Synthesis of Benzopyrrolizidinones by Norrish–Yang Cyclization of 3-(Acylmethyl)-2-alkylisoindol-1-ones

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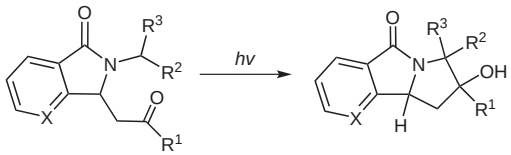
Received 25 November 2008; revised 12 February 2009

Abstract: An efficient synthesis of 2-hydroxy-1,2,3,9b-tetrahydropyrrolo[2,1-*a*]isoindol-5-ones has been achieved by the Norrish–Yang cyclization of 3-(acylmethyl)-2-alkylisoindol-1-ones in benzene in moderate to high yields.

Key words: Norrish–Yang cyclization, 3-(acylmethyl)-2-alkylisoindol-1-one, pyrrolo[2,1-*a*]isoindol-5-one, intramolecular ring formation, C–C bond formation

The synthetic utility of the Norrish–Yang cyclization has been studied extensively since Yang reported on an intriguing intramolecular C–C bond-forming reaction resulting from intramolecular hydrogen abstraction by a photoexcited ketone in 1958.[1] The stereoselectivity of the reaction has attracted considerable attention and has also been closely studied in recent years in the photocyclization of ketone-amino acids.[2,3] Different cyclic compounds can be obtained by Norrish–Yang cyclization. From a product-oriented point of view, the site of hydrogen abstraction determines the size of the resulting ring. The facile γ-hydrogen abstraction, proceeding through a six-membered transition state, affords four-membered rings (cyclobutanes, oxetanes, azetidines, etc.).[3] If there is no hydrogen atom available in the γ-position to the carbonyl group, δ- or ε-hydrogen atom abstractions occur. The subsequent cyclization results in five- or six-membered rings. For example, Giese and Wyss reported the stereoselective formation of substituted pyrrolidin-2-ones and piperidin-2-ones by Norrish–Yang cyclization of *N*-(2-benzoylethyl)glycinamides and *N*-(benzoylmethyl)glycinamides, respectively.[2–4]

We are interested in the intramolecular photocyclization reactions of ketones with amides in relation to the synthesis of alkaloid-like compounds benzopyrrolizidines because benzopyrrolizidines possess a wide range of biological activities, such as potential kinase inhibitory,[5a] antitumor, and antiviral activities.[5b,c] They could be used as versatile key intermediates in the synthesis of alkaloid gephyrotoxins.[6] Hence the chemistry and synthesis of these compounds have also received considerable attention. A literature survey shows that many methodologies have been reported for the synthesis of benzopyrrolizidines, for example, photocyclization of phthalimide derivatives;[7] tributyltin hydride mediated radical cyclization;[8] hydrogen fluoride catalyzed cyclization of azomethine ylides produced from the addition of organolithium to *N*-[(tributylstannyl)methyl]phthalimide;[9] palladium-catalyzed tandem cyclization of *N*-allyl-2-iodobenzamides;[10] flash vacuum pyrolysis of 1-[2-(methoxycarbonyl)phenyl]pyrrole;[11] base-catalyzed cyclization and O-alkylation of *N*-phthalimido-substituted keto esters;[12] metal-mediated intramolecular Heck reactions;[13] intramolecular Wittig reactions.[14] Despite these advances, room still exists for developing alternative approaches to the synthesis of such compounds. We report herein a new synthesis of benzopyrrolizidinones by Norrish–Yang cyclization of 3-(acylmethyl)-2-alkylisoindol-1-ones in benzene solution (Scheme 1).



1a–**d** X = C, R^1 = Ph
1e–**h** X = C, R^1 = Me
1i,**j** X = C, R^1 = CO_2Me
1k,**l** X = N, R^1 = Ph

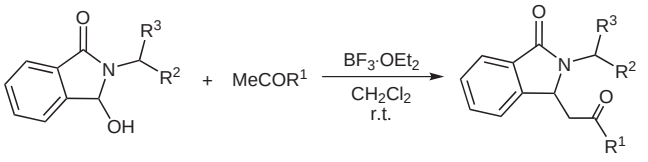
2a–**l**

Scheme 1 Photocyclization of 3-(acylmethyl)-2-alkylisoindol-1-ones

Our new synthetic method for the formation of benzopyrrolizidines reported in this paper is based on the construction of the pyrrolo[2,1-*a*]isoindol-5-one skeleton by photocyclization of four kinds of ketone-amides such as 2-alkyl-3-(benzoylmethyl)isoindol-1-ones **1a**–**d**, 3-(acetylmethyl)-2-alkylisoindol-1-ones **1e**–**h**, methyl 3-(2-alkyl-3-oxoisoindol-1-yl)pyruvates **1i**,**j**, and 7-(acetylmethyl)pyrrolo[3,4-*b*]pyridin-5-ones **1k**,**l**.

Reactants **1a**–**d** and **1i**,**j** were synthesized by the coupling reactions of carbonyl compounds [acetophenone (**4a**) and methyl pyruvate (**4b**)] with 2-alkyl-3-hydroxyisoindol-1-ones **3a**–**d**[15a] under the catalysis of boron trifluoride–diethyl ether complex at room temperature (Scheme 2); reactants **1e**–**h** were synthesized by a modified literature method[16] from the coupling reactions of ethyl acetoacetate (**4c**) with 2-alkyl-3-hydroxyisoindol-1-ones **3a**–**d** firstly under the catalysis of boron trifluoride–diethyl ether complex at room temperature to give **5a**–**d** and then hydroxylation and decarboxylation (Scheme 3); **1k**,**l** were synthesized by the coupling reaction of acetophenone (**4a**)

SYNTHESIS 2009, No. 11, pp 1821–1828
Advanced online publication: 27.04.2009
DOI: 10.1055/s-0028-1088065; Art ID: F23708SS

