A Stereoselective Synthesis of (*Z*)-3-Aryl and Alkylmethylidene-1*H*-isoindolin-1-ones

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Abstract: A series of *Z*-configured (hetero)aryl and alkylmethylideneisoindolin-1-ones has been efficiently prepared by treatment of *N*-methoxycarbonylisoindolin-1-ones with a base and reaction with selected aldehydes. The parent N-acylated isoindolin-1-ones have been assembled by a Parham-type cyclization of *N*-(2-iodoarylmethyl)dicarbamates.

Key words: carbanions, Mitsonobu reaction, cyclizations, olefination, stereoselectivity

In recent years the 3-(hetero)aryl and alkylmethylideneisoindolin-1-one systems have become a desirable synthetic target since they represent the central structural feature of a very large number of natural and synthetic products displaying a wide range of biological activities.[1–5] For example, compound **1** (AKS 186) has been reported to inhibit vasoconstriction induced by the thromboxane A2 analogue (U–46619)[1] and the 4-acetoxyphenylmethylidene derivative **2** has been claimed to exhibit local anesthetic activity superior to that of procaine.[2] Moreover, 3-(hetero)aryl and alkylmethylideneisoindolin-1-ones possess potential utility as versatile key building blocks in the synthesis of various classes of alkaloids such as isoindolobenzazepines, e.g. lennoxamine (**3**),[6] and aristolactames, e.g. cepharanone A (**4**),[7] aristoyagonine (**5**)[8] (Figure 1).

The existing methods for the synthesis of aryl and/or alkylmethylideneisoindolin-1-ones (general formula **I**) differ mainly but not merely in the type of reaction giving rise to the pendant ylidene unit. Thus, these highly conjugated lactams can be obtained from the corresponding phthalides by treatment with a primary amine.[7,9] The most convenient routes, however, rely upon the reaction of an N-substituted phthalimide either with a Grignard reagent[1,10] or with arylacetates involved in a photodecarboxylative addition process.[11] They are also accessible by making use of a Wittig reagent[12] which offers the advantage of avoiding the dehydration step. More recently, several groups have developed elegant synthetic methods that hinge upon the intramolecular cyclization of *o*-(1-alkynyl)benzamide[13] or benzonitrile derivatives[14] and upon the domino reaction of aromatic ynamides.[15] Besides these main synthetic approaches, other procedures involving the Horner reaction of phosphorylated isoindolinones,[5] a combination of carbonylation and nitrogenation applied to *o*-halophenylalkylketones[16] and a Parham-type cyclization of *N*-acyl-2-bromo-benzamides[17] have been used occasionally. Most of these methods involve simple experimental procedures and the corresponding reactions proceed in good yields. Unfortunately, their applicability is generally unsatisfactory mainly because of restrictions in the choice of substituents, namely in their number and position on the aromatic nucleus, and in the selection of the aromatic, heteroaromatic and aliphatic units connected to the pendant methylidene moiety. The issues raised by elaboration of aryl and alkylmethylideneisoindolinones may not be simply addressed by comparison with phthalides as precursors. The synthetic methods relying upon nucleophilic additions to phthalimides lack regiose-

