

Two Concurrent Pathways of the Reaction of Pyrrolobenzoxazinetriones with Cyclic Alkoxyolefins. Synthesis of Alkaloid-Like Pentacyclic 6/6/5/6/5- and 6/6/5/6/6-Angularly Fused Heterocycles

E. E. Stepanova^a, M. V. Dmitriev^a, P. A. Slepukhin^b, and A. N. Maslivets^{a*}

^a Perm State National Research University, ul. Bukireva 15, Perm, 614990 Russia

*e-mail: koh2@psu.ru

^b Postovskii Institute of Organic Synthesis, Ural Branch, Russian Academy of Sciences, ul. S. Kovalevskoi/Akademicheskaya 22/20, Yekaterinburg, 620990 Russia

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Abstract—3-Aroylpyrrolo[2,1-*c*][1,4]benzoxazine-1,2,4-triones reacted with 2,3-dihydrofuran and 3,4-dihydro-2*H*-pyran to give mixtures of the corresponding hetero-Diels–Alder and Michael addition products.

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A combination of high stereoselectivity, atom economy, and experimental simplicity makes hetero-Diels–Alder reactions a valuable synthetic approach to polyheterocyclic compounds [1–3]. It was shown previously that monocyclic 4-acyl-1*H*-pyrrole-2,3-diones act as heterodienes in [4+2]-cycloadditions to cyclic and acyclic alkoxyolefins to afford substituted pyrano-[4,3-*b*]pyrroles with high diastereoselectivity (only *endo*-cycloadducts were obtained) [4–10].

4-Acyl-substituted 1*H*-pyrrole-2,3-diones fused through the N¹–C⁵ bond to a benzo[*b*][1,4]oxazin-2-one fragment (3-arylpyrrolo[2,1-*c*][1,4]benzoxazine-1,2,4-triones) are interesting model structures for studying hetero-Diels–Alder reactions due to the presence in their molecules of a nonplanar heterodiene fragment (C^{3a}=C³–C=O), which hinders cycloaddition according to the concerted mechanism or makes it impossible. The cycloadditions of 3-arylpyrrolo[2,1-*c*][1,4]benzoxazine-1,2,4-triones and their aza analogs, 3-arylpyrrolo[1,2-*a*]quinoxaline-1,2,4(5*H*)-triones and 3-aryl-4-arylpyrrolo[1,2-*a*]quinoxaline-1,2-diones, with acyclic alkoxyolefins lead to the formation of mixtures of diastereoisomeric [4+2]-cycloadducts, the *exo* adducts being the major products [11–14]. In order to find out how the dienophile structure affects the direction and diastereoselectivity of the above cycloadditions, we studied reactions of 3-aryl-

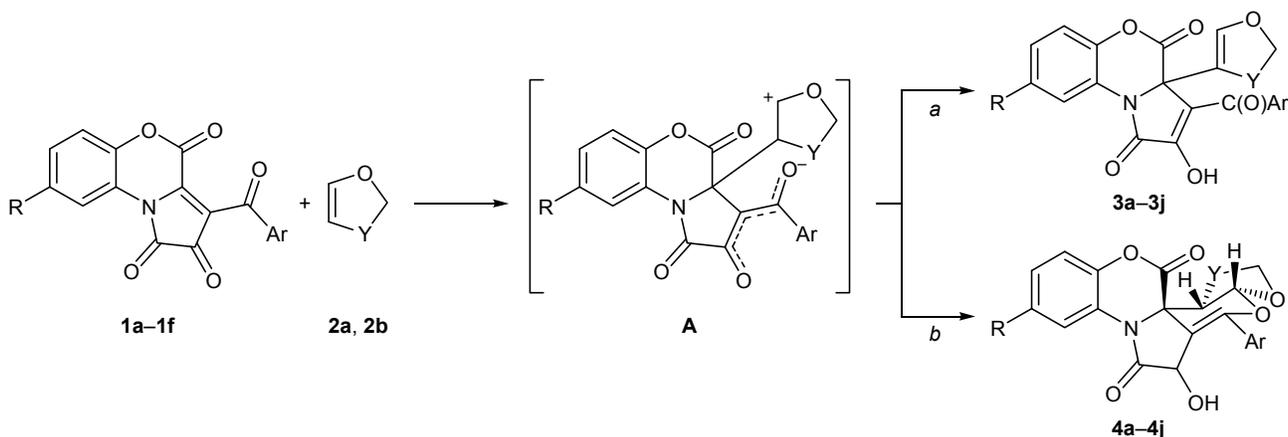
pyrrolo[2,1-*c*][1,4]benzoxazine-1,2,4-triones with cyclic alkoxyolefins.

The reactions of aroylpyrrolobenzoxazinetriones **1a–1f** with 2,3-dihydrofuran (**2a**) and 3,4-dihydro-2*H*-pyran (**2b**) were carried out at a reactant ratio of 1:1.5 in benzene under reflux until disappearance of the violet color typical of compounds **1a–1f** (2–90 min). As a result, mixtures of Michael adducts **3a–3j** and cycloadducts **4a–4j** were formed, which were separated by fractional crystallization [15] (Scheme 1).

Compounds **3a–3j** are high-melting (with decomposition) yellow crystalline substances that are readily soluble in DMSO, aromatic and halogenated hydrocarbons, ethyl acetate, acetone, 1,4-dioxane, and alcohols, poorly soluble in carbon tetrachloride and saturated hydrocarbons, and insoluble in water. They showed a positive color test (cherry color) for enolic hydroxyl on treatment with an alcoholic solution of iron(III) chloride.

The IR spectra of **3a–3j** (mineral oil) contained absorption bands due to stretching vibrations of the hydroxy group (3087–3200 cm⁻¹) and lactone (C⁴=O, 1766–1785 cm⁻¹), lactam (C¹=O, 1690–1727 cm⁻¹), and ketone carbonyl groups (3-C=O, 1641–1664 cm⁻¹). Compounds **3a** and **3e–3j** showed in the ¹H NMR spectra (DMSO-*d*₆) signals from protons of the methylene groups, aromatic rings and substituents therein,

Scheme 1.



1, R = H, Ar = Ph (**a**), 4-EtOC₆H₄ (**c**), 4-BrC₆H₄ (**d**), 4-MeOC₆H₄ (**e**), 4-O₂NC₆H₄ (**f**); R = Cl, Ar = Ph (**b**); **2**, Y = CH₂ (**a**), CH₂CH₂ (**b**); **3**, **4**, Y = CH₂, Ar = Ph, R = H (**a**), Cl (**b**); R = H, Ar = 4-EtOC₆H₄ (**c**), 4-BrC₆H₄ (**d**); Y = CH₂CH₂, Ar = Ph, R = H (**e**), Cl (**f**); R = H, Ar = 4-MeOC₆H₄ (**g**), 4-EtOC₆H₄ (**h**), 4-BrC₆H₄ (**i**), 4-O₂NC₆H₄ (**j**).

a signal from the vinylic proton in the furan or pyran ring [multiplet (**3a**) or singlet (**3e-3j**)] in the region δ 6.23–6.46 ppm, and a broadened singlet from the enolic hydroxy group at δ 11.54–11.93 ppm. In the ¹H NMR spectra of **3b-3d** recorded from solutions in CDCl₃, the vinylic proton signal appeared as a multiplet in the region δ 6.30–6.35 ppm, and the OH signal was a broadened singlet at δ 9.34–9.87 ppm.