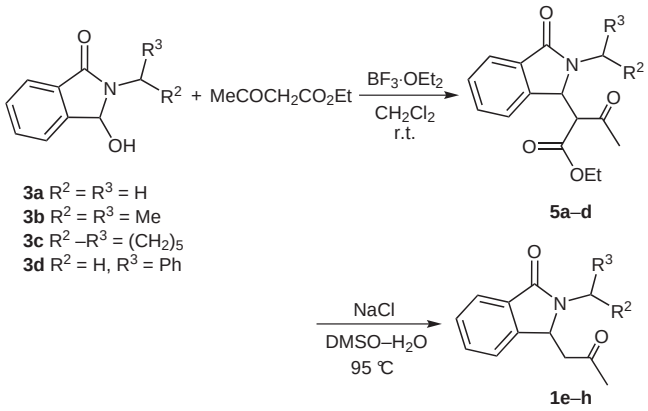


3a $R^2 = R^3 = H$
3b $R^2 = R^3 = Me$
3c $R^2 - R^3 = (CH_2)_5$
3d $R^2 = H$, $R^3 = Ph$

4a $R^1 = Ph$
4b $R^1 = CO_2Me$

1a-d $R^1 = Ph$
1i,**j** $R^1 = CO_2Me$

Scheme 2

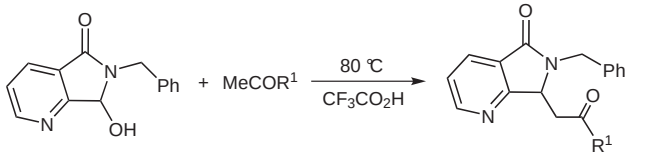


3a $R^2 = R^3 = H$
3b $R^2 = R^3 = Me$
3c $R^2 - R^3 = (CH_2)_5$
3d $R^2 = H$, $R^3 = Ph$

5a–d

1e–h

Scheme 3



6

4a $R^1 = Ph$
4d $R^1 = Me$

1k,**l**

Scheme 4

or acetone (**4d**) with 7-hydroxypyrrolo[3,4-*b*]pyridin-5-one **6**[15b] in trifluoroacetic acid under heating (Scheme 4).

Feasibility of the photocyclization reaction of **1a** was investigated in three different solvents under irradiation in Pyrex tubes with a high-pressure mercury lamp (500 W) at ambient temperature and the results are shown in Table 1. Obviously the photoreaction in benzene is more efficient than those in acetonitrile and dichloromethane, hence benzene was selected as solvent in the photoreactions of all other reactants **1b**–**l** (Table 2). The products benzopyrrolizidines **2a**–**l** were obtained by separation using silica gel column chromatography or repeated preparative layer chromatography (silica gel plate, acetone–hexane, 1:10) and further purified by recrystallization. All products were fully identified by ^1H, ^{13}C NMR, MS, and NOE correlations (Figure 1) and the structures of **2a**, **2h**, and **2j** were further confirmed by the X-ray crystal structures as depicted in Figures 2–4.

Table 1 Photocyclization of **1a** in Different Solvents

Solvent	Time (h)	Conversion (%)	Yield (%)
CH_2Cl_2	48	35	32
MeCN	32	75	37
benzene	20	81	45

The photoreactions of **1a**–**l** showed high stereoselectivity. Although two stereoisomers were produced in most cases, only one isomer was the dominant product as depicted in Table 2. In the case of **1g**, two pure stereoisomers *cis*-**2g** and *trans*-**2g** could be separated by repeated preparative thin layer chromatography and their stereostructures were evaluated by NOE correlation as shown in Figure 1. In NOE correlation of *cis*-**2g**, the reciprocal interaction between H10 and protons of methyl group on C2 is observed, which indicates that the configuration of H10 and the methyl group on C2 is *cis*; while no reciprocal interaction between H10 and protons of methyl group on C2 is observed in the NOE correlation of *trans*-**2g**, indicating that the configuration of H10 and the methyl group is *trans*. The configurations of other products have also been examined by NOE correlation. For example, no reciprocal interaction between H10 and protons of phenyl group on C2 in products **2a**–**d** indicated that H10 and phenyl group on C2 is *trans*; the reciprocal interaction between H10 and protons of the methyl group on C2 in products **2e**–**h** indicate that H10 and methyl group on C2 are *cis*; no reciprocal interaction between H10 and protons of methoxy group on C2 in products **2i**,**j** indicate that H10 and CO_2Me group on C2 are *trans*. All these deduction are consistent with the X-ray crystal structure analysis of **2a**, **2h**, and **2j** as shown in Figures 2– 4.

