A Concise Synthesis of the DNA-Intercalating and Antimalarial Alkaloid Cryptolepine and Its Fluorescence Behaviour in Solvents of Different Polarities

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A microwave-induced rapid and facile synthesis of the DNA-intercalating and antimalarial drug cryptolepine is described. The key step in this synthesis involves the aqueous-phase base-catalyzed condensation of isatin and 1-acetyl-1*H*-indol-3-yl acetate which has been simplified and expedited by dielectric heating, employing an ordinary domestic microwave oven. The method transforms the synthesis of an important drug molecule from a prohibitively lengthy process to a matter of a few minutes with a much improved yield. Dual absorption and fluorescence is observed from the molecular system in solvents of different polarity thus providing valuable insight into its binding modes toward protein or DNA.

1. Introduction. – Malaria is one of the planet's deadliest diseases and a leading cause of sickness and death in the developing world. It kills over a million people each year – mostly children, with an estimated 300–500 million clinical cases per year and some 3.2 billion people living in 107 countries or territories are at risk. Africa alone accounts for over 90% of reported cases [1]. Cryptolepine (5-methyl-5*H*-indolo[3,2-*b*]quinoline; **1**), found in the decoction of the roots of the West African climbing shrubs *Cryptolepis sanguinolenta* and *Cryptolepis triangularis* N. E. BR. (Periplocaceae), is a rare example of a natural product whose synthesis was reported prior to its isolation [2]. Pharmacological investigations on cryptolepine and its analogues have revealed diverse biological activities of this polyheteroaromatic alkaloid that include its DNA-intercalating, cytotoxic, hypotensive, antipyretic, anti-inflammatory, antibacterial, and antihyperglycemic activities in addition to its more celebrated role in the control of various forms of malaria [3–8]. The skeleton also serves as a template for the development of newer antimalarials so urgently required to combat the growing menace of malaria caused by various resistant strains of the parasite *Plasmodium falciperum*. The interest in this polyheteroaromatic skeleton has grