7.09 7.10	6.68	6.53		
0.98				3.12		1.00	1.02		1.00

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

3 Feb 2009



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

25.05.2012 21:37:41

Acquisition Time (sec)	2.1837	Comment	single pulse	Date	22 May 2012	09:25:03		Date Stamp	22 May 2012 13:14:16
File Name	D:\NMR\21.0	05.12\FZ2408-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	4
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	20.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	1984.6338	Sweep Width (Hz)	7503.00	Temperature (degree C	24.500
				Compo	ounds 2	0Ab/20Bb	2 1 2 1 2 1 2 1 2 1 2 1 2 1 2 1 2 1 2 1	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	$\begin{array}{c} 12a & \begin{array}{c} 6 & N \\ 6a & 6ab \\ 12 & 0 \\ 11 & 10a0 \\ 11 & 10a0 \\ 10 & 9 \\ 42 \\ HO \\ 44 \\ 46 \end{array}$
Ab/20Bb = 3	57/43							22 02 22 05 22	45
								8 (DIM 20	
								1.57	1.55
							420		
							¥		
62. C							3.33		
				19	.86	5.46		140.00	-
COOH				- <u>-</u>	5			MIEZ	
				5 5 68 68	34	21 5.09 88	16	33.07	
-12.38			8.44	7.69 7.61 7.26 7.26 7.07 6 6 6		5.24 5.06 4.95	<u>د</u>	-2.08	
						1.00 1.00	2		

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

25.05.2012 21:37:55

cquisition Time (sec)	2.1837	Comment	single_pulse	Date	22 May 2012	2 09:25:03		Date Stamp	22 May 2012 13:14:16
ile Name	D:\NMR\21.	05.12\FZ2408-1.jdf	0 _	Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	4
rigin	ECS 400	Original Points Cour	t 16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
eceiver Gain	20.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	1984.6338	Sweep Width (Hz)	7503.00	Temperature (degree C)	24.500
				Comp	ounds	20Ab/20Bb	= = = = = = = = =	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	$\begin{array}{c} & & & & & H \\ & & & & & 6a \\ & & & & & & 6a \\ & & & & & & & 6a \\ & & & & & & & & & & & \\ & & & & & & $
2408-1.jdf					L_	2-1.	Û.	0 22	45
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11 1			PC-	2				M-4	D.
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+ N				66		0		6.7	co co
8.4				7.6		28 26 7.0		1 11	
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0.91				0.99		5.42 1.05		1.00 0.98	1.06 0.99
						1.1.			the second se

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

25.05.2012 21:38:59

cquisition Time (sec)	2.1837	Comment	single_pulse	Date	22 May 2012	09:25:03		Date Stamp	22 May 2012 13:14:16
ile Name	D:\NMR\21.05	.12\FZ2408-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	4
rigin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
eceiver Gain	20.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	1984.6338	Sweep Width (Hz)	7503.00	Temperature (degree C)	24.500
				Comp	oounds	20Ab/20Bb	$ \begin{array}{c} 3 \\ 1 \\ 2 \\ 1 \\ 1 \\ 2 \\ 1 \\ 1 \\ 2 \\ 1 \\ 1$	$\begin{array}{c c} O & & & & & & \\ & & & & & & \\ & & & & &$	$\begin{array}{c} 4a & O \\ 12a & 6a & 6ab \\ 12a & 6a & 6ab \\ 12 & 6b & 7 \\ 11 & 10aO \\ 0 & 13 & 9 \\ 42 & 10 & 9 \\ 42 & HO & 44 \\ HO & 44 & 10 \\ \end{array}$
2408-1.jdf							57 12	43 0 22 43	46 O 45
							M-		
							d	d (151)	
							(151)	(12.2	
	H- 69			H-69		2	C.	1	
	2			3		d))
80 20 20				5.46		(12.1)			15.1)
						5.24	5.14	5.06 4.98	
1.0	0			1.03		1.13	2.18	8 1.03	
General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

25.05.2012 21:39:09



3 Feb 2009

Acquisition Time (sec)	0.9044	Comment	fraction 24	Date	02 Feb 2009 13:4	1:20	
File Name	D:\NMR\C_13\Жен	я и Инга (ІОС конец 2008))\7nik (DMSO)\nik1d6	600\nik1d600_131000fid		Frequency (MHz)	150.94
Nucleus	13C	Number of Transients	778	Original Points Count	32768	Points Count	32768
Pulse Sequence	zgpg30	Solvent	CHLOROFORM-D			Sweep Width (Hz)	36231.88
Temperature (degree C) 21.000						



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

3 Feb 2009

Acquisition Time (sec)	0.9044	Comment	fraction 24	Date	02 Feb 2009 13:41	:20		
File Name	D:\NMR\C_13\Xer	я и Инга (ІОС конец 2008)	\7nik (DMSO)\nik1de	600\nik1d600_131000fid		Frequency (MHz)	150.94	
Nucleus	13C	Number of Transients	778	Original Points Count	32768	Points Count	32768	
Pulse Sequence	zgpg30	Solvent	CHLOROFORM-D			Sweep Width (Hz)	36231.88	
Temperature (degree C) 21.000							

Compounds 20Ab/20Bb





3 Feb 2009

Acquisition Time (sec)	0.9044	Comment	fraction 24	Date	02 Feb 2009 13:41:20				
File Name	D:\NMR\C_13\XKens	я и Инга (ІОС конец 2008)	\7nik (DMSO)\nik1d6	00\nik1d600_131000fid		Frequency (MHz)	150.94	18. C	
Nucleus	13C	Number of Transients	778	Original Points Count	32768	Points Count	32768		
Pulse Sequence	zgpg30	Solvent	CHLOROFORM-D			Sweep Width (Hz)	36231.88		
Temperature (degree C)	21.000								





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3 Feb 2009



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

3 Feb 2009



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

3 Feb 2009



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.06.2012 16:22:52



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.06.2012 16:27:06



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

FW 378.1742 Formula C16H12BrNO5 Comment Acquisition Time (sec) 1.5729 5 mm QNP 1H/15N/13C/31P Z3379/0400 Date 08 Jun 2012 14:56:00 08 Jun 2012 14:56:00 Date Stamp C:\Users\Fedor\Desktop\C13 Рома Для Статьи в JOC 25.05.12\rudn-250512-20c_1\rudn-250512-20c_1_001000fid File Name Frequency (MHz) 400.14 Nucleus Number of Transients 48 Origin 1H spect **Original Points Count** 16384 Owner root **Points Count** 16384 **Pulse Sequence** zg **Receiver Gain** 128.00 SW(cyclical) (Hz) 10416.67 Solvent DMSO-d6 Spectrum Offset (Hz) 2712.0542 Sweep Width (Hz) 10416.03 Temperature (degree C) 32.000

Compounds 20Ac/20Bc Ó 19 Br HO-23 20 0 22 -3.31 -3.30 rudn-250512-20c_1_001000fid 8 H-5 me 34 3 ė 3.11 C3.08 mai ma 0 5.03 12 18:3 S 4.99 MIN 5.16 0 MIM 3.15 5.27 -5.23 5.08

Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

4.0

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May

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

03.07.2012 20:39:55

Br

Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	31 May 2012 16:10:40
Date Stamp	31 May 2012 16	:10:40	1				
File Name	C:\Users\Fedor\	Desktop\C13 Рома Для Стат	тыя в JOC 25.05.12\	rudn-250512-20c-c13dec\rudn-2	250512-20c-c13de	c_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	2369	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10548.9570
Sweep Width (Hz)	29409.97	Temperature (degree	C) 27.000				
							4 6



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

03.07.2012 20:33:49



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

01.06.2012 15:46:21

ormula C32H24Br2N2O10	,?	FW 756.3484+? (37	78.1742+378.1742+?+?				
cquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15	V/13C/31P Z3379/0400		Date	31 May 2012 16:10:40
ate Stamp	31 May 2012 16:	10:40	1			N	
ile Name	C:\Users\Fedor\I	Desktop\C13 Рома Для Стат	гыя в JOC 25.05.12\rud	n-250512-20c-c13dec\rudn-2	50512-20c-c13dec_0	001000fid	
requency (MHz)	100.62	Nucleus	13C	Number of Transients	2369	Origin	spect
riginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10548.9570
weep Width (Hz)	29409.97	Temperature (degree	e C) 27.000				
				Compounds	20Ac/20B	C $3 4 4a 5 0$ 2 1 2a 6 6 0 12 a 6 0	$\begin{array}{cccccccccccccccccccccccccccccccccccc$
		69				$HO_{47} = 21$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$
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		and the second second	dentry all and a	the second se			the discount of strengthe starts
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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

01.06.2012 15:30:10

Date Stamp						Duto	of may zonz foresizo
	31 May 2012 16:	53:20					
-ile Name	C:\Users\Fedor\E	Desktop\C13 Рома Для Статы	в JOC 25.05.12\ru	dn-250512-20c-dept135\rudn-2	50512-20c-dept135_00	01000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	380	Origin	spect
Driginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9098.5479
weep Width (Hz)	29409.97	Temperature (degree (27.000				
dn-250512-20c-dent135	001000fid				35		
		127.43 124.86 123.56	Compou	inds 20AC/20BC			53.57

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.



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9 Oct 2009



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

9 Oct 2009



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

23 Jan 2009



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

23 Jan 2009



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

23 Jan 2009



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23 Jan 2009



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.02.2012 18:41:13 Formula C.-H. NO. FW 313.3047 Acquisition Time (sec) 2.1837 Comment single pulse Date 28 Feb 2012 10:56:58 Date Stamp 28 Feb 2012 15:31:31 File Name D:\NMR\21.02.12\FZ2241-1.idf Frequency (MHz) 399.78 Nucleus 1H Number of Transients 1 Origin ECS 400 **Original Points Count** 16384 Owner delta Points Count 16384 Pulse Sequence single pulse.ex2 Receiver Gain 24.00 Solvent DMSO-d6 Spectrum Offset (Hz) 1998.9109 Sweep Width (Hz) 7503 00 Temperature (degree C) 24.300 0 Compounds 22Ab/22Bb 0 /19 21 20 before crystallization -OH 23 2a_ \cap CH₃ 13 5 22Ab/22Bb = 86/142.46 before crystallization M-10 H-48,5 23 maj may Å min min (16.9) d 69 9 16 10 35 (0.14) 6.80 6.39 2.61 N 7.19 2.93 3.96 1.37 r-3.35 3.33 86 37 C 5.97 92 81 N 33 23 6.72 6.02 5.99 4 . O 84 4.29 5 3 9.42 1.14 0.26 0.76 0.88 1.83 1.00 8 1.12 1.05 0.14 2.84 7.0 6.5 6.0 5.5 5.0 4.0 4.5 3.5 3.0 2.5 2.0 1.5 Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

0.2

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C.-H. NO. FW 313 3047 Acquisition Time (sec) 2.1837 Comment single pulse Date 06 Mar 2012 09:39:46 Date Stamp 06 Mar 2012 14:14:34 File Name D:\NMR\05.03.12\FZ2260.jdf Frequency (MHz) 399.78 Nucleus 1H Number of Transients 8 Origin ECS 400 **Original Points Count** 16384 Owner delta Points Count 16384 Pulse Sequence single pulse.ex2 Receiver Gain 18.00 Solvent CHLOROFORM-d Spectrum Offset (Hz) 1998 9109 Sweep Width (Hz) 7503.00 Temperature (degree C) 24.000 0 0 22 Compound 22Ab OH 23 after crystallization 12a_ •0 CH2 13 5 Ĥ 22Ab after crystallization 4ba 1.74 * CHCS 7.25 MezCO 59 * 2.16 S TMS 6.30 2.99 0.01 93 92 3.01 87 6.6 7.01 5.06 43 N 50 7.24 5.00 62 5.14 0.91 0.76 0.95 1.00 0.58 1.95 0.64 2.78 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladin P. Zaysev, (Wetter N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

10.03.2012 21:56:06

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.03.2012 21:56:17



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.03.2012 21:56:30

Formula	C.H.NO.	FW	313.3047
rormula	C., H., NO.	FVV	313.3047

Acquisition Time (sec)	2.1837	Comment	single_pulse	Date 06 Mar 2012 09:39:46				Date Stamp	06 Mar 2012 14:14:34
File Name	D:\NMR\05.03	.12\FZ2260.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	8
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	18.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	1998.9109	Sweep Width (Hz)	7503.00
and the state of the									

Temperature (degree C) 24.000





FZ2260.jdf



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.03.2012 14:48:04



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.03.2012 14:48:38



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16 Aug 2011

Acquisition Time (sec)	1.6056	Comment	Imported from UXNN	AR.		Date	10 Aug 2011 17:06:08
File Name	C:\Users\Fedd	or\Desktop\05.08.11\rudn-050811-	-N2\rudn-050811-N2-1	\rudn-050811-N2-1_00100	Ofid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	16	Original Points Count	16384	Points Count	16384
Pulse Sequence	zg	Solvent	CHLOROFORM-D	Sweep Width (Hz)	10204.08	Temperature (degree	C) 27.000

Compound 22Aa





209

16 Aug 2011



16 Aug 2011

Acquisition Time (sec)	1.6056	Comment	Imported from UXNN	IR.		Date	10 Aug 2011 17:06:08
File Name	C:\Users\Fedor\Des	ktop\05.08.11\rudn-050811-	N2\rudn-050811-N2-1	\rudn-050811-N2-1_00100	Ofid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	16	Original Points Count	16384	Points Count	16384
Pulse Sequence	zg	Solvent	CHLOROFORM-D	Sweep Width (Hz)	10204.08	Temperature (degree C	;) 27.000

Compound 22Aa





211

16 Aug 2011



16 Aug 2011



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212

16 Aug 2011

Acquisition Time (sec)	0.5571	Comment	Imported from UXNMF	۲.		Date	10 Aug 2011 17:36:00
File Name	C:\Users\Fedor\Deskto	op\05.08.11\rudn-050811-N	2\rudn-050811-N2-c13d	dec\rudn-050811-N2-c13de	c_001000fid	Frequency (MHz)	100.62
Nucleus	13C	Number of Transients	2500	Original Points Count	16384	Points Count	16384
Pulse Sequence	zgpg	Solvent	CHLOROFORM-D	Sweep Width (Hz)	29411.77	Temperature (degree	C) 27.000

Compound 22Aa





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16 Aug 2011



8 Sep 2011

Acquisition Time (sec)	1.6056	Comment	Imported from	1 UXNMR.		Date	07 Sep 20	11 14:00:32		
File Name	D:\NMR\02.09	.11\991\991_001000fid		Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	12	
Original Points Count	16384	Points Count	16384	Pulse Sequence	zg	Solvent	CHLORO	FORM-D		
Sweep Width (Hz)	10204.08	Temperature (degree C)	27.000							

Compound 23




8 Sep 2011 Acquisition Time (sec) 1.6056 Comment Imported from UXNMR. Date 07 Sep 2011 14:00:32 1H Number of Transients 12 File Name D:\NMR\02.09.11\991\991 001000fid Frequency (MHz) 400.14 Nucleus **Original Points Count** 16384 Points Count 16384 Pulse Sequence Solvent CHLOROFORM-D zg Sweep Width (Hz) 10204.08 Temperature (degree C) 27.000 7,00 0 6,9 U-R 19 H-6 Compound 23 (1,2;8,3) 12a_ 17.5, 0 5 H 4ba 7,09 47 4-40 10 4-3 (1,2;7,5;8,3) 2 to A 16 7.25 (7, 5)4-2 A 6.77 (16.8) 1,5;56 6.79 6.45 .02 7.00 6.98 4.97 5.01 6.43 18 5.19 6.96 . 2 2 7.09 7.19 10 17 21 0.96 0.95 0.93 0.93 0.92 1.00 1.05 1.95 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 6.1 6.0 5.9 5.8 5.7 5.6 5.5 5.4 5.3 5.2 5.1 5.0 4.9 Chemical Shift (ppm)

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8 Sep 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

8 Sep 2011



8 Sep 2011

cquisition Time (sec)	0.5571	Comment	Imported from U	IXNMR.		Date	07 Sep 2011 15	:28:00
e Name	D:\NMR\02.09	.11\991-dept135\991-dept13	5_001000fid	Frequency (MHz)	100.62	Nucleus	13C	
imber of Transients	403	Original Points Count	16384	Points Count	16384	Pulse Sequence	dept135	
Ivent	CHLOROFOR	M-D		Sweep Width (Hz)	29411.77	Temperature (degre	ee C) 27.000	
136.12	131.84	122.26		83.31	Compo	ound 23	45.78	
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Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

25.07.2011 16:08:07

Η

Formula C7H9NOS	FW 155.2175						
Acquisition Time (sec)	1.6056	Comment	5 mm QNP 11	H/15N/13C/31P Z3379/0400		Date	21 Jul 2011 15:51:28
Date Stamp	21 Jul 2011 15:5	1:28		File Name	D:\NMR\19.07.11	(Рома)\rudn-190711-N	3\rudn-190711-N3_002000fid
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	12	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	512.00	SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-	ł	
Spectrum Offset (Hz)	2602.0486	Sweep Width (Hz)	10203.46	Temperature (degree C)	27.000		

Initial for compound 24a



25.07.2011 16:08:17 155.2175 5 mm QNP 1H/15N/13C/31P Z3379/0400 Comment Date 21 Jul 2011 15:51:28 21 Jul 2011 15:51:28 File Name D:\NMR\19.07.11 (Poma)\rudn-190711-N3\rudn-190711-N3_002000fid Number of Transients Nucleus 1H 12 Origin spect Pulse Sequence Owner root Points Count 16384 zg SW(cyclical) (Hz) 10204.08 Solvent CHLOROFORM-d Sweep Width (Hz) 10203.46 Temperature (degree C) 27.000



221

Formula C7HaNOS

Frequency (MHz)

Receiver Gain

Original Points Count

Date Stamp

Acquisition Time (sec) 1.6056

FW

400.14

16384

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25.07.2011 16:12:49

	V 155.2175	and the second sec					
Acquisition Time (sec)	0.5898	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	21 Jul 2011 16:08:32
Date Stamp	21 Jul 2011 16:08:3	2		File Name	D:\NMR\19.07.11 (Рома)\rudn-190711-N3-	-c13dec\rudn-190711-N3-c13dec_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	203	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	27777.78	Solvent	CHLOROFORM-d		
Spectrum Offset (Hz)	9613.9424	Sweep Width (Hz)	27776.08	Temperature (degree C)	27.000		



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

25.07.2011 16:11:51

Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	21 Jul 2011	16:12:48
Date Stamp	21 Jul 2011 16:12	2:48		File Name	D:\NMR\19.07.11 ((Рома)\rudn-190711-	N3-dept135\rudn-190	0711-N3-dept135_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	209	Origin	spect	
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135	
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	1		
Spectrum Offset (Hz)	9614.0205	Sweep Width (Hz)	29409.97	Temperature (degree (C) 27.000			
udn-190711-N3-dept135_	_001000fid			110.11		65,26	Initial for	compound 24a
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13.06.2012 17:49:33

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Acquisition Time (sec)	2.1837	Comment	single_pulse	Date	06 Jun 2012	11:54:17		Date Stamp	06 Jun 2012 15:43:34
File Name	D:\NMR\04.0	06.12\FZ2444-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	9
Drigin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	36.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	2413.7561	Sweep Width (Hz)	7503.00	Temperature (degree C) 22.900
						$\begin{array}{c} OH \\ O \\ 0 \\ 0 \\ 10 \\ 9a \\ 9b \\ 9b \\ 9b \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ $	naj	$ \begin{array}{c} 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\$	min
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Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.06.2012 17:49:51



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.06.2012 17:50:07



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.06.2012 16:54:03

cquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	26 Jun 2012 16:53:20
Date Stamp	26 Jun 2012 16:53:2	0					
File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Стать	и в ЈОС 25.05.12\гис	in-140612-24Aa-24Ba-c13dec	rudn-140612-24Aa	-24Ba-c13dec_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	628	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.3486
Sweep Width (Hz)	29409.97	Temperature (degree	C) 27.000				
udn-140612-24Aa-24Ba-	c13dec_001000fid	137.74 133.87		Com	pounds 2	24Aa/24Ba	$0 \\ 104 \\ 9 \\ 9 \\ 9 \\ 9 \\ 9 \\ 9 \\ 9 \\ 9 \\ 9 \\ $
						-52	45.81 44.97
174.79 172.12172.67 172.12		-137.35		-92.14	- 82.47	65.56	49.68 40.16 44.42 39.96 39.75 39.53 39.53 32.97

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.06.2012 16:54:13

Formula C11H11NO4S	FW 253.2743						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N/	13C/31P Z3379/0400		Date	26 Jun 2012 16:53:20
Date Stamp	26 Jun 2012 16:53:20						
File Name	C:\Users\Fedor\Deskt	ор\С13 Рома Для Статьи	в JOC 25.05.12\rudn-1	40612-24Aa-24Ba-c13dec\	rudn-140612-24Aa-24B	a-c13dec_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	628	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.3486
Sweep Width (Hz)	29409.97	Temperature (degree C	27.000				
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udn-140612-24Aa-24Ba-0	c13dec_001000fid		137.74	33.87		Cor	npounds 24Aa/24Ba
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4.79			-137.35	135.60			92
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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.06.2012 16:54:26

cquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/	15N/13C/31P Z3379/0400		Date	26 Jun 2012 16:53:20
ate Stamp	26 Jun 2012 16:53:	20	,				
ile Name	C:\Users\Fedor\Des	sktop\C13 Рома Для Стат	ьи в JOC 25.05.12\ru	dn-140612-24Aa-24Ba-c13	dec\rudn-140612-24Aa	-24Ba-c13dec_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transier	nts 628	Origin	spect
Driginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.3486
weep Width (Hz)	29409.97	Temperature (degree	e C) 27.000				
			96	Co	ompounds	24Aa/24Ba	$ \begin{array}{c} 0 \\ 104 \\ 9 \\ 9 \\ 9 \\ 9 \\ 9 \\ 9 \\ 9 \\ 9 \\ 9 \\ 9$
dp_140612-2442-2482	c13dec 001000fid		0		Sq		S 1
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						6 0	80
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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.06.2012 16:51:22

cauisition Time (sec)	0.5571	Comment	5 mm ONP 1H/15	N/13C/31P 73379/0400		Date	26 Jun 2012 17:04	00
ate Stamp	26 Jun 2012 17:04:0	0				Date	20 0011 2012 11:04	
le Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Стать	и в JOC 25.05.12\rudn	-140612-24Aa-24Ba-dept135	rudn-140612-24Aa-24	Ba-dept135 001000fid		
equency (MHz)	100.62	Nucleus	13C	Number of Transients	347	Origin	spect	
iginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135	
ceiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9103.9404	
eep Width (Hz)	29409.97	Temperature (degree	C) 27.000					
In-140612-24Aa-24Ba- 72.200 1000000000000000000000000000000000	dept135_001000fid				pounds 24	Aa/24Ba	4224 4224 4224 4224 4224 4224 42664 42664 42664 42664 4266666 426666666666	3
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10 Apr 2012



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10 Apr 2012



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.07.2012 16:14:27

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Formula C12H13NO4S	FW 267.3009	9					
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/1	15N/13C/31P Z3379/0400		Date	06 Jul 2012 16:14:56
Date Stamp	06 Jul 2012 16:14	:56					
File Name	C:\Users\Fedor\D	esktop\C13 Рома Для Статі	ы в ЈОС 25.05.12	\rudn-060712-24b\rudn-0607	12-24b_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	96	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Temperature (degree C) 32.000			en de la companya de			