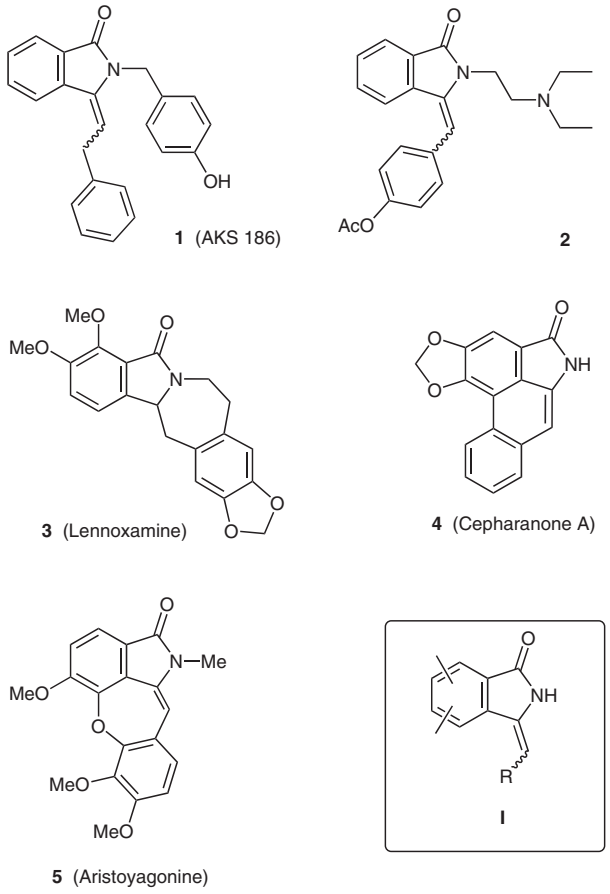

Figure 1

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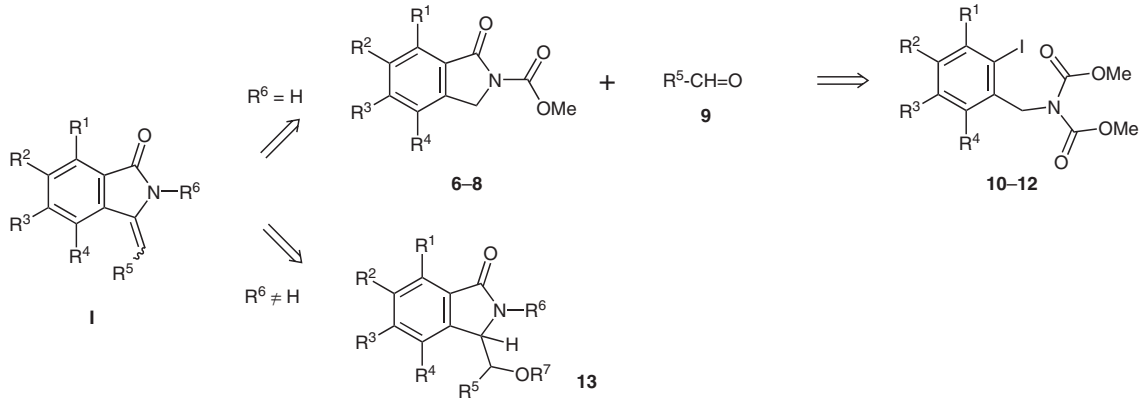


Scheme 1 For the definitions of R groups, see Scheme 2.

lectivity and hence have been confined to the elaboration of bare or symmetrically substituted models. Furthermore, most of these methods require a rather problematic deprotection step[18] in order to deliver the N-unsubstituted products of general formula **I**.

We wish to delineate in this paper an alternative, efficient and tactically new approach to these highly conjugated lactams that is based on the retrosynthetic analysis shown in Scheme 1. This new synthetic strategy relies upon the treatment of *N*-methoxycarbonyl-isoindolin-1-ones **6–8** with a base and subsequent reaction with an appropriate aldehyde **9**. The acylated isoindolin-1-ones **6–8** could be conceivably assembled by a Parham-type cyclization applied to the dimethyl dicarbamates **10–12**. The present work originated from the following premises. (i) Isoindolin-1-ones have been successfully metalated at the benzylic position of the heterocyclic five-membered ring thus permitting the connection of a range of electrophiles and, in particular, aldehydes that allow the installation of an hydroxyalkyl appendage.[19] (ii) The N-acylated carbamate group is resistant towards basic and nucleophilic reagents intermolecularly[20] but is endowed with a remarkable propensity to react with nucleophiles in an intramolecular fashion.[21] This operation releases an alkoxide that can further act as a base. (iii) It has been shown that the base-induced dehydration of *erythro* and *threo* adducts structurally related to **13** through an E1cb mechanism gives rise stereoselectively to the arylideneisoindolin-1-ones **I** ($R^6 \neq H$) (Scheme 1).[22]