The ¹³C NMR spectra of **3a-3e** and **3g-3i** in DMSO-*d*₆ or CDCl₃ displayed signals at δ_c 67.4–71.1 (C^{3a}), 106.4–109.3 (C^{4'} in **3a-3d** or C^{5'} in **3e** and **3g-3i**), 113.1–116.9 (C³), 141.8–143.3 (C^{5'} in **3a-3d** or C^{6'} in **3e** and **3g-3i**), 148.0–153.3 (C²), 160.9–163.1 (C⁴), 162.4–164.3 (C¹), and 187.6–190.2 ppm (3-C=O).

Compounds **4a-4j** are yellow crystalline substances melting with decomposition at high temperature. They are poorly soluble in DMSO, aromatic and halogenated hydrocarbons, ethyl acetate, acetone, and 1,4-dioxane, and insoluble in alkanes and water.

The IR spectra of **4a-4j** dispersed in mineral oil displayed absorption bands at 1766–1793 (lactone carbonyl) and 1710–1741 cm⁻¹ (lactam and ketone carbonyls). In the ¹H NMR spectra of **4a-4d** in DMSO-*d*₆ or CDCl₃ we observed signals from methylene groups and aromatic rings and substituents in the latter, a multiplet at δ 3.02–3.22 ppm due to 2-H, and a doublet at δ 6.00–6.05 ppm ($J = 5.3$ Hz) due to 6-H. The 2-H and 7-H protons of **4e-4j** resonated in the ¹H NMR spectra (DMSO-*d*₆) at δ 2.44–2.60 (m) and 5.71–5.80 ppm (d, $J = 3.5$ –4.0 Hz), respectively.

The ¹³C NMR spectra of **4a-4c** in DMSO-*d*₆ or CDCl₃ contained the following signals, δ_c , ppm: 42.2–

43.6 (C²), 58.8–59.3 (C¹), 68.6–69.3 (C⁴), 96.7–99.4 (C⁶), 103.1–104.1 (C⁹), 159.2–159.9 (C¹¹), 162.0–163.1 (C²⁰=O), 167.0–170.4 (C⁸), 173.9–176.1 (C¹⁰). In the ¹³C NMR spectra of **4e-4i** in DMSO-*d*₆, the corresponding signals were located at δ_c 32.5–33.9 (C²), 60.6–61.0 (C¹), 60.8–61.7 (C⁵), 96.9–97.6 (C⁷), 97.8–99.4 (C¹⁰), 160.0–160.6 (C¹²), 162.9–166.1 (C²¹), 165.7–167.4 ppm (C⁹), and 175.5–176.2 ppm (C¹¹). Also, signals of the methylene carbon atoms and aromatic substituents were present in the spectra of **4a-4c** and **4e-4i**.

According to the X-ray diffraction data (Fig. 1), compound **3i** crystallized in space group *Ia-3d* belonging to the cubic crystal system, which is very rare for organic compounds. The bond lengths and bond angles

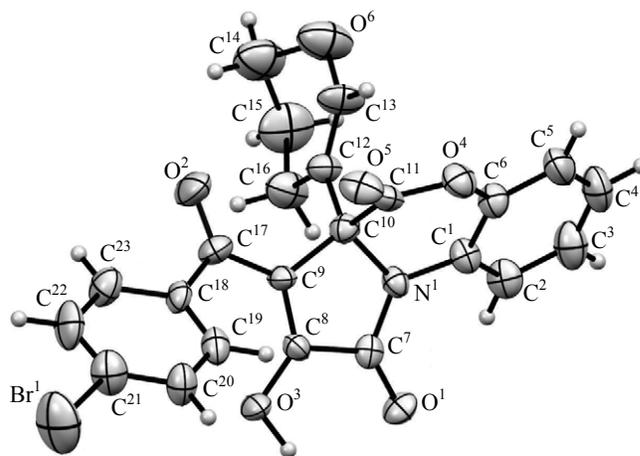


Fig. 1. Structure of the molecule of 3-(4-bromobenzoyl)-3a-(3,4-dihydro-2H-pyran-5-yl)-2-hydroxypyrrolo[2,1-*c*][1,4]-benzoxazine-1,4(3aH)-dione (**3i**) according to the X-ray diffraction data.

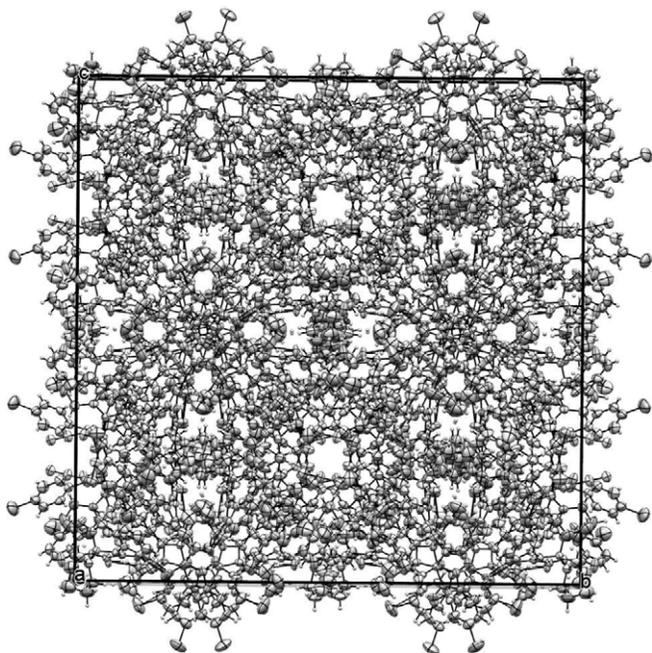


Fig. 2. A fragment of crystal packing of 3-(4-bromobenzoyl)-3a-(3,4-dihydro-2*H*-pyran-5-yl)-2-hydroxypyrrolo[2,1-*c*]-[1,4]benzoxazine-1,4(3*aH*)-dione (**3i**).