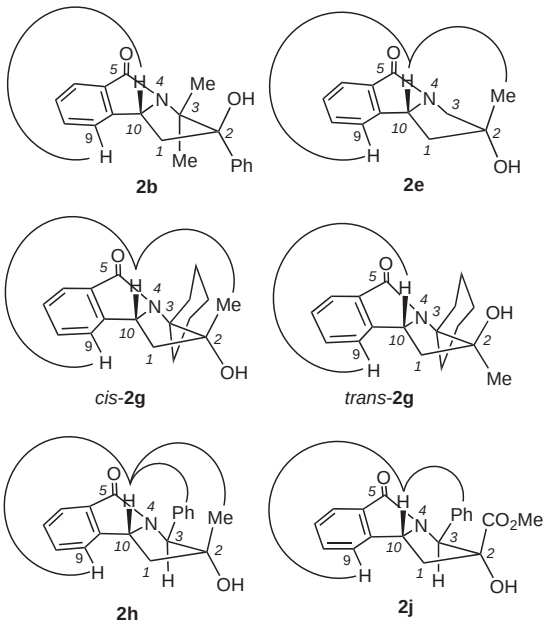


Figure 1 NOE correlations of **2b**, **2e**, **2g**, **2h**, and **2j**

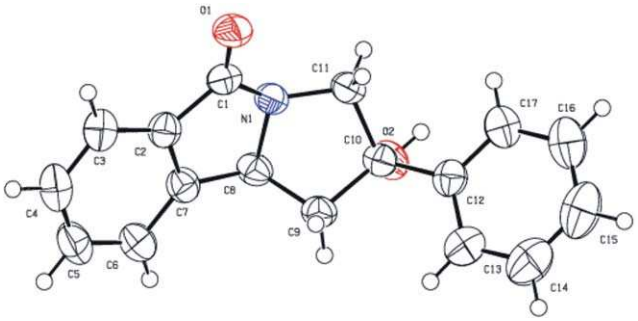


Figure 2 X-ray crystal structure (ORTEP drawing) of **2a**

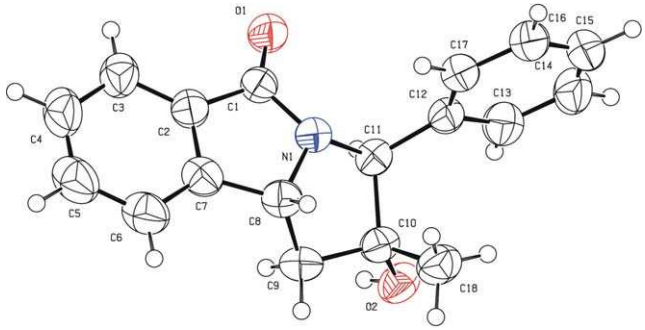


Figure 3 X-ray crystal structure (ORTEP drawing) of **2h**

It could be observed from Table 2 that the acyl groups in reactants **1a**–**l** have a great effect on the stereoselectivity of the photoreaction. For example, the photoreactions of **1a**–**d** and **1k** in which the acyl groups are all benzoyl group give mainly the *trans*-isomers, in other words, the configuration of the hydrogen atom on C10 and the hydroxy group on C2 is *cis*; but the photoreactions of **1e**–**j** and **1l** in which the acyl groups are acetyl or methoxyoxalyl groups afford mainly the *cis*-isomers in which the configuration of the hydrogen atom on C10 and the hydroxy group on C2 is *trans*. Meanwhile, *N*-alkyl groups have also effects on the structures of photocyclization products. For example, the configuration of the hydrogen atom on C10 and the phenyl group on C3 in products **2d**, **2h**, **2j**, **2k**, and **2l** are all *cis* when the *N*-alkyl is the benzyl group.

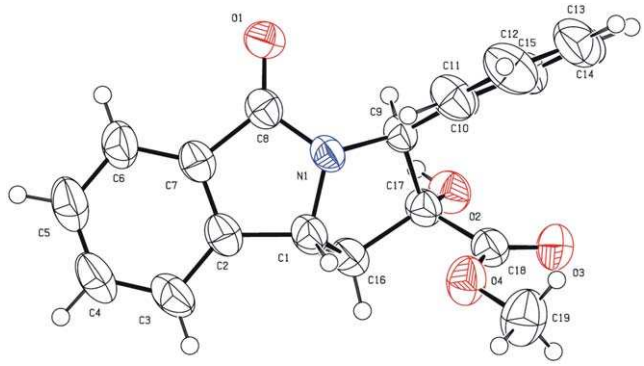


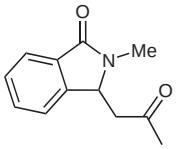
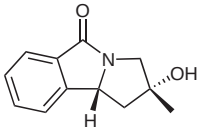
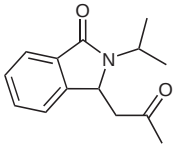
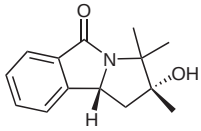
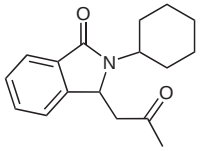
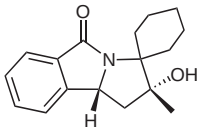
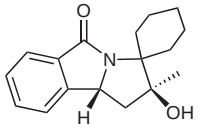
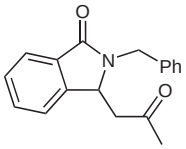
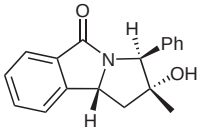
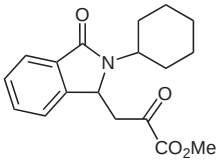
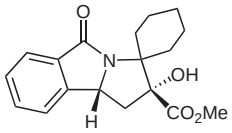
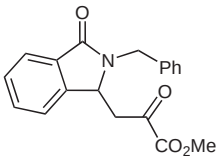
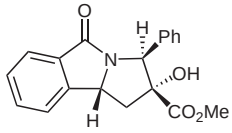
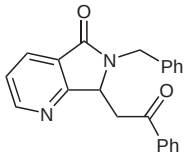
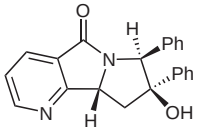
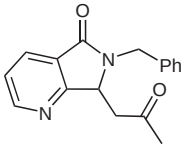
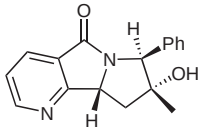
Figure 4 X-ray crystal structure (ORTEP drawing) of **2j**

In summary, an efficient synthesis of 2-hydroxypyrrolo[2,1-*a*]isoindol-5-ones has been achieved by the Norrish–Yang cyclization of 3-(acylmethyl)-2-alkylisoindol-1-ones in benzene.

Table 2 Photocyclization of 3-(Acylmethyl)-2-alkylisoindol-1-ones and 6-Alkyl-7-(benzoylmethyl)pyrrolo[3,4-*b*]pyridin-5-ones

Entry	Substrate	Time (h)	Conversion (%)	Product[a]	Yield[b] (%)	Mp (°C)
1	**1a**	20	81	**2a**	45	141–142
2	**1b**	20	76	**2b**	42	253–254
3	**1c**	18	85	**2c**	56	269–270
4	**1d**	24	74	**2d**	39	197–199

Table 2 Photocyclization of 3-(Acylmethyl)-2-alkylisoindol-1-ones and 6-Alkyl-7-(benzoylmethyl)pyrrolo[3,4-*b*]pyridin-5-ones (continued)

Entry	Substrate	Time (h)	Conversion (%)	Product[a]	Yield[b] (%)	Mp (°C)
5	**1e** 	20	75	**2e** 	51	149–150
6	**1f** 	20	68	**2f** 	48	dense oil
7	**1g** 	18	80	**2g**  (*cis*)	63	242–243
				2g  (*trans*)	15	dense oil
8	**1h** 	26	62	**2h** 	47	218–219
9	**1i** 	18	65	**2i** 	45	183–184
10	**1j** 	20	48	**2j** 	61	156–157
11	**1k** 	18	93	**2k** 	87	130–131
12	**1l** 	18	82	**2l** 	78	166–168

[a] Isolated pure isomers.
[b] Yields based on conversion of **1a**–**l**.