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.07.2012 16:14:55

Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	06 Jul 2012 16:14:56
Date Stamp	06 Jul 2012 16:14	:56					
File Name	C:\Users\Fedor\De	esktop\C13 Рома Для Стать	и в ЈОС 25.05.12\г	udn-060712-24b\rudn-0607	12-24b_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	96	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Temperature (degree C) 32.000						
			H-C	Con afte	mpounds er crystalli	24Ab/24Bb ₁ zation	$H_{3C} = \begin{pmatrix} 0 & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 &$
udn-060712-24b_001000	ofid H-8		À				S 1
(7.2)	d (5,7))	-5.60	~ 90/10			
+B	(A) 978						H-3A
6.63	6.26				$\left(p\right)$		e de de
231 231	6.36				- 5.16		25 422 422 420 420 420 420 420 420
10.50 1.	25 9.35		8.75		1.01		1.19 11.84

10.07.2012 16:15:14



10.07.2012 15:55:11

OH 18

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Formula C ₁₂ H ₁₃ NO ₄ S	FW 267.3009						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	06 Jul 2012 16:17:04
Date Stamp	06 Jul 2012 16:17:04						
File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Статьи	в JOC 25.05.12\rud	n-060712-24b-c13dec\rudn-0	060712-24b-c13dec_0	01000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	2170	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10548.5127
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				



239

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10.07.2012 15:57:23

Formula C ₁₂ H ₁₃ NO ₄ S	FW 267.3009						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	06 Jul 2012 16:17:04
Date Stamp	06 Jul 2012 16:17:0	4	•				
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Статьи	в JOC 25.05.12\rud	n-060712-24b-c13dec\rudn-	060712-24b-c13dec	_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	2170	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10548.5127
Sweep Width (Hz)	29409 97	Temperature (degree C)	27 000				-





rudn-060712-24b-c13dec_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C12H13NO4S FW 267.3009 Acquisition Time (sec) 0.5571 Comment 5 mm QNP 1H/15N/13C/31P Z3379/0400 06 Jul 2012 16:17:04 Date 06 Jul 2012 16:17:04 Date Stamp C:\Users\Fedor\Desktop\C13 Рома Для Статьи в JOC 25.05.12\rudn-060712-24b-c13dec\rudn-060712-24b-c13dec_001000fid File Name 100.62 Frequency (MHz) Nucleus 13C Number of Transients 2170 Origin spect **Original Points Count** 16384 16384 Owner root Points Count **Pulse Sequence** zgpg **Receiver Gain** 32768.00 SW(cyclical) (Hz) 29411.77 DMSO-d6 Spectrum Offset (Hz) 10548.5127 Solvent Temperature (degree C) 27.000 Sweep Width (Hz) 29409.97



10.07.2012 15:59:34

OH

18

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112

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F141

007 0000

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

21.07.2011 20:05:55

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Formula C ₁₂ H ₁₃ NO ₄ S	FW 207.3009						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15N	/13C/31P Z3379/0400		Date	12 Jul 2011 09:27:28
Date Stamp	12 Jul 2011 09:27:28	3	8 . 80			25	
File Name	D:\NMR\19.05.2011	С 13 Для Иры Статья в Т	етраэдрон\N-3\rudn-	-0611-N3\rudn-0611-N3\ru	Idn-0611-N3_001000	Dfid	
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	16	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	128.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542
Sweep Width (Hz)	10416.03	Temperature (degree C)	32.000				



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007 0000

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

21.07.2011 20:06:07

Formula C ₁₂ H ₁₃ NO ₄ S	FW 267.3009						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	12 Jul 2011 09:27:28
Date Stamp	12 Jul 2011 09:27:2	В					
File Name	D:\NMR\19.05.2011	С 13 Для Иры Статья в	Тетраздрон\N-3\rud	n-0611-N3\rudn-0611-N3\ru	dn-0611-N3_00100	Ofid	
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	16	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	128.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542
Sweep Width (Hz)	10416.03	Temperature (degree C) 32.000				





rudn-0611-N3_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

21.07.2011 20:06:24

Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H	1/15N/13C/31P Z3379/0400		Date	12 Jul 2011 09:27:28
Date Stamp	12 Jul 2011 09:2	27:28	•				
File Name	D:\NMR\19.05.2	2011 С 13 Для Иры Статья	в Тетраэдрон\N-3	rudn-0611-N3\rudn-0611-N3\ru	dn-0611-N3_001	000fid	
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	16	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	128.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542
Sweep Width (Hz)	10416.03	Temperature (degree	C) 32.000				



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

21.07.2011 20:15:21

Formula C ₁₂ H ₁₃ NO ₄ S	FW 267.3009						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/	15N/13C/31P Z3379/0400		Date	12 Jul 2011 09:38:08
Date Stamp	12 Jul 2011 09:38:08		*				
File Name	D:\NMR\19.05.2011	С 13 Для Иры Статья в Те	граэдрон\N-3\rud	In-0611-N3-c13dec\rudn-0611-N	3-c13dec\rudn-06	11-N3-c13dec_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	2000	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10547.6182
Sween Width (Hz)	29409 97	Temperature (degree C)	27 000				

Compound 24Ab



rudn-0611-N3-c13dec 001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

21.07.2011 20:13:40

Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N/	13C/31P Z3379/0400		Date	12 Jul 2011 10:46:24
Date Stamp	12 Jul 2011 10:46:24	File Name	D:\NMR\19.05.2011 Тетраздрон\N-3\rudr	С 13 Для Иры Статья в n-0611-N3-dept135\rudn-06	611-N3-dept135\rudn-	0611-N3-dept135_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	2000	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cvclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9097.1982
Sween Width (Hz)	29409.97	Temperature (degree C	27.000				
idn-0611-N3-dept135_00	1000fid				Compound	l 24Ab	Mick
	alion 1 - ani na Kananasi kana kanakananak	1000 Lancon and Lancon and Lancon and Lancon and the	han an a bind a specific speci	02.78	52.72 52.72 47.98	galant James J. Lagard with J. James any any state of the st	15.67
					45.37	32.67	
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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10 Apr 2012

Acquisition Time (sec)	1.6056	Comment	Imported from	m UXNMR.		Date	15 Jul 200	9 12:50:08
File Name	D:\Timur\Тимур	(лето 2009)\rudn8\rudn8	_001000fid	Frequency (MHz)	400.14	Nucleus	1H	Number of Transients 4
Original Points Count	16384	Points Count	16384	Pulse Sequence	zg	Solvent	CHLOROF	FORM-D
Sweep Width (Hz)	10204.08	Temperature (degree (27.000					







General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

26.05.2012 18:30:37 Formula C10H11NO2S FW 209.2648 Acquisition Time (sec) 1.6056 Comment 5 mm QNP 1H/15N/13C/31P Z3379/0400 Date 15 Jul 2009 12:50:08 Date Stamp 15 Jul 2009 12:50:08 File Name D:\NMR\C_13\Тимур (лето 2009)\rudn8\rudn8_001000fid Frequency (MHz) 400.14 Nucleus 1H Number of Transients 4 Origin spect **Original Points Count** 16384 Owner root **Points Count** 16384 Pulse Sequence zg 512.00 **Receiver Gain** SW(cyclical) (Hz) 10204.08 Solvent CHLOROFORM-d Spectrum Offset (Hz) 2612.4158 Sweep Width (Hz) 10203.46 Temperature (degree C) 27.000 0 Compound 25a •0 H-96 10. 2 Η 9ba rudn8_001000fid 59 5 4-9 H-8 dd 6.41 H-3A, (5.5) (1.8;5.5) 1.8;6.4;11.9) (+.8:3.7) 6.44 6.40 5.09 .09 5.08 6.46 10 1.98 0.97 0.99 1.00 6.5 6.4 6.3 6.2 6.1 6.0 5.9 5.8 5.6 5.5 5.7 5.4 5.3 5.2 5.1 5.0 4.9 4.8 4.6 4.7 4.5 4.4 Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

Formula C10H11NO2S FW

Acquisition Time (sec) 1.6056

209.2648

Comment

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Date

5 mm QNP 1H/15N/13C/31P Z3379/0400

26.05.2012 18:30:47

15 Jul 2009 12:50:08

Date Stamp	15 Jul 2009 12:	60:08		File Name	D:\NMR\C_1	13\Тимур (лето 2009)\rudn8	3\rudn8_001000fid	
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	4	Origin	spect	
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg	
Receiver Gain	512.00	SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFO	DRM-d		
pectrum Offset (Hz)	2612.4158	Sweep Width (Hz)	10203.46	Temperature (degree	C) 27.000			
H-2A ddd t.8;6.4;H.0 dn8_001000fid	H-2 (6.') dt	B 1;11.6)			Compo	ound 25a		$ \begin{array}{c} O\\/14\\ -5a\\ -9a\\ -9a\\ -9b\\ -9b\\ -9b\\ -2\\ -2\\ -2\\ -2\\ -2\\ -2\\ -2\\ -2\\ -2\\ -2$
ß	1	H-Sa Bzda (3.7	endo d ; 8.3)	H-6 dd (3.7;	s.0;1	1.3)		H-6endo dd (8.3;11.9)
ş	02 0 1 2.96 1 2.92 2.92 2.90 2.90	8 77 75		. m 09			×	-1.58 -1.56 -1.55 -1.53
4	2.94	2.7		2.32 2.33 2.33 2.33 2.33 2.33 2.33 2.33	2.29		H2O	
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15 Jul 2009 15:04:32

Imported from UXNMR.

10 Apr 2012

ile Name	D:\Timur\Tи	лур (лето 2009)\rudn8c13dec\ru	dn8c13dec_001000	Ofid		Frequency (MHz)	100.62	
lucleus	13C	Number of Transients	282	Original Points Count	16384	Points Count	16384	
ulse Sequence	zgpg	Solvent	CHLOROFORM-	2		Sweep Width (Hz)	26315.79	
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176 168	160 152	144 136 128	120 112	104 96 88 Chamical Shift (a	80 72	64 56 4	40 32	24 16 8
	Fed	or I. Zubkov, Eugeniya V. Nikitii	na, Timur R. Galee	v, Vladimir P. Zaytsev, Vic	tor N. Khrustalev,	Roman A. Novikov, and A	Alexey V. Varlamov	

Acquisition Time (sec) 0.6226

Comment

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10 Apr 2012


General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.



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H₃C

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Formula C ₁₁ H ₁₃ NO ₂ S	FW	223.2914						
Acquisition Time (sec)	1.6056		Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	07 Apr 2010 16:51:12
Date Stamp	07 Apr	2010 16:51:1	12		File Name	D:\NMR\C_13\07.0	4.10 (В основном аддукт	ъ с DMAD на пиперидонах)\N848\N848_001000fid
Frequency (MHz)	400.14		Nucleus	1H	Number of Transients	12	Origin	spect
Original Points Count	16384		Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	1024.00)	SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d		
Spectrum Offset (Hz)	2608.99	912	Sweep Width (Hz)	10203.46	Temperature (degree C)	27.000		

Compound 25b







General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

03.05.2012 15:10:01

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Formula C11H13NO2S	FW 223.2914						
Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	07 Apr 2010 16:51:12
Date Stamp	07 Apr 2010 16:51:	12		File Name	D:\NMR\C_13\07.0	4.10 (В основном аддукть	и с DMAD на пиперидонах)\N848\N848_001000fid
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	12	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	1024.00	SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d		
Spectrum Offset (Hz)	2608.9912	Sweep Width (Hz)	10203.46	Temperature (degree C)	27.000		



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

03.05.2012 15:10:57

Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	07 Apr 2010 16:51:12
Date Stamp	07 Apr 2010 16:51:	12		File Name	D:\NMR\C_13\07.0	04.10 (В основном адд	укты с DMAD на пиперидонах)\N848\N848_001000fic
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	12	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	1024.00	SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d		
Spectrum Offset (Hz)	2608.9912	Sweep Width (Hz)	10203.46	Temperature (degree C) 27.000		
				Corr	pound 25	ih	$H_{3}C_{1} - 7 - 6 - 5a - 5'_{1}$
	H-	59		Com	pound 25	ib	$H_{3}C_{1}$ 7 0 $5a$ 5 N_{4} 3 $9a$ $9b$ 4 3
	H-	59 dad		Com	pound 25	бb	$H_{3}C_{1} = 7 = 6 = 5a = 5 = 5 = 10$ $B_{9} = 9a = 9b = 1 = 10$ $H_{3}C_{1} = 7 = 6 = 5a = 5 = 5$ $N_{1} = 3 = 5 = 10$ $H_{3}C_{1} = 7 = 6 = 5a = 5$ $N_{1} = 3 = 5$ $H_{3}C_{1} = 7 = 6 = 5a = 5$ $N_{1} = 3 = 5$ $H_{3}C_{1} = 7 = 5a = 5a = 5$ $H_{3}C_{1} = 7 = 5a $



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C11H13NO2S FW 223.2914 Acquisition Time (sec) 0.6226 Comment 07 Apr 2010 17:44:32 5 mm QNP 1H/15N/13C/31P Z3379/0400 Date 07 Apr 2010 17:44:32 Date Stamp D:\NMR\C_13\07.04.10 (В основном аддукты с DMAD на пиперидонах)\N848c13dec\N848c13dec_001000fid File Name 100.62 Frequency (MHz) Nucleus 13C Number of Transients 16384 128 Origin spect **Original Points Count** Owner root Points Count 16384 Pulse Sequence Receiver Gain 32768.00 zgpg SW(cyclical) (Hz) 26315.79 Solvent CHLOROFORM-d 9619.4717 Sweep Width (Hz) 26314.18 Spectrum Offset (Hz) Temperature (degree C) 27.000 0 H₂(Compound 25b -0 104 0 Η 9ba 2 132.29 N848c13dec_001000fid 6 Sa 32.27 141.05 53.63 45.25 Me-7 34.65 9B 8.70 ga 65.42 5 86.83 174.16 79 92.53 20 alabet interferent and a fare interferent alternation alternation alternation and a fare and a fare and a fare والمتناغات الانتحاب والمعالية المسترية فتعالمه والأطور لمتابر ومعاده فالمعادية الغني ومعالمة وتوسيل القدامانية أرياب فاجتباله والغارافين 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 56 40 32 24 16 64 48 8 0



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

03.05.2012 15:22:39

07 Apr 2010 17:48						
	:48					
D:\NMR\C_13\07.0	04.10 (В основном аддукты с	с DMAD на пиперидон	ax)\N848dept135\N848de	pt135_001000fid	Frequency (MHz)	100.62
13C	Number of Transients	74	Origin	spect	Original Points Count	16384
root	Points Count	16384	Pulse Sequence	dept135	Receiver Gain	32768.00
26315.79	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9619.4814	Sweep Width (Hz)	26314.18
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09.07.2012 21:03:54

Formula C12H13NO4S	FW 267.3009						
Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H/15N	1/13C/31P Z3379/0400		Date	06 Jul 2012 15:17:20
Date Stamp	06 Jul 2012 15:17:2	0					
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Стать	и в ЈОС 25.05.12\ruc	In-060712-26a\rudn-06071	2-26a_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	18	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	512.00
SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2602.0486	Sweep Width (Hz)	10203.46
Temperature (degree C	27.000			1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 -			



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.07.2012 21:04:17

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Formula C ₁₂ H ₁₃ NO ₄ S	FW 267.30	09					
Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H/15M	V/13C/31P Z3379/0400		Date	06 Jul 2012 15:17:20
Date Stamp	06 Jul 2012 15:	17:20	*				
File Name	C:\Users\Fedor	Desktop\C13 Рома Для Стать	и в ЈОС 25.05.12\гис	dn-060712-26a\rudn-06071	2-26a_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	18	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	512.00
SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2602.0486	Sweep Width (Hz)	10203.46
Temperature (degree C	27.000						

Compounds 26Aa/26Ba after crystallization



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

cquisition Time (sec)	1.6056	Comment	5 mm QNP 1H/15N	/13C/31P Z3379/0400		Date	06 Jul 2012 15:17:20
ate Stamp	06 Jul 2012 15:	17:20					
ile Name	C:\Users\Fedor\	Desktop\C13 Рома Для Стать	и в JOC 25.05.12\rud	In-060712-26a\rudn-06071	2-26a_001000fid	Frequency (MHz)	400.14
ucleus	1H	Number of Transients	18	Origin	spect	Original Points Count	16384
wner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	512.00
N(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2602.0486	Sweep Width (Hz)	10203.46
<u>, </u>				Co aft	ompounds er crystall	26Aa/26Ba ization	$ \begin{array}{c} CH_{3} \\ O \\ 16 \\ 15 \\ 17 \\ 0 \\ 14 \\ 5 \\ 5 \\ 5 \\ 5 \\ 5 \\ 5 \\ 5 \\ 5 \\ 5 \\ 5$
				B			$\begin{bmatrix} 7 & 0 & 5a \\ 10 & 9a & 9b \\ 8 & 9 & 9b \\ 9 & 9 & 9b \\ 8 & 5 \\ 1 \end{bmatrix}$
in-060712-26a_001000	fid		3.75	2		$(\cap$	11-6.H-59
				00-No		(b)	4-0/11
			1	Co-2 Me			
H-3A			1	3.14		H-3B	d d
B						H-2 3H, M	(8,7)
,g; 6.2; 1	1,2)						92 2.93 -2.71 .69
Ţ						~3.10 3.08	89 1 2
4.48 4.45 4.45 4.45 4.41 4.41 4.41 4.40	4.39					3.16 3.15 3.15 3.13 3.13 3.13 3.13	2.96 2.91 2.91 2.81 2.81 2.83 2.83 2.83 2.83 2.83 2.83 2.83 2.83
1 00 0 05			11.0	-			100 201

3.7 3.6 3.5 Chemical Shift (ppm)

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.07.2012 21:22:02

CH₃

Formula C ₁₂ H ₁₃ NO ₄ S	FW 267.3009						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	06 Jul 2012 15:21:36
Date Stamp	06 Jul 2012 15:21:36		<i>.</i>				
File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Статьи	B JOC 25.05.12\rud	n-060712-26a-c13dec\rudn-0	60712-26a-c13dec_00	1000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	1724	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9618.5391
Sween Width (Hz)	29409 97	Temperature (degree C)	27 000				



Formula C ₁₂ H ₁₃ NO ₄ S	<i>FW</i> 267.3009						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N	/13C/31P Z3379/0400		Date	06 Jul 2012 15:21:36
Date Stamp	06 Jul 2012 15:21:36						
File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Статьи	B JOC 25.05.12\rudn-	-060712-26a-c13dec\rudn-0	60712-26a-c13dec_00	1000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	1724	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9618.5391
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				

Compounds 26Aa/26Ba after crystallization



CH₃



rudn-060712-26a-c13dec_001000fid

09.07.2012 21:22:11

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.07.2012 21:22:23

0

Formula C ₁₂ H ₁₃ NO ₄ S	FW 267.3009						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	06 Jul 2012 15:21:36
Date Stamp	06 Jul 2012 15:21:36		*				
File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Стать	и в ЈОС 25.05.12	rudn-060712-26a-c13dec\rudn-0	60712-26a-c13dec_00	1000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	1724	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9618.5391
Sweep Width (Hz)	29409.97	Temperature (degree	C) 27.000			-1	
							CH ₃

Compounds 26Aa/26Ba

after crystallization



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

09.07.2012 21:17:41

FW Formula C12H13NO4S 267.3009 Acquisition Time (sec) 0.5571 Comment 5 mm QNP 1H/15N/13C/31P Z3379/0400 Date 06 Jul 2012 15:53:36 Date Stamp 06 Jul 2012 15:53:36 C:\Users\Fedor\Desktop\C13 Рома Для Статьи в JOC 25.05.12\rudn-060712-26a-dept135\rudn-060712-26a-dept135_001000fid File Name Frequency (MHz) 100.62 Nucleus 13C Number of Transients 597 Origin spect **Original Points Count** 16384 Owner root **Points Count** 16384 **Pulse Sequence** dept135 32768.00 SW(cyclical) (Hz) **Receiver Gain** 29411.77 Solvent CHLOROFORM-d Spectrum Offset (Hz) 9618.5283 Sweep Width (Hz) 29409.97 Temperature (degree C) 27.000



10 Apr 2012

Acquisition Time (sec)	1.6056	Comment	Imported from	UXNMR.		Date	15 Jul 2009 13:00:48	
File Name	D:\Timur\Тимур	(лето 2009)\rudn10\rudn10	_001000fid	Frequency (MHz)	400.14	Nucleus	1H	
Number of Transients	4	Original Points Count	16384	Points Count	16384	Pulse Sequence	zg	
Solvent	CHLOROFORM	I-D		Sweep Width (Hz)	10204.08	Temperature (degree C	27.000	







3.77

10 Apr 2012

Acquisition Time (sec)	1.6056	Comment	Imported from	UXNMR.		Date	15 Jul 2009 13:00:48	
File Name	D:\Timur\Тимур	(лето 2009)\rudn10\rudn10	_001000fid	Frequency (MHz)	400.14	Nucleus	1H	
Number of Transients	4	Original Points Count	16384	Points Count	16384	Pulse Sequence	zg	
olvent	CHLOROFORM	I-D		Sweep Width (Hz)	10204.08	Temperature (degree C	c) 27.000	-
					Compou	ind 26Ba	$ \begin{array}{c} & & & & \\ & & & & \\ & & & & \\ & & & &$	
							90a 1	
							H-3A	
-8				41-96)	H-7		
d	H-9			S		d	ddd	
(8,2)	d (5.8)			5.54		(112)	(2,5;4.4: 1	1.2
6.52						5.22		
6.53 6.53 6.45							444 444 434	4.42
2.03				0.98		1.02		1.00

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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10 Apr 2012



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10 Apr 2012

Acquisition Time (sec)	0.6226	Comment	Imported from UXN	MR.		Date	15 Jul 2009 15:13:0	4
File Name	D:\Timur\Тимур (л	ето 2009)\rudn10c13dec\r	udn10c13dec_00100	OOfid		Frequency (MHz)	100.62	
Nucleus	13C	Number of Transients	292	Original Points Count	16384	Points Count	16384	
Pulse Sequence	zgpg	Solvent	CHLOROFORM-D)		Sweep Width (Hz)	26315.79	
Temperature (degree C)	27.000			С	ompound	l 26Ba	O 16 15 0 17 0 17 0 17 0 17	0 //14
		Q						ba 1
		138.0						
	9					36	35.34	
		8		7		64.22	₩ 2	
					-80.79	CO2Me	45.8	
		133.57				37 8 6		
5, colle				<u>3</u> a	107008.4	52.1		
72.04 —170.81				92.67	77.44 77.12 76.80			×
litikitingan ang mananan kanalaka kanalaka kanalaka kanalaka kanalaka kanalaka kanalaka kanalaka kanalaka kanal	n malakan manalan kaban manan man	ende frijenend welterfrijeteterfrijeterwelteterfrij). Wither in the second	hour and the state of the state	al water al approximition to	ห่าน พระระว่าเวลาสุดเล่าถึงการสุดสุด	u ⁿ teantista aise kakistateko utean teantistatean	langan na ana ang kana na kana kana kana
176 168 160	152 144	136 128 12	0 112 104	4 96 88 Chemical Shift (pr	80 72 om)	64 56 48	40 32	24 16 8 0

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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10 Apr 2012



269

03.05.2012 18:19:22

Acquisition Time (sec)	1.4549	Comment	single_pulse	Date	23 Nov 2010	14:38:05		Date Stamp	23 Nov 2010 13:50:36
ile Name	D:\NMR\22.11.	10\FZ1481-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	: 10
rigin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
eceiver Gain	50.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	3601.0339	Sweep Width (Hz)	11261.26
CH_3 O O O O O 0 0 0 0 0 0 0 0	H ₃ C_	CH_3 O O O O O O O O	3			C	ompou	nds 26Ab/26	Bp
H [°] S ⁻ 1	_2	H S	2			3.72			-1.68
7.25	6.63	6.25 6.25	5.47	5.23			∽3.13 12 2.99	2.85	
	0.65	1.60	0.52 1	4.51	4.50 4.40 4.39 4.39 4.39 4.39 4.39		3.16 3.1	2.73 2.73 2.73	

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

03.05.2012 18:19:43

Acquisition Time (sec)	1.4549	Comment	single_pulse	Date	23 Nov 2010 1	4:38:05		Date Stamp	23 Nov 2010 13:50:36
File Name	D:\NMR\22.11	.10\FZ1481-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	s 10
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	50.00	Solvent	CHLOROFOR	M-d		Spectrum Offset (Hz)	3601.0339	Sweep Width (Hz)	11261.26
.'1481-1.jdf				Compo	ounds 26 <i>1</i>	Ab/26Bb	O H ₃ C		
µ-2	۳, ۶	8			F	5.23 S	8	05a - 5a -	7 0 5a 5 10 10 10 10 10 10 10 10 10 10 10 10 10 1
(5.5))	(S,S)			4				H-3A
6.63		6.25			-5.47			(2,0;6.9	ddd s;11.7)
6.53		6.23			4				51 4.50 9
the	-	A.C.		S.	t				4,40
1.03 0.65		1.60			0.52	1.00			1.03 0

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

03.05.2012 18:19:59



Formula C U NO C

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201 2275

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.04.2011 21:53:13

Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	14 Apr 2011 05:56:16
Date Stamp	14 Apr 2011 05:56	16		File Name	D:\NMR\13.04.201	1 C-13\rudn-130411-N2-c	13dec\rudn-130411-N2-c13dec_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	650	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411:77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10547.6182
Sweep Width (Hz)	29409.97	Temperature (degree C) 32.000				

Compounds 26Ab/26Bb

H₃C

-0



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.04.2011 21:53:22

Formula C ₁₃ H ₁₅ NO ₄ S	FW	281.3275	
Acquisition Time (see)	0.5571		C

Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	14 Apr 2011 05:56:16
Date Stamp	14 Apr 2011 05:56	5:16		File Name	D:\NMR\13.04.201	1 C-13\rudn-130411-N2-c	13dec\rudn-130411-N2-c13dec_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	650	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10547.6182
Sweep Width (Hz)	29409.97	Temperature (degree C) 32.000				

Compounds 26Ab/26Bb



rudn-130411-N2-c13dec_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.04.2011 21:53:37

4 Apr 2011 05:56: 00.62 6384 2768 00	16 Nucleus Owner	13C	File Name Number of Transie	D:\NMR\13.04.20	011 C-13\rudn-130411-N2-c	:13dec\rudn-130411-N2-c13dec_001000fid
00.62 6384	Nucleus Owner	13C	Number of Transie	-1- 050		
6384	Owner	10.000 C		nts 650	Origin	spect
2760 00		root	Points Count	16384	Pulse Sequence	zgpg
2708.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10547.6182
	7		(Compounds	26Ab/26Bb	$H_{3}C \xrightarrow{0}_{16} H_{3}C \xrightarrow{0}_{16} H_{15} H_{16} H$
υουπα	52.86	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	30.75	- 39.32 39.0	26 32.77	1
	20	-48.05	45.40 74		- 32	15.59
	с Х	51.02	44.	39.13		
			40.16	38.91		
	9409.97	9409.97 Temperature (degree 0 1000fid 6 1000fid 7 1000fid 7 10	10000fid 6 2 59 10000fid 6 2 59 10000fid 6 2 59 10000fid 7 59	1000fid 6 3 59 1000fid 7 40 1000fid 7 40 1000fi	$10000fid \qquad \qquad$	Pados 97 Temperature (degree C) 32.000 100001d 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0

Formula C H NO

281.3275

FW

C

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.04.2011 21:51:51

CH-

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H₃C.