in molecule **3i** are close to the corresponding reference values, except for some deviations related, in particular, to the presence of sp^3 -hybridized C^{10} atom among bridgehead carbon atoms of the conjugated tricyclic system. The enolic proton in the $O^2C^{17}C^9C^8O^3$ fragment is located on the O^3 atom of the dihydropyrrole ring, and this structure is stabilized by strong intramolecular hydrogen bond with the O^1 atom of the neighboring carbonyl group. The 3-oxacyclohexene ring adopts a *sofa* conformation with strong thermal disordering of the C^{14} atom. Molecules **3i** in crystal are packed through a system of intermolecular hydrogen

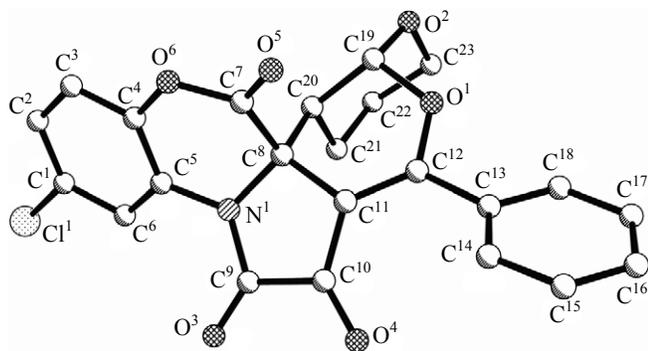


Fig. 3. Structure of the molecule of (2*S**,1*R**,7*R**)-16-chloro-9-phenyl-6,8,20-trioxa-13-azapentacyclo-[11.8.0.0^{1,10}.0^{2,7}.0^{14,19}]henicososa-9,14,16,18-tetraene-11,12,21-trione (**4f**) according to the X-ray diffraction data. Hydrogen atoms are not shown for clarity.

bonds which link them to three-dimensional tetramers, and the latter give rise eventually to a non-primitive cubic unit cell. The atoms involved in intermolecular hydrogen bonding form fairly wide pores oriented along one of the fourth-order symmetry axes (Fig. 2).

Compound **4f** (Fig. 3) crystallized in *P*-1 centrosymmetric space group belonging to the triclinic crystal system. All bond lengths and bond angles in molecule **4f** fall within the corresponding reference ranges. The pyrrole ring is planar, the tetrahydropyran ring appears in a *chair* conformation, and the dihydropyran and oxazine rings adopt a *distorted sofa* conformation bent through dihedral angles of 41.2 and 49.4° along the $C^{19}C^8$ and C^7N^1 lines, respectively. The crystal packing of **4f** is stabilized by van der Waals interactions without essential contribution of specific short contacts.

Compounds **3a–3j** were isolated as the major products, while **4a–4j** were the minor ones. The ratio **3/4** depended on the solvent used. When the reaction was carried out in benzene, the ratio **3/4** was ~2:1 (according to the 1H NMR data for reaction mixtures), whereas in 1,4-dioxane and acetonitrile the product ratio was ~2:1 and ~10:1, respectively. Furthermore, the violet color of compounds **1a–1f** in the reaction carrier out in acetonitrile disappeared more rapidly than in benzene.

Hetareno[*e*]pyrrole-2,3-diones **1a–1f** reacted with 2,3-dihydrofuran (**2a**) appreciably more readily than with 3,4-dihydro-2*H*-pyran **2b**, which is very consistent with the Mayr–Patz nucleophilicity scale [16, 17].

We previously believed [15, 18] that compounds **3a–3j** are formed as a result of either nucleophilic addition of cyclic alkoxyolefins **2a** and **2b** to hetareno[*e*]pyrrole-2,3-diones **1a–1f** or hydrolysis of diastereoisomeric compounds **4a–4j** with subsequent dehydration or isomerization of **4a–4j**. However, no signals assignable to diastereoisomers of **4a–4j** were observed in the 1H NMR spectra of the reaction mixtures. The corresponding compounds **3a–3j** and **4a–4j** were not converted into each other on heating in organic solvents (benzene, 1,4-dioxane, acetonitrile) under reflux or on melting (TLC, UHPLC). Hydrolysis of **4a–4j** on heating in organic solvents (benzene, 1,4-dioxane, acetonitrile, DMSO) in the presence of water, as well as of acid catalysts (acetic acid or HCl) or by keeping these compounds in the same media at room temperature for several weeks did not produce compounds **3a–3j** (TLC, UHPLC). These findings indicated that the most probable way of formation of compounds **3a–3j**

and **4a–4j** is nucleophilic addition of cyclic alkoxyolefins **2a** and **2b** to the C^{3a} atom of hetareno[*e*]pyrrole-2,3-diones **1a–1f** to give zwitterionic intermediate **A** (Scheme 1). The latter undergoes either 1,5-prototropic shift leading to adducts **3a–3j** (pathway *a*) or intramolecular cyclization involving the cationic carbon atom of the cyclic alkoxyolefin residue and carbonyl oxygen atom of the aroyl substituent, yielding **4a–4j** (pathway *b*). Compounds **4a–4j** are formed as a single diastereoisomer (*endo* cycloadduct) which is not typical of reactions of hetareno[*e*]pyrrole-2,3-diones like **1** with olefins [11–14, 19].

Molecules **4a–4j** possess a pentacyclic alkaloid-like skeleton based on perhydrobenzo[*c*]indene structure. Interest in compounds containing such structural fragment continuously increases since many structurally related natural compounds (dankasterone [20], pretazettine [21], erythroidine [22, 23], etc.) possess pronounced biological activity.

EXPERIMENTAL

The ¹H and ¹³C NMR spectra were recorded on Bruker Avance III 500, Bruker Avance DRX-400, Bruker Avance DRX-500 equipped with a dual carbon/proton (CPDUL) cryoprobe, and Bruker Avance III HD 400 spectrometers using tetramethylsilane or hexamethyldisiloxane as internal standards. The IR spectra were recorded on FSM-1201 and Perkin Elmer Spectrum Two spectrometers. The elemental analyses were obtained on Perkin Elmer 2400 Series II and Vario MICRO cube analyzers. The reaction conditions were optimized by ¹H NMR (Bruker Avance III HD 400) and UHPLC monitoring (Waters ACQUITY UPLC I-Class; Acquity UPLC BEH C18 column, grain size 1.7 μm; eluent acetonitrile–water, flow rate 0.6 mL/min; ACQUITY UPLC PDA eλ UV detector; Xevo TQD detector; positive electrospray ionization; ion source temperature 150°C; capillary voltage 3500–4000 V; cone voltage 20–70 V; vaporizer temperature 150–300°C). The purity of the isolated compounds was checked by TLC on Silufol plates using benzene–ethyl acetate (5 : 1) as eluent; development with iodine vapor.