All the reactions were monitored by TLC (silica gel coated TLC plates). Silica gel 230–400 mesh was used for column chromatography. EI-MS were recorded with a HP 5988 A mass spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DRX-300 NMR spectrometers in $CDCl_3$ and DMSO-d_6 with TMS as an internal standard. Melting points were determined on a Yanagimoto melting point apparatus. All solvents were dried and distilled before use.

3-(Benzoylmethyl)-2-isopropyl-2,3-dihydro-1*H*-isoindol-1-one (1b); Typical Procedure

To a stirred CH_2Cl_2 soln (100 mL) of 3-hydroxy-2-isopropylisoindol-1-one **3b** (0.01 mol) and acetophenone (0.015 mol) [or methyl pyruvate (0.015 mol) for **1f**] was added $BF_3{\cdot}OEt_2$ (0.015 mol) at r.t. The mixture was stirred for 24–36 h until **3b** disappeared (monitored by TLC). The mixture was washed with H_2O (2 × 100 mL) and the organic phase was dried (anhyd Na_2SO_4) and concentrated in vacuo. The residuals were separated by column chromatography (silica gel, hexane–acetone) to afford **1b** (2.3 g, 78%).

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.44–1.51 (m, 6 H, 2 CH_3), 3.29 (dd, *J* = 17.7, 9.0 Hz, 1 H), 3.61 (dd, *J* = 17.7, 3.6 Hz, 1 H), 4.24 (m, 1 H), 5.38 (dd, *J* = 9.0, 3.6 Hz, 1 H), 7.35–7.39 (m, 1 H), 7.44 (m, 2 H), 7.47–7.52 (m, 2 H), 7.62 (t, *J* = 7.2 Hz, 1 H), 7.81–7.84 (m, 1 H), 7.96 (d, *J* = 6.9 Hz, 2 H, 2 CH).

3-(Benzoylmethyl)-2-cyclohexyl-2,3-dihydro-1*H*-isoindol-1-one (1c)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.17–1.40 (m, 3 H), 1.67 (d, *J* = 11.7 Hz, 1 H), 1.82–2.02 (m, 5 H), 2.03–2.16 (m, 1 H), 3.22–3.32 (m, 1 H), 3.65 (dd, *J* = 17.4, 3.6 Hz, 1 H), 3.68–3.84 (m, 1 H), 5.37 (dd, *J* = 8.7, 3.3 Hz, 1 H), 7.29–7.40 (m, 3 H), 7.42–7.51 (m, 2 H), 7.58–7.63 (m, 1 H), 7.78–7.81 (m, 1 H), 7.94–7.96 (m, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 25.4 (CH_2), 26.0 (CH_2), 26.1 (CH_2), 30.8 (CH_2), 31.2 (CH_2), 43.2 (CH_2), 54.0 (CH), 56.2 (CH), 122.7 (CH), 123.4 (C), 128.1 (2 CH), 128.3 (CH), 128.8 (2 CH), 131.5 (CH), 132.4 (C), 133.8 (CH), 146.2 (CH), 168.6 (C=O), 197.5 (C=O).

Methyl 3-(2-Cyclohexyl-3-oxo-2,3-dihydro-1*H*-isoindol-1-yl)pyruvate (1i)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.13–1.28 (m, 1 H), 1.31–1.45 (m, 2 H), 1.61–1.78 (m, 1 H), 1.83–2.09 (m, 6 H), 3.18 (dd, *J* = 18.9, 8.7 Hz, 1 H), 3.61 (dd, *J* = 18.3, 3.0 Hz, 1 H), 3.71–3.80 (m, 1 H), 3.88 (s, 3 H, CH_3), 5.11 (dd, *J* = 8.7, 3.6 Hz, 1 H), 7.31 (d, *J* = 6.3 Hz, 1 H), 7.50 (t, *J* = 6.3 Hz, 2 H, 2 CH), 7.81 (d, *J* = 6.3 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 25.3 (CH_2), 25.9 (CH_2), 26.1 (CH_2), 30.6 (CH_2), 31.2 (CH_2), 43.9 (CH_2), 53.3 (CH), 53.9 (CH), 55.1 (CH_3), 122.3 (CH), 123.5 (CH), 128.5 (CH), 131.5 (CH), 132.4 (C), 144.9 (C), 160.7 (C=O), 168.3 (C=O), 191.5 (C=O).

Methyl 3-(2-Benzyl-3-oxo-2,3-dihydro-1*H*-isoindol-1-yl)pyruvate (1j)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.12 (dd, *J* = 18.3, 6.3 Hz, 1 H), 3.38 (dd, *J* = 18.9, 6.0 Hz, 1 H), 3.77 (s, 3 H, CH_3), 4.55 (d, *J* = 15.3 Hz, 1 H), 4.98 (d, *J* = 11.7 Hz, 1 H, CH), 5.01 (d, *J* = 15.0 Hz, 1 H), 7.22–7.31 (m, 5 H), 7.35 (d, *J* = 6.3 Hz, 1 H), 7.47–7.55 (m, 2 H), 7.90 (d, *J* = 7.5 Hz, 1 H).

3-(Acetylmethyl)-2-cyclohexyl-2,3-dihydro-1*H*-isoindol-1-one (1g); Typical Procedure

To a stirred CH_2Cl_2 soln (100 mL) of 2-cyclohexyl-3-hydroxyisoindol-1-one **3c** (0.01 mol) and ethyl acetoacetate (0.011 mol), $BF_3{\cdot}OEt_2$ (0.015 mol) was added at r.t. The mixture was stirred for 2 h until **3c** disappeared (monitored by TLC). The mixture was washed with H_2O (2 × 100 mL)and the organic phase was dried (anhyd Na_2SO_4) and concentrated under reduced pressure. The residuals were separated by column chromatography (silica gel, hexane–acetone) to afford the coupling product ethyl 2-(2-cyclohexyl-3-oxo-2,3-dihydro-1*H*-isoindol-1-yl)acetoacetate (**5c**). To a soln of **5c** (5 mmol) in DMSO (5 mL) was added NaCl (6.5 mmol) and H_2O (40 μL, 2.2 mmol). The mixture was heated at 100 °C for 5 h and then H_2O was added and the mixture was extracted with CH_2Cl_2 (4 × 20 mL). The combined organic phases were dried (anhyd Na_2SO_4) and concentrated in vacuo. The residuals were separated by column chromatography (silica gel, hexane–acetone) to afford the decarboxylated product **1g** (1.2 g, 89%).