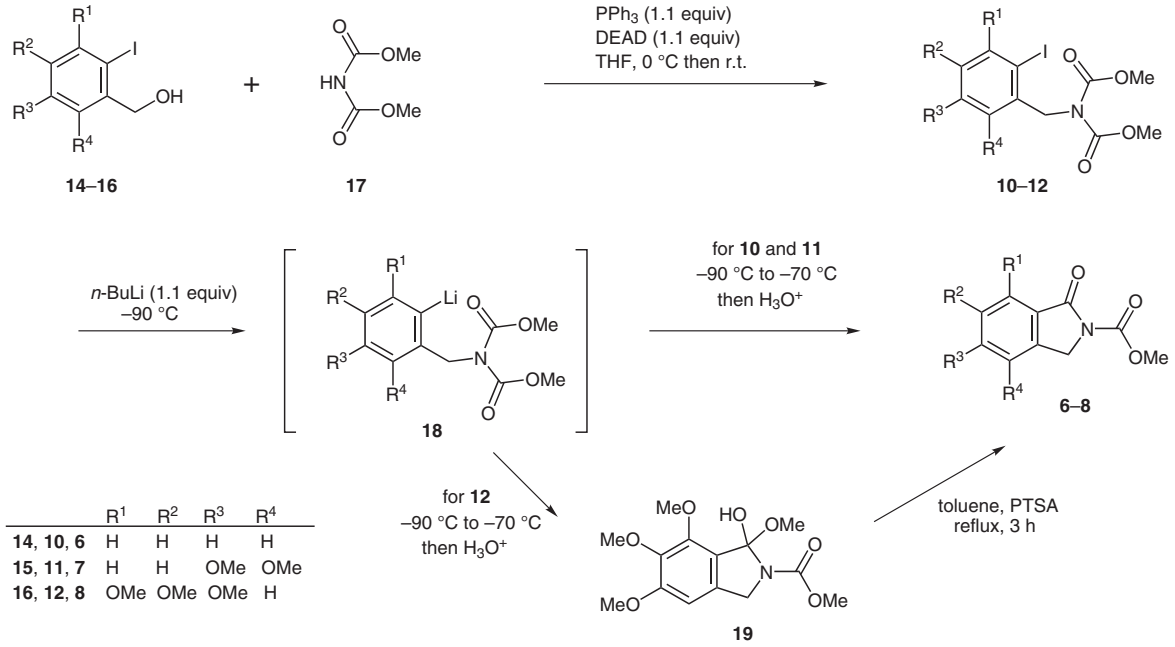
The first facet of the synthesis was the elaboration of the parent N-acylated isoindolin-1-ones **6–8**. We conjectured that these functionalized isoindolinones would be readily obtained by the Parham cyclization protocol[23] applied to the dicarbamates **10–12** (Scheme 1). This concept, which is based upon aromatic lithiation, usually by lithium–halogen exchange and subsequent reaction with an internal electrophile, has not yet been applied to the diacylamine functionality.

Initially, the parent dicarbamates **10–12** were readily accessible, albeit in moderate yield, by Mitsonobu reaction involving the suitably substituted benzylic alcohols **14–16** with dimethyl iminodicarboxylate **17** (Scheme 2, Table 1).We were then pleased to observe that treatment of **10–12** with *n*-BuLi at –90 °C spared the carbamate functionalities and that the initially formed aromatic anion **18** was trapped intramolecularly to afford the desired *N*-methoxycarbonylisoindolin-1-ones **6–8** with very satisfactory yields (Scheme 2, Table 1).

Interestingly, the presence of a large amount of the hemiketal-type intermediate **19** was observed upon annulation of **12**, but it was quantitatively converted into the desired parent model **8** under acidic conditions. The formation of this compound may be tentatively explained by coordination of lithium by the adjacent aromatic methoxy group after anionic cyclization.

We next planned to install the pendant (hetero)aryl and alkylmethylidene unit on the isoindolin-1-one framework by treatment of the parent isoindolinone **6–8** with a base and subsequent reaction with the appropriate aldehyde **9a–h**. After considerable experimentation with various temperatures, solvents (THF, Et_2O, EtOH) and bases (KHMDS, LHMDS, EtONa) it was found that the best results were obtained by treatment of the N-acylated isoindolin-1-ones **6–8** with NaH in DMF followed by slow addition of the appropriate aldehyde **9a–h** and ultimate refluxing in DMF for eight hours (Scheme 3). The method tolerates a wide variety of carbonyl compounds and a representative series of compounds which have been prepared by this method are presented in Table 2. This simple procedure thus gives indiscriminate access to aryl, heteroaryl and alkylmethylidene isoindolin-1-ones **20a–h**, **21a**, **22a** with moderate to good yields (Table 3). Compounds **20–22** were obtained as mixtures of geometric isomers with the chromatographically separable *Z* isomer predominating by a large margin. The stereochemistry of the exocyclic double bond was unambiguously assigned from the ^1H NMR spectra both by the chemical shift of the olefinic proton and by comparison with authentic samples. In all cases the vinylic proton of the *Z* isomers appears at lower field than the vinylic proton of the corresponding *E* isomer.[6,13c]