3-Benzoyl-3a-(2,3-dihydrofuran-4-yl)-2-hydroxypyrrolo[2,1-*c*][1,4]benzoxazine-1,4(3a*H*)-dione (3a). A solution of 0.33 g (4.7 mmol) of 2,3-dihydrofuran (**2a**) in 5 mL of anhydrous benzene was added to a solution of 1.00 g (3.1 mmol) of compound **1a** in 10 mL of anhydrous benzene, and the mixture was refluxed for 5 min (until the original violet color disappeared). The mixture was cooled, and the yellow precipitate

(compound **4a**) was filtered off. The mother liquor was diluted with 70 mL of hexane, and the yellow solid (compound **3a**) was filtered off. Yield 0.73 g (60%), mp 262–263°C (decomp., from hexane). IR spectrum, ν , cm⁻¹: 3200 br (O–H), 1766 (C⁴=O), 1697 (C¹=O), 1651 (3-C=O). ¹H NMR spectrum (400 MHz, DMSO-*d*₆), δ , ppm: 2.34 m and 2.51 m (1H each, 3'-H), 4.16 m and 4.37 m (1H each, 2'-H), 6.38 m (1H, 5'-H), 7.33–7.92 m (9H, H_{arom}), 11.74 br.s (1H, OH). ¹³C NMR spectrum (100 MHz, DMSO-*d*₆), δ _C, ppm: 27.5 (C^{3'}), 63.3 (C^{2'}), 70.2 (C^{3a}), 109.3 (C^{4'}), 114.9 (C³), 116.4–137.6 (C_{arom}), 143.3 (C^{6'}), 145.6 (C_{arom}), 150.2 (C²), 160.9 (C⁴), 162.4 (C¹), 189.9 (3-C=O). Found, %: C 67.75; H 3.93; N 3.68. C₂₂H₁₅NO₆. Calculated, %: C 67.86; H 3.88; N 3.60.

Compounds **3b–3j** and **4b–4j** were synthesized in a similar way.

3-Benzoyl-8-chloro-3a-(2,3-dihydrofuran-4-yl)-2-hydroxypyrrolo[2,1-*c*][1,4]benzoxazine-1,4(3a*H*)-dione (3b). Yield 0.77 g (58%), mp 243–244°C (decomp., from hexane). IR spectrum, ν , cm⁻¹: 3188 br (O–H), 1783 (C⁴=O), 1714 (C¹=O), 1650 (3-C=O). ¹H NMR spectrum (400 MHz, CDCl₃), δ , ppm: 2.31 m and 2.46 m (1H each, 3'-H), 4.24 m and 4.35 m (1H each, 2'-H), 6.30 m (1H, 5'-H), 7.29–7.88 m (8H, H_{arom}), 9.87 br.s (1H, OH). ¹³C NMR spectrum (100 MHz, CDCl₃), δ _C, ppm: 27.6 (C^{3'}), 64.1 (C^{2'}), 71.1 (C^{3a}), 106.9 (C^{4'}), 115.3 (C³), 117.9–136.6 (C_{arom}), 141.8 (C^{6'}), 142.4 (C_{arom}), 148.2 (C²), 161.3 (C⁴), 162.6 (C¹), 189.8 (3-C=O). Found, %: C 62.27; H 3.49; N 3.35. C₂₂H₁₄ClNO₆. Calculated, %: C 62.35; H 3.33; N 3.31.

3a-(2,3-Dihydrofuran-4-yl)-3-(4-ethoxybenzoyl)-2-hydroxypyrrolo[2,1-*c*][1,4]benzoxazine-1,4(3a*H*)-dione (3c). Yield 0.83 g (62%), mp 238–239°C (decomp., from CCl₄). IR spectrum, ν , cm⁻¹: 3188 br (O–H), 1782 (C⁴=O), 1690 (C¹=O), 1641 (3-C=O). ¹H NMR spectrum (400 MHz, CDCl₃), δ , ppm: 1.45 t (3H, Me, *J* = 7.0 Hz), 2.31 m and 2.45 m (1H each, 3'-H), 4.12 q (2H, CH₂Me, *J* = 7.0 Hz), 4.20 m and 4.32 m (1H each, 2'-H), 6.35 m (1H, 5'-H), 6.93–7.93 m (8H, H_{arom}), 9.67 br.s (1H, OH). ¹³C NMR spectrum (100 MHz, CDCl₃), δ _C, ppm: 14.6 (Me), 27.7 (C^{3'}), 63.9 (CH₂Me), 64.0 (C^{2'}), 71.0 (C^{3a}), 107.4 (C^{4'}), 114.2 (C³), 116.8–133.5 (C_{arom}), 143.3 (C^{6'}), 144.0 (C_{arom}), 148.0 (C²), 161.2 (C⁴), 163.3 (C¹), 164.4 (C_{arom}), 187.6 (3-C=O). Found, %: C 66.58; H 4.35; N 3.36. C₂₄H₁₉NO₇. Calculated, %: C 66.51; H 4.42; N 3.23.

3-(4-Bromobenzoyl)-3a-(2,3-dihydrofuran-4-yl)-2-hydroxypyrrolo[2,1-*c*][1,4]benzoxazine-1,4(3a*H*)-

dione (3d). Yield 0.79 g (55%), mp 200–201°C (decomp., from hexane). IR spectrum, ν , cm^{-1} : 3200 (O–H), 1780 ($\text{C}^4=\text{O}$), 1715 ($\text{C}^1=\text{O}$), 1651 (3-C=O). ^1H NMR spectrum (400 MHz, CDCl_3), δ , ppm: 2.32 m and 2.44 m (1H each, 3'-H), 4.22 m and 4.36 m (1H each, 2'-H), 6.32 m (1H, 5'-H), 7.21–7.76 m (8H, H_{arom}), 9.34 br.s (1H, OH). ^{13}C NMR spectrum (100 MHz, CDCl_3), δ_{C} , ppm: 27.6 (C^3), 64.3 (C^2), 71.0 ($\text{C}^{3\text{a}}$), 107.99 (C^4), 114.8 (C^3), 116.9–135.4 (C_{arom}), 143.3 (C^6), 143.9 (C_{arom}), 148.2 (C^2), 161.1 (C^4), 164.3 (C^1), 188.6 (3-C=O). Found, %: C 56.58; H 2.93; N 2.87. $\text{C}_{22}\text{H}_{14}\text{BrNO}_6$. Calculated, %: C 56.43; H 3.01; N 2.99.