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.18–1.29 (m, 1 H), 1.34–1.44 (m, 2 H), 1.69 (d, *J* = 12.3 Hz, 1 H), 1.77–1.97 (m, 5 H), 2.00–2.09 (m, 1 H), 2.22 (s, 3 H, CH_3), 2.65–2.75 (m, 1 H), 3.21 (dd, *J* = 17.7 Hz, 1 H), 3.68–3.77 (m, 1 H), 5.10 (dd, *J* = 8.7, 3.6 Hz, 1 H), 7.31–7.34 (m, 1 H), 7.40–7.49 (m, 2 H), 7.77–7.80 (m, 1 H).

7-(Benzoylmethyl)-6-benzyl-6,7-dihydro-5*H*-pyrrolo[3,4-*b*]pyridin-5-one (1k)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.31 (dd, *J* = 18.0, 6.9 Hz, 1 H), 3.67 (dd, *J* = 18.0, 3.3 Hz, 1 H), 4.70 (d, *J* = 15.3 Hz, 1 H), 4.91 (d, *J* = 15.0 Hz, 1 H), 5.32 (dd, *J* = 6.9, 3.6 Hz, 1 H), 7.08–7.17 (m, 4 H), 7.25 (d, *J* = 6.6 Hz, 2 H), 7.35–7.44 (m, 3 H), 7.53 (t, *J* = 7.2 Hz, 1 H), 7.73–7.76 (m, 1 H), 8.18 (d, *J* = 7.8 Hz, 1 H), 8.70 (d, *J* = 8.1 Hz, 1 H).

7-(Acetylmethyl)-6-benzyl-6,7-dihydro-5*H*-pyrrolo[3,4-*b*]pyridin-5-one (1l)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.81 (s, 3 H, CH_3), 2.71 (dd, *J* = 18.0, 7.5 Hz, 1 H), 3.13 (dd, *J* = 18.0, 3.6 Hz, 1 H), 4.72 (d, *J* = 15.3 Hz, 1 H), 4.86 (d, *J* = 15.3 Hz, 1 H), 5.08 (dd, *J* = 7.2, 3.6 Hz, 1 H), 7.25–7.31 (m, 5 H), 7.41 (dd, *J* = 7.5, 4.5 Hz, 1 H), 8.16 (dd, *J* = 7.5, 1.4 Hz, 1 H), 8.70 (dd, *J* = 4.8, 1.4 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 29.8 (CH_3), 45.0 (CH_2), 45.0 (CH_2), 57.3 (CH), 123.4 (CH), 125.5 (C), 127.5 (CH), 128.0 (2 CH), 128.6 (2 CH), 131.9 (CH), 137.1 (C), 152.6 (CH), 164.9 (C=O), 166.9 (C), 204.6 (C=O).

2-Hydroxy-2-phenyl-1,2,3,9b-tetrahydro-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (2a); Typical Procedure for Photochemical Reactions

To dry benzene (100 mL) was added **1a** (1.0 mmol). The soln was distributed into 5 20-mL quartz tubes and deaerated by bubbling argon for 30 min and irradiated with a high-pressure Hg lamp (500 W) at r.t. for the appropriate time. The solvent was removed in vacuo and the products were separated by column chromatography (silica gel, hexane–acetone, 10:1) to afford **2a**. The solid was further purified by recrystallization (hexane–acetone) to give pure **2a** as colorless needles.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.73 (t, *J* = 11.7 Hz, 1 H), 2.01 (s, 1 H, OH), 2.61 (dd, *J* = 12.3,5.4 Hz, 1 H), 3.75 (d, *J* = 12.9 Hz, 1 H), 4.10 (d, *J* = 12.3 Hz, 1 H), 5.32 (dd, *J* = 11.1, 5.4 Hz, 1 H), 7.24–7.36 (m, 3 H), 7.41–7.51 (m, 4 H), 7.53–7.58 (m, 1 H), 7.81 (d, *J* = 4.8 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 45.6 (CH_2), 57.8 (CH_2), 63.9 (CH), 80.8 (C), 122.8 (CH), 124.1 (CH), 124.9 (2 CH), 127.8 (CH), 128.4 (2 CH), 128.5 (CH), 131.8 (CH), 133.3 (C), 143.5 (C), 143.6 (C), 171.7 (C=O).

MS (EI, 70 eV): *m*/*z* (%) = 265 (M^+, 15), 175 (65), 146 (76), 77 (100).

HRMS: *m*/*z* $[M + H]^+$ calcd for $C_{17}H_{16}NO_2$: 266.1176; found: 266.1170.

X-ray crystal data for **2a**: Recrystallized (acetone–hexane). $C_{17}H_{15}NO_2$, M_r = 265.30, monoclinic, *a* = 11.971(3) Å, *b* = 10.876(3) Å, *c* = 11.991(3) Å, β = 118.643(4), *V* = 1370.2(6) Å^3, colorless plates, ρ = 1.286 g cm^{-3}, *T* = 293(2) K, space group

*P*2(1)/*c*, *Z* = 4, μ (MoKα) = 0.085 mm^{-1}, 2 θ_{max} = 51°, 7039 reflections measured, 2555 unique (R_{int} = 0.0291), which were used in all calculations. The final *wR*(F^2) was 0.1144 (for all data), R_1 = 0.0413. CCDC file No. 707695.

3,3-Dimethyl-2-hydroxy-2-phenyl-1,2,3,9b-tetrahydro-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (2b)
Colorless needles.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.18 (s, 3 H, CH_3), 1.59 (s, 1 H, OH), 1.71 (s, 3 H, CH_3), 2.09 (dd, *J* = 14.1, 4.2 Hz, 1 H), 3.35 (dd, *J* = 14.1, 10.5 Hz, 1 H), 5.11 (dd, *J* = 10.5, 4.8 Hz, 1 H), 7.30–7.47 (m, 5 H), 7.52–7.57 (m, 3 H), 7.81 (d, *J* = 7.5 Hz, 1 H).