1 011101 0131 1151 0040							
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/1	15N/13C/31P Z3379/0400		Date	14 Apr 2011 06:11:12
Date Stamp	14 Apr 2011 06:11	:12		File Name	D:\NMR\13.04.201	1 C-13\rudn-130411-N2-d	ept135\rudn-130411-N2-dept135_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	575	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9097.1982
Sweep Width (Hz)	29409.97	Temperature (degree C) 32.000				

Compounds 26Ab/26Bb





10 Apr 2012

Acquisition Time (sec)	1.6056	Comment	Imported from L	JXNMR.		Date	15 Jul 2009 12:41:36
File Name	D:\Timur\Тимур	(лето 2009)\rudn11\rudn11	_001000fid	Frequency (MHz)	400.14	Nucleus	1H
Number of Transients	4	Original Points Count	16384	Points Count	16384	Pulse Sequence	zg
Solvent	CHLOROFORM	-D		Sweep Width (Hz)	10204.08	Temperature (degree	C) 27.000

Compound 27





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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10 Apr 2012

Acquisition Time (sec)	1.6056	Comment	Imported from	UXNMR.		Date	15 Jul 2009 12:41:36	
File Name	D:\Timur\Тимур	(лето 2009)\rudn11\rudn11	_001000fid	Frequency (MHz)	400.14	Nucleus	1H	
Number of Transients	4	Original Points Count	16384	Points Count	16384	Pulse Sequence	zg	
Solvent	CHLOROFORM	-D		Sweep Width (Hz)	10204.08	Temperature (degree	C) 27.000	
							0	

Compound 27





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

10 Apr 2012



10 Apr 2012

cquisition Time (sec)	0.6226	Comment	Imported from UX	NMR.		Date Frequency (MHz)	15 Jul 2009 16:32	::00
ucleus	13C	Number of Transients	276	Original Points Count	16384	Points Count	16384	
ulse Sequence	ZQDQ	Solvent	CHLOROFORM-	D		Sweep Width (Hz)	26315.79	
emperature (degree C)	27.000		on control of an			Cheep maar (ng	20010110	
		4	,5	Com	pound 27	3		SCH_3
		-136.75	1,44	3	352 52))	CH ₃	7,21
			.		8		2	27.16
	гЭ				.23		45.04	11.82 38.11
Ne	OLH	e		2	80 77.43	76.81		30.60
		huganhanin phanyan dika dunid.	ingen an war ne far before and see in	a far factor of the factor of		nan kalimentun susatan sisi di kata susat		
ويسيبه ويعطيها الأنافيقال بفأبيتك يعابأ لاستاب	وليلج يعرهم القينية مسابق يعتر مسيه	وأول يعربا لينار المار لمار فرحمه ويريد أهليا الاركاف فينادا ا	وملايتها والشاريك ويتقاونه فالشرق	ade illinged as the successive successive state of the state of the successive	and the second second states of	A D. L. BRANNES CO. MAN AND A DATASE	الانتجاب أيتيا يتجر المتلك أنقالا بأمرين يدغل بن	والراهية والمطاور معاركة والمناقبات والمناهية والمرامية والمراجع

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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10 Apr 2012

Acquisition Time (sec)	0.6226	Comment	Imported from UXNM	R.		Date	15 Jul 2009 16:	40:32	
ile Name	D:\Timur\Тиму	р (лето 2009)\rudn11dept13	35\rudn11dept135_001000)fid		Frequency (MHz)	100.62		
icleus	13C	Number of Transient	s 176 (Driginal Points Count	16384	Points Count	16384		
Ise Sequence	dept135	Solvent	CHLOROFORM-D			Sweep Width (Hz)	26315.79		
mperature (degree C)	27.000								
-136.75 			82.75			90.55 6	pound 27	- 21.02	
hipsipsipsipsipsipsipsipsipsipsipsipsipsi	unnunnun	allansi kata kata kata kata kata kata kata kat	aidanhayhahasalalannaharinnaharinnah	ubahlananpaparannahlarahn	analisia kalaanati nijettaan mad	nt+thing the hospital that the the the	glattavosida, a kanvisada	ra ing delaterishing ng managerishing ng managerishing ng managerishing ng managerishing ng managerishing ng ma	int for the second second
						41.56	28.12 27.1		

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

25.07.2011 16:21:21

NH

-ormula C ₈ H ₁₁ NOS FW 109.2440
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Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H/15N/13C/31P Z3379/0400			Date	21 Jul 2011 16:00:00
Date Stamp	21 Jul 2011 16:00:00			File Name	D:\NMR\19.07.11	4\rudn-190711-N4_001000fid	
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	16	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	512.00	SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM	-d	
Spectrum Offset (Hz)	2602.0486	Sweep Width (Hz)	10203.46	Temperature (degree C)	27.000		



169.2440

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Formula C.H. NOS FW

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

25.07.2011 16:29:18

Acquisition Time (sec)	0.5898	Comment	5 mm QNP 1H/15N/13C/31P Z3379/0400			Date	21 Jul 2011 16:19:12
Date Stamp	21 Jul 2011 16:19:1	12	File Name D:\NMR\19.07.11 (Рома)\rudn-190711-N4-c13dec\rudn-190711-N4-c				-c13dec\rudn-190711-N4-c13dec_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	219	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	27777.78	Solvent	CHLOROFORM-d		
Spectrum Offset (Hz)	9608.4277	Sweep Width (Hz)	27776.08	Temperature (degree C) 27.000		



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

25.07.2011 16:27:56



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

21.07.2011 18:43:11

OH 18

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Formula C12H13NO4S	FW 267.3009									
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15N/13C/31P Z3379/0400			Date	08 Jul 2011 07:45:04			
Date Stamp	08 Jul 2011 07:45:0	4								
File Name	D:\NMR\19.05.2011	D:\NMR\19.05.2011 C 13 Для Иры Статья в Тетраэдрон\rudn-0611-N13\rudn-0611-N13\rudn-0611-N13\rudn-0611-N13_001000fid								
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	8	Origin	spect			
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg			
Receiver Gain	128.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542			
Sweep Width (Hz)	10416.03	Temperature (degree C)	32.000							

Compound 31a



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

21.07.2011 18:43:39

Formula C12H13NO4S	FW 267.3009										
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15N/13C/31P Z3379/0400			Date	08 Jul 2011 07:45:04				
Date Stamp	08 Jul 2011 07:45:0	4									
File Name	D:\NMR\19.05.2011	D:\NMR\19.05.2011 С 13 Для Иры Статья в Тетраэдрон\rudn-0611-N13\rudn-0611-N13\rudn-0611-N13\rudn-0611-N13_001000fid									
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	8	Origin	spect				
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg				
Receiver Gain	128.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	St			
Sween Width (Hz)	10416 03	Temperature (degree C) 32,000								







rudn-0611-N13_001000fid


General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

21.07.2011 19:01:28



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

21.07.2011 18:59:13

0.

17

OH

Formula C ₁₂ H ₁₃ NO ₄ S	FW 267.3009						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP	1H/15N/13C/31P Z3379/0400		Date	08 Jul 2011 08:29:52
Date Stamp	08 Jul 2011 08:29:52						
File Name	D:\NMR\19.05.2011 C	13 Для Иры Статья в Тет	раздрон\rudn	-0611-N13\rudn-0611-N13-dept135\ru	dn-0611-N13-dept1	35\rudn-0611-N13-dept135_00	1000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	750	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9102.5918
Sweep Width (Hz)	29409.97	Temperature (degree C	27.000				



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

15.07.2011 17:56:44

Formula C13H15NO4S FW 281.3275

Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15N/13C/31P Z3379/0400			Date	14 Jul 2011 10:05:52
Date Stamp	14 Jul 2011 10	0:05:52					
File Name	D:\NMR\19.05	.2011 С 13 Для Иры Статья в	Тетраэдрон\rud	n-0611-N41\rudn-0611-N41_0	Frequency (MHz)	400.14	
Nucleus	1H	Number of Transients	12	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	256.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03

Temperature (degree C) 32.000

Compound 31b



31b



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

15.07.2011 17:57:12

Formula C., H., NO.S	FW	281.3275
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Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	14 Jul 2011 10:05:52
Date Stamp	14 Jul 2011 10:0	05:52					
File Name	D:\NMR\19.05.2	2011 С 13 Для Иры Статья в	Тетраздрон\rud	Frequency (MHz)	400.14		
Nucleus	1H	Number of Transients	12	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	256.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03

Temperature (degree C) 32.000

Compound 31b



rudn-0611-N41_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

15.07.2011 17:57:21

Formula C13H15NO4S	FW	281.3275						
Acquisition Time (sec)	1.5729		Comment	5 mm QNP	1H/15N/13C/31P Z3379/0400		Date	14 Jul 2011 10:05:52
Date Stamp	14 Jul	2011 10:05:5	52					
File Name	D:\NM	R\19.05.201	1 С 13 Для Иры Статья в	в Тетраздрон\	rudn-0611-N41\rudn-0611-N41_0	001000fid	Frequency (MHz)	400.14
Nucleus	1H		Number of Transients	12	Origin	spect	Original Points Count	16384
Owner	root		Points Count	16384	Pulse Sequence	zg	Receiver Gain	256.00
SW(cyclical) (Hz)	10416.	67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Temperature (degree C) 32.000							and the second



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

15.07.2011 18:11:33

FW 281.3275						
0.5898	Comment	5 mm QNP 1H/15	5N/13C/31P Z3379/0400		Date	14 Jul 2011 10:12:16
14 Jul 2011 10:12:10	6					
D:\NMR\19.05.2011	С 13 Для Иры Статья в	Тетраэдрон\rudn-06	11-N41-c13dec\rudn-0611-N	41-c13dec_001000	Dfid	
100.62	Nucleus	13C	Number of Transients	435	Origin	spect
16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
32768.00	SW(cyclical) (Hz)	27777.78	Solvent	DMSO-d6	Spectrum Offset (Hz)	9099.9072
27776.08	Temperature (degree	C) 27.000				
01000fid			Com 8,10a	pound 31	р ⁸⁵⁰²⁹ 106 ⁸⁴⁷⁴ У ⁸⁴⁷⁴ У ⁸⁴⁷⁴	$\begin{array}{c} 17 \\ 17 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\$
G			-89.16		52.4	14 9.32 27.38 0 27.38
						24.82
						40.17 39.95 38.90 39.10
	0.5898 14 Jul 2011 10:12:1 D:\NMR\19.05.2011 100.62 16384 32768.00 27776.08 01000fid	0.5898 Comment 14 Jul 2011 10:12:16 D:\NMR\19.05.2011 C 13 Для Иры Статья в 100.62 Nucleus 16384 Owner 32768.00 SW(cyclical) (Hz) 27776.08 Temperature (degree of 1000fid 9, 10 1000fid 9, 10 1000fid 6, 10 1000fid 76 1000fid	O.5898 Comment 5 mm QNP 1H/15 14 Jul 2011 10:12:16 D:NMR(19.05.2011 C 13 Для Иры Статья в Тетраздрон\rudn-06 100.62 Nucleus 13C 16384 Owner root 32768.00 SW(cyclical) (Hz) 27777.78 27776.08 Temperature (degree C) 27.000	V 201.02.10 0.5898 Comment 5 mm QNP 1H/15N/13C/31P Z3379/0400 14 Jul 2011 10:12:16 Diversion Number of Transients 100.62 Nucleus 13C Number of Transients 16384 Owner root Points Count 32768.00 SW(cyclical) (Hz) 27777.78 Solvent	0.5898 Comment 5 mm QNP 1H/15N/13C/31P Z3379/0400 14 Jul 2011 10:12:16 D:INMR19.05.2011 C 13 Для Иры Crars в в TerpaagoorVudn-0611-N41-c13dec_00100 100.62 Nucleus 13C Number of Transients 435 13584 Owner root Points Count 15884 32768.00 SW(cyclical) (Hz) 27777.78 Solvent DMSO-d6 27776.08 Temperature (degree C) 27.000 Compound 31 01000fid 9, 10 1000fid 9,	Y 2013013 0.5898 Comment 5 mm QNP 1H/15N/13C/31P Z3379/0400 Date 0.5898 Comment 5 mm QNP 1H/15N/13C/31P Z3379/0400 Date DINMR19.05.2011 C 13 [Ins Mpb Crars is Terpesapportrudin-0611-N41-c13declrudin-0611-

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

15.07.2011 18:10:19

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Formula C ₁₃ H ₁₅ NO ₄ S	<i>FW</i> 281.3275						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	14 Jul 2011 10:22:56
Date Stamp	14 Jul 2011 10:22:5	6					
File Name	D:\NMR\19.05.2011	С 13 Для Иры Статья в Те	траэдрон\rudn-06	11-N41-dept135\rudn-0611-N4	41-dept135_001000fid		
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	462	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9099.9863
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				







General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

15.07.2011 18:42:32

Formula C ₁₁ H ₁₃ NO ₂ S	FW 223.2914						
Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400	í	Date	14 Jul 2011 08:53:20
Date Stamp	14 Jul 2011 08:53:	20					
File Name	D:\NMR\19.05.201	1 С 13 Для Иры Статья в	Тетраздрон\rudn-06	11-N14\rudn-0611-N14	_002000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	512.00
SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d			Spectrum Offset (Hz)	2602.0486
Sweep Width (Hz)	10203.46	Temperature (degree C) 27.000				

Compound 32





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

15.07.2011 18:42:48

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Formula C ₁₁ H ₁₃ NO ₂ S	FW 223.2914						
Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H/15M	V/13C/31P Z3379/0400		Date	14 Jul 2011 08:53:20
Date Stamp	14 Jul 2011 08:53:2	0					
File Name	D:\NMR\19.05.2011	С 13 Для Иры Статья в 7	Гетраэдрон\rudn-06	11-N14\rudn-0611-N14_	002000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	512.00
SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d			Spectrum Offset (Hz)	2602.0486
Sweep Width (Hz)	10203.46	Temperature (degree C)	27.000				



Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

Compound 32

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

15.07.2011 18:43:05

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Formula C ₁₁ H ₁₃ NO ₂ S	FW 223.2914						
Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400	j.	Date	14 Jul 2011 08:53:20
Date Stamp	14 Jul 2011 08:53:	20					
File Name	D:\NMR\19.05.201	1 С 13 Для Иры Статья в	Тетраэдрон\rudn-06	11-N14\rudn-0611-N14	_002000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	16	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	512.00
SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d			Spectrum Offset (Hz)	2602.0486
Sweep Width (Hz)	10203.46	Temperature (degree C)	27.000				





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

15.07.2011 18:57:31 Formula C11H13NO2S FW 223.2914 Acquisition Time (sec) 0.5898 Comment 5 mm QNP 1H/15N/13C/31P Z3379/0400 Date 14 Jul 2011 08:59:44 Date Stamp 14 Jul 2011 08:59:44 D:\NMR\19.05.2011 С 13 Для Иры Статья в Тетраэдрон\rudn-0611-N14-c13dec\rudn-0611-N14-c13dec_001000fid Frequency (MHz) 100.62 File Name Nucleus 13C Number of Transients 523 Origin **Original Points Count** 16384 spect Points Count 16384 Pulse Sequence **Receiver Gain** 32768.00 Owner root zgpg SW(cyclical) (Hz) 27777.78 Solvent CHLOROFORM-d 9622.8369 Sweep Width (Hz) 27776.08 Spectrum Offset (Hz) Temperature (degree C) 27.000 0 Compound 32 11 10 1 10ba 79.45 rudn-0611-N14-c13dec_001000fid 131.58 0 2. 106 27.79 28.61 25.14 136.69 0.46 3 60.95 60 10a 46.41 90.62 77.44 77.12 76.80 41 72 184 176 168 152 144 136 128 120 96 88 80 72 64 56 48 32 192 160 112 104 40 24 16 Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

Formula C11H13NO2S

223.2914

Comment

FW

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Date

5 mm QNP 1H/15N/13C/31P Z3379/0400

15.07.2011 18:54:39

14 Jul 2011 09:10:24

Acquisition Time (sec) 0.5571 14 Jul 2011 09:10:24 Date Stamp D:\NMR\19.05.2011 С 13 Для Иры Статья в Тетраэдрон\rudn-0611-N14-dept135\rudn-0611-N14-dept135_001000fid Frequency (MHz) 100.62 File Name 13C Number of Transients 1058 Origin spect **Original Points Count** 16384 Nucleus root Points Count 16384 Pulse Sequence dept135 **Receiver Gain** 32768.00 Owner SW(cyclical) (Hz) 29411.77 Solvent CHLOROFORM-d Spectrum Offset (Hz) 9622.9170 Sweep Width (Hz) 29409.97 Temperature (degree C) 27.000 \cap Compound 32 10ba 36.69 rudn-0611-N14-dept135_001000fid 46.40 60.96 79.44 131.57 40.46 28,62 25 4 24 56 96 72 32 184 176 168 160 152 144 136 128 120 112 104 88 80 64 48 40 16 8

General Synthetic Approach towards Annelated 3a.6-Epoxylsoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

15.10.2010 14:37:28 Formula C., H., NO,S FW 301.3171 Acquisition Time (sec) 1.4549 Comment single pulse Date 13 Oct 2010 11:00:43 Date Stamp 13 Oct 2010 10:13:08 D:\NMR\11.10.2010\FZ1390-1.jdf File Name Frequency (MHz) 600.17 Nucleus 1H Number of Transients 8 Origin ECA 600 **Original Points Count** 16384 Owner delta Points Count 16384 Pulse Sequence single pulse.ex2 Receiver Gain 20.00 Solvent DMSO-d6 Spectrum Offset (Hz) 3000.8616 Sweep Width (Hz) 11261.26 Temperature (degree C) 18.800 .OH 21 0 20 19 0 18 Compound 33a la -0 124 H 5 4ba 5 33a 50 N 6.68 Camad CO2H brd S 6.61 6.60 5.09 2.60 3.39 6.53 7.08 6.52 & Merco 04 20140 60 7.02 12.32 2.05 46 N 33 N e 1.01 1.05 2.23 1.03 1.01 1.07 1.02 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 3.5 4.5 4.0 3.0 2.5 2.0 1.5 1.0 0.5 0 Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

15.10.2010 14:37:39

Formula C ₁₅ H ₁₁ NO ₄ S	FW 301.	.3171							
Acquisition Time (sec)	1.4549	Comment	single_pulse	Date	13 Oct 2010	11:00:43		Date Stamp	13 Oct 2010 10:13:08
File Name	D:\NMR\11.1	0.2010\FZ1390-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	8
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single pulse.ex2
Receiver Gain	20.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	3000.8616	Sweep Width (Hz)	11261.26	Temperature (degree C	18.800

Compound 33a



FZ1390-1.jdf



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

15.10.2010 14:37:49

Formula	C15H11NO4S	FW	301.3171
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Acquisition Time (sec)	1.4549	Comment	single_pulse	Date	13 Oct 2010 11:00:43			Date Stamp	13 Oct 2010 10:13:08
File Name	D:\NMR\11.1	0.2010\FZ1390-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	8
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single pulse ex2
Receiver Gain	20.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	3000.8616	Sweep Width (Hz)	11261.26	Temperature (degree C	18 800

Compound 33a



H

3 -

2.9

2.60

GZMQ

P

2.46

FZ1390-1.jdf

5.2

H-2,d (1,6)



3.33



1.01 1.07 1.02 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.6 3.4 3.7 3.5 3.3 3.1 2.6 3.2 3.0 2.9 2.8 2.7 2.5 2.4 Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C15H11NO4S	FW 301.3	3171							
Acquisition Time (sec)	0.6921	Comment	single pulse de	ecoupled gated NOE		Date	27 Oct 201	1 08.52.02	
Date Stamp	27 Oct 2010 0	8:04:42	.	File Name	D:\NMR\22.1	0.10\FZ1423 13C-1 idf	27 001 201	Frequency (MHz)	150.01
Nucleus	13C	Number of Transients	10000	Origin	ECA 600	Original Points Count	32768	Owner	dolta
Points Count	32768	Pulse Sequence	single pulse of	lec		Receiver Gain	56.00	Solvent	DMCO de
Spectrum Offset (Hz)	15091.3428	Sweep Width (Hz)	47348.49	Temperature (degree (22 300	incontrol Cam	50.00	Solvent	DIVISO-06

Compound 33a



FZ1423_13C-1.jdf



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.10.2010 10:06:04

10.