3-Benzoyl-3a-(3,4-dihydro-2H-pyran-5-yl)-2-hydroxypyrrolo[2,1-c][1,4]benzoxazine-1,4(3aH)-dione (3e). Yield 0.78 g (62%), mp 120–122°C (decomp., from isooctane). IR spectrum, ν , cm^{-1} : 3200 (O–H), 1776 ($\text{C}^4=\text{O}$), 1715 ($\text{C}^1=\text{O}$), 1654 (3-C=O). ^1H NMR spectrum (400 MHz, $\text{DMSO}-d_6$), δ , ppm: 1.60 m (2H, 3'-H), 1.80 m (2H, 4'-H), 3.70 m and 3.87 m (1H each, 2'-H), 6.26 s (1H, 6'-H), 7.33–7.90 m (9H, H_{arom}), 11.76 br.s (1H, OH). ^{13}C NMR spectrum (126 MHz, $\text{DMSO}-d_6$), δ_{C} , ppm: 17.1 (C^4), 20.9 (C^3), 65.1 (C^2), 67.4 ($\text{C}^{3\text{a}}$), 106.5 (C^5), 113.1 (C^3), 116.5–137.7 (C_{arom}), 142.8 (C^6), 143.4 (C_{arom}), 150.9 (C^2), 161.4 (C^4), 162.8 (C^1), 190.0 (3-C=O). Found, %: C 68.32; H 4.35; N 3.26. $\text{C}_{23}\text{H}_{17}\text{NO}_6$. Calculated, %: C 68.48; H 4.25; N 3.47.

3-Benzoyl-8-chloro-3a-(3,4-dihydro-2H-pyran-5-yl)-2-hydroxypyrrolo[2,1-c][1,4]benzoxazine-1,4(3aH)-dione (3f). Yield 0.91 g (66%), mp 235–237°C (decomp., from isooctane). IR spectrum, ν , cm^{-1} : 3175 (O–H), 1770 ($\text{C}^4=\text{O}$), 1727 ($\text{C}^1=\text{O}$), 1664 (3-C=O). ^1H NMR spectrum (400 MHz, $\text{DMSO}-d_6$), δ , ppm: 1.64 m (2H, 3'-H), 1.80 m (2H, 4'-H), 3.74 m and 3.89 m (1H each, 5'-H), 6.27 s (1H, 6'-H), 7.40–7.90 m (8H, H_{arom}), 11.84 br.s (1H, OH). Found, %: C 63.24; H 3.47; N 3.53. $\text{C}_{23}\text{H}_{16}\text{ClNO}_6$. Calculated, %: C 63.09; H 3.68; N 3.20.

3a-(3,4-Dihydro-2H-pyran-5-yl)-2-hydroxy-3-(4-methoxybenzoyl)pyrrolo[2,1-c][1,4]benzoxazine-1,4(3aH)-dione (3g). Yield 0.83 g (61%), mp 104–105°C (decomp., from isooctane). IR spectrum, ν , cm^{-1} : 3187 (O–H), 1776 ($\text{C}^4=\text{O}$), 1715 ($\text{C}^1=\text{O}$), 1651 (3-C=O). ^1H NMR spectrum (500 MHz, $\text{DMSO}-d_6$), δ , ppm: 1.59 m (2H, 3'-H), 1.80 m (2H, 4'-H), 3.69 m (1H, 5'-H), 3.86 m (4H, MeO, 5'-H), 6.23 s (1H, 6'-H), 7.02–7.88 m (8H, H_{arom}), 11.56 br.s (1H, OH). ^{13}C NMR spectrum (126 MHz, $\text{DMSO}-d_6$), δ_{C} , ppm: 17.0 (C^4), 20.9 (C^3), 55.5 (Me), 65.1 (C^2), 67.5 ($\text{C}^{3\text{a}}$),

106.6 (C^5), 113.5 (C^3), 115.2–131.9 (C_{arom}), 142.7 (C^6), 143.4 (C_{arom}), 153.0 (C^2), 161.4 (C^4), 162.8 (C^1), 163.2 (C_{arom}), 188.4 (3-C=O). Found, %: C 66.68; H 4.38; N 3.52. $\text{C}_{24}\text{H}_{19}\text{NO}_7$. Calculated, %: C 66.51; H 4.42; N 3.23.

3a-(3,4-Dihydro-2H-pyran-5-yl)-3-(4-ethoxybenzoyl)-2-hydroxypyrrolo[2,1-c][1,4]benzoxazine-1,4(3aH)-dione (3h). Yield 0.94 g (68%), mp 119–121°C (decomp., from isooctane). IR spectrum, ν , cm^{-1} : 3200 (O–H), 1774 ($\text{C}^4=\text{O}$), 1723 ($\text{C}^1=\text{O}$), 1654 (3-C=O). ^1H NMR spectrum (400 MHz, $\text{DMSO}-d_6$), δ , ppm: 1.36 t (3H, Me, $J = 6.9$ Hz), 1.59 m (2H, 3'-H), 1.81 m (2H, 4'-H), 3.69 m and 3.87 m (1H each, 2'-H), 4.14 q (2H, CH_2Me , $J = 6.9$ Hz), 6.23 s (1H, 6'-H), 7.00–7.87 m (8H, H_{arom}), 11.54 br.s (1H, OH). ^{13}C NMR spectrum (126 MHz, $\text{DMSO}-d_6$), δ_{C} , ppm: 14.5 (Me), 17.1 (C^4), 20.9 (C^3), 63.5 (C^2), 65.1 (CH_2Me), 67.5 ($\text{C}^{3\text{a}}$), 106.6 (C^5), 113.9 (C^3), 114.2–131.9 (C_{arom}), 142.7 (C^6), 143.0 (C_{arom}), 143.4 (C_{arom}), 150.0 (C^2), 161.4 (C^4), 162.5 (C^1), 162.9 (C_{arom}), 188.4 (3-C=O). Found, %: C 67.27; H 4.76; N 3.15. $\text{C}_{25}\text{H}_{21}\text{NO}_7$. Calculated, %: C 67.11; H 4.73; N 3.13.

3-(4-Bromobenzoyl)-3a-(3,4-dihydro-2H-pyran-5-yl)-2-hydroxypyrrolo[2,1-c][1,4]benzoxazine-1,4(3aH)-dione (3i). Yield 0.79 g (53%), mp 170–172°C (decomp., from toluene). IR spectrum, ν , cm^{-1} : 3087 (O–H), 1778 ($\text{C}^4=\text{O}$), 1703 ($\text{C}^1=\text{O}$), 1650 (3-C=O). ^1H NMR spectrum (500 MHz, $\text{DMSO}-d_6$), δ , ppm: 1.59 m (2H, 3'-H), 1.79 m (2H, 4'-H), 3.70 m and 3.87 m (1H each, 5'-H), 6.27 s (1H, 6'-H), 7.34–7.84 m (8H, H_{arom}), 11.93 br.s (1H, OH). ^{13}C NMR spectrum (126 MHz, $\text{DMSO}-d_6$), δ_{C} , ppm: 17.1 (C^4), 20.9 (C^3), 65.2 (C^2), 67.4 ($\text{C}^{3\text{a}}$), 106.4 (C^5), 114.3 (C^3), 116.5–136.7 (C_{arom}), 142.9 (C^6), 143.4 (C_{arom}), 151.0 (C^2), 161.4 (C^4), 162.7 (C^1), 189.2 (3-C=O). Found, %: C 57.36; H 3.53; N 2.78. $\text{C}_{23}\text{H}_{16}\text{BrNO}_6$. Calculated, %: C 57.28; H 3.34; N 2.90.