^{13}C NMR (75 MHz, DMSO-d_6): δ = 17.4 (CH_3), 25.8 (CH_3), 59.9 (CH_2), 68.6 (CH), 86.2 (C), 122.7 (CH), 122.9 (CH), 126.9 (2 CH), 127.0 (C), 127.4 (2 CH), 127.6 (CH), 131.7 (CH), 133.9 (C), 142.2 (C), 148.1 (C), 171.3 (C=O).

MS (EI, 70 eV): *m*/*z* (%) = 293 (M^+, 2), 250 (9), 173 (100).

HRMS: *m*/*z* [M + H]$^+$ calcd for $C_{19}H_{20}NO_2$: 294.1489; found: 294.1495.

2′-Hydroxy-2′-phenyl-1′,9b′-dihydrospiro[cyclohexane-1,3′-pyrrolo[2′,1′-*a*]isoindol]-5′(2′*H*)-one (2c)
Colorless needles.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.88–1.08 (m, 2 H), 1.10 (td, *J* = 13.5, 4.2 Hz, 1 H), 1.18–1.36 (m, 1 H), 1.54–1.65 (m, 4 H), 1.72–1.77 (m, 1 H), 1.93–2.01 (m, 1 H), 2.25–2.34 (m, 1 H), 2.43–2.89 (m, 1 H), 2.87 (d, *J* = 13.5 Hz, 1 H), 3.26 (t, *J* = 14.1 Hz, 1 H), 5.03 (dd, *J* = 10.5, 4.2 Hz, 1 H), 7.11–7.14 (m, 1 H), 7.33–7.48 (m, 4 H), 7.54–7.72 (m, 2 H), 7.73 (d, *J* = 5.8 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 22.2 (CH_2), 23.8 (CH_2), 24.6 (CH_2), 25.3 (CH_2), 33.5 (CH_2), 40.7 (C), 59.9 (CH_2), 73.6 (CH), 88.3 (C), 122.0 (CH), 123.9 (CH), 126.8 (2 CH), 127.5 (CH), 127.8 (2 CH),1 27.9 (CH), 131.8 (CH), 134.1 (C), 140.8 (C), 147.2 (C), 173.6 (C=O).

MS (EI, 70 eV): *m*/*z* (%) = 333 (M^+, 2), 251 (3), 213 (100).

HRMS: *m*/*z* [M + H]$^+$ calcd for $C_{22}H_{24}NO_2$: 334.1802; found: 334.1798.

2-Hydroxy-2,3-diphenyl-1,2,3,9b-tetrahydro-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (2d)
Colorless needles.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.05 (s, 1 H, OH), 2.22 (d, *J* = 13.5 Hz, 1 H), 3.08 (t, *J* = 10.5 Hz, 1 H), 5.47 (s, 2 H, 2 CH), 6.93–6.96 (m, 2 H), 7.11–7.19 (m, 8 H), 7.45–7.49 (m, 2 H), 7.53–7.64 (m, 1 H), 7.91 (d, *J* = 6.5 Hz, 1 H).

^{13}C NMR (75 MHz, DMSO-d_6): δ = 39.5 (CH_2), 63.2 (C), 73.4 (CH), 87.4 (CH), 123.3 (CH), 123.6 (CH), 126.5 (2 CH), 126.8 (CH), 127.4 (2 CH), 127.5 (2 CH), 127.6 (CH), 127.7 (2 CH), 128.1 (CH), 132.3 (CH), 132.5 (C), 138.5 (C), 142.2 (C), 150.1 (C), 174.4 (C=O).

MS (EI, 70 eV): *m*/*z* (%) = 323 ($M^+ - H_2O$, 2), 221 (82), 57 (100).

HRMS: *m*/*z* [M + Na]$^+$ calcd for $C_{23}H_{19}NNaO_2$: 364.1308; found: 364.1305.

2-Hydroxy-3-methyl-1,2,3,9b-tetrahydro-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (2e)
Colorless needles.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.54 (s, 3 H, CH_3), 1.82 (dd, *J* = 12.3, 7.5 Hz, 1 H), 2.31 (dd, *J* = 12.3, 8.4 Hz, 1 H), 2.77 (s, 1 H, OH), 3.22 (d, *J* = 12.3 Hz, 1 H), 3.90 (d, *J* = 12.6 Hz, 1 H), 4.80 (t, *J* = 8.4 Hz, 1 H), 7.41 (t, *J* = 7.5 Hz, 2 H, 2 CH), 7.51 (d, *J* = 7.2 Hz, 1 H), 7.75 (d, *J* = 7.8 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 26.7 (CH_3), 43.8 (CH_2), 57.3 (CH_2), 63.1 (C), 82.3 (CH), 122.5 (CH), 124.2 (CH), 128.2 (CH), 132.1 (CH), 132.6 (C), 147.6 (C), 173.3 (C=O).

MS (EI, 70 eV): *m*/*z* (%) = 203 (M^+, 26), 174 (4), 160 (17), 145 (100).

HRMS: *m*/*z* [M + H]$^+$ calcd for $C_{12}H_{14}NO_2$: 204.1090; found: 204.1020.

2-Hydroxy-2,3,3-trimethyl-1,2,3,9b-tetrahydro-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (2f)
Pale dense oil.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.41 (s, 3 H, CH_3), 1.42 (s, 3 H, CH_3), 1.61 (s, 3 H, CH_3), 1.93 (dd, *J* = 12.8, 8.0 Hz, 1 H), 2.00 (s, 1 H, OH), 2.38 (dd, *J* = 12.8, 8.8 Hz, 1 H), 4.79 (t, *J* = 8.4 Hz, 1 H), 7.35 (d, *J* = 7.6 Hz, 1 H), 7.40 (t, *J* = 7.2 Hz, 1 H), 7.50 (t, *J* = 7.2 Hz, 1 H), 7.75 (d, *J* = 7.6 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 18.7 (CH_3), 23.3 (CH_3), 24.8 (CH_3), 41.3 (CH_2), 60.5 (C), 66.3 (C), 84.3 (CH), 122.1 (CH), 123.9 (CH), 128.1 (CH), 131.6 (CH), 134.5 (C), 146.5 (C), 171.0 (C=O).