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Formula	C15H11NO4S	FW	301.3171
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Acquisition Time (sec)	0.6921	Comment	single pulse de	coupled gated NOE		Date	27 Oct 2010	0 08:52:03	
Date Stamp	27 Oct 2010 0	8:04:42		File Name	D:\NMR\22.1	0.10\FZ1423_13C-1.jdf		Frequency (MHz)	150.91
Nucleus	13C	Number of Transients	10000	Origin	ECA 600	Original Points Count	32768	Owner	delta
Points Count	32768	Pulse Sequence	single_pulse_d	ec		Receiver Gain	56.00	Solvent	DMSO-de
Spectrum Offset (Hz)	15091.3428	Sweep Width (Hz)	47348 49	Temperature (degre	e C) 22 300				

Compound 33a



301.3171

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.10.2010 10:06:27

Acquisition Time (sec)	0.6921	Comment	single pulse de	ecoupled gated NO	E	Date	27 Oct 2010	0 08:52:03	
Date Stamp	27 Oct 2010 08	8:04:42		File Name	D:\NMR\22.1	0.10\FZ1423_13C-1.jdf		Frequency (MHz)	150.91
Vucleus	13C	Number of Transients	10000	Origin	ECA 600	Original Points Count	32768	Owner	delta
Points Count	32768	Pulse Sequence	single_pulse_	dec		Receiver Gain	56.00	Solvent	DMSO-d6
pectrum Offset (Hz)	15091.3428	Sweep Width (Hz)	47348.49	Temperature (d	degree C) 22.300			.6. e H	
					Compour	nd 33a	7	$5a^{-5}$ (4ba $4b^{-4a}$ $4b^{-4a}$ $4a^{-4}$ $4a^{-4}$ $4a^{-4}$ $4a^{-4}$ $4a^{-4}$ 10^{-11} $11a^{-12}$	3
1423_13C-1.jdf								0 18 HO 21	≥ _O 20
49								1,1	La
~								\sim	
1.68								C)
თ		2			46			54,85	
		2							
		81.9			67.29			^	
						145			

General Synthetic Approach towards Annelated 3a,6-Epoxylsoindoles by Tandem Acylation/IMDAF Reaction

08.12.2010 20:10:48

07 Dec 2010 11:34:24

single_pulse.ex2

5 5 55

Formula C16H13NO4S	FW 31	5.3437							
Acquisition Time (sec)	1.0905	Comment	single_pulse	Date	07 Dec 2010	12:22:41		Date Stamp	0
File Name	D:\NMR\06.1	2.10\FZ1525-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transient	s 10
Origin	ECA 600	Original Points Cour	nt 16384	Owner	delta	Points Count	16384	Pulse Sequence	si
Receiver Gain	44.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	4201.2061	Sweep Width (Hz)	15024.04		0.
								OOH	1027
								HC	O /,18
				8 -	Compo	ound 33b		$\frac{1}{2} - \frac{1}{2} - \frac{1}$	11
								3 12 4a	1 / 4b
								⁴ H	TO I
33b								4ba	1
and the second second second									
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					6.63	с. С		-2.5	
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andy				5.65 5.65	33		42	64	
				7.4.7.2	0.0		ri v	in 19	
620 S				000				1	
34				7.02					
12									

1 1 14 0.95 1.02 2.19 1.97 1.03 1.00 0.95 3.01 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 2.5 3.0 2.0 1.5 1.0 0.5 0 Chemical Shift (ppm)

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

08.12.2010 20:11:00

cquisition Time (sec)	1.0905	Comment	single_pulse	Date	07 Dec 2010	12:22:41		Date Stamp	07 Dec 2010 11:34:24
le Name	D:\NMR\06.12	.10\FZ1525-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	10
rigin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
ceiver Gain	44.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	4201.2061	Sweep Width (Hz)	15024.04		
525_1 idf					Compo	ound 33b	1	$H_{3}C$ J_{1} J_{2} J_{2} J_{1} J_{2} J_{2} J_{1} J_{2} $J_{$	$ \begin{array}{c} 0 \\ 18 \\ 1 \\ 1 \\ 9a \\ 9a \\ 9a \\ 8 \\ 5 \\ 5 \\ 6 \\ 7 \\ 5 \\ 6 \\ 7 \\ 9a \\ 8 \\ 8 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10$
:5-1.jdi									
				2			H-46	2,5	
H-9	cel	H-6, d	Н	-8, t H-7	,t		5.63	4-3,4	
(7,7))	(7,7)	(7,7) (7.7)		(d 5,8)		d. (5,8)
7.47 7.45	2	-7.29 7.28		7.08 7.03			6.65		6.33 6.33
				7.09					
1.02		1.08		2.19			1.97		1.03

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

equisition Time (sec)	1.0905	Comment	single_pulse	Date	07 Dec 2010	12:22:41		Date Stamp	07 Dec 2010 11:34:24
le Name	D:\NMR\06.12	2.10\FZ1525-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	10
rigin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
ceiver Gain	44.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	4201.2061	Sweep Width (Hz)	15024.04	O 21 20 OH 22	O /18
				(Compo	und 33b]	H_3C 2 O $1a$ O $1a$	N 92 9 8
									5^{4b} 10 10 10 10 10 10 10 10
525-1.jdf								4ba	1.55
								Ş	s, CH3-2
				1.1					34
2, d				25a, d	\$ ØW(20			
$(1, \mathcal{O})$)			(g,1)	2.5				
3.42							14-1		
					S				, jt
1.00				0.95					3.01

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.04.2011 19:33:13



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.04.2011 19:31:58

Formula C ₁₅ H ₁₁ NO ₄ S	FW 301.3171						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	14 Apr 2011 06:56:00
Date Stamp	14 Apr 2011 06:56	:00		File Name	D:\NMR\13.04.201	1 C-13\rudn-130411-N8-d	lept135\rudn-130411-N8-dept135_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	201	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9102.1396
Sween Width (Hz)	29409 97	Temperature (degree C	32 000				

Compound 33b



rudn-130411-N8-dept135_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

5 Feb 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

5 Feb 2011



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

5 Feb 2011



Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.04.2011 20:42:51 FW 380.2132 Formula C., H., BrNO,S 15 Apr 2011 08:12:48 Acquisition Time (sec) 0.5571 5 mm QNP 1H/15N/13C/31P Z3379/0400 Date Comment D:\NMR\13.04.2011 C-13\rudn-130411-N9-c13dec\rudn-130411-N9-c13dec 001000fid Date Stamp 15 Apr 2011 08:12:48 File Name 13C 977 Frequency (MHz) 100.62 Nucleus Number of Transients Origin spect Points Count 16384 Pulse Sequence **Original Points Count** 16384 Owner root zgpg 29411.77 DMSO-d6 Spectrum Offset (Hz) 10548,9658 32768.00 SW(cyclical) (Hz) Solvent **Receiver Gain** Sweep Width (Hz) 29409 97 Temperature (degree C) 90.000 .OH O. 21 20' Compound 33c Br 19 -0 6,7,8 53 rudn-130411-N9-c13dec_001000fid 5 57.28 25.34 ; 125.40 150 2,49 39.75 39.32 00,H:11 07 52. 46 89.45 3 169.26 122.83 140.74 136.03 115.44 50,99 66.47 DMF 39.94 39.12 35.75 米 134.67 31.71 OMF 30.75 × 167.89 90.45 62.23 40.16 38.91 Unclassing Windschahl Albert Washingtoold sabirin Weismuch along an 160 152 136 128 120 112 96 88 80 72 64 56 48 40 32 168 144 104

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.04.2011 20:49:18

Formula C ₁₅ H ₁₀ BrNO ₄ S		FVV 3	80.2132				
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1	H/15N/13C/31P Z3379/0400		Date	15 Apr 2011 08:12:48
Date Stamp	15 Apr 2011 0	8:12:48		File Name	D:\NMR\13.04	.2011 C-13\rudn-130411-N9-c	c13dec\rudn-130411-N9-c13dec_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	977	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10548.9658
Sween Width (Hz)	29409 97	Temperature (deo	aree C) 90 000				

Compound 33c



rudn-130411-N9-c13dec_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.04.2011 20:41:47 Formula C15H10BrNO4S FW 380.2132 Acquisition Time (sec) 0.5571 Comment 5 mm QNP 1H/15N/13C/31P Z3379/0400 Date 15 Apr 2011 08:36:16 15 Apr 2011 08:36:16 D:\NMR\13.04.2011 C-13\rudn-130411-N9-dept135\rudn-130411-N9-dept135_001000fid Date Stamp File Name Frequency (MHz) 100.62 Nucleus 13C 443 Number of Transients Origin spect **Original Points Count** 16384 Owner root **Points Count** 16384 **Pulse Sequence** dept135 **Receiver Gain** 32768.00 SW(cyclical) (Hz) 29411.77 Solvent DMSO-d6 Spectrum Offset (Hz) 9098.5479 Sweep Width (Hz) 29409.97 Temperature (degree C) 90.000 .OH 0 Br Compound 33c 19 57.28 115.42 rudn-130411-N9-dept135_001000fid 125.40 122.83 66.47 52.06 36.03 140.74 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 Chemical Shift (ppm)

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

1 Feb 2011

Acquisition Time (sec)	1.6056	Comment	Imported from UXNMR.			Date	31 Jan 2011 14:06:56	
File Name	C:\Users\Fea	dor\Desktop\31.01.11-1\fz1614\	fz1614_001000fid			Frequency (MHz)	400.14	
Nucleus	1H	Number of Transients	32	Original Points Count	16384	Points Count	16384	
Pulse Sequence	zg	Solvent	CHLOROFORM	-D		Sweep Width (Hz)	10204.08	
Temperature (degree C	27.000							

Compound 34*





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

1 Feb 2011

quisition Time (sec)	1.6056	Comment	Imported from	n UXNMR.		Date	31 Jan 2011 14:06:56	11002
le Name	C:\Users\Fed	or\Desktop\31.01.11-1\fz	1614\fz1614_001000	Jfid	·	Frequency (MHz)	400.14	
icleus	1H	Number of Transie	ents 32	Original Points Co	unt 16384	Points Count	16384	
Ilse Sequence	zg	Solvent	CHLOROFO	RM-D		Sweep Width (Hz)	10204.08	
				C LI-6	compound a	34*	6 7 2 2 5 4 5 4 1	3' 4' 5' 5' 5' 5' 5' 5' 5' 5' 5' 5' 5' 5' 5'
		7	1-7 1,21 1d	7,13 dt (1.0;7.5)			0 11 11 11 11 11 11 11 11 11 11 11 11 11	⁸ —H CIS
		(1	.0;7,5)	H-5			ω	
		H-S' bzds CH	ces (7.07 dt (2.0;7.5)	E H	H-trans	H-3', H-4' M. 2H	Hais,
		7.33 7.25 🏈			dd	(1,6',16.8)	9 - 11	(1.6;10.
H-4	,		21 7.13 11 7.09 7.07	4-2	(10.3;16 8)	0		5.86 5.83
0.1017	2	CC 1	7.15 7.05	88 88	5.70 5.67 6.66 6.63	6.5		
7.65			VE JEHN	Q				
0.78		0.96	0.97 2.10	0.90	2.15	5	2.00	1.07

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

1 Feb 2011

cquisition Time (sec)	0.5898 C:\Llsers\Fedor\De	Comment	Imported from UXN	IMR.	Date Erequency (MHz)	31 Jan 2011 14:47:28
ucleus	13C	Number of Transients	360	Original Points Count 16384	Points Count	16384
ulse Sequence	ząpą	Solvent	CHLOROFORM-D		Sweep Width (Hz)	27777.78
emperature (degree C)	27.000					
			5,6,7	Compound 34	1*	$ \begin{array}{c} $
			3".			O 11 CH ₂ " CH ₂ "
			-130.27 	3',41		ί.
		1				
		5		10.38		
		142.89	28.08	1		
			12	- ¹¹		2
(1")			L	1,2		
00	2)			470		61.03
r			22.92	1		
64.09	152.02	301		9.03		0 2
<u>-</u>		13		-		77.44
		-137				
here and the second	والمتحاريق والمارية والمحمون والمراح فالمعاوم	skins respectively and an and and	and the stand of the second of the	monoral management and a direction where and	งางสังหากกระบบการทรงสาวรักษาสาสกัจหาเป็นระบัการใช้สุดสำคัญสราวรักษา	record and the second

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

1 Feb 2011

Acquisition Time (sec)	0.5571 C:\Users\Fedor\Des	Comment sktop\31.01.11-1\fz1614de	Imported from UXN pt135\fz1614dept135	MR. 002000fid	Date Frequency (MHz)	31 Jan 2011 14:56:00 100.62
lucleus	13C	Number of Transients	539	Original Points Count 16384	Points Count	16384
ulse Sequence	dept135	Solvent	CHLOROFORM-D		Sweep Width (Hz)	29411.77
emperature (degree C)	27.000	5,44		Compound 34*	6	7 7 7 7 7 7 7 7
	142.89	128.07 125.18 1291	113.03	110.39		
		130.28				

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

26 Jan 2011

Acquisition Time (sec)	1.6056	Comment	Imported from I	VNMP		1 2/2/	10 A	
File Name	C:\Llsers\Fedor\Deskton\27.12.10\fractCt2\textcol27.12.10\fractCt2\text				Date		24 Jan 2011 18:05:52	
Number of Taxain	C. 10 Sets (1 edot 10 esktop 127.12.10 (12 16 12 (12 16 12) 001000 fid			Frequency (MHz) 400.1	400.14	Nucleus	1H	
Number of Transients	40	Original Points Count	16384	Points Count	16384	Pulse Seguence	70	
Solvent	CHLOROFORM-D			Sugar Medite (11-)	10001 00	i uise sequence	29	
				Sweep width (Hz)	10204.08	Temperature (degree C) 7	e C) 27.000	

Compound 34





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

26 Jan 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

26 Jan 2011



Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

18 Jul 2011



325

18 Jul 2011



18 Jul 2011

Acquisition Time (sec)	0.5898	Comment	Imported from UXNMF	R.		Date	14 Jul 2011 09:36:00
File Name	D:\NMR\19.05.2011 C	13 Для Иры Статья в Тет	раэдрон\rudn-0611-N2	-c13dec\rudn-0611-N2-c13	dec_001000fid	Frequency (MHz)	100.62
Nucleus	13C	Number of Transients	746	Original Points Count	16384	Points Count	16384
Pulse Sequence	zgpg	Solvent	CHLOROFORM-D	Sweep Width (Hz)	27777.78	Temperature (degree (c) 27.000

Compound 34



122.52

122.5

123.0

122.0

25.40

125.5

126.0

.2C

125.0

124.5

Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

123.5

124.0

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

20.10.2010 15:53:22

uisition Time (sec)	2.9098	Comment	single_pulse	Date	18 Oct 2010	15:17:16		Date Stamp	18 Oct 2010 14:29:45
Name	D:\NMR\18.1	0.2010\fz1399-1.jdf	22700	Frequency (MHz)	600.17	Nucleus Deinte Compt	1H	Number of Transients	2 ciacle sules sul
gin Joiwar Gain	36 00	Solvent	DMSO de	Spectrum Offect (Hz)	3000 9616	Swoon Width (Hz)	32/08	Puise Sequence	single_pulse.exz
					Comp	oound 35		$\begin{bmatrix} & & & & \\ & & & & \\ & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & $	
								18 20	0 21 CH ₃
A							56		
							ë		
	6.69								2.50 &
		6.62		133			8.47 3.46	2.76	2.74
7.40 7.28 7.28 7.28 7.08 7.08	7.05 7.05 7.04 7.04	6.53 6.53 6.52 6.52		2 2			3.54		
		1					llla		
1.010.99 2.2	21 1.0	1 1.03		1.00			1.04	1.0	8
7.5	7.0	6.5 6.0	0	5.5 5.0 Chemic	al Shift (ppm)	4.5 4.0	3.5	3.0	2.5

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

cquisition Time (sec) ile Name	2.9098 D:\NMR\18.1	Comment 10.2010\fz1399-1.jdf	single_pulse	Date Frequency (MHz)	18 Oct 2010 600.17	15:17:16 Nucleus	1H	Date Stamp Number of Transients	18 Oct 2010 14:29:45
rigin	ECA 600	Original Points Count	32768	Owner	delta	Points Count	32768	Pulse Sequence	single pulse.ex2
eceiver Gain	36.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	3000.8616	Sweep Width (Hz)	11261.26	Temperature (degree C	21.100
					Comj	pound 35		$S_{a} = S_{b} = S_{b$	
						s, 3H, Ce	ZMe	// 0 18 20	0 21 CH ₃
399-1.jdf						3.56			1
		/							
11-2							٨	14-	10
MC						4-110	d	Pr	2114
1						11 119	0	CO	(x)
~						(q, l)	2		(,2)
(1,7)						CSII	1)	On
									Å.
									2.50
0° 00								6	4
5.1.						3.47		2.7	2.7
						3.54			
						A H			
1.00						a sea a su			u fur

Formula C LI NO C EW

315 3437

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

20.10.2010 15:53:31

Acquisition Time (sec)	2.9098	Comment	single_pulse	Date	18 Oct 2010	15:17:16		Date Stamp	18 Oct 2010 14:29:45
File Name	D:\NMR\18.	10.2010\fz1399-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	2
Origin	ECA 600	Original Points Count	32768	Owner	delta	Points Count	32768	Pulse Sequence	single pulse.ex2
Receiver Gain	36.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	3000.8616	Sweep Width (Hz)	11261.26	Temperature (degree C) 21,100

Compound 35



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

08.11.2010 18:53:39



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

08.11.2010 18:53:49

Formula	C.,H.,NO,S	FW	315.3437
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Acquisition Time (sec)	0.6921	Comment	single pulse d	ecoupled gated NOE		Date	03 Nov 201	0 13:36:58	
Date Stamp	03 Nov 2010 1	2:49:03		File Name	D:\NMR\22.1	0.10\FZ1424_13C-1.jdf		Frequency (MHz)	150.91
Nucleus	13C	Number of Transients	130	Origin	ECA 600	Original Points Count	32768	Owner	delta
Points Count	32768	Pulse Sequence	single_pulse_	dec		Receiver Gain	56.00	Solvent	DMSO-d6
Spectrum Offset (Hz)	15091.3428	Sweep Width (Hz)	47348.49	Temperature (degree	e C) 24.100				

Compound 35



FZ1424_13C-1.jdf

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

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08.11.2010 18:53:59

ate Stamp ucleus oints Count vectrum Offset (Hz)	03 Nov 2010 12 13C 32768	2:49:03 Number of Transients		File Name	D:\NMR\22 10	101E71404 120 1 H		Examples ou (MUa)	150.01
ucleus pints Count pectrum Offset (Hz)	13C 32768	Number of Transients			D. 11 11 11 122. 10.	10/FZ1424_130-1.jur		Frequency (WHZ)	150.91
oints Count bectrum Offset (Hz)	32768		130	Origin	ECA 600	Original Points Count	32768	Owner	delta
pectrum Offset (Hz)		Pulse Sequence	single_pulse_d	lec		Receiver Gain	56.00	Solvent	DMSO-d6
	15091.3428	Sweep Width (Hz)	47348.49	Temperature (degree	C) 24.100				
1					Compo	ound 35		$S_{a} = S + H_{a} + $	3
49								21 CH ₃	20
424_1 3 C-1.jdf ත									
				1.			1,150	9	(0
				48					0.0
									4
						(20
				2					40
				37.3			N.17	5	
							OS M	33	
		0					+	46	
		L				0	<u>~</u>		
						55.0	2.0		
		22				Ť	2		34
		20							40.
									- S. F.
									48
									.97
									40
1.1									
and the fore and a second s	Marsh Breaktonder What Sales	a province the second second second second second second	And the second	and a second state in the strange of the strange of the strange	An al source to be taken on a submersion of	Devent in strand sold and an and a strand strand strand strand strand	AND Sectored as which We was sectored	with the burnship of the with	Although the state of the
Hard Party Party	and the second second second second	A STATE OF A	the second states	Hard and a star of the second	and the second	2010 10 10 10 10 10 10 10 10 10 10 10 10	and a second second	A REAL PROPERTY OF A REA	and a starting of the start of



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations. 334 17 73.85 73.85 73.85 73.83 73.81 -3.81 -3.81 -3.81 -3.69 -3.69 -3.67 -3.67 -3.67 -3.63 -3.57 -2.4 444444444 -2.3 H-ga, -2.2 4-2 ÇH3 Compound 36a -2.1 COZME, S, 3H -2.0 -1.9 H-29 H-9 -1.8 -1.7 3.3) 3 3 -1.6 -1.5 -1.4 -1.3 -1.2 -1.1 -1.0 4-29 H-8e + H-6e-m, 2H -0.9 -0.8 -0.7 2,2/12.8 -0.6 -0.5 -0.4 (22:50:12,6) 7:5,0:13,2 -0.3 -0.2 -0.1-0.0 --0.1 Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov 0.92-1.03 0.93 8 2.79 0.89 93 --0.2



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

23 Dec 2008



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

23 Dec 2008



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23 Dec 2008



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22 Jun 2009



22 Jun 2009



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22 Jun 2009



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22 Jun 2009

equisition Time (sec)	0.6226	Comment	Imported from UX	NMR.	Date	19 Jun 2009 16:59:44	
le Name	C:\Users\Fedor\	Desktop\19.06.09\laz20c13de	c\laz20c13dec_00	1000fid	Frequency (MHz)	100.62	
icleus	13C	Number of Transients	144	Original Points Count 16384	Points Count	16384	
Ilse Sequence	zgpg	Solvent	CHLOROFORM-	D	Sweep Width (Hz)	26315.79	
inperatore (degree C)	21.000			Compou	nd 36b		
36		8		19,3,30,9c, Cozh	le =	$H^{*}_{9aa} \xrightarrow{9b}{0} 3a$ $9c \xrightarrow{10}{0} 12$ $10 \xrightarrow{10}{18} 0$ $20 \xrightarrow{11}{18} 0$ $20 \xrightarrow{11}{18} 0$ $20 \xrightarrow{11}{18} 0$	3
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85.8	77.7						
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13 May 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13 May 2011



13 May 2011

Acquisition Time (sec)	1.5729	Comment	Imported from UX	NMR.		Date	06 May 2011 10:20:48
File Name	D:\NMR\C_13\29.	04.11 (C-13 Рома)\rudn-29	0411-N15\rudn-29	0411-N15 002000fid		Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	12	Original Points Count	16384	Points Count	16384
Pulse Sequence	zg	Solvent	DMSO-D6	Sweep Width (Hz)	10416.67	Temperature (degree C)	32.000

Compound 36c





13 May 2011

cquisition Time (sec)	0.5571 DUNIMENC 12020.04	Comment	Imported from UXN	MR.	0054	Date	06 May 2011 11:24:48	
	13C	Number of Transients	230	Original Points Count	16384	Prequency (WHZ)	16384	
cieus Iso Soguenco	7000	Solvent	Z39 DMSO D6	Swoon Width (Hz)	20/11 77	Tomporature (degree)	C) 27 000	
				Comp	ound 36c	2 a	$\begin{array}{c} & O \\ & & 0 \\ & & 0 \\ & & 0 \\ & & 0 \\ & & 0 \\ & & 0 \\ & & 0 \\ & & 9 \\ & & 9 \\ & & 9 \\ & & 9 \\ & & 9 \\ & & 9 \\ & & & 9 \\ \end{array}$	6 _7
						0. 1 0.66.65	88.18 H, 0 H, 0 H, 0 H, 0 H, 0 H, 0 H, 0 H, 0	30.47
					84.72		47.02	N
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Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladiff 中. Zaytsev, Vietov N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13 May 2011



13 May 2011 Acquisition Time (sec) Imported from UXNMR. Date 06 May 2011 11:31:12 0.5571 Comment Frequency (MHz) D:\NMR\C_13\29.04.11 (C-13 Poma)\rudn-290411-N15-dept135\rudn-290411-N15-dept135_001000fid 100.62 File Name **Original Points Count** 16384 Points Count 16384 Nucleus 13C Number of Transients 184 Temperature (degree C) 32.000 DMSO-D6 Sweep Width (Hz) 29411.77 Pulse Sequence dept135 Solvent 47.03 Compound 36c 75.68 49.10 46.42 84.73 38.16 66.65 24.99 30.47 30 Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir Przytsewift Vistor, N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov 25 20 85 80