The X-ray diffraction data for compound 3i were obtained from a $0.31 \times 0.25 \times 0.10$ -mm colorless single crystal on an Xcalibur S automated diffractometer according to standard procedure [MoK radiation, graphite monochromator, ω -scanning with a step of 1° , 295(2) K]. The structure was solved and refined using SHELXTL software package [24] in anisotropic approximation for non-hydrogen atoms (isotropic for hydrogens). Cubic crystal system, space group $Ia\bar{3}d$; unit cell parameters: $a = 37.8244(14)$ Å; $V = 54115(3)$ Å³; $Z = 96$; $d = 1.421$ g/cm³; $\mu = 1.861$ mm⁻¹. Total of 21 787 reflection intensities were measured in the range $2.75 < \theta < 26.40^\circ$, including 4541 indepen-

dent reflections ($R_{\text{int}} = 0.0888$) and 1394 reflections with $I > 2\sigma(I)$; completeness 97.9%. A correction for absorption was applied analytically. Final divergence factors: $R_1 = 0.0601$, $wR_2 = 0.1064$ [for reflections with $I > 2\sigma(I)$], $R_1 = 0.2428$, $wR_2 = 0.1244$ (all reflections); goodness of fit 1.004, $\Delta\rho = 0.398/-0.419 \text{ e} \text{ \AA}^{-3}$. The crystallographic data for compound **3i** were deposited to the Cambridge Crystallographic Data Centre (CCDC entry no. 1519733).

3a-(3,4-Dihydro-2H-pyran-5-yl)-2-hydroxy-3-(4-nitrobenzoyl)pyrrolo[2,1-c][1,4]benzoxazine-1,4(3aH)-dione (3j). Yield 0.78 g (59%), mp 198–200°C (decomp., from isoctane). IR spectrum, ν , cm^{-1} : 3182 (O–H), 1785 ($\text{C}^4=\text{O}$), 1710 ($\text{C}^1=\text{O}$), 1650 ($3-\text{C}=\text{O}$). ^1H NMR spectrum (400 MHz, DMSO- d_6), δ , ppm: 1.57 m (2H, 3'-H), 1.75 m (2H, 4'-H), 3.69 m and 3.84 m (1H each, 5'-H), 6.30 s (1H, 6'-H), 7.34–8.31 m (8H, H_{arom}), 11.76 br.s (1H, OH). Found, %: C 61.35; H 3.66; N 6.51. $\text{C}_{23}\text{H}_{16}\text{N}_2\text{O}_8$. Calculated, %: C 61.61; H 3.60; N 6.25.

(2S*,1R*,6R*)-8-Phenyl-5,7,19-trioxa-12-azapentacyclo[10.8.0.0^{1,9}.0^{2,6}.0^{13,18}]jicosa-8,13,15,17-tetraene-10,11,20-trione (4a). Yield 0.34 g (28%), mp 271–272°C (decomp., from benzene). IR spectrum, ν , cm^{-1} : 1779 ($\text{C}^{20}=\text{O}$), 1728 ($\text{C}^{10}=\text{O}$, $\text{C}^{11}=\text{O}$). ^1H NMR spectrum (400 MHz, DMSO- d_6), δ , ppm: 1.57–1.67 m and 1.77–1.85 m (1H each, 3-H), 3.22 m (1H, 2-H), 3.97 q (1H, 4-H, $J = 7.9$ Hz), 4.13 t.d (1H, 4-H, $J = 9.0, 4.0$ Hz), 6.00 d (1H, 6-H, $J = 5.3$ Hz), 7.36–7.87 m (9H, H_{arom}). ^{13}C NMR spectrum (100 MHz, DMSO- d_6), δ_{C} , ppm: 22.8 (C^3), 42.2 (C^2), 58.8 (C^1), 68.9 (C^4), 99.4 (C^6), 104.1 (C^9), 116.9–143.1 (C_{arom}), 159.9 (C^{11}), 163.1 (C^{20}), 167.0 (C^8), 176.1 (C^{10}). Found, %: C 67.99; H 4.01; N 3.66. $\text{C}_{22}\text{H}_{15}\text{NO}_6$. Calculated, %: C 67.86; H 3.88; N 3.60.

(2S*,1R*,6R*)-15-Chloro-8-phenyl-5,7,19-trioxa-12-azapentacyclo[10.8.0.0^{1,9}.0^{2,6}.0^{13,18}]jicosa-8,13,15,17-tetraene-10,11,20-trione (4b). Yield 0.41 g (31%), mp 278–280°C (decomp., from benzene). IR spectrum, ν , cm^{-1} : 1773 ($\text{C}^{20}=\text{O}$), 1738 ($\text{C}^{10}=\text{O}$), 1713 ($\text{C}^{11}=\text{O}$). ^1H NMR spectrum (400 MHz, CDCl_3), δ , ppm: 1.68–1.78 m and 1.91–2.00 m (1H each, 3-H), 3.02 t.d (1H, 2-H, $J = 9.5, 5.3$ Hz), 4.12 q (1H, 4-H, $J = 8.0$ Hz), 4.13 t.d (1H, 4-H, $J = 9.2, 4.6$ Hz), 6.05 d (1H, 6-H, $J = 5.3$ Hz), 7.24–8.04 m (8H, H_{arom}). ^{13}C NMR spectrum (100 MHz, CDCl_3), δ_{C} , ppm: 23.2 (C^3), 43.6 (C^2), 59.3 (C^1), 69.3 (C^4), 98.1 (C^6), 104.0 (C^9), 118.3–141.8 (C_{arom}), 159.2 (C^{11}), 162.0 (C^{20}), 170.4 (C^8), 173.9 (C^{10}). Found, %: C 62.48; H 3.41; N 3.22. $\text{C}_{22}\text{H}_{14}\text{ClNO}_6$. Calculated, %: C 62.35; H 3.33; N 3.31.