From a mechanistic point of view one can reasonably assume that deprotonation of the benzylic position and interception with the appropriate aldehyde allows the



Scheme 2

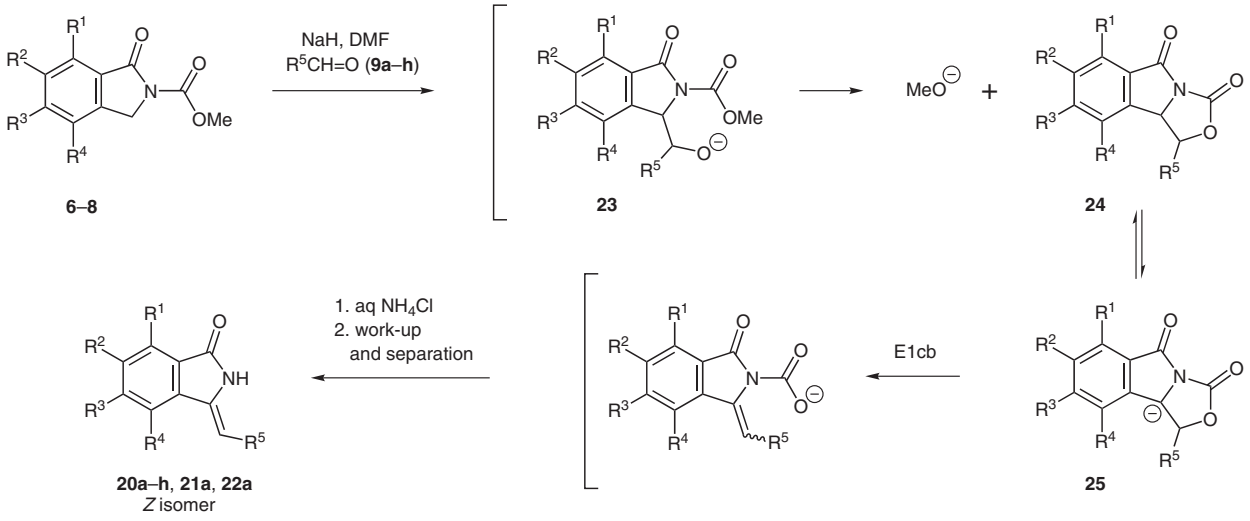
connection of a transient aryl (or alkyl)methine alkoxide appendage (Scheme 3). Intramolecular attack of the resulting oxanion **23** may generate the highly strained isoindolin-1-one derivative **24** accompanied by the release of sodium methoxide which is of sufficient kinetic basicity to deprotonate the latent benzylic lactam position. As anticipated, E1cb elimination from the resulting carbanionic species **25**[17] followed by decarboxylation delivers the target NH-free aryl, alkyl and heteroarylmethylideneisoindolin-1-ones **20**–**22** as the thermodynamically favored *Z* isomers, either predominantly or exclusively.

To summarize, the goal of our program has been to develop a new general and versatile approach for the synthesis of (hetero)aryl- and alkylmethylideneisoindolin-1-ones. This new synthetic route offers special advantages including good stereoselectivity, high efficiency and experimental simplicity.

Table 1 Spectroscopic and Physical Data of the Parent Dicarbamates **10**–**12** and *N*-Acylisoindolin-1-ones **6**–**8**