11.05.2012 15:43:54

Date Stamn Dec	40 0007						
Date Stamp Dec	- 13 2007 F	-ile Name	D:\NMR\C_13	ТУРЧИН зима 2007-08\Гузе	ль № 15\Guzel	15\FZGuz15-h_131207	
Frequency (MHz) 399.9	.96 ^	Vucleus	1H	Number of Transients	32	Original Points Count	29999
Points Count 3276	68 F	Pulse Sequence	s2pul	Receiver Gain	40.00	Solvent	CHLOROFORM-d
Spectrum Offset (Hz) 1611	1.3262 S	Sweep Width (Hz)	5999.70	Temperature (degree C	29.000		



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.05.2012 15:44:09

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Acquisition Time (sec)	5.0001	Comment	ENOTHERA-	56_02.12.05		Date	Dec 13 2007
Date Stamp	Dec 13 2007	File Name	D:\NMR\C_1	3\ТУРЧИН зима 2007-08\Гузе	ль № 15\Guzel15	5\FZGuz15-h_131207	
Frequency (MHz)	399.96	Nucleus	1H	Number of Transients	32	Original Points Count	29999
Points Count	32768	Pulse Sequence	s2pul	Receiver Gain	40.00	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	1611.3262	Sweep Width (Hz)	5999.70	Temperature (degree C) 29.000		
Spectrum onset (nz)	1011.5202		0000.10	Co	ompound	1 37a	H ₃ C 0 9 9b





11.05.2012 15:44:27

quisition Time (sec)	5.0001	Comment	ENOTHERA	-56_02.12.05		Date	Dec 13 2007	
te Stamp	Dec 13 2007	File Name	D:\NMR\C_1	13\ТУРЧИН зима 2007-08\Гузе	ль № 15\Guzel	15\FZGuz15-h_131207		
equency (MHz)	399.96	Nucleus	1H	Number of Transients	32	Original Points Count	29999	
ints Count	32768	Pulse Sequence	s2pul	Receiver Gain	40.00	Solvent	CHLOROFORM-d	
ectrum Offset (Hz)	1611.3262	Sweep Width (Hz)	5999.70	Temperature (degree C) 29.000			
	3.10, H	-6a 4,5; 11,5)		Co	ompoun	s, $COMe \times 2$	$\begin{array}{c} H_{3}C \\ 0 \\ H_{3}C \\ 0 \\ 0 \\ H_{3}C \\ H_{3}C \\ 0 \\ H_{3}C \\ 0 \\ H_{3}C \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ $	
						\sim	$\begin{array}{c} \begin{array}{c} 0 \\ 19 \end{array} \begin{array}{c} 1 \\ 2 \end{array} \begin{array}{c} 1 \\ 10aa \\ 3 \end{array} \begin{array}{c} 1 \\ 3 \end{array}$	23
uz15-h_131207						1.99		
3.09	3.09 dd	, H-7	1120				H-39x	4-30
3.10	(1.5; 11.3	5) H-40 dt	10				ha	(13.6)
10		(0.01)	2 1				1.41	m
2		(3,5)	21)				1	1
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3.17		2.74 2.74 2.71 2.68	-2.67			1.85 1.85	1.84 1.83 1.83 1.79 1.76 1.76	1.45 1.45 1.45 1.45
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Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.05.2012 15:55:26

lie Name D.VMR(C, 1317):PH41 awa 2007.06/17.926. № 15/02.02/15-200.131207 Prequency (MHz) 100.58 Unders 513C Number of Transients 8192 Original Polints Count 32768 25.00 Solvent CPULOROCORNA peedrum Offset (Hz) 9466.8164 Sweep Width (Hz) 27008.78 Temperature (degree C) 29.000 Compound 37a H12 (-) (-) (-) (-) (-) (-) (-) (-) (-) (-)	cquisition Time (sec)	1.0000	Comment	13C OBSERVE	Date	Dec 13 2007	Date Stamp	Dec 13 2007	
Underson 13C Number of Transients 8192 Original Points Count 27009 Points Count 33783 ubles Sequence 4504 Receiver Gall 3000 Solvent CHURPCORMA 33783	ile Name	D:\NMR\C_13\	ТУРЧИН зима 2007-08\Гузе	ль № 15\Guzel15\	FZGuz15-cpp_131207		Frequency (MHz)	100.58	
$\frac{1}{26uc} \frac{82pul}{16e} 8$	ucleus	13C	Number of Transients	8192	Original Points Count	27009	Points Count	32768	
Spectrum Offset (Hz) 9466.8164 Sweep Width (Hz) 27008.78 Temperature (degree C) 29.000 Compound 37a $H_{2}^{c} (f_{2}^{c}) (f_{1}^{c}) (f_{1}^{c}) (f_{2}^{c}) (f_{1}^{c}) (f_{1}^{c}) (f_{2}^{c}) (f_{1}^{c}) (f_{1}^{c}) (f_{2}^{c}) (f_{1}^{c}) ($	ulse Sequence	s2pul	Receiver Gain	58.00	Solvent	CHLOROFORM-	d		
Securits-copp_131207 $Compound 37a \qquad \qquad$	pectrum Offset (Hz)	9466.8164	Sweep Width (Hz)	27008.78	Temperature (degree C) 29.000			
2G0212-cob ⁻ 131502					C	ompound	. 37a	$\begin{array}{c} H_{3}C_{2} & 0 & H_{3}C_{2} \\ 0 & 0 & 9 & 9b \\ 0 & 22 & 0 & 8 & 7a & 0 \\ 0 & 10 & 9a & 6a & 0 \\ 0 & 10 & 9a & 6a & 0 \\ 0 & 10 & 0 & 24 & CH_{3} \\ 0 & 0 & 10 & 0 & 24 \\ 0 & 0 & 10 & 10 & 0 \\ 0 & 19 & 1 & H & 5 & 23 \\ 0 & 19 & 1 & H & 5 & 23 \\ 0 & 10 & 10 & 10 & 10 \\ 0 & 10 & 10 &$	
analaction 13150								-3-	
	Guz15-cpp_131207							.24	
						25		45	
						76.			
						31.5			
163.70 163.70 164.72 164.72 164.74 164.74 164.74 164.44 17.22 164.44 17.22 164.44 17.22 164.44 17.22 164.44 17.25 164.44 17.25 164.44 17.25 164.44 17.25 165.44 17.75 165.46 17.75						8			
							4	80	
							8 [.] 4 [.]	24.8	
							9	4.7.94	
						4		33	
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102.18 102.18 102.18 102.18 102.18 102.18						8		0.37	
		70						5	
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		14.4			8			21	
		167			5.				
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						22 6.9			
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	and a state of the second s	a sund del si forma si fiel es ci stan	าระวดที่ เหมือนที่มารถใน พัตร์ และสาวารถายสารที่สารที่สาวที่	all i Chengra mila Ma Lauran all agas.	erale editeration attraction and and a second second	بطرير وملططمة بالقيمين بالرجيد أحاكيه	dente de la construcción de la cons La construcción de la construcción d	يطابية وميدوليو أواد مادهات عاديا أندار بالدياب وموادلة فالمالية فالمطالبة فالعظينا بغردانا بما التصاد المتباطعة رتبت	North Andrews Adds

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.05.2012 15:55:32

Formula C ₁₇ H ₂₁ NO ₉	FW 383.349	9						
Acquisition Time (sec)	1.0000	Comment	13C OBSERVE	Date	Dec 13 2007	Date Stamp	Dec 13 2007	
File Name	D:\NMR\C_13\T	УРЧИН зима 2007-08\Гузе	ль № 15\Guzel15\	FZGuz15-cpp_131207		Frequency (MHz)	100.58	
Nucleus	13C	Number of Transients	8192	Original Points Count	27009	Points Count	32768	
Pulse Sequence	s2pul	Receiver Gain	58.00	Solvent	CHLOROFORM	Λ-d		
Spectrum Offset (Hz)	9466.8164	Sweep Width (Hz)	27008.78	Temperature (degree C)	29.000			

Compound 37a



FZGuz15-cpp_131207



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173.5 173.0 172.5 172.0 171.5 171.0 170.5 170.0 169.5 169.0 168.5 168.0 167.5 167.0 166.5 166.0 165.5 165.0 164.5 164.0 163.5 163.0 162.5 162.0 161.5 161.0 160.5 Chemical Shift (ppm)

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.05.2012 15:55:41

cquisition Time (sec)	1.0000	Comment	13C OBSERVE	Date	Dec 13 2007	Date Stamp	Dec 13 2007	
le Name	D:\NMR\C_13\T}	/РЧИН зима 2007-08\Гузе	ель № 15\Guzel15\	FZGuz15-cpp_131207		Frequency (MHz)	100.58	
ıcleus	13C	Number of Transients	8192	Original Points Count	27009	Points Count	32768	
Ise Sequence	s2pul	Receiver Gain	58.00	Solvent	CHLOROFORM-	d		
ectrum Offset (Hz)	9466.8164	Sweep Width (Hz)	27008.78	Temperature (degree C)) 29.000			
				Co	ompound	. 37a	$\begin{array}{c} H_{3}C & O \\ & 0 \\ & 0 \\ & 0 \\ & 0 \\ & 0 \\ & 0 \\ & 0 \\ H_{3}C \\ & 10 \\ &$	$ \begin{array}{c} H \\ 9b \\ 9a \\ 9a \\ 6a \\ 0 \\ 24 \\ N \\ 0 \end{array} \begin{array}{c} H \\ 0 \\ 0 \\ 0 \\ 0 \\ 1 \end{array} \begin{array}{c} 0 \\ 0 \\ 0 \\ 0 \\ 1 \end{array} $
					0	9	$\begin{array}{c} 0 & 1 & H \\ 19 & 2 & 10a \\ 3 & 3 \end{array}$	5 23 a 4
uz15-cpp 131207					X	25		-
					25	-76.		2
					81.5			
				100				44
				109				68.
				24				
10				85.				
10								
~								
2.18								
10				1.12		0		
						6.9		
						.77		
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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.05.2012 15:55:53



37b

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

21.05.2011 20:40:34

Formula	C15H19NO7	FW	325.3139
	10 10 1	Les a les e	

Acquisition Time (sec)	2.1837	Comment	single_pulse	Date 17 May 2011 11:43:12				Date Stamp	17 May 2011 15:31:23
File Name	D:\NMR\16.05	.11\FZ1857-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	10
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	38.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	7503.00
	10.000								

Temperature (degree C) 19.000

Compound 37b

H₂C H-C 15 H.u., 10aa O H Qan ö 21 2.08 2.04



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

15 Jun 2011



358

15 Jun 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

15 Jun 2011 Acquisition Time (sec) 1.6056 Comment Imported from UXNMR. Date 09 Jun 2011 17:53:04 C:\Users\Fedor\Desktop\19.05.2011 C 13 Для Иры Статья в Тетраэдрон\N-10\rudn-300511-N10\rudn-300511-N10\rudn-300511-N10_001000fid File Name Frequency (MHz) 400.14 Nucleus 1H Number of Transients 32 **Original Points Count** 16384 **Points Count** 16384 **Pulse Sequence** zg Solvent CHLOROFORM-D Sweep Width (Hz) 10204.08 Temperature (degree C) 27.000 2.07 2.02 ,Η $H_3($ "H Me-10 6 \cap " H O Compound 37b H₃C 0 H H-ga, brd m H-3eg H-4ax (3.1 - Fendo 000 60 3ax 13.1 (3.1) 0 13.7 12.1: 3 2 7. 3. 3.21 2.14 2, 2.77 1.48 2.06 45 2.92 2.91 2.89 2.87 2.88 2.10 2.80 2.79 2.73 2.17 88 1.99 94 2.08 2.23 2.18 83 95
General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.05.2012 21:52:37

Formula C15H19NO7	FW 325.3139						
Acquisition Time (sec)	0.5898	Comment	5 mm QNP 1H/1	15N/13C/31P Z3379/0400		Date	09 Jun 2011 18:08:00
Date Stamp	09 Jun 2011 18:08:00						
File Name	D:\NMR\19.05.2011 C	13 Для Иры Статья в Тетр	аэдрон\N-10\rudr	n-300511-N10-c13dec\rudn-3005	11-N10-c13dec\rudn-30	0511-N10-c13dec_001000	fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	3000	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	27777.78	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9637.6885
Sweep Width (Hz)	27776.08	Temperature (degree C)	27.000				



0.000.00

Automatic Management

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.05.2012 21:53:02

Formula C ₁₅ H ₁₉ NO ₇	FW 325.3139				ŕ		
Acquisition Time (sec)	0.5898	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	09 Jun 2011 18:08:00
Date Stamp	09 Jun 2011 18:08:00						
File Name	D:\NMR\19.05.2011 C	13 Для Иры Статья в Тетр	аэдрон\N-10\ru	dn-300511-N10-c13dec\rudn-3005	11-N10-c13dec\rudn-30	00511-N10-c13dec_001000	fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	3000	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	27777.78	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9637.6885
Sweep Width (Hz)	27776.08	Temperature (degree C)	27.000				



Compound 37b

rudn-300511-N10-c13dec_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.05.2012 21:53:17

H2(

Formula C15H19NO7	FW 325.3139						
Acquisition Time (sec)	0.5898	Comment	5 mm QNP 1H/15N	/13C/31P Z3379/0400		Date	09 Jun 2011 18:08:00
Date Stamp	09 Jun 2011 18:08:00						
File Name	D:\NMR\19.05.2011 C	13 Для Иры Статья в Тет	раэдрон\N-10\rudn-30	00511-N10-c13dec\rudn-3005	11-N10-c13dec\rudn-30	0511-N10-c13dec_001000	fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	3000	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	27777.78	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9637.6885
Sweep Width (Hz)	27776.08	Temperature (degree C) 27.000				

Compound 37b



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.05.2012 13:30:20



General Synthetic Approach towards Annelated 3a,6-Epoxylsoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.04.2011 16:05:28

cquisition Time (sec)	1.4549	Comment	single_pulse	Date	16 Dec 2010	14:33:23		Date Stamp	16 Dec 2010 13:45:15
ile Name	D:\NMR\13.1	2.10\FZ1541-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	10
rigin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
eceiver Gain	54.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	3601.0339	Sweep Width (Hz)	11261.26		
1541-1.jdf 15	-89 \$	4			Со	mpound 38a H-2	d	$\begin{array}{c} 2 \\ 0 \\ 1 \\ 0 \\ 0 \\ 1 \\ 0 \\ 0$	
						(4.8)		H-6 ddd	A
					з	4.61		(2,8;	6:12.6)
							έŝ,		4.18 4.17 4.16 4.16 4.16 4.15 4.14
0.93						0.95			1.01

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.04.2011 16:06:19

equisition Time (sec)	1.4549	Comment	single_pulse	Date	16 Dec 2010	14:33:23		Date Stamp	16 Dec 2010 13:45:15
e Name	D:\NMR\13.12	.10\FZ1541-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	10
igin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
ceiver Gain	54.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	3601.0339	Sweep Width (Hz)	11261.26		
\$ MISO					Со	mpound 38a		$\begin{array}{c} 2 \\ 3 \\ 0 \\ 1 \\ 0 \\ 1 \\ 0 \\ 8 \\ 8 \\ 8 \\ 0 \\ 1 \\ 0 \\ 0$	N_{-4} N_{-6} S_{-8a} S_{-5} S_{-5} S_{-5} S_{-16} S_{-16}
l1⊈jdf									x
, cq									00
									zeva
								0	K-Id
								-3ex	12
							ł	In her	(22)
								0, 8;12,1)	(3"
							(3.	4; 4,0	
								122	
								2 일 문	
									69
								80	1.67
						140		3 1.82 1.79 1.79	- The second
		26						1.8	
1		-21							
								J. L	AM
									1.83

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16.04.2011 16:06:06

Formula C10H11NO5S	FW	257.2630							
Acquisition Time (sec)	1.4549	Comment	single_pulse	Date	16 Dec 2010	14:33:23		Date Stamp	16 Dec 2010 13:45:15
File Name	D:\NMR	13.12.10\FZ1541-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	10
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single pulse.ex2
Receiver Gain	54.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	3601.0339	Sweep Width (Hz)	11261.26		5 <u>–</u>
									0



73.79

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16 Apr 2011

Acquisition Time (sec)	0.5571	Comment	Imported from UX	NMR.		Date	14 Apr 2011 07:04:32
File Name	D:\NMR\13.04.201	1 C-13\rudn-130411-N4-c1	3dec\rudn-130411-	N4-c13dec_001000fid		Frequency (MHz)	100.62
Nucleus	13C	Number of Transients	384	Original Points Count	16384	Points Count	16384
Pulse Sequence	zgpg	Solvent	DMSO-D6	Sweep Width (Hz)	29411.77	Temperature (degree C) 32.000
							0

Compound 38a

86.21

75.61



Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16 Apr 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

16 Apr 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

27.05.2010 20:37:28

Ne-7-

Formula C11H13NO3S	FW	239.2908

Acquisition Time (sec)	1.8193	Comment	single_pulse	Date	24 May 2010	15:57:57		Date Stamp	24 May 2010 15:56:38
File Name	D:\NMR\11.0	5.10\fz1236-1.jdf		Frequency (MHz) ,	600.17	Nucleus	1H	Number of Transients	2
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	30.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	3601.0339	Sweep Width (Hz)	9005.76	Temperature (degree C) 25.000

Compound 38b



38b



074	
3/1	

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Acquisition Time (sec)	1.8193	Comment	single_pulse	Date	24 May 2010	15:57:57		Date Stamp	24 May 2010 15:56:38
ile Name	D:\NMR\11.05	5.10\fz1236-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	2
Drigin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single pulse.ex2
Receiver Gain	30.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	3601.0339	Sweep Width (Hz)	9005.76	Temperature (degree C)) 25.000
				- Sin 1	Compo	und 38b		$\begin{bmatrix} 1 & 2 & 0 \\ 0 & 9 & 8b \end{bmatrix}$	N-6



141.20

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

27.05.2010 20:37:35

FW 239	1.2908							
1.8193	Comment	single pulse	Date	24 May 2010	15:57:57		Date Stamp	24 May 2010 15:56:38
D:\NMR\11.0	05.10\fz1236-1.jdf	·	Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	2
ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
30.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	3601.0339	Sweep Width (Hz)	9005.76	Temperature (degree C) 25.000
	1.8193 D:\NMR\11.0 ECA 600 30.00	FW 239.2908 1.8193 Comment D:\NMR\11.05.10\fz1236-1.jdf ECA 600 Original Points Count 30.00 Solvent	FW 239.2908 1.8193 Comment single_pulse D:\NMR\11.05.10\fz1236-1.jdf ECA 600 Original Points Count 16384 30.00 Solvent DMSO-d6	FW 239.2908 1.8193 Comment single_pulse Date D:\NMR\11.05.10\fz1236-1.jdf Frequency (MHz) ECA 600 Original Points Count 16384 Owner 30.00 Solvent DMSO-d6 Spectrum Offset (Hz)	FW 239.2908 1.8193 Comment single_pulse Date 24 May 2010 D:\NMR\11.05.10\fz1236-1.jdf Frequency (MHz) 600.17 ECA 600 Original Points Count 16384 Owner delta 30.00 Solvent DMSO-d6 Spectrum Offset (Hz) 3601.0339	FW 239.2908 1.8193 Comment single_pulse Date 24 May 2010 15:57:57 D:\NMR\11.05.10\fz1236-1.jdf Frequency (MHz) 600.17 Nucleus ECA 600 Original Points Count 16384 Owner delta Points Count 30.00 Solvent DMSO-d6 Spectrum Offset (Hz) 3601.0339 Sweep Width (Hz)	FW 239.2908 1.8193 Comment single_pulse Date 24 May 2010 15:57:57 D:\NMR\11.05.10\fz1236-1.jdf Frequency (MHz) 600.17 Nucleus 1H ECA 600 Original Points Count 16384 Owner delta Points Count 16384 30.00 Solvent DMSO-d6 Spectrum Offset (Hz) 3601.0339 Sweep Width (Hz) 9005.76	FW 239.2908 1.8193 Comment single_pulse Date 24 May 2010 15:57:57 Date Stamp D:\NMR\11.05.10\fz1236-1.jdf Frequency (MHz) 600.17 Nucleus 1H Number of Transients ECA 600 Original Points Count 16384 Owner delta Points Count 16384 Pulse Sequence 30.00 Solvent DMSO-d6 Spectrum Offset (Hz) 3601.0339 Sweep Width (Hz) 9005.76 Temperature (degree C)





fz1236-1.jdf



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.05.2012 15:03:22

0

15

16

H₃C

Formula C ₁₁ H ₁₃ NO ₅ S	FW 271.2	896					
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1	H/15N/13C/31P Z3379/0400		Date	13 Apr 2011 17:21:04
Date Stamp	13 Apr 2011 17	7:21:04		File Name	D:\NMR\C_13\	13.04.11 C-13\rudn-130411-N	3-c13dec\rudn-130411-N3-c13dec_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	305	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10548.5234
Sweep Width (Hz)	29409.97	Temperature (degree	C) 32.000				

Compound 38b

rudn-130411-N3-c13dec_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.05.2012 15:03:38



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.03.2011 21:21:58

Formula C. H. NO.S	FW 359	9.3517							
14.17.080				Data	10 Mar 201	11 13:48:45		Date Stamp	11 Mar 2011 00:14:31
Acquisition Time (sec)	2.1837	.1837 Comment		Date	1011101 201	Alucious	1H	Number of Transients	10
Eile Name D:\NMR\9.03.11\FZ1702-1.jdf			Frequency (MHz)	399.78	Nucleus	10004	Pulso Sequence	single pulse ex2	
Outatin	ECS 400	ECS 400 Original Points Count		Owner	delta	Points Count	10304	Fuise Sequence	
Origin	rigin ECS 400 Original forme count			PMd		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	7503.00
Receiver Gain	38.00	Solvent	CHLOROFO	T T T T T T T T T T T T T T T T T T T					

Temperature (degree C) 21.500





39a



Formula C14H17NO8S

FW

359.3517

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

26.04.2011 19:31:39

		1							
Acquisition Time (sec)	2.1837	Comment	single pulse	Date	10 Mar 201	1 12.40.46			
File Name	D-\NMR\9.03	11\E71702 1 idf	single_pulse	E .	10 Mai 201	1 13.40.45		Date Stamp	11 Mar 2011 00:14:31
Ontente	5.00.000	1111 Z 1702-1.jul		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	10
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	10204		10
Receiver Gain	38.00	Solvent		ODOFODM /		roms count	10384	Pulse Sequence	single_pulse.ex2
Temperature (degree C	21.500	convent	CHLOROFOR	KIVI-O		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	7503.00





FZ1702-1.jdf



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

26.04.2011 19:32:52

0

Formula C14H17NO8S	FW 359	.3517							
Acquisition Time (sec)	2.1837	Comment	single_pulse	Date	10 Mar 201	1 13:48:45		Date Stamp	11 Mar 2011 00:14:31
File Name	D:\NMR\9.03	3.11\FZ1702-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	10
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	38.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	7503.00
Temperature (degree C,) 21.500								

Compound 39a



Formula C H NOS FW

359 3517

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

26.04.2011 19:33:03

Acquisition Time (sec)	2.1837	Comment	single_pulse	Date	10 Mar 201	1 13:48:45		Date Stamp	11 Mar 2011 00:14:3
File Name	D:\NMR\9.03.1	11\FZ1702-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	10
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	38.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	7503.00
Temperature (degree C) 21.500								



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

26.04.2011 20:52:50

1 011101a 0 ₁₄ Π ₁₇ Ν0 ₈ 5							
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/	15N/13C/31P Z3379/0400		Date	18 Apr 2011 16:55:28
Date Stamp	18 Apr 2011 16:55:2	8					
File Name	D:\NMR\4.04.2011 (I	Рома Димер Женя + Ир	а перегруп. с серой)	\rudn-fz-180411-N3-c13dec\rud	In-fz-180411-N3-c13	dec_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	762	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10535.4922
Sweep Width (Hz)	29409.97	Temperature (degree	e C) 50.000				
				Com	pound 39	a	H ₃ C ²¹³ H ₃ C ⁴ 19 8
					a. e 1	С 1 Н ₃	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$
					Ja, 0, +		



379

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

26.04.2011 20:51:37



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

08.12.2010 19:26:08

Formula C16H21NO8S	FW	387.4048
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Acquisition Time (sec)	1.4549	Comment	single_pulse	Date	07 Dec 201	0 10:39:04		Date Stamp	07 Dec 2010 09:50:47
File Name	D:\NMR\06.12	10\FZ1519-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	10
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	sinale pulse.ex2
Receiver Gain	46.00	Solvent	CHLOROFOR	M-d		Spectrum Offset (Hz)	3601.0339	Sweep Width (Hz)	11261.26





H₃C.





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

08.12.2010 19:26:36

Acquisition Time (sec)	1.4549	Comment	single_pulse	Date	07 Dec 201	0 10:39:04		Date Stamp	07 Dec 2010 09:50:47
File Name	D:\NMR\06	12.10\FZ1519-1.jdf		Frequency (MHz)	600.17	Nucleus	1H	Number of Transients	10
Origin	ECA 600	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	46.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	3601.0339	Sweep Width (Hz)	11261.26



Formula C16H21NO8S FW

387.4048

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Acquisition Time (sec) 1.4549 Comment single_pulse Date 07 Dec 2010 10:39:04 Date Stamp File Name D:\NMR\06.12.10\FZ1519-1.jdf 07 Dec 2010 09:50:47 Frequency (MHz) 600.17 Nucleus 1H Number of Transients 10 Origin ECA 600 Original Points Count 16384 Owner delta Points Count 16384 **Receiver Gain Pulse Sequence** single_pulse.ex2 46.00 Solvent CHLOROFORM-d Spectrum Offset (Hz) 3601.0339 Sweep Width (Hz) 11261.26 H₃C Compound 39b CH-3.27-3,16 24 H-2 FZ1519-1.jdf H-3B m, 2H H-59 8.3: +3,1) 3.20 ; 11,7) 41 -3.24 -3.24 -3.23 3.23 3.49 3.46 3.00 3.01 3.18 3.18 3.21 3.48 3.47 45 3.50 3.01 3.02 2.99 3.27 3.26 3.25 3.19 3.16 1.00 1.94 0.97 3.50 3.45 3.40 3.35 3.30 3.25 3.20 3.15 3.10 3.05 3.00 2.95 Chemical Shift (ppm)

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

08.12.2010 19:27:09

Formula C., Ha, NO, S FW

387 4048

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

08.12.2010 19:27:33



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

ct3, OMSO

equisition Time (sec)	0.5571	Comment	Imported from U	JXNMR.		Date	18 Apr 2011 17:21:04	
le Name	C:\Users\Fedor	\Desktop\4.04.2011 (Рома)	\rudn-fz-180411-N4-c1	3dec\rudn-fz-180411-N4-c13d	ec_001000fid	Frequency (MHz)	100.62	
icleus	13C	Number of Transie	ents 793	Original Points Count	16384	Points Count	16384	
Ilse Sequence	zgpg	Solvent	DMSO-D6	Sweep Width (Hz)	29411.77	Temperature (degree	e C) 50.000	
				Comp	ound 39b	0~	0 22 H_3C 2' 0 16 $9^{/10.8a}$	CH3
						1.5	H_2C Q P_a N	
					8		14 14 15 15 14 12 15 12 14 12 15 12 12 13 12 13 12 13 13 13 13 13 13 13 13	23
					40		1.5:	
					76.(7.30	5
							4. 9.54	21.2
							35 33	14
							38.	
					0		44	
					Ja		38.33	
					2			
				0	0.2			53
				7	2			20.
29.2.7				t				
9.46				3.84				
169				¹⁰			9	
				7.8			6.65	
e				ω.				
38.8		14 (14 (14 (14 (14 (14 (14 (14 (14 (14 (
10								
						1		1
والمسلسط المتعلين والألمط للالمان والمتعال	huidhal air as dao àire	الاسترابة والمعادة والمتعادية أوروك والمتقاد والمتعادية المتعادية والمتعادية والمتعادية والمتعادية والمتعادية و	A Maninething the balance of bala	and the fight of the state of the	Alighter text to short be used and it had took	addition with addited the addition	attactives Replacedalises	AL ECHALICS MUSICACION
altra man a chilana kasheyini n	in te construction de la const	a li data a canta da sa	e als distantisti riska deleva Håled, dela padri	if its all say to see a district a store of taking the big	al new later and the state of the	विराय के प्रियंत के रायर के रायर के साम के साम कि	an all i chikan da in an a tha all an	विश्वविद्यानुक्रियरा । वस्त्रिक महिल्ला रा

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

19 Apr 2011



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

19 Apr 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

06.10.2011 16:57:25

Formula C ₁₅ H ₁₂ N ₂ O	FW 236.2	586							
Acquisition Time (sec)	1.8193	Comment	single_pulse	-Date	06 Oct 2011	09:01:49		Date Stamp	06 Oct 2011 12:50:43
File Name	D:\NMR\03.10.	11\FZ1958-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	8
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	38.00	Solvent	CHLOROFOR	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	9005.76



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

06.10.2011 16:57:33 Formula C15H12N2O FW 236.2686 Acquisition Time (sec) 1.8193 Comment single_pulse Date 06 Oct 2011 09:01:49 Date Stamp 06 Oct 2011 12:50:43 File Name D:\NMR\03.10.11\FZ1958-1.jdf Frequency (MHz) 399.78 Nucleus 1H Number of Transients 8 Origin ECS 400 Original Points Count 16384 Owner delta Points Count 16384 Pulse Sequence single_pulse.ex2 **Receiver Gain** 38.00 Solvent CHLOROFORM-d Spectrum Offset (Hz) 2398.6931 Sweep Width (Hz) 9005.76 Compound 40a HN NH H-4 and H-9 FZ1958-1.jdf 7.24 4-4 H-S 7.26 (-1,6;7,2) 3,3) LiH- Waphtyl (1.6;3.3) 1. 6 m 6.59 6.58 6.56 7.42 6.40 7.25 6.35 6.41 7.28 21 (O) ~ 6.35 0.82 3.97 1.75 0.88 0.91 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75 6.70 6.65 6.60 6.55 6.50 6.45 6.40 6.35

General Synthetic Approach towards Annelated 3a,6-Epoxylsoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 16:44:29

H

Formula C ₁₅ H ₁₂ N ₂ O	FW 236.26	86					
Acquisition Time (sec)	1.0433	Comment	single pulse decou	pled gated NOE		Date	18 Jun 2012 11:51:51
Date Stamp	18 Jun 2012 15	5:41:34		File Name	C:\Users\Fedor\	Desktop\13.06.12\FZ_2459-	-1.jdf
Frequency (MHz)	100.53	Nucleus	13C	Number of Transients	241	Origin	ECS 400
Original Points Count	32768	Owner	delta	Points Count	32768	Pulse Sequence	single_pulse_dec
Receiver Gain	60.00	Solvent	CHLOROFORM-	1		Spectrum Offset (Hz)	10052.5303
Sweep Width (Hz)	31407.04	Temperature (degree C)	23.200				

Compound 40a



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 16:44:38

Η

Formula C ₁₅ H ₁₂ N ₂ O	FW 236.2686	i					
Acquisition Time (sec)	1.0433 Comment single p		single pulse decou	pled gated NOE		Date	18 Jun 2012 11:51:51
Date Stamp	18 Jun 2012 15:4	1:34		File Name	C:\Users\Fedor\I	Desktop\13.06.12\FZ_2459	-1.jdf
Frequency (MHz)	100.53	Nucleus	13C	Number of Transients	241	Origin	ECS 400
Original Points Count	32768	Owner	delta	Points Count	32768	Pulse Sequence	single_pulse_dec
Receiver Gain	60.00	Solvent	CHLOROFORM-C	1		Spectrum Offset (Hz)	10052.5303
Sweep Width (Hz)	31407.04	Temperature (degree C)	23.200				





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

06.10.2011 17:04:49

2.31

CH₂

HN

Formula C16H14N2O FW 250.2952

Acquisition Time (sec)	1.8193	Comment	single_pulse	Date	06 Oct 2011 08:11:57			Date Stamp	06 Oct 2011 12:00:52
File Name	D:\NMR\03.10.	11\FZ1962-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	8
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	38.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	9005.76



40b



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

06.10.2011 17:04:58

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Formula C ₁₆ H ₁₄ N ₂ O	FW 250.2	2952							
Acquisition Time (sec)	1.8193	Comment	single_pulse	Date	06 Oct 201	1 08:11:57	Date Stamp	06 Oct 2011 12:00:52	
File Name	D:\NMR\03.10.11\FZ1962-1.jdf			Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	8
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	38.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	9005.76

Compound 40b

FZ1962-1.jdf



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

17.11.2011 15:52:26

Formula C ₁₆ H ₁₄ N ₂ O	FW 250.29	52							
Acquisition Time (sec)	1.0433	Comment	single pulse deco	oupled gated NOE		Date	16 Nov 2011	13:50:02	
Date Stamp	17 Nov 2011 00:13:11			File Name	D:\NMR\14.11.	11\FZ2052_13C-1.jdf		Frequency (MHz)	100.53
Nucleus	13C	Number of Transients	82	Origin	ECS 400	Original Points Count	32768	Owner	delta
Points Count	32768	Pulse Sequence	single_pulse_de	C		Receiver Gain	60.00		
Solvent	CHLOROFOR	M-d		Spectrum Offset (Hz)	12063.0361	Sweep Width (Hz)	31407.04	Temperature (degree	C) 26.000






General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

17.11.2011 15:53:14

Acquisition Time (sec)	1.0433	Comment	single pulse dec	oupled gated NOE		Date	16 Nov 2011	13:50:02	
Date Stamp	17 Nov 2011 00:	13:11		File Name	D:\NMR\14.11.	11\FZ2052_13C-1.jdf		Frequency (MHz)	100.53
Nucleus	13C	Number of Transients	82	Origin	ECS 400	Original Points Count	32768	Owner	delta
Points Count	32768	Pulse Sequence	single_pulse_de	c		Receiver Gain	60.00		
Solvent	CHLOROFORM	-d		Spectrum Offset (Hz)	12063.0361	Sweep Width (Hz)	31407.04	Temperature (degree C)	26.000
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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

17 Oct 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

17 Oct 2011



398

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

17 Oct 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

17 Oct 2011



400

Formula C H N O

FW

334 3255

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

14.05.2012 11:07:58

39.39

- 19: 14: 2 - 4							
Acquisition Time (sec)	0.5243	Comment	5 mm QNP 11	H/15N/13C/31P Z3379/0400		Date	26 Oct 2011 17:50:56
Date Stamp	26 Oct 2011 1	7:50:56		File Name	D:\NMR\14.10	.11\rudn-141011-N3_1-c13de	c\rudn-141011-N3_1-c13dec_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	10000	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	31250.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	10547.7090
Sween Width (Hz)	31248 09	Temperature (degree	C) 27 000				

Compounds 41Aa/41Ba reaction mixture



rudn-141011-N3 1-c13dec 001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

14.05.2012 11:09:50

Acquisition Time (sec)	0.5243	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	26 Oct 2011 17:50:56
Date Stamp	26 Oct 2011 17:50	0:56		File Name	D:\NMR\14.10.1	1\rudn-141011-N3_1-c13ded	c\rudn-141011-N3_1-c13dec_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	10000	Origin	spect
Driginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	31250.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	10547.7090
weep Width (Hz)	31248.09	Temperature (degree	e C) 27.000				
							H H γ_{aa} γ_{b} $\gamma_{$





rudn-141011-N1 2 001000fid

41Aa

COOH

12.38

0.94

12.0

11.0

11.5

10.5

12.5

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C ₁₉ H ₁₄ N ₂ O ₄	FW 334.	3255					N11000A (CH2Ce2) 0,20
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1	H/15N/13C/31P Z3379/0400		Date	24 Oct 2011 17:46:40
Date Stamp	24 Oct 2011	17:46:40		File Name	D:\NMR\14.10	0.11\rudn-141011-N1_2\rudn	-141011-N1_2_001000fid
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	40	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	1024.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542
Sweep Width (Hz)	10416.03	Temperature (degree	C) 32.000				



14.05.2012 10:29:06

Chemical Shift (ppm)

rudn-141011-N1_2_001000fid

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

14.05.2012 10:29:33

Formula C ₁₉ H ₁₄ N ₂ O ₄	FW	334.3255	
Acquisition Time (sec)	1.5729	Comn	nent

Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1	IH/15N/13C/31P Z3379/0400		Date	24 Oct 2011 17:46:40
Date Stamp	24 Oct 2011 17	7:46:40		File Name	D:\NMR\14.10).11\rudn-141011-N1_2\rudn-	-141011-N1 2 001000fid
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	40	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zq
Receiver Gain	1024.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542
Sweep Width (Hz)	10416.03	Temperature (degree	C) 32.000				



H-5



Compound 41Aa



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

14.05.2012 10:29:43

Date Stamp	24 Oct 2011	17:46:40	o nin qiti i	File Name	D:\NMR\14 10) 11\rudn-141011-N1 2\rudn	-141011-N1 2 001000fid
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	40	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	1024.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542
Sweep Width (Hz)	10416.03	Temperature (degree	C) 32.000				



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

14.05.2012 10:56:01

Formula C ₁₉ H ₁₄ N ₂ O ₄	FW 334.3255						
Acquisition Time (sec)	0.5243	Comment	5 mm QNP 1H/15	5N/13C/31P Z3379/0400		Date	24 Oct 2011 18:01:36
Date Stamp	24 Oct 2011 18:01	:36		File Name	D:\NMR\14.10.11\	rudn-141011-N1_2-c13dec	c\rudn-141011-N1_2-c13dec_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	7000	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	31250.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	10547.7090
Sweep Width (Hz)	31248.09	Temperature (degree C) 27.000				

Compound 41Aa



rudn-141011-N1_2-c13dec_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 14:14:29

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Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15	5N/13C/31P Z3379/0400		Date	21 Jun 2012	2 13:24:16	· · ·	····· ···· ·
Date Stamp	21 Jun 2012 13	:24:16						N 2 N 2		· · · · · ·
File Name	C:\Users\Fedor	Desktop\C13 Рома Для Стат	ъи в JOC 25.05.12\r	udn-250512-41b\rudn-2505	12-41b_001000fid	Frequency (MHz)	400.14	1 . 16	e .	
Nucleus	1H	Number of Transients	20	Origin	spect	Original Points Count	16384	·** -	*	
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00	* 1 may 2		Acres 6
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03		8.8.8	1. 1 12 18
Temperature (degree C	32.000							in the section	19.25	has marine and







1.62

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 14:14:50

6.42 6.38 6.36

2.24

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6.3

Formula C ₂₀ H ₁₆ N ₂ O ₄	FW 348.3520						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15M	V/13C/31P Z3379/0400		Date	21 Jun 2012 13:24:16
Date Stamp	21 Jun 2012 13:24:	16					
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Стат	ы в JOC 25.05.12\ru	dn-250512-41b\rudn-2505	12-41b_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	20	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Towns anothers (downso C	1 22 000						

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Temperature (degree C) 32.000



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 14:14:59

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Formula C ₂₀ H ₁₆ N ₂ O ₄	<i>FW</i> 348.3520						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	21 Jun 2012 13:24:16
Date Stamp	21 Jun 2012 13:24:	:16					
File Name	C:\Users\Fedor\De	sktop\C13 Рома Для Стат	ы в JOC 25.05.12\	rudn-250512-41b\rudn-2505	12-41b_001000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	20	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Temperature (degree 0	32.000						



3.5

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

1.36

3.0

2.32

2.5

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0.99

5.5

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 13:38:41

Formula C ₂₀ H ₁₆ N ₂ O ₄	FW 348.3520						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N	13C/31P Z3379/0400		Date	21 Jun 2012 13:24:16
Date Stamp	21 Jun 2012 13:24:1	16	1				
ile Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Стат	ы в JOC 25.05.12\rudn-	250512-41b-c13dec\rudn-2	250512-41b-c13dec_00	01000fid	
requency (MHz)	100.62	Nucleus	13C	Number of Transients	1123	Origin	spect
riginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
eceiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10548.2051
weep Width (Hz)	29409.97	Temperature (degree	C) 27.000				



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 13:40:21

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Formula C ₂₀ H ₁₆ N ₂ O ₄	FW 348.3520						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N/	13C/31P Z3379/0400		Date	21 Jun 2012 13:24:16
Date Stamp	21 Jun 2012 13:24:16	1					
File Name	C:\Users\Fedor\Deskt	ор\С13 Рома Для Статьи	в JOC 25.05.12\rudn-2	250512-41b-c13dec\rudn-2	50512-41b-c13dec_00)1000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	1123	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10548.2051
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 13:39:35

Formula C ₂₀ H ₁₆ N ₂ O ₄	<i>FW</i> 348.3520						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	21 Jun 2012 13:24:16
Date Stamp	21 Jun 2012 13:24:1	6					
File Name	C:\Users\Fedor\Des	top\C13 Рома Для Статьи	в ЈОС 25.05.12\ги	dn-250512-41b-c13dec\rudn-2	250512-41b-c13dec	_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	1123	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10548.2051
Sweep Width (Hz)	29409.97	Temperature (degree C	27.000				

Compounds 41Ab/41Bb



rudn-250512-41b-c13dec_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 13:39:14

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3b

Formula C ₂₀ H ₁₆ N ₂ O ₄	FW 348.3520						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N	/13C/31P Z3379/0400		Date	21 Jun 2012 13:24:16
Date Stamp	21 Jun 2012 13:24:1	6	,				
File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Статьи	в JOC 25.05.12\rudn	-250512-41b-c13dec\rudn-2	250512-41b-c13dec_0	01000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	1123	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10548.2051
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C ₂₀ H ₁₆ N ₂ O ₄	FW 348.3520							
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	I/15N/13C/31P Z3379/0400		Date	21 Jun 2012 13:47:44	
Date Stamp	21 Jun 2012 13:47:4	4	<i>,</i>					
File Name	C:\Users\Fedor\Des	top\C13 Рома Для Статьи	в ЈОС 25.05.12	\rudn-250512-41b-dept135\rudn-2	250512-41b-dept1	35_001000fid		
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	641	Origin	spect	
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135	
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9099.0557	
Sween Width (Hz)	29409 97	Temperature (degree C	27 000					

Compounds 41Ab/41Bb



rudn-250512-41b-dept135_001000fid



22.06.2012 13:52:02

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.06.2012 13:52:09

Formula C ₂₀ H ₁₆ N ₂ O ₄	FW 348.3520						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N/	13C/31P Z3379/0400		Date	21 Jun 2012 13:47:44
Date Stamp	21 Jun 2012 13:47:4	4	e				
File Name	C:\Users\Fedor\Dest	top\C13 Рома Для Статьи в	в JOC 25.05.12\rudn-2	250512-41b-dept135\rudn-2	250512-41b-dept135_0	01000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	641	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9099.0557
Sweep Width (Hz)	29409.97	Temperature (degree C)	27.000				

Compounds 41Ab/41Bb



rudn-250512-41b-dept135_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

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Formula C ₂₀ H ₁₆ N ₂ O ₄	FW 348.3520						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H	H/15N/13C/31P Z3379/0400		Date	30 Dec 2011 11:01:20
Date Stamp	30 Dec 2011 11:0	1:20		File Name	D:\NMR\19.12.11	(Рома)\rudn-191211-N11-	10031\rudn-191211-N11-10031_001000fid
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	96	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	1024.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542
Sweep Width (Hz)	10416.03	Temperature (degree C	32.000				

Compound 41Ab



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rudn-191211-N11-1003-_1_001000fid

41Ab



FW

348.3520

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

12.05.2012 19:43:19

Formula C ₂₀ H ₁₆ N ₂ O ₄	FW 348.352	0					
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1	H/15N/13C/31P Z3379/0400		Date	30 Dec 2011 11:01:20
Date Stamp	30 Dec 2011 11:	01:20		File Name	D:\NMR\19.12.	11 (Рома)\rudn-191211-N11-	10031\rudn-191211-N11-10031_001000fid
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	96	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	1024.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542
Sweep Width (Hz)	10416.03	Temperature (degree	C) 32.000				



rudn-191211-N11-1003-_1_001000fid







General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

12.05.2012 19:43:32



Chemical Shift (ppm)

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction

12.05.2012 20:00:51

-CH3

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24

H 7aa

13 13a

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HO

26

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3b

39.52

Formula C ₂₀ H ₁₆ N ₂ O ₄	FW 348.3520						
Acquisition Time (sec)	0.5243	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	29 Dec 2011 18:57:04
Date Stamp	29 Dec 2011 18:57	:04					
File Name	D:\NMR\19.12.11 (Рома)\rudn-191211-N11-1	003c13dec\rudn-19	1211-N11-1003c13dec_0	01000fid	Frequency (MHz)	100.62
Nucleus	13C	Number of Transients	7000	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zgpg	Receiver Gain	32768.00
SW(cyclical) (Hz)	31250.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	9099.1699	Sweep Width (Hz)	31248.09
Temperature (degree C	27.000						

Compound 41Ab

rudn-191211-N11-1003--c13dec_001000fid

FW



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

12.05.2012 20:01:05

Formula	C20H16N2O4	FW	348.3520	
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Acquisition Time (sec)	0.5243	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	29 Dec 2011 18:57:04
Date Stamp	29 Dec 2011 18:5	7:04					
File Name	D:\NMR\19.12.11	(Рома)\rudn-191211-N11-10	003c13dec\rudn-19	1211-N11-1003c13dec_0	01000fid	Frequency (MHz)	100.62
Nucleus	13C	Number of Transients	7000	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zgpg	Receiver Gain	32768.00
SW(cyclical) (Hz)	31250.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	9099.1699	Sweep Width (Hz)	31248.09
Temperature (degree C)	27.000						

Compound 41Ab



rudn-191211-N11-1003--c13dec_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

12.05.2012 20:01:16

Η

Formula C ₂₀ H ₁₆ N ₂ O ₄	<i>FW</i> 348.3520						
Acquisition Time (sec)	0.5243	Comment	5 mm QNP 1H/15M	N/13C/31P Z3379/0400		Date	29 Dec 2011 18:57:04
Date Stamp	29 Dec 2011 18:57	04					
File Name	D:\NMR\19.12.11 (I	Рома)\rudn-191211-N11-10	003c13dec\rudn-191	1211-N11-1003c13dec_0	01000fid	Frequency (MHz)	100.62
Nucleus	13C	Number of Transients	7000	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zgpg	Receiver Gain	32768.00
SW(cyclical) (Hz)	31250.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	9099.1699	Sweep Width (Hz)	31248.09
Temperature (degree C) 27.000						



Chemical Shift (ppm)

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

12.05.2012 20:44:16

15.58

Acquisition Time (sec)	0.5243	Comment 5 mm QNP 1H/15N/13C/31P Z3379/0400				Date	30 Dec 2011 00:08:32
Date Stamp	30 Dec 2011 00:08:	32					
File Name	D:\NMR\19.12.11 (F	ома)\rudn-191211-N11-10	03dept135\rudn-1	91211-N11-1003dept135_	001000fid	Frequency (MHz)	100.62
Nucleus	13C	Number of Transients	5000	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384 -	Pulse Sequence	dept135	Receiver Gain	32768.00
SW(cyclical) (Hz)	31250.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	9099.1699	Sweep Width (Hz)	31248.09
Temperature (degree C)	27.000						

rudn-191211-N11-1003-dept135_001000fid





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.07.2012 16:10:18

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Formula C ₁₈ H ₁₄ N ₂ O ₂	<i>FW</i> 290.3160									
Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H	//15N/13C/31P Z3379/0400		Date	11 Jul 2012 13:37:04			
Date Stamp	11 Jul 2012 13:37:0	4								
File Name	C:\Users\Fedor\Des	:\Users\Fedor\Desktop\C13 Рома Для Статьи в JOC 25.05.12\rudn-060712-42-42_61\rudn-060712-42-42_61_001000fid								
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	20	Origin	spect			
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg			
Receiver Gain	512.00	SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2602.0486			
Sweep Width (Hz)	10203.46	Temperature (degree C) 27.000							