(2S*,1R*,6R*)-8-(4-Ethoxyphenyl)-5,7,19-trioxa-12-azapentacyclo[10.8.0.0^{1,9}.0^{2,6}.0^{13,18}]jicosa-8,13,15,17-tetraene-10,11,20-trione (4c). Yield 0.47 g (35%), mp 274–275°C (decomp., from benzene). IR spectrum, ν , cm^{-1} : 1766 ($\text{C}^{20}=\text{O}$), 1741 ($\text{C}^{10}=\text{O}$), 1710 ($\text{C}^{11}=\text{O}$). ^1H NMR spectrum (400 MHz, CDCl_3), δ , ppm: 1.47 t (3H, Me, $J = 7.0$ Hz), 1.67–1.77 m and 1.84–1.93 m (1H each, 3-H), 3.03 t.d (1H, 2-H, $J = 9.5, 5.5$ Hz), 4.06 q (1H, 4-H, $J = 8.0$ Hz), 4.11–4.23 m (3H, 4-H, OCH_2Me), 6.02 d (1H, 6-H, $J = 5.3$ Hz), 6.99–8.11 m (8H, H_{arom}). ^{13}C NMR spectrum (100 MHz, CDCl_3), δ_{C} , ppm: 14.1 (Me), 22.8 (C^3), 43.2 (C^2), 59.0 (C^1), 63.5 (CH_2Me), 68.6 (C^4), 96.7 (C^6), 103.1 (C^9), 113.6–142.8 (C_{arom}), 159.8 (C^{11}), 162.8 (C^{20}), 163.9 (C_{arom}), 169.8 (C^8), 174.0 (C^{10}). Found, %: C 66.40; H 4.46; N 3.37. $\text{C}_{24}\text{H}_{19}\text{NO}_7$. Calculated, %: C 66.51; H 4.42; N 3.23.

(2S*,1R*,6R*)-8-(Bromophenyl)-5,7,19-trioxa-12-azapentacyclo[10.8.0.0^{1,9}.0^{2,6}.0^{13,18}]jicosa-8,13,15,17-tetraene-10,11,20-trione (4d). Yield 0.43 g (30%), mp 280–281°C (decomp., from benzene). IR spectrum, ν , cm^{-1} : 1779 ($\text{C}^{20}=\text{O}$), 1739 ($\text{C}^{10}=\text{O}$, $\text{C}^{11}=\text{O}$). ^1H NMR spectrum (400 MHz, CDCl_3), δ , ppm: 1.67–1.77 m and 1.87–1.96 m (1H each, 3-H), 3.02 t.d (1H, 2-H, $J = 9.7, 5.1$ Hz), 4.08 q (1H, 4-H, $J = 8.0$ Hz), 4.19 t.d (1H, 4-H, $J = 9.2, 4.4$ Hz), 6.04 d (1H, 6-H, $J = 5.3$ Hz), 7.30–7.95 m (8H, H_{arom}). Found, %: C 56.50; H 3.11; N 3.12. $\text{C}_{22}\text{H}_{14}\text{BrNO}_6$. Calculated, %: C 56.43; H 3.01; N 2.99.

(2S*,1R*,7R*)-9-Phenyl-6,8,20-trioxa-13-azapentacyclo[11.8.0.0^{1,10}.0^{2,7}.0^{14,19}]henicosa-9,14,16,18-tetraene-11,12,21-trione (4e). Yield 0.40 g (32%), mp 299–300°C (decomp., from benzene). IR spectrum, ν , cm^{-1} : 1778 ($\text{C}^{21}=\text{O}$), 1727 ($\text{C}^{11}=\text{O}$, $\text{C}^{12}=\text{O}$). ^1H NMR spectrum (400 MHz, DMSO- d_6), δ , ppm: 1.10–1.27 m and 1.43 m (1H each, 3-H), 1.48–1.68 m (2H, 4-H), 2.44 m (1H, 2-H), 3.80 m (2H, 5-H), 5.74 m (1H, 7-H), 7.38–7.89 m (9H, H_{arom}). ^{13}C NMR spectrum (126 MHz, DMSO- d_6), δ_{C} , ppm: 17.7 (C^3), 22.2 (C^4), 32.6 (C^2), 60.7 (C^1), 61.0 (C^5), 97.2 (C^7), 99.0 (C^{10}), 117.0–143.5 (C_{arom}), 160.2 (C^{12}), 163.4 (C^{21}), 167.0 (C^9), 176.1 (C^{11}). Found, %: C 68.50; H 4.22; N 3.73. $\text{C}_{23}\text{H}_{17}\text{NO}_6$. Calculated, %: C 68.48; H 4.25; N 3.47.

(2S*,1R*,7R*)-16-Chloro-9-phenyl-6,8,20-trioxa-13-azapentacyclo[11.8.0.0^{1,10}.0^{2,7}.0^{14,19}]henicosa-9,14,16,18-tetraene-11,12,21-trione (4f). Yield 0.33 g (24%), mp 269–270°C (decomp., from benzene). IR spectrum, ν , cm^{-1} : 1785 ($\text{C}^{21}=\text{O}$), 1736 ($\text{C}^{11}=\text{O}$, $\text{C}^{12}=\text{O}$). ^1H NMR spectrum (500 MHz, DMSO- d_6), δ , ppm: 1.07–1.16 m and 1.50 m (1H each, 3-H), 1.55–1.66 m (2H, 4-H), 2.54 m (1H, 2-H), 3.80 m (2H, 5-H),

5.73 d (1H, 7-H, $J = 3.9$ Hz), 7.42–7.90 m (8H, H_{arom}). ^{13}C NMR spectrum (126 MHz, DMSO- d_6), δ_{C} , ppm: 17.6 (C^3), 22.2 (C^4), 32.5 (C^2), 60.6 (C^1), 61.0 (C^5), 97.2 (C^7), 98.8 (C^{10}), 118.7–142.3 (C_{arom}), 160.1 (C^{12}), 162.9 (C^{21}), 167.3 (C^9), 175.6 (C^{11}). Found, %: C 63.40; H 3.28; N 3.33. $\text{C}_{23}\text{H}_{16}\text{ClNO}_6$. Calculated, %: C 63.09; H 3.68; N 3.20.

X-Ray analysis of compound 4f. A single crystal of **4f** (yellow plate) for X-ray analysis was obtained by crystallization from toluene. The X-ray diffraction data were acquired on an Xcalibur R automated four-circle diffractometer with χ -geometry (monochromatized MoK_α radiation, ω -scanning) using CrysAlisPro software [21]. Triclinic crystal system, space group $P-1$; $\text{C}_{23}\text{H}_{16}\text{ClNO}_6$; unit cell parameters: $a = 9.2253(13)$, $b = 10.7101(13)$, $c = 11.3059(15)$ Å; $\alpha = 73.195(11)$, $\beta = 81.299(11)$, $\gamma = 69.205(12)^\circ$; $V = 998.2(2)$ Å 3 ; $M = 437.82$; $d_{\text{calc}} = 1.457$ g/cm 3 ; $Z = 2$. Total of 7726 reflection intensities were collected in the range $2.94 < \theta < 29.40^\circ$, including 4592 independent reflections ($R_{\text{int}} = 0.0450$) and 3403 reflections with $I > 2\sigma(I)$; completeness 99.6% for $\theta < 26^\circ$. Absorption by the crystal was taken into account using SCALE3 ABSPACK algorithm [25]. The structure was solved and refined using SHELX97 [24]. The positions of all non-hydrogen atoms were refined independently in anisotropic approximation; hydrogen atoms were placed in geometrically calculated positions and refined according to the riding model with dependent thermal parameters. Final divergence factors: $R_1 = 0.0649$, $wR_2 = 0.1648$ [reflections with $I > 2\sigma(I)$]; $R_1 = 0.0866$, $wR_2 = 0.1828$ (all reflections); goodness of fit $S = 1.068$; $\Delta\rho = 0.362/-0.563$ e Å $^{-3}$. The crystallographic data for compound **4f** were deposited to the Cambridge Crystallographic Data Centre (CCDC entry no. 1519734).