Product[a,b]	Yield (%)	Mp (°C)	^{1}H NMR ($CDCl_3$–TMS) δ (ppm), *J* (Hz)	^{13}C NMR ($CDCl_3$–TMS) δ (ppm)
10	83	oil	3.80 (s, 6 H, 2 × OCH_3), 4.87 (s, 2 H, OCH_2), 6.94 (t, *J* = 7.5, 1 H, H_{arom}), 7.04 (d, *J* = 7.7, 1 H, H_{arom}), 7.29 (t, *J* = 7.5, 1 H, H_{arom}), 7.81 (d, *J* = 7.7, 1 H, H_{arom})	54.2, 54.9, 97.2, 127.8, 128.5, 128.8, 139.0, 139.5, 153.9
11	53	57–59	3.78 (s, 3 H, OCH_3), 3.82 (s, 6 H, 2 × OCH_3), 3.84 (s, 3 H, OCH_3), 5.06 (s, 2 H, CH_2), 6.61 (d, *J* = 8.7, 1 H, H_{arom}), 7.54 (d, *J* = 8.7, 1 H, H_{arom})	51.9, 53.8, 55.8, 60.8, 89.2, 113.7, 134.6, 132.7, 148.5, 153.2, 154.2
12	50	75–77	3.80 (s, 3 H, OCH_3), 3.84 (s, 6 H, 2 × OCH_3), 3.85 (s, 3 H, OCH_3), 3.87 (s, 3 H, OCH_3), 4.87 (s, 2 H, CH_2), 6.46 (s, 1 H, H_{arom})	54.3, 55.0, 56.2, 60.8, 60.9, 85.0, 105.5, 134.8, 141.1, 153.1, 154.1
6	76	136–138	3.93 (s, 3 H, OCH_3), 4.78 (s, 2 H, CH_2), 7.47 (t, *J* = 6.1, 2 H, H_{arom}), 7.62 (t, *J* = 7.4, 1 H, H_{arom}), 7.88 (d, *J* = 8.3, 1 H, H_{arom})	49.1, 53.7, 123.2, 125.2, 128.6, 130.9, 133.8, 140.8, 152.5, 166.2
7	44	168–170	3.96 (s, 9 H, 3 × OCH_3), 4.81 (s, 2 H, CH_2), 7.07 (d, *J* = 8.3, 1 H, H_{arom}), 7.66 (d, *J* = 8.3, 1 H, H_{arom})	46.9, 53.6, 56.3, 60.4, 113.4, 121.4, 124.1, 133.1, 143.4, 152.5, 156.5, 165.8
8	64	129–131	3.96 (s, 3 H, OCH_3), 3.92 (s, 6 H, 2 × OCH_3), 4.07 (s, 3 H, OCH_3), 4.66 (s, 2 H, CH_2), 6.69 (s, 1 H, H_{arom})	48.5, 53.5, 56.5, 61.5, 62.6, 101.2, 115.7, 138.6, 142.0, 152.4, 152.6, 159.3, 164.1
19	53	76–78	3.66 (s, 3 H, OCH_3), 3.86 (s, 3 H, OCH_3), 3.90 (s, 9 H, 3 × OCH_3), 4.24 (d, *J* = 5.5, 2 H, CH_2), 5.47 (br s, 1 H, OH), 6.76 (s, 1 H, H_{arom})	52.2, 52.4, 56.1, 60.9, 61.8, 109.0, 120.0, 133.4, 141.6, 152.1, 155.1, 156.5, 157.0, 168.0

[a] Satisfactory microanalyses obtained: C ± 0.27, H ± 0.32, N ± 0.27.
[b] IR (KBr): ν_{CO} two bands 1790–1735 and 1695–1685 cm^{-1}.



Scheme 3

Table 2 Aryl(Alkyl)methylideneisoindolin-1-ones **20**–**22**

Compd	R^1	R^2	R^3	R^4	R^5
20a	H	H	H	H	Ph
20b	H	H	H	H	2-$MeOC_6H_4$
20c	H	H	H	H	3,4-$(MeO)_2C_6H_3$
20d	H	H	H	H	2,3,4-$(MeO)_3C_6H_2$
20e	H	H	H	H	3,4-$(OCH_2O)C_6H_3$
20f	H	H	H	H	2-naphthyl
20g	H	H	H	H	2-thienyl
20h	H	H	H	H	*i*-Pr
21a	H	H	OMe	OMe	Ph
22a	OMe	OMe	OMe	H	Ph

Melting point determinations were carried out on a Reichert–Thermopan apparatus and are uncorrected. ^1H and ^{13}C NMR spectra were measured at 300 MHz and 75 MHz, respectively, on a Bruker AM 300 spectrometer as solutions in $CDCl_3$ with TMS as internal standard. Elemental analyses were determined by the CNRS microanalysis centre. For flash chromatography, silica gel 60 M (230–400 mesh ASTM) was used. All solvents were dried and distilled according to standard procedures. Dry glassware for moisture-sensitive reactions was obtained by oven drying and assembly under Ar. An inert atmosphere was obtained with a stream of Ar and glassware equipped with rubber septa; reagent transfer was performed by syringe.