MS (EI, 70 eV): *m*/*z* (%) = 231 (M^+, 4), 196 (42), 173 (100).

HRMS: *m*/*z* [M + H]$^+$ calcd for $C_{14}H_{18}NO_2$: 232.1332; found: 232.1332.

***trans*-2′-Hydroxy-2′-methyl-1′,9b′-dihydrospiro[cyclohexane-1,3′-pyrrolo[2′,1′-*a*]isoindol]-5′(2′*H*)-one (2g)**
Pale dense oil.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.05 (s, 3 H, CH_3), 1.35–1.39 (m, 2 H), 1.63–1.68 (m, 2 H), 1.71–1.78 (m, 2 H), 1.81–1.89 (m, 2 H), 2.25–2.29 (m, 2 H), 2.45 (dd, *J* = 12.8, 10.8 Hz, 1 H), 2.66 (d, *J* = 13.6 Hz, 1 H), 4.89 (dd, *J* = 10.0, 5.6 Hz, 1 H), 7.37 (d, *J* = 7.6 Hz, 1 H), 7.43 (t, *J* = 7.2 Hz, 1 H),7.52 (t, *J* = 7.6 Hz, 1 H), 7.76 (d, *J* = 7.6 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 22.4 (CH_3), 24.2 (CH_2), 25.6 (CH_2), 26.3 (CH_2), 26.5 (CH_2), 31.6 (CH_2), 41.5 (CH_2), 59.0 (C), 69.5 (C), 82.6 (CH), 122.2 (CH), 123.8 (CH), 128.1 (CH), 131.7 (CH), 134.5 (C), 146.3 (C), 170.7 (C=O).

MS (EI, 70 eV): *m*/*z* (%) = 271 (M^+, 11), 251 (19), 228 (5), 213 (100).

HRMS: *m*/*z* [M + H]$^+$ calcd for $C_{17}H_{22}NO_2$: 272.1645; found: 272.1649.

***cis*-2′-Hydroxy-2′-phenyl-1′,9b′-dihydrospiro[cyclohexane-1,3′-pyrrolo[2′,1′-*a*]isoindol]-5′(2′*H*)-one (3g)**
Colorless needles.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.31 (s, 3 H, CH_3), 1.33–1.40 (m, 2 H), 1.61–1.64 (m, 2 H), 1.66–1.70 (m, 3 H), 1.93 (dd, *J* = 13.6, 4.8 Hz, 2 H), 2.36–2.43 (m, 2 H), 2.74 (dd, *J* = 13.2, 6.0 Hz, 1 H), 4.81 (dd, *J* = 10.4, 4.8 Hz, 1 H), 7.34 (d, *J* = 7.6 Hz, 1 H), 7.40 (t, *J* = 7.6 Hz, 1 H), 7.50 (t, *J* = 7.6 Hz, 1 H), 7.74 (d, *J* = 7.2 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 22.5 (CH_3), 22.7 (CH_2), 24.0 (CH_2), 25.1 (CH_2), 25.8 (CH_2), 33.6 (CH_2), 41.4 (CH_2), 60.0 (C), 72.4 (C), 85.4 (CH), 122.0 (CH), 123.9 (CH), 128.0 (CH), 131.8 (CH), 134.1 (C), 147.1 (C), 173.0 (C=O).

MS (EI, 70 eV): *m*/*z* (%) = 271 (M^+, 11), 251 (13), 228 (16), 213 (100).

HRMS: *m*/*z* [M + H]$^+$ calcd for $C_{17}H_{22}NO_2$: 272.1645; found: 272.1651.

2-Hydroxy-2-methyl-3-phenyl-1,2,3,9b-tetrahydro-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (2h)
Colorless needles.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.10 (s, 3 H), 1.58 (s, 1 H, OH), 1.87 (t, *J* = 11.7 Hz, 1 H), 2.45 (dd, *J* = 12.0, 6.0 Hz, 1 H), 4.92 (s, 1 H), 5.02 (dd, *J* = 12.3, 6.6 Hz, 1 H), 7.29–7.36 (m, 5 H), 7.46–7.57 (m, 2 H), 1.80 (t, *J* = 6.3 Hz, 1 H), 7.89 (d, *J* = 7.8 Hz, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 24.4 (CH_3), 44.5 (CH_2), 62.3 (CH), 69.4 (CH), 83.6 (C), 123.3 (CH), 123.4 (CH), 126.4 (2 CH), 126.9 (CH), 128.0 (2 CH), 128.3 (CH), 132.1 (CH), 132.3 (C), 139.5 (C), 147.8 (C), 172.0 (C=O).

MS (EI, 70 eV): *m*/*z* (%) = 279 (M^+, 3), 236 (2), 221 (100).

HRMS: *m*/*z* $[M + H]^+$ calcd for $C_{18}H_{17}NNaO_2$: 302.1151; found: 302.1146.

C*rystal data for* **2h**: Recrystallized (acetone–hexane). $C_{18}H_{17}NO_2$, M_r = 279.33, monoclinic, *a* = 20.782(19) Å, *b* = 8.375(7) Å, *c* = 16.846(14) Å, β = 94.00(3), *V* = 2925(4) Å^3, colorless plates, ρ = 1.269 g cm^{-3}, *T* = 296(2) K, space group *C*2/*c*, *Z* = 8, μ(MoKα) = 0.083 mm^{-1}, 2 θ_{max} = 54.40°, 8312 reflections measured, 3212 unique (R_{int} = 0.0688), which were used in all calculations. The final *wR*(F^2) was 0.1460 (for all data), R_1 = 0.0926. CCDC file No. 707696.