(2S*,1R*,7R*)-9-(4-Methoxyphenyl)-6,8,20-trioxo-13-azapentacyclo[11.8.0.0 1,10 .0 2,7 .0 14,19]henicosa-9,14,16,18-tetraene-11,12,21-trione (4g). Yield 0.34 g (25%), mp 272–374°C (decomp., from benzene). IR spectrum, ν , cm $^{-1}$: 1780 ($\text{C}^{21}=\text{O}$), 1737 ($\text{C}^{11}=\text{O}$, $\text{C}^{12}=\text{O}$). ^1H NMR spectrum (500 MHz, DMSO- d_6), δ , ppm: 1.06–1.13 m and 1.42 m (1H each, 3-H), 1.50–1.62 m (2H, 4-H), 2.44 d.t (1H, 2-H, $J = 12.5$, 4.0 Hz), 3.77 m (2H, 5-H), 3.89 s (3H, Me), 5.71 d (1H, 7-H, $J = 4.0$ Hz), 7.12–7.94 m (8H, H_{arom}). ^{13}C NMR spectrum (126 MHz, DMSO- d_6), δ_{C} , ppm: 17.6 (C^3), 22.1 (C^4), 32.8 (C^2), 55.6 (Me), 60.8 (C^1), 60.9 (C^5), 96.9 (C^7), 97.9 (C^{10}), 113.4–143.4 (C_{arom}), 160.4 (C^{12}), 163.3 (C_{arom}), 163.4 (C^{21}), 167.0 (C^9), 175.5 (C^{11}).

Found, %: C 66.42; H 4.28; N 3.37. $\text{C}_{24}\text{H}_{19}\text{NO}_7$. Calculated, %: C 66.51; H 4.42; N 3.23.

(2S*,1R*,7R*)-9-(4-Ethoxyphenyl)-6,8,20-trioxo-13-azapentacyclo[11.8.0.0 1,10 .0 2,7 .0 14,19]henicosa-9,14,16,18-tetraene-11,12,21-trione (4h). Yield 0.37 g (27%), mp 269–270°C (decomp., from benzene). IR spectrum, ν , cm $^{-1}$: 1778 ($\text{C}^{21}=\text{O}$), 1731 ($\text{C}^{11}=\text{O}$, $\text{C}^{12}=\text{O}$). ^1H NMR spectrum (400 MHz, DMSO- d_6), δ , ppm: 1.04–1.15 m (1H, 3-H), 1.38 t (3H, Me, $J = 6.9$ Hz), 1.42 m (1H, 3-H), 1.49–1.63 m (2H, 4-H), 2.44 d.t (1H, 2-H, $^2J = 12.8$, $^3J = 4.4$ Hz), 3.70–3.81 m (2H, 5-H), 4.17 d.q (2H, CH_2Me , $J = 6.9$, 2.0 Hz), 5.71 d (1H, 7-H, $J = 3.7$ Hz), 7.11–7.92 m (8H, H_{arom}). ^{13}C NMR spectrum (126 MHz, DMSO- d_6), δ_{C} , ppm: 14.5 (Me), 17.6 (C^3), 22.1 (C^4), 32.8 (C^2), 60.8 (C^1), 60.8 (C^5), 63.6 (CH_2Me), 96.9 (C^7), 97.8 (C^{10}), 113.7–143.4 (C_{arom}), 160.4 (C^{12}), 162.6 (C_{arom}), 163.4 (C^{21}), 167.0 (C^9), 175.5 (C^{11}). Found, %: C 67.30; H 4.68; N 3.31. $\text{C}_{25}\text{H}_{21}\text{NO}_7$. Calculated, %: C 67.11; H 4.73; N 3.13.

(2S*,1R*,7R*)-9-(4-Bromophenyl)-6,8,20-trioxo-13-azapentacyclo[11.8.0.0 1,10 .0 2,7 .0 14,19]henicosa-9,14,16,18-tetraene-11,12,21-trione (4i). Yield 0.25 g (17%), mp 270–271°C (decomp., from benzene). IR spectrum, ν , cm $^{-1}$: 1782 ($\text{C}^{21}=\text{O}$), 1738 ($\text{C}^{11}=\text{O}$, $\text{C}^{12}=\text{O}$). ^1H NMR spectrum (500 MHz, DMSO- d_6), δ , ppm: 1.11–1.23 m and 1.42 m (1H each, 3-H), 1.49–1.63 m (2H, 4-H), 2.48 m (1H, 2-H), 3.74–3.82 m (2H, 5-H), 5.74 d (1H, 7-H, $J = 3.7$ Hz), 7.38–7.82 m (8H, H_{arom}). ^{13}C NMR spectrum (126 MHz, DMSO- d_6), δ_{C} , ppm: 17.6 (C^3), 22.2 (C^4), 32.6 (C^2), 60.7 (C^1), 61.0 (C^5), 97.4 (C^7), 99.4 (C^{10}), 117.0–143.4 (C_{arom}), 160.0 (C^{12}), 163.2 (C^{21}), 165.7 (C^9), 176.1 (C^{11}). Found, %: C 57.43; H 3.23; N 2.63. $\text{C}_{23}\text{H}_{16}\text{BrNO}_6$. Calculated, %: C 57.28; H 3.34; N 2.90.

(2S*,1R*,7R*)-9-(4-Nitrophenyl)-6,8,20-trioxo-13-azapentacyclo[11.8.0.0 1,10 .0 2,7 .0 14,19]henicosa-9,14,16,18-tetraene-11,12,21-trione (4j). Yield 0.25 g (19%), mp 265–266°C (decomp., from benzene). IR spectrum, ν , cm $^{-1}$: 1793 ($\text{C}^{21}=\text{O}$), 1732 ($\text{C}^{11}=\text{O}$, $\text{C}^{12}=\text{O}$). ^1H NMR spectrum (400 MHz, DMSO- d_6), δ , ppm: 1.20–1.28 m and 1.43 m (1H each, 3-H), 1.52–1.66 m (2H, 4-H), 2.55 m (1H, 2-H), 3.81 m (2H, 5-H), 5.79 d (1H, 7-H, $J = 3.5$ Hz), 7.37–8.43 m (8H, H_{arom}). Found, %: C 61.89; H 3.48; N 6.38. $\text{C}_{23}\text{H}_{16}\text{N}_2\text{O}_8$. Calculated, %: C 61.61; H 3.60; N 6.25.

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