Compounds 42A/42B





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.07.2012 16:10:28

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Formula C ₁₈ H ₁₄ N ₂ O ₂	FW 290.3160											
Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H/15N/13C/31P		3C/31P Z3379/0400		11 Jul 2012 13:37:04					
Date Stamp	11 Jul 2012 13:37:04		1									
File Name	C:\Users\Fedor\Desk	Users\Fedor\Desktop\C13 Рома Для Статьи в JOC 25.05.12\rudn-060712-42-42_61\rudn-060712-42-42_61_001000fid										
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	20	Origin	spect					
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg					
Receiver Gain	512.00	SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	2602.0486					
Sweep Width (Hz)	10203.46	Temperature (degree C)	27.000									

Compounds 42A/42B



5.5

5.0

4.5

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.07.2012 16:10:52

2.0

2.5

Formula C ₁₈ H ₁₄ N ₂ O ₂	FW 290.3160							
Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	11 Jul 2012 13:37:04	1
Date Stamp	11 Jul 2012 13:37:0	4						
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Стат	ьи в JOC 25.05.12\ru	dn-060712-42-42_61\rudn-06	50712-42-42_61_0010	OOfid		
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	20	Origin	spect	
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg	
Receiver Gain	512.00	SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d	Spectrum Offset (H	Iz) 2602.0486	
H-79 rudn-060712-42-42_61_0	01000fid H-10			Comp	ounds 42A H - (1q	/42B	$H = \frac{H}{7aa}$ $N = \frac{7}{7a}$ $K = \frac{1}{7a}$ $K =$	H-llende
-5.49	(H.8;	4.5)			(3,7;8	(7)	(A+B) ddd (S;7;4,5;11.8) 1	(A+B) dd (8,7; 11,8)
524 5.17 5.17	5.16					2.73 2.72 2.69 2.71 2.68 2.70	2.38 2.37 2.36 2.36 2.35 2.34 2.33	
7.27 7	7.20					8.64	8.77	8.46

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

3.5

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4.0

Formula C H N O

EW/

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

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Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N/	13C/31P Z3379/0400		Date	11 Jul 2012 13:37:04				
Date Stamp	11 Jul 2012 13:37:04	File Name	C:\Users\Fedor\Desk	C:\Users\Fedor\Desktop\C13 Рома Для Статьи в JOC 25.05.12\rudn-060712-42-42_61-c13dec\rudn-060712-42-42_61-c13dec_001							
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	1624	Origin	spect				
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg				
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9632.4561				
Sweep Width (Hz)	29409.97	Temperature (degree (C) 27.000								

Compounds 42A/42B



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.07.2012 17:49:29

ition Time (cool 0 5571							
nuon nine (sec) 0.5571	Comment	5 mm QNP 1H/15N/13	3C/31P Z3379/0400		Date	11 Jul 2012 13:37:04	
tamp 11 Jul 2012 13:37:0	4 File Name	C:\Users\Fedor\Deskto	р\С13 Рома Для Статьи и	в JOC 25.05.12\rudn-0	50712-42-42_61-c13dec\ru	dn-060712-42-42_61-c13de	_001000fid
ncy (MHz) 100.62	Nucleus	13C	Number of Transients	1624	Origin	spect	
al Points Count 16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg	
er Gain 32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9632.4561	
Width (Hz) 29409.97	Temperature (degree C)	27.000				H 8-9	
					5 6 6	H 7aa / 1	10
				o VI		$11a^{14}11$	1.0
			25	15,01		$ \begin{bmatrix} 13a & 13 \\ 1 \end{bmatrix} $	20
	(A+6)		412	\sim	3	22 ···	\sim
J712-42-42_61-c13dec_001000fid	35		$\sim\sim\sim$	3.48	Compound	ds 42A/42B	-113.54
8	-132.		5.46	-12			(
			126				
5	No.	(F)					
6.39		(\mathbf{A})	(2 A)	Λ			
14		226	126.5	-1		136	
137.95	Ja Ba				00		
33.09	5 0	D	(A	-1515 -	A	-113.29
T T	-134.5	CH		24.16	22	5.82	
137.43	34.41				119.		
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428

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.07.2012 17:44:19

Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N/13	3C/31P Z3379/0400		Date	11 Jul 2012 14:28:16
Date Stamp	11 Jul 2012 14:28:16	File Name	C:\Users\Fedor\Deskto	р\С13 Рома Для Статьи и	в JOC 25.05.12\rudn-06	0712-42-42_61-dept135\ru	udn-060712-42-42_61-dept135_001000
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	948	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9632.4453
Sweep Width (Hz)	29409.97	Temperature (degree	C) 27.000				1
dn-060712-42-42_61-de	pt135_001000fid					Coi	npounds 42A/42B
37.96	126.65 3.48 .28	-113.54			67.03		
-137.43 -137.43 -131.08		113.29	interpretation and the second second	79.89	69.05	18 30	
							50.50
							28.60

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

14.05.2012 12:11:42

	Formula	C18H14N2O2	FW	290.3160
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Acquisition Time (sec)	2.1837	Comment	single_pulse	Date 14 Dec 2011 10:20:06			Date Stamp	14 Dec 2011 14:54:45	
File Name D:\NMR\12.12.11\FZ2125-1.jdf .				Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	8
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	46.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	7503.00
Temperature (degree C)	23.100								







General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

14.05.2012 12:11:53

Acquisition Time (sec)	2.1837	Comment	single_pulse	Date	14 Dec 2011	10:20:06	10000	Date Stamp	14 Dec 2011 14:54:45
File Name	D:\NMR\12.1	2.11\FZ2125-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	8
Drigin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	46.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	7503.00
emperature (degree C)	23.100	1.	0						
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2125-1.jdf			25				(Compound 42	B
			7					Joinpound 12	
					1.	Q			1
		10		16	H-	6		M	H-10
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0,9;7,3)		(012) 412)				1	~	4	
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0.80		4.08		0.97	1.9	1		0.95	1.00
	0.0	7.5		7.0		CE	6.0	5	E

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

14.05.2012 12:12:09

Formula C₁₈H₁₄N₂O₂ FW 290.3160

Acquisition Time (sec)	2.1837	Comment	single_pulse	Date 14 Dec 2011 10:20:06				Date Stamp	14 Dec 2011 14:54:45
File Name D:\NMR\12.12.11\FZ2125-1.jdf				Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	8
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	46.00	Solvent	CHLOROFOR	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	7503.00
Temperature (degree C,) 23.100								



Compound 42B

FZ2125-1.jdf


General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.07.2012 18:11:05

Formula C ₁₈ H ₁₄ N ₂ O ₂	FW 290.3160						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N/	13C/31P Z3379/0400		Date	11 Jul 2012 21:54:08
Date Stamp	11 Jul 2012 21:54:08	File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Статьи	в JOC 25.05.12\rudn-0	60712-42-36_41c13dec\ru	dn-060712-42-36_41c13dec_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	34633	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9638.7344
Sweep Width (Hz)	29409.97	Temperature (degree C	27.000				



76.80 77.12

rudn-060712-42-36_41c13dec_001000fid



13.07.2012 15:27:33

Formula C ₁₈ H ₁₄ N ₂ O ₂	FW 290.3160						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	11 Jul 2012 21:54:08
Date Stamp	11 Jul 2012 21:54:08	File Name	C:\Users\Fedor	Desktop\C13 Рома Для Статьи	в JOC 25.05.12\rudn-0	060712-42-36_41c13dec\ru	dn-060712-42-36_41c13dec_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	34633	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9638.7344
Sween Width (Hz)	29409 97	Temperature (degree C	27 000				



Compound 42B



rudn-060712-42-36_41c13dec_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

13.07.2012 18:07:49

Formula C ₁₈ H ₁₄ N ₂ O ₂	FW 290.3160						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	11 Jul 2012 18:18:40
Date Stamp	11 Jul 2012 18:18:40	File Name	C:\Users\Fedor\D	esktop\C13 Рома Для Статьи	в JOC 25.05.12\rudn-0	60712-42-36_41-dept135\ru	udn-060712-42-36_41-dept135_001000fid
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	9000	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9638.7344
Sween Width (Hz)	29409 97	Temperature (degree C) 27 000				

Compound 42B





Acquisition Time (sec) 1.6056 Comment Imported from UXNMR. Date 23 Aug 2011 10:31:28 File Name C:\Users\Fedor\Desktop\12.08.11\rudn-190811-N14\rudn-190811-N14 001000fid Frequency (MHz) 400.14 Nucleus 1H Number of Transients 16 Original Points Count 16384 Points Count 16384 Pulse Sequence zg Solvent CHLOROFORM-D Sweep Width (Hz) 10204.08 Temperature (degree C) 27.000 Compound 43a NH reaction mixture 43a (reaction Н mixture) 1 H-2,5 7.25 5.35 4-4 6.72 76.8) 40 6.35 6.38 N 66 4.15 é - 6.60 6.58 6.73 6.39 7.04 6.92 6.90 6.70 3.94 19 4 7.02 7.05 5.28 35 1.19 1.23 1.09 1.30 1.16 1.92 1.00 1.19 1.13 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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23 Aug 2011

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23 Aug 2011

isition Time (sec)	1.6056	Comment	Imported from UXN	MR.		Date	23 Aug 2011 10:31:28	
Name	C:\Users\Fedor\De	esktop\12.08.11\rudn-1908	11-N14\rudn-190811-N	N14_001000fid		Frequency (MHz)	400.14	
eus	1H	Number of Transients	16	Original Points Count 163	384	Points Count	16384	
e Sequence	zg	Solvent	CHLOROFORM-D			Sweep Width (Hz)	10204.08	
perature (degree C) 5' b2 d	27.000	CH CS & 52.7	H-7 Br &d	Compound reaction mi H-S B2d	l 43a ixture H- X (1.2;7	-6 t (5) 67	H = 8	H $2 \xrightarrow{3} \xrightarrow{4} \xrightarrow{0} \xrightarrow{1} \xrightarrow{4} \xrightarrow{1} \xrightarrow{1} \xrightarrow{1} \xrightarrow{1} \xrightarrow{1} \xrightarrow{1} \xrightarrow{1} 1$
(4.6)			20 7.5,8,2,5	(2, 7) (2, 7)	م ۲ ل	6.72 6.72 6.72	(8,1) (8:9)	3 4 dd 3.1) 8:9 (1.6 9:3 9:3
1.19		mann	50.7 1.30	1.16		1.23	1.09	1.92

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

24 Aug 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

24 Aug 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

23 Aug 2011



23 Aug 2011



Chemical Shift (ppm)

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

23 Aug 2011

Acquisition Time (sec)	1.5729	Comment	Imported from UX	NMR.		Date	23 Aug 2011 09:14:40
File Name	C:\Users\Fedor\D	esktop\12.08.11\rudn-1208	11-N3\rudn-120811	-N3_001000fid		Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	16	Original Points Count	16384	Points Count	16384
Pulse Sequence	zg	Solvent	DMSO-D6	Sweep Width (Hz)	10416.67	Temperature (degree	C) 32.000

Compounds 44Aa/44Ba reaction mixture before recrystallization





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

24 Aug 2011





Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

24 Aug 2011



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

24 Aug 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

29.05.2012 10:54:23

Formula C ₁₆ H ₁₄ N ₂ O ₄	<i>FW</i> 298.2934						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	28 May 2012 16:49:04
Date Stamp	28 May 2012 16:49	:04					
File Name	C:\Users\Fedor\De	sktop\C13 Рома Для Стать	и в ЈОС 25.05.12\ги	Idn-250212-44Aa\rudn-250	212-44Aa_002000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	4	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Taman anatuma (damaa (21 22 000						

Temperature (degree C) 32.000





rudn-250212-44Aa_002000fid

44Aa



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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

29.05.2012 10:54:46

Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H	1/15N/13C/31P Z3379/0400		Date	28 May 2012 16:49:04
Date Stamp	28 May 2012 16:4	49:04					
File Name	C:\Users\Fedor\D	esktop\C13 Рома Для Стать	и в ЈОС 25.05.1	2\rudn-250212-44Aa\rudn-250	212-44Aa_002000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	4	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
Temperature (degree C) 32.000						



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

29.05.2012 10:55:02

Formula C ₁₆ H ₁₄ N ₂ O ₄	FW 298.2934						
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	28 May 2012 16:49:04
Date Stamp	28 May 2012 16:49	:04					
File Name	C:\Users\Fedor\Des	sktop\C13 Рома Для Стать	и в ЈОС 25.05.12\ги	dn-250212-44Aa\rudn-250	212-44Aa_002000fid	Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	4	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zg	Receiver Gain	128.00
SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	Sweep Width (Hz)	10416.03
_							

Temperature (degree C) 32.000

rudn-250212-44Aa_002000fid

H-10A

A

H-10B

Compound 44Aa





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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

29.05.2012 11:21:25

Formula C ₁₆ H ₁₄ N ₂ O ₄	FW 298.2934						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	I/15N/13C/31P Z3379/0400		Date	28 May 2012 16:51:12
Date Stamp	28 May 2012 16:51:1	2					
File Name	C:\Users\Fedor\Desk	top\C13 Рома Для Статы	и в ЈОС 25.05.12	\rudn-250512-44Aa-c13dec\rudn-	250512-44Aa-c13de	c_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	730	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.3594
Sweep Width (Hz)	29409.97	Temperature (degree (27.000				





rudn-250512-44Aa-c13dec_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

29.05.2012 11:21:36

Formula C ₁₆ H ₁₄ N ₂ O ₄	FW 298.2934						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	15N/13C/31P Z3379/0400		Date	28 May 2012 16:51:12
Date Stamp	28 May 2012 16:51:	12					
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Статьи	в ЈОС 25.05.12\	rudn-250512-44Aa-c13dec\rudn-	250512-44Aa-c13de	ec_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	730	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.3594
Sweep Width (Hz)	29409.97	Temperature (degree C	27.000				



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

caulsition Time (sec)	0 5571	Comment	5 mm ONP 14/15	N/13C/31P 73379/0400		Data	28 May 2012 16:51:12	
ate Stamn	28 May 2012 16:51	12	Shin Que nais	11/130/311 233/3/0400		Date	20 Way 2012 10.01.12	
le Name	C:\Users\Fedor\Des	ktop\C13 Рома Лля Стат	и в ЮС 25 05 12\гис	n-250512-44Aa-c13dec\rudn	-250512-44Aa-c13d	lec 001000fid		
requency (MHz)	100.62	Nucleus	13C	Number of Transients	730	Origin	spect	
riginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	Zapa	
eceiver Gain	32768.00	SW(cvclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10554.3594	
veen Width (Hz)	29409.97	Temperature (degree	C) 27 000		51100 00	opeca and encort (inc)		
				Compound	d 44Aa			20 - OH 22
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n-250512-44Aa-c13de	ec_001000fid			1.0			6	1C
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68 66 64 62 Chemical Shift (ppm)

quisition mine (see)	0.5571	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	28 May 2012 17:04:00	
te Stamp	28 May 2012 17:04:	:00						
Name	C:\Users\Fedor\Des	sktop\C13 Рома Для Стать	и в ЈОС 25.05.12\ги	dn-250512-44Aa-dept135\rudn	-250512-44Aa-dept	t135_001000fid		
quency (MHz)	100.62	Nucleus	13C	Number of Transients	342	Origin	spect	
ginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	dept135	
ceiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	9103.9404	
eep Width (Hz)	29409.97	Temperature (degree	C) 27.000					
-250512-44Aa-dept13	85_001000fid			Compound 4	4Aa	65.58	48.97	
44 4.12 27.18 27.18	00.021	116.26					44.01	
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1 Sep 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

1 Sep 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

1 Sep 2011



1 Sep 2011

quisition Time (sec)	0.5243	Comment	Imported from UXN	MR.		Date	25 Aug 2011 18:01	:36
e Name	C:\Users\Fedor\Des	ktop\12.08.11\rudn-120811	-N4-c13dec\rudn-120	811-N4-c13dec_001000fic		Frequency (MHz)	100.62	
cleus	13C	Number of Transients	4000	Original Points Count	16384	Points Count	16384	
lse Sequence	zgpg	Solvent	DMSO-D6	Sweep Width (Hz)	31250.00	Temperature (degree C	27.000	
				Compound	44Ba		$ \begin{array}{c} 10 \\ 11 \\ 11 \\ 1 \\ 10 \\ 11 \\ 1 \\ 10 \\ 10$	$0 \\ 19 \\ 20 \\ 12a \\ 1 \\ 1 \\ 0 \\ 13 \\ 2 \\ 4 \\ 3 \\ 3 \\ 2 \\ 4 \\ 3 \\ 2 \\ 3 \\ 2 \\ 4 \\ 3 \\ 3 \\ 2 \\ 4 \\ 3 \\ 3 \\ 2 \\ 4 \\ 3 \\ 3 \\ 2 \\ 4 \\ 3 \\ 3 \\ 2 \\ 4 \\ 3 \\ 3 \\ 2 \\ 4 \\ 3 \\ 3 \\ 2 \\ 4 \\ 3 \\ 3 \\ 2 \\ 4 \\ 3 \\ 3 \\ 2 \\ 4 \\ 3 \\ 3 \\ 3 \\ 3 \\ 3 \\ 3 \\ 3 \\ 3 \\ 3$
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1 Sep 2011

Acquisition Time (sec)	0.5243	Comment	Imported from UXNMR.			Date	25 Aug 2011 18:01:36
File Name	C:\Users\Fedor\Des	ktop\12.08.11\rudn-120811	8.11\rudn-120811-N4-c13dec\rudn-120811-N4-c13dec_001000fid			Frequency (MHz)	100.62
Nucleus	13C	Number of Transients	4000	Original Points Count	16384	Points Count	16384
Pulse Sequence	zgpg	Solvent	DMSO-D6	Sweep Width (Hz)	31250.00	Temperature (degree C) 27.000

Compound 44Ba







17 Oct 2011



Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

17 Oct 2011

Acquisition Time (sec)	1.5729	Comment	Imported from UXNMR.			Date	14 Oct 2011 16:23:28
File Name	C:\Users\Fedor\De	esktop\14.10.11\rudn-1410	11-N5\rudn-141011-	-N5_001000fid	Frequency (MHz)	400.14	
Nucleus	1H	Number of Transients	32	Original Points Count	16384	Points Count	16384
Pulse Sequence	zg	Solvent	DMSO-D6	Sweep Width (Hz)	10416.67	Temperature (degree C) 32.000

Compound 44Ab





17 Oct 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.10.2011 21:45:58

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Formula C17H16N2O4	<i>FW</i> 312.3199						
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15	5N/13C/31P Z3379/0400		Date	21 Oct 2011 08:08:32
Date Stamp	21 Oct 2011 08:08:	32					
File Name	C:\Users\Fedor\Des	sktop\14.10.11\rudn-14101	1-N5_1-c13dec\rudr	n-141011-N5_1-c13dec_001000	Ofid	Frequency (MHz)	100.62
Nucleus	13C	Number of Transients	6000	Origin sp	pect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence zg	pgg	Receiver Gain	32768.00
SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz) 10	0053.6455	Sweep Width (Hz)	29409.97
Temperature (degree C	27.000				la la		



Chemical Shift (ppm)

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

22.10.2011 21:50:22

Formula C ₁₇ H ₁₆ N ₂ O ₄	FW 31	2.3199					
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	21 Oct 2011 08:08:32
Date Stamp	21 Oct 201	1 08:08:32					
File Name	C:\Users\F	edor\Desktop\14.10.11\rudn-14101	1-N5_1-c13dec\rud	n-141011-N5_1-c13dec_00	1000fid	Frequency (MHz)	100.62
Nucleus	13C	Number of Transients	6000	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zgpg	Receiver Gain	32768.00
SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10053.6455	Sweep Width (Hz)	29409.97
Temperature (degree C	27.000						





rudn-141011-N5_1-c13dec_001000fid



22.10.2011 21:42:36

Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N/13C/31P Z3379/0400			Date	21 Oct 2011 11:29:04
Date Stamp	21 Oct 2011 1	1:29:04					
File Name	C:\Users\Fedo	r\Desktop\14.10.11\rudn-14101	1-N5_1-dept135\r	Frequency (MHz)	100.62		
Nucleus	13C	Number of Transients	5000	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	dept135	Receiver Gain	32768.00
SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10053.6455	Sweep Width (Hz)	29409.97
Temperature (degree C) 27.000						
rudn-141011-N5_1-dept13	35_001000fid				Compo	und 44Ab	25.03



.42 Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C ₁₆ H ₁₃ BrN ₂ O ₄		FW 377.189	4					11.06.2012 17:47:34
Acquisition Time (sec)	1.5729	Comment	5 mm ONP 1H	1/15N/13C/31P 73370/0400				
Date Stamp	05 Jun 2012 17:36	5:00		10101000111 20079/0400		Date	05 Jun 2012 17:36:00	
File Name	C:\Users\Fedor\De	esktop\C13 Рома Для Ста	ты в ЈОС 25 05 12	Vrudp-250512-44Ac 44Bc 1)-	- 050510 444			
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	n-250512-44Ac-44	4Bc_1_001000fid		
Original Points Count	16384	Owner	root	Painte Count	12	Origin	spect	
Receiver Gain	128.00	SIA/(avaliant) (11-)	1001	Points Count	16384	Pulse Sequence	zg	
Swoon Width (U-)	120.00	Sw(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712 0542	
Sweep width (Hz)	10416.03	Temperature (degree	C) 32.000					







min

OH

Br

0


Formula C ₁₆ H ₁₃ BrN ₂ O ₄		FW 377.189	4				
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H	/15N/13C/31P Z3379/0400		Date	05 Jun 2012 17:36:00
Date Stamp	05 Jun 2012 17:36:	00					
File Name	C:\Users\Fedor\Des	ktop\C13 Рома Для Стат	тыи в JOC 25.05.12	\rudn-250512-44Ac-44Bc_1\rud	n-250512-44Ac-4	4Bc_1_001000fid	
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	12	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	128.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542
Sweep Width (Hz)	10416.03	Temperature (degree	C) 32.000				

11.06.2012 17:47:44



Formula C16H13BrN2O4		FW 377.1894					
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1H/15N	/13C/31P Z3379/0400		Date	05 Jun 2012 17:36:00
Date Stamp	05 Jun 2012 17:36:00		·				
File Name	C:\Users\Fedor\Deskto	р\С13 Рома Для Статы	и в JOC 25.05.12\rudn	-250512-44Ac-44Bc_1\rud	n-250512-44Ac-44Bc	1_001000fid	
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	12	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg
Receiver Gain	128.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542
Sweep Width (Hz)	10416.03	Temperature (degree C) 32,000				

Compounds 44Ac/44Bc





11.06.2012 17:48:05

0

11

12a_

0 21

²⁰-OH 23

Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

469

16.7

76

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1.12

4.6

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4.5

4.4

4.81

3.54

4.7

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.06.2012 17:58:33

Formula C16H13BrN2O4		FW 377.1894					
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	05 Jun 2012 16:25:36
Date Stamp	05 Jun 2012 16:25:36		. e.				
File Name	C:\Users\Fedor\Deskto	р\С13 Рома Для Статьи	в JOC 25.05.12\rud	n-250512-44Ac-44Bc-c13dec	rudn-250512-44Ac	-44Bc-c13dec_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	3812	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10549.4092
Sweep Width (Hz)	29409.97	Temperature (degree C	27.000				