Dimethyl iminodicarboxylate **17**[24] was prepared according to a reported procedure.[25] Alcohol **14** is commercially available. The 2-iodobenzyl alcohols **15**[26] and **16**[27] were synthesized according to the literature.

Synthesis of Dicarbamates 10–12; General Procedure

DEAD (1.5 mL, 40% in toluene, 3.3 mmol) was added dropwise at 0 °C under Ar to a stirred solution of the suitably substituted benzyl alcohol **14**–**16** (3 mmol), triphenylphosphine (867 mg, 3.3 mmol) and iminodicarboxylate **17** (400 mg, 3 mmol) in dry THF (50 mL). The mixture was then stirred at r.t. for an additional 24 h, quenched with H_2O (3 mL) and concentrated under vacuum. The residual oil was purified by column chromatography (CH_2Cl_2–Et_2O–hexanes, 50:30:20) and finally recrystallized from hexane–toluene (for compounds **11** and **12**).

N-Acylated Isoindolin-1-ones 6–8; General Procedure

A solution of *n*-BuLi (1.4 mL, 1.6 M in hexanes, 2.2 mmol) was added dropwise at –90 °C under Ar to a solution of dicarbamate **10**–**12** (2 mmol) in dry THF (30 mL). The reaction mixture was stirred at –90 °C for an additional 5 min, slowly warmed to –70 °C over 15 min and then quenched by addition of a dilute HCl solution in EtOH (15%, 5 mL). The reaction mixture was warmed to r.t., extracted with Et_2O (3 × 10 mL) and the combined organic extracts were washed with brine (5 mL) and dried (Na_2SO_4). After evaporation of the solvent, the crude residual oil was purified by column chromatography (EtOAc–hexanes, 30:70) to afford the annulated compounds **6**–**8**.

Column chromatography of the reaction mixture obtained upon Parham cyclization of **12** delivered **8** (11%) and the primarily annulated compound **19** which was readily converted into the desired isoindolin-1-one **8**. Thus, compound **19** was refluxed in toluene (20 mL) in the presence of a catalytic amount of PTSA for 3 h. Usual work-up delivered the N-acylated isoindolin-1-one **8** quantitatively. Combined crops of **8** were recrystallized from hexane–toluene.

Synthesis of (*Z*)-3-Aryl- and Alkylmethylideneisoindolin-1-ones 20–22; General Procedure

A solution of isoindolin-1-one **6**–**8** (1 mmol) in dry DMF (10 mL) was added dropwise to a stirred suspension of NaH (1.1 mmol) in DMF (40 mL) at 0 °C under Ar. The mixture was stirred at r.t. for an additional 1 h and a solution of the appropriate aldehyde **9a**–**h** (1.1 mmol) in DMF (10 mL) was added dropwise. The reaction mixture was refluxed for 9 h, cooled and quenched with sat. aq NH_4Cl (10 mL). The mixture was diluted with H_2O (30 mL), extracted with Et_2O (3 × 30 mL) and the combined organic layers were dried (Na_2SO_4). Evaporation of the solvent in vacuo left an oily residue which was purified by column chromatography (acetone–hexanes, 20:80). The *Z*-configured stereomers of **20**–**22** were invariably eluted first and were finally recrystallized from hexane–toluene.

Table 3 Spectroscopic and Physical Data of the Aryl(Alkyl)methylideneisoindolin-1-ones **20**–**22**