Methyl 2′-Hydroxy-5′-oxo-1′,2′,5′,9b′-dihydrospiro[cyclohexane-1,3′-pyrrolo[2′,1′-*a*]isoindole]-2′-carboxylate (2i)

Colorless needles.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.12–1.28 (m, 2 H), 1.34–1.42 (m, 1 H), 1.59–1.66 (m, 2 H), 1.76 (d, *J* = 12.4 Hz, 1 H), 1.87–1.93 (m, 2 H), 1.94–2.05 (m, 1 H), 2.33–2.37 (m, 1 H), 2.86 (dd, *J* = 13.6, 3.2 Hz, 1 H), 3.19 (dd, *J* = 14.0, 10.4 Hz, 1 H), 3.85 (s, 3 H, CH_3), 4.95 (dd, *J* = 10.4, 5.2 Hz, 1 H), 7.37 (d, *J* = 7.6 Hz, 1 H), 7.43 (t, *J* = 7.6 Hz, 1 H), 7.52 (m, 1 H), 7.75 (d, *J* = 3.6 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 22.3 (CH_2), 23.9 (CH_2), 25.4 (CH_2), 25.5 (CH_2), 34.1 (CH_2), 37.7 (CH_2), 52.9 (CH_3), 60.2 (C), 72.6 (C), 88.5 (CH), 122.1 (CH), 123.9 (CH), 128.2 (CH), 132.0 (CH), 133.8 (C), 146.5 (C), 172.4 (C=O), 172.8 (C=O).

MS (EI, 70 eV): *m*/*z* (%) = 315 (M^+, 6), 227 (3), 213 (100).

HRMS: *m*/*z* $[M + H]^+$ calcd for $C_{18}H_{22}NO_4$: 316.1543; found: 316.1540.

Methyl 2-Hydroxy-5-oxo-3-phenyl-2,3,5,9b-tetrahydro-1*H*-pyrrolo[2,1-*a*]isoindole-2-carboxylate (2j)

Colorless needles.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.92 (t, *J* = 11.7 Hz, 1 H), 2.80 (dd, *J* = 12.9, 6.3 Hz, 1 H), 3.33 (s, 3 H, CH_3), 4.17 (s, 1 H, OH), 5.24 (s, 1 H), 5.43 (dd, *J* = 10.8, 6.3 Hz, 1 H), 7.25–7.46 (m, 5 H), 7.46–7.57 (m, 2 H), 7.60 (t, *J* = 6.6 Hz, 1 H), 7.88–7.91 (m, 1 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 41.3 (CH_3), 52.6 (CH_2), 64.1 (CH), 68.9 (C), 89.9 (CH), 122.6 (CH), 124.6 (CH), 125.6 (2 CH), 127.6 (CH), 128.2 (2 CH), 128.7 (CH), 132.3 (CH), 132.4 (C), 137.3 (C), 146.5 (C), 172.2 (C=O), 173.4 (C=O).

MS (EI, 70 eV): *m*/*z* (%) = 323 (M^+, 2), 234 (3), 221 (100).

HRMS: *m*/*z* $[M + H]^+$ calcd for $C_{19}H_{18}NO_4$: 324.1231; found: 324.1236.

C*rystal data for* **2j**: Recrystallized (acetone–hexane). $C_{19}H_{17}NO_4$, M_r = 323.34, triclinic, *a* = 8.608(5) Å, *b* = 10.207(6) Å, *c* = 10.802(5) Å, β = 107.91(3), *V* = 808.9(8) Å^3, colorless plates, ρ = 1.327 g cm^{-3}, *T* = 296(2) K, space group $P\bar{1}$, *Z* = 2, μ(MoKα) = 0.094 mm^{-1}, 2 θ_{max} = 54.40°, 3840 reflections measured, 2643 unique (R_{int} = 0.0284) which were used in all calculations. The final *wR*(F^2) was 0.1460 (for all data), R_1 = 0.0926. CCDC file no. 722237.

8-Hydroxy-7,8-diphenyl-7,8,9,9a-tetrahydro-5*H*-pyrido[2,3-*a*]pyrrolizin-5-one (2k)

Colorless needles.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.03 (t, *J* = 12.3 Hz, 1 H), 2.84 (dd, *J* = 12.6, 5.1 Hz, 1 H), 5.50 (s, 1 H), 5.58 (dd, *J* = 11.1, 5.1 Hz, 1 H), 7.07–7.10 (m, 2 H), 7.27–7.32 (m, 4 H), 7.33–7.47 (m, 5 H), 8.16 (dd, *J* = 7.8, 1.5 Hz, 1 H), 8.75 (dd, *J* = 4.8, 1.5 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 45.5 (CH_2), 65.7 (CH), 70.1 (CH), 87.2 (C), 123.6 (C), 125.1 (2 CH), 126.9 (2 CH), 127.1 (C), 127.7 (CH), 128.2 (CH), 128.5 (2 CH), 128.7 (2 CH), 132.6 (CH), 134.9 (C), 142.5 (C), 152.9 (CH), 166.3 (CH), 170.2 (C=O).

MS (EI, 70 eV): *m*/*z* (%) = 342 (M^+, 3), 237 (6), 84 (100).

HRMS: *m*/*z* $[M + H]^+$ calcd for $C_{22}H_{18}N_2NaO_2$: 365.1260; found: 365.1254.

8-Hydroxy-8-methyl-7-phenyl-7,8,9,9a-tetrahydro-5*H*-pyrido[2,3-*a*]pyrrolizin-5-one (2l)

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.15 (s, 3 H, CH_3), 2.05 (dd, *J* = 12.8, 8.0 Hz, 1 H), 2.54 (dd, *J* = 12.4, 7.6 Hz, 1 H), 5.07 (s, 1 H), 5.14 (t, *J* = 8.0 Hz, 1 H), 7.28–7.31 (m, 1 H), 7.32–7.39 (m, 4 H), 7.44 (dd, *J* = 7.6, 4.8 Hz, 1 H), 8.16 (d, *J* = 8.0 Hz, 1 H), 8.77 (d, *J* = 4.0 Hz, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 26.5 (CH_3), 43.0 (CH_2), 65.3 (CH), 68.9 (CH), 83.6 (C), 123.6 (CH), 127.5 (2 CH), 128.4 (CH), 128.5 (C), 128.9 (2 CH), 132.7 (CH), 135.9 (C), 152.8 (C), 166.5 (C=O), 170.3 (C=O).

MS (EI, 70 eV): *m*/*z* (%) = 280 (M^+, 23), 237 (21), 194 (37), 106 (100).

HRMS: *m*/*z* $[M + H]^+$ calcd for $C_{17}H_{17}N_2O_2$: 281.1285; found: 281.1290.

Acknowledgment

We thank the National Natural Science Foundation of China (20872056) for financial support.

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