Compounds 44Ac/44Bc



rudn-250512-44Ac-44Bc-c13dec_001000fid



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.06.2012 17:59:09

-OH 23

Br 22

0

11

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19

12a_

13

0

Formula C16H13BrN2O4		FW 377.1894	N				
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H	H/15N/13C/31P Z3379/0400		Date	05 Jun 2012 16:25:36
Date Stamp	05 Jun 2012 16:25:36		6 . %				
File Name	C:\Users\Fedor\Deskto	р\С13 Рома Для Стат	ы в ЈОС 25.05.12	rudn-250512-44Ac-44Bc-c13dec\r	rudn-250512-44Ac	-44Bc-c13dec_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	3812	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10549.4092
Sweep Width (Hz)	29409.97	Temperature (degree	C) 27.000				

Compounds 44Ac/44Bc



11.06.2012 17:59:56

Formula C16H13BrN2O4		FW 377.1894					
Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N/1	13C/31P Z3379/0400		Date	05 Jun 2012 16:25:36
Date Stamp	05 Jun 2012 16:25:36						
File Name	C:\Users\Fedor\Deskto	р\С13 Рома Для Статьи	в JOC 25.05.12\rudn-2	50512-44Ac-44Bc-c13dec	rudn-250512-44Ac-4	4Bc-c13dec_001000fid	
Frequency (MHz)	100.62	Nucleus	13C	Number of Transients	3812	Origin	spect
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
Receiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10549.4092
Sween Width (Hz)	29409 97	Temperature (degree (C) 27 000				

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

11.06.2012 18:00:50

cquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15	N/13C/31P Z3379/0400		Date	05 Jun 2012 16:25:36
ate Stamp	05 Jun 2012 16:25:3	6					
le Name	C:\Users\Fedor\Desk	top\C13 Рома Для Статьи	в JOC 25.05.12\rudr	-250512-44Ac-44Bc-c13dec\r	rudn-250512-44A	c-44Bc-c13dec 001000fid	
requency (MHz)	100.62	Nucleus	13C	Number of Transients	3812	Origin	spect
riginal Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zgpg
eceiver Gain	32768.00	SW(cyclical) (Hz)	29411.77	Solvent	DMSO-d6	Spectrum Offset (Hz)	10549.4092
weep Width (Hz)	29409.97	Temperature (degree (C) 27.000				
				1. 12a			$N = \begin{bmatrix} 12 & -0 \\ 12 & -0 \\ 23 \\ -0 & -0 \\ -0 $
n-250512-44Ac-44Bc-0	c13dec_001000fid			-/			4 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2
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077 4004

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C ₁₆ H ₁₃ BrN ₂ O ₄		FW 377.18	594					
Acquisition Time (sec)	1.5729	Comment	5 mm QNP	1H/15N/13C/31P Z3379/0400		Date	14 Oct 2011 16:40:32	
Date Stamp	14 Oct 2011 16:	:40:32		File Name	D:\NMR\14.10	.11 (Рома)\rudn-141011-N9\	rudn-141011-N9_001000fid	
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	48	Origin	spect	
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg	
Receiver Gain	512.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	
Sweep Width (Hz)	10416.03	Temperature (degree (32.000					



rudn-141011-N9_001000fid

44Bc



17.05.2012 12:41:00

Formula C ₁₆ H ₁₃ BrN ₂ O ₄		FW 3	377.1894					
Acquisition Time (sec)	1.5729	Comment	5 mm QNP 1	H/15N/13C/31P Z3379/0400		Date	14 Oct 2011 16:40:32	
Date Stamp	14 Oct 2011	16:40:32		File Name	D:\NMR\14.10).11 (Рома)\rudn-141011-N9\	\rudn-141011-N9_001000fid	
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	48	Origin	spect	
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg	
Receiver Gain	512.00	SW(cyclical) (Hz)	10416.67	Solvent	DMSO-d6	Spectrum Offset (Hz)	2712.0542	
Sweep Width (Hz)	10416.03	Temperature (dec	ree C) 32 000					

H-6, d H-8, b2t (8,0) ~(7,4)





2 Nov 2011

Acquisition Time (sec)	1.5729	Comment	Imported from UXI	NMR.		Date	14 Oct 2011 16:40:32
File Name	C:\Users\Fedor\De	esktop\14.10.11\rudn-1410*	11-N9\rudn-141011-	-N9_001000fid		Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	48 -	Original Points Count	16384	Points Count	16384
Pulse Sequence	zg	Solvent	DMSO-D6	Sweep Width (Hz)	10416.67	Temperature (degree C)	32.000

Compound 44Bc





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

2 Nov 2011



2 Nov 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

23 Aug 2011

Acquisition Time (sec)	1,6056	Comment	Imported fro	om UXNMR.	Date	23 Aug 2011 10:01:36	
File Name	C:\Users\Fee	dor\Desktop\12.08.11\rudn-19081	1-N11\rudn-1	90811-N11_001000fid	Frequency (MHz)	400.14	
Nucleus	1H	Number of Transients	24	Original Points Count 16384	Points Count	16384	
Pulse Sequence	zg	Solvent	CHLOROF	ORM-D	Sweep Width (Hz)	10204.08	

Temperature (degree C) 27.000

Compound 45A





Acquisition Time (sec)	1 6056	Comment					23 Aug 2011
File Name	C:\Ucore\Eee	Comment	Imported fr	om UXNMR.	Date	23 Aug 2011 10:01:36	
Nuclous	C. IUSEISIFEC	Ion Desktop 12.08.11 \rudn-19081	1-N11\rudn-1	190811-N11_001000fid	Frequency (MHz)	400 14	
Rucieus	П	Number of Transients	24	Original Points Count 16384	Points Count	16384	
Pulse Sequence	zg	Solvent	CHLOROF	ORM-D	Sween Width (H-)	10004	
Temperature (degree C)	27.000				Sweep Width (Hz)	10204.08	



5.6 5.5 5.4 Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

5.3

5.2

5.1

5.0

4.9

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

23 Aug 2011

Acquisition Time (sec)	1.6056	Comment	Imported from UXN	MR.		Date	23 Aug 2011 10:01:36
File Name	C:\Users\Fedor\[Desktop\12.08.11\rudn-19081	311-N11\rudn-190811-N11 001000fid			Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	24	Original Points Count	16384	Points Count	16384
Pulse Sequence	zg	Solvent	CHLOROFORM-D			Sweep Width (Hz)	10204 08
Temperature (degree C)	27.000					oncep main (m)	10204.00

Compound 45A





482

24 Aug 2011



483

24 Aug 2011



45B

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.11.2011 20:01:54

Formula C ₁₅ H ₁₄ N ₂ O ₂	FW 254.2	2839						\oplus	
Acquisition Time (sec)	1.8193	Comment	single_pulse	Date	23 Nov 201	1 11:52:55		Date Stamp	23 Nov 2011 16:28:34
File Name	D:\NMR\21.1	1.11\FZ2064-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	8
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	38.00	Solvent	CHLOROFOF	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	9005.76
Temperature (degree C)	27.700								1.001

Compound 45B





General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.11.2011 20:02:27



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.11.2011 20:02:21



FW

254 2839

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28.11.2011 20:02:14

0 19

Formula C ₁₅ H ₁₄ N ₂ O ₂	FW 254	2839							
Acquisition Time (sec)	1.8193	Comment	single_pulse	Date	23 Nov 201	1 11:52:55		Date Stamp	23 Nov 2011 16:28:34
File Name	D:\NMR\21.1	11.11\FZ2064-1.jdf		Frequency (MHz)	399.78	Nucleus	1H	Number of Transients	8
Origin	ECS 400	Original Points Count	16384	Owner	delta	Points Count	16384	Pulse Sequence	single_pulse.ex2
Receiver Gain	38.00	Solvent	CHLOROFOR	RM-d		Spectrum Offset (Hz)	2398.6931	Sweep Width (Hz)	9005.76
Temperature (degree C) 27.700								

Compound 45B 12a_ E H H 5 4ba H-1 endo, dd (3,0;120) FZ2064-1.jdf H-S exo ddd (12.0; 4.4; 3.3) H-12g endo bid dd 1.63 (3.3; 9.0) 1.66 60 2.29 2.26 2.28 2.28 2.25 2.58 2.57 2.56 2.55 2.15 1.42 0.93 1.05 1.50 2.40 2.20 2.15 2.00 1.80 1.60 1.55 2.65 2.60 2.50 2.45 2.30 2.25 1.95 1.90 1.85 1.75 1.65 2.55 2.35 2.10 2.05 1.70

Chemical Shift (ppm)

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

01.12.2011 16:26:45

quisition Time (sec)	1.0433	Comment	single pulse deco	oupled gated NOE		Date	30 Nov 2011 10:48:13	\$		
te Stamp	30 Nov 2011 1	5:24:08		File Name	D:\NMR\28.11.11	I\FZ2085_13C-1.jdf				
equency (MHz)	100.53	Nucleus	13C	Number of Transients	162	Origin	ECS 400			
iginal Points Count	32768	Owner	delta	Points Count	32768	Pulse Sequence	single_pulse_dec			
ceiver Gain	60.00	Solvent	CHLOROFORM	-d		Spectrum Offset (Hz)	12063.0361			
eep Width (Hz)	31407.04	Temperature (deg	ree C) 26.100							
				Ca			0 1/	\int_{1}^{C}) 9	
				Co	mpouna	45B	8 9 9a	N-12		
							7. 5a	11 4b	12a_1	
							~6~~N	4a-4a-	13 2	
							H 5	H 4ba	4=3	
085_13C-1.jdf						77.12			E.	
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		7.60	27.71 127 127.7					2	00.0	
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168 160	152	144 136	128 120	112 104 Chemical Shi	96 88 ft (ppm)	80 72	64 56	48	40 32	NUT T
	Eada	r I. Zuhlany Eugenius)	/ Nikiting Timur D. Co	loov Madimir D. Zoutoou	Vistan NL Khaustel					

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

01.12.2011 16:27:09

Formula C ₁₅ H ₁₄ N ₂ O ₂	FW 254.28	39					
Acquisition Time (sec)	1.0433	Comment	single pulse deco	upled gated NOE		Date	30 Nov 2011 10:48:13
Date Stamp	30 Nov 2011 15	:24:08		File Name	D:\NMR\28.11.1	1\FZ2085_13C-1.jdf	
Frequency (MHz)	100.53	Nucleus	13C	Number of Transients	162	Origin	ECS 400
Original Points Count	32768	Owner	delta	Points Count	32768	Pulse Sequence	single_pulse_dec
Receiver Gain	60.00	Solvent	CHLOROFORM-	d		Spectrum Offset (Hz)	12063.0361
Sweep Width (Hz)	31407.04	Temperature (degree C)	26.100				







Formula C H N O

FW

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

01.12.2011 16:27:00

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Acquisition Time (sec)	1.0433	Comment	single pulse dec	oupled gated NOE		Date	30 Nov 2011 10:48:13
Date Stamp	30 Nov 2011	15:24:08		File Name	D:\NMR\28.1	11.11\FZ2085 13C-1.idf	
Frequency (MHz)	100.53	Nucleus	13C	Number of Transients	162	Origin	ECS 400
Original Points Count	32768	Owner	delta	Points Count	32768	Pulse Sequence	single pulse dec
Receiver Gain	60.00	Solvent	CHLOROFORM	-d		Spectrum Offset (Hz)	12063 0361
Swoon Width (Ha)	21407.04	Tama anatuma (damaa Ol	00 100				

Sweep Width (Hz) 31407.04 Temperature (degree C) 26.100

254 2839

Compound 45B



FZ2085_13C-1.jdf



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

17 Oct 2011

1.6056	Comment	Imported from	UXNMR.	Date	14 Oct 2011 16:59:44	
C:\Users\Fedd	or\Desktop\14.10.11\rudn-14101	1-N10\rudn-1410	011-N10_001000fid	Frequency (MHz)	400.14	
1H	Number of Transients	20	Original Points Count 16384	Points Count	16384	
zg	Solvent	CHLOROFOR	M-D	Sweep Width (Hz)	10204.08	
27.000						
						0
i	1.6056 C:\Users\Fedd 1H 2g 27.000	1.6056 Comment C:\Users\Fedor\Desktop\14.10.11\rudn-14101 1H Number of Transients zg Solvent 27.000	1.6056CommentImported fromC:\Users\Fedor\Desktop\14.10.11\rudn-141011-N10\rudn-1411HNumber of Transients20ZgSolventCHLOROFOR27.000CHLOROFOR	1.6056 Comment Imported from UXNMR. C:\Users\Fedor\Desktop\14.10.11\rudn-141011-N10\rudn-141011-V10_001000fid 01000fid 1H Number of Transients 20 Original Points Count 16384 2g Solvent CHLOROFORM-D 16384 27.000 1 1 1	1.6056 Comment Imported from UXNMR. Date C:\Users\Fedor\Desktop\14.10.11\rudn-141011-N10\rudn-141011-N10_001000fid Frequency (MHz) 1H Number of Transients 20 Original Points Count 16384 Points Count 2g Solvent CHLOROFORM-D Sweep Width (Hz) Sweep Width (Hz) 27.000 Sweep Width (Hz)	1.6056 Comment Imported from UXNMR. Date 14 Oct 2011 16:59:44 C:\Users\Fedor\Desktop\14.10.11\rudn-141011-N10\rudn-141011-N10_001000fid Frequency (MHz) 400.14 1H Number of Transients 20 Original Points Count 16384 Points Count 16384 2g Solvent CHLOROFORM-D Sweep Width (Hz) 10204.08 27.000 Sweep Width (Hz) 10204.08

Compound 46







492

17 Oct 2011



17 Oct 2011

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Acquisition Time (sec)	1.6056	Comment	Imported from UXNMR.			Date	14 Oct 2011 16:59:44
File Name	C:\Users\Fedor\De	sktop\14.10.11\rudn-14101	1-N10\rudn-141011-N10_001000fid			Frequency (MHz)	400.14
Nucleus	1H	Number of Transients	20	Original Points Count	16384	Points Count	16384
Pulse Sequence	zg	Solvent	CHLOROFORM-D			Sweep Width (Hz)	10204.08
Temperature (degree C)	27.000						

Compound 46



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28 Oct 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28 Oct 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28 Oct 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

28 Oct 2011



Acquisition Time (sec)	1.6056	Comment	Imported from UXN	MR.		Date	23 Aug 2011	10:35:44
File Name	C:\Users\Fedor\I	Desktop\12.08.11\rudn-1908	11-N15\rudn-190811-	N15_001000fid		Frequency (MHz)	400.14	
Nucleus	1H	Number of Transients	20	Original Points Count	16384	Points Count	16384	
Pulse Sequence	zg	Solvent	CHLOROFORM-D			Sweep Width (Hz)	10204.08	
Temperature (degree C)	27.000							
				(H-2)	4		5 4 N H 3	NH 2 2 2 3 4 0 5
				.67		Co	mnound	1 48/49/50
18/10/50 = 58/1	1/28			4			mpound	10 10/ 17/ 50
10/49/30 - 30/1	7/20							
	25					64		
3.07	2	28				ri ri		
u l		5.25						
	7.47	0 4						
		6.6						
		6.7						Σ.
							3.95	2
		24				3.2	2.9(
2 - C		9				66 33.24	8	m m
						e e	1 2.9	.51
							2.92	55
						3.2	16	2.01 2.01 1.7 50
						e.	5 5	85 85
		i		90		59	2.7	922
				2.2		e e	0	1 1
							1	
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	d Allen			A	dl	- I have have		
1.07 1	.02 0.97	1.05 1.09 1.99		1.00		2.26 2.17	2.11	0.94 2.69
8.0 7	.5 7.0	6.5	6.0 5.5	5.0 4.5	4.0	3.5	3.0	2.5 2.0 1.5

Chemical Shift (ppm) Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

25 Aug 2011 23 Aug 2011 10:35:44 Acquisition Time (sec) 1.6056 Comment Imported from UXNMR. Date C:\Users\Fedor\Desktop\12.08.11\rudn-190811-N15\rudn-190811-N15_001000fid File Name 400.14 Frequency (MHz) Original Points Count 16384 16384 Nucleus 1H Number of Transients 20 **Points Count** Sweep Width (Hz) Solvent CHLOROFORM-D 10204.08 Pulse Sequence zg Temperature (degree C) 27.000 Compounds 48/49/50 9 H-8;1.8 6.28 12 24 3 6.25 6.44 8.07 7.31 6.43 2 9 50 47 N 6.70 7.32 6.29 7.48 6.44 8.07 6.44 6.25 25 1.01 1.01 0.90 1.00 1.03 1.98 7.3 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.



Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

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25 Aug 2011



General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

25 Aug 2011 24 Aug 2011 08:12:48 Acquisition Time (sec) 0.5898 Comment Imported from UXNMR. Date File Name C:\Users\Fedor\Desktop\12.08.11\rudn-190811-N15-c13dec\rudn-190811-N15-c13dec_001000fid 100.62 Frequency (MHz) Nucleus 13C 16384 **Points Count** 16384 Number of Transients 3099 **Original Points Count Pulse Sequence** Solvent CHLOROFORM-D Sweep Width (Hz) 27777.78 Temperature (degree C) 27.000 zgpg 3 Compounds 48/49/50 X ×2 C=N 113.79 105.33 50.07 10.11 111.60 48 50 (48) x 2 5 144.69 0 2 41.78 151.68 155.04 49.81 annon the stand where the stand of the stand 158 154 152 150 148 140 138 136 134 132 130 128 126 124 156 146 144 142 120 118 116 110 122 114 112 108 106 104 Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimi P. Zavtsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

503

25 Aug 2011

Acquisition Time (sec)	0.5898	Comment	Imported from UXNN	IR.		Date	24 Aug 2011 08:12:48
File Name	C:\Users\Fedor\Des	ktop\12.08.11\rudn-190811-	N15-c13dec\rudn-190	811-N15-c13dec_001000fic	10001	Frequency (MHz)	100.62
Nucleus	130	Number of Transients	3099	Original Points Count	16384	Points Count	16384 CL 27.000
	4). B	50.35 = N-0	X2 R2-CU2-N	45.71	5 18)	Compounds 48/49/50
77.12 76.80		68.70	A.				= N-042 042-N- (48)
77.44		-N-Cu	2-alz-cuzt	142		NH2 CH CH	2) C42-N 5 € (1) > (43)
		4	F (E			0	31.86
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30 78 76	74 72 70	68 66 64 6	60 58	56 54 52 50 Chemical Shift (ppm)	9 48 46 4	4 42 40 38	8 36 34 32 30 28 :
General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

25.08.2011 18:31:13

Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N	I/13C/31P Z3379/0400		Date	24 Aug 2011 09:14:40	
Date Stamp	24 Aug 2011 0	9:14:40						
File Name	C:\Users\Fedor	Desktop\12.08.11\rudn-190811	-N15-dept135\rudn-1	90811-N15-dept135_0010	00fid	Frequency (MHz)	100.62	
Nucleus	13C	Number of Transients	2007	Origin	spect	Original Points Count	16384	
Owner	root	Points Count	16384	Pulse Sequence	dept135	Receiver Gain	32768.00	
SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9636.4912	Sweep Width (Hz)	29409.97	
a hard of the second provident in a static second second	Contraction of the second s							

Temperature (degree C) 27.000

rudn-190811-N15-dept135_001000fid



504

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150 1040

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

01.08.2013 14:18:11

Formula C ₁₀ H ₉ NO	159.1840							
Acquisition Time (sec)	1.6056	Comment	5 mm QNP 1H/1	5N/13C/31P Z3379/0400		Date	31 Jul 2013 14:56:00	
Date Stamp	31 Jul 2013 14:56:0	00		File Name	C:\Users\asus\De: 2013\nmrlastsamp	sktop\Рома лето lles\FZ-310713-N1\FZ-31	10713-N1_001000fid	
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	8	Origin	spect	
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	zg	
Receiver Gain	256.00	SW(cyclical) (Hz)	10204.08	Solvent	CHLOROFORM-d	1		
Spectrum Offset (Hz)	2602.0486	Sweep Width (Hz)	10203.46	Temperature (degree C	27.000			







C:\Users\asus\Desktop\Poмa лето 2013\nmrlastsamples\FZ-310713-N1\FZ-310713-N1_001000fid

Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

Formula C ₁₀ H ₉ NO	FW 159.1846							01.08.2013 14:18:0
Acquisition Time (see	1.6056	Comment	5 mm QNP 1	1/15N/13C/31P 73379/0400		Data		
Date Stamp	31 Jul 2013 14:5	6:00		File Name	C:\Users\asus\De	esktop\Poma лето	31 Jul 2013 14:56:00	
Frequency (MHz)	400.14	Nucleus	1H	Number of Transients	8	Origin	10/13-N1_001000fid	
Original Points Count	16384	Owner	root	Points Count	16384	Pulse Sequence	spect	
Receiver Gain	256.00	SW(cyclical) (Hz)	10204.08	Solvent	CHI OBOEORM-	d	Zg	
Spectrum Offset (Hz)	2602.0486	Sweep Width (Hz)	10203.46	Temperature (dearee C	27 000	u		



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Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

01.08.2013 14:23:38

Formula C ₁₀ H ₉ NO	FW 159.1846						
Acquisition Time (sec	.) 0.5571	Comment	5 mm QNP 1H/15N	I/13C/31P Z3379/0400		Date	31 Jul 2013 15:02:24
Date Stamp	31 Jul 2013 15:02:24	4	,				
File Name	C:\Users\asus\Desk	top\Poмa лето 2013\nmrla	stsamples\FZ-310713	-N1-c13dec\FZ-310713-N1	-c13dec_001000fid	Frequency (MHz)	100.62
Nucleus	13C	Number of Transients	318	Origin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zgpg	Receiver Gain	32768.00
SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9630.2021	Sweep Width (Hz)	29409.97
Temperature (degree	CI 27 000						





C:\Users\asus\Desktop\Poma лето 2013\nmrlastsamples\FZ-310713-N1-c13dec\FZ-310713-N1-c13dec_001000fid Fedor I. Zubkov, Eugeniya V. Nikitina, Timur R. Galeev, Vladimir P. Zaytsev, Victor N. Khrustalev, Roman A. Novikov, and Alexey V. Varlamov

เป็นสู่สุดที่สาวใหญ่ เหล่างหลางการเหล่างเกม

General Synthetic Approach towards Annelated 3a.6-Epoxylsoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

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Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N	13C/31P Z3379/0400		Date	31 Jul 2013 15:02:24
Date Stamp	31 Jul 2013 15:02:2	24				1	
File Name	C:\Users\asus\Des	ktop\Poмa пето 2013\nmrla	stsamples\FZ-310713	N1-c13dec\EZ-310713-N1	-c13dec 001000fid	Frequency (MHz)	100.62
Nucleus	13C	Number of Transients	318	Oriain	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	zapa	Receiver Gain	32768.00
SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	9630.2021	Sweep Width (Hz)	29409.97
Temperature (degree C	27.000						/
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FZ-310713-N1-c13dec_00	D1000fid				5.93 125.94 50	4	-109.61
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155.21			-139.90 -136.78			08 58	

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General Synthetic Approach towards Annelated 3a,6-Epoxyisoindoles by Tandem Acylation/IMDAF Reaction of Furylazaheterocycles. Scope and Limitations.

01.08.2013 14:22:02

Acquisition Time (sec)	0.5571	Comment	5 mm QNP 1H/15N	/13Ci31P Z3379/0400		Date	31 Jul 2013 15:04:32
Date Stamp	31 Jul 2013 15:04:3	2					
File Name	C:\Users\asus\Des	ktop\Poma лето 2013\nmrla	stsamples\FZ-310713-	N1-dept135\FZ-310713-N1	-dept135_001000fid	Frequency (MHz)	100.62
Nucleus	13C	Number of Transients	355	Orgin	spect	Original Points Count	16384
Owner	root	Points Count	16384	Pulse Sequence	dept135	Receiver Gain	32768.00
SW(cyclical) (Hz)	29411.77	Solvent	CHLOROFORM-d	Spectrum Offset (Hiz)	9630.2119	Sweep Width (Hz)	29409.97
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