Product[a,b]	*Z*/*E* (%)[c]	Yield (%)[d] of *Z* isomer	Mp (°C)	^{1}H NMR ($CDCl_3$–TMS)[e] δ (ppm), *J* (Hz)	^{13}C NMR ($CDCl_3$–TMS)[e] δ (ppm)
20a	90:10	75	183–184 (Lit.[10b] 181–182)		
20b	90:10	69	162–163 (Lit.[28] 163)		
20c	90:10	73	163–164	3.77 (s, 3 H, OCH_3), 3.85 (s, 3 H, OCH_3), 6.61 (s, 1 H, H_{vinyl}), 6.96 (d, *J* = 8.5, 1 H, H_{arom}), 7.18 (m, 2 H, H_{arom}), 7.50 (t, *J* = 7.3, 1 H, H_{arom}), 7.65 (t, *J* = 7.3, 1 H, H_{arom}), 7.75 (d, *J* = 7.3, 1 H, H_{arom}), 7.98 (d, *J* = 7.3, 1 H, H_{arom}), 10.75 (br s, 1 H, NH)	60.6, 60.7, 111.6 117.0, 117.6, 125.2, 127.5, 127.8, 132.6, 133.3, 133.8, 135.9, 137.2 (C), 137.2 (CH), 153.6, 153.9, 174.2
20d	85:15	73	169–170	3.68 (s, 3 H, OCH_3), 3.86 (s, 6 H, 2 × OCH_3), 6.70 (s, 1 H, H_{vinyl}), 6.85 (s, 2 H, H_{arom}), 7.52 (t, *J* = 7.3, 1 H, H_{arom}), 7.67 (t, *J* = 7.3, 1 H, H_{arom}), 7.75 (d, *J* = 7.3, 1 H, H_{arom}), 7.99 (d, *J* = 7.3, 1 H, H_{arom}), 10.80 (br s, 1 H, NH)	61.1, 62.2, 111.6, 111.7, 125.3, 127.9, 133.4, 134.1, 135.4, 137.0, 137.4, 142.3, 143.9, 158.2, 174.3
20e	90:10	70	226–227	6.05 (s, 2 H, CH_2), 6.69 (s, 1 H, H_{vinyl}), 6.95 (d, *J* = 8.1, 1 H, H_{arom}), 7.11 (dd, *J* = 1.7, 8.1, 1 H, H_{arom}), 7.28 (t, *J* = 1.7, 1 H, H_{arom}), 7.51 (t, *J* = 7.3, 1 H, H_{arom}), 7.67 (d, *J* = 7.3, 1 H, H_{arom}), 7.73 (d, *J* = 7.3, 1 H, H_{arom}), 7.99 (d, *J* = 7.3, 1 H, H_{arom}), 10.68 (br s, 1 H, NH)	111.2, 113.8, 113.9, 125.3, 127.8, 129.1, 133.3, 133.9, 136.1, 137.2, 144.1, 151.8, 152.9, 174.1
20f	85:15	70	215–216	6.90 (s, 1 H, H_{vinyl}), 7.40–7.60 (m, 3 H, H_{arom}), 7.65–8.00 (m, 6 H, H_{arom}), 8.08 (d, *J* = 7.3, 1 H, H_{arom}), 8.25 (s, 1 H, H_{arom}), 10.95 (br s, 1 H, NH)	110.0, 125.6, 127.9, 131.4, 131.5, 132.5, 132.6, 132.8, 133.2, 133.4, 137.2, 137.4, 137.5, 138.0, 138.5, 144.0, 174.4
20g	90:10	69	144–146 (Lit.[14] 144–145)		
20h	90:10	75	179–181 (Lit.[29] 180–181)		
21a	100:0	54	209–210	4.00 (s, 3 H, OCH_3), 4.04 (s, 3 H, OCH_3), 7.07 (d, *J* = 8.3, 1 H, H_{arom}), 7.08 (s, 1 H, H_{vinyl}), 7.31–7.33 (m, 1 H, H_{arom}), 7.44–7.46 (m, 4 H, H_{arom}), 7.63 (d, *J* = 8.3, 1 H, H_{arom}), 7.97 (br s, 1 H, NH)	56.4, 60.1, 110.4, 113.4, 120.0, 127.5, 128.5, 129.2, 122.8, 131.9, 135.8, 144.5, 156.4, 168.3
22a	95:5	71	63–164	3.94 (s, 3 H, OCH_3), 4.03 (s, 3 H, OCH_3), 4.17 (s, 3 H, OCH_3), 6.44 (s, 1 H, H_{vinyl}), 7.04 (s, 1 H, H_{arom}), 7.30 (d, *J* = 9.0, 1 H, H_{arom}), 7.45–7.47 (m, 4 H, H_{arom}), 8.21 (br s, 1 H, NH)	56.4, 61.6, 62.5, 97.8, 104.8, 127.5, 128.3, 129.3, 113.6, 132.9, 135.1, 135.6, 142.7, 151.3, 157.9, 167.4

[a] Satisfactory microanalyses obtained: C ± 0.25, H ± 0.30, N ± 0.21.
[b] IR (KBr): ν_{NH} 3215–3150 cm^{-1}; ν_{CO} 1695–1675 cm^{-1}.
[c] Estimated from ^{1}H NMR spectra.
[d] Yield of purified product.
[e] DMSO-d_6 as the solvent for **20e**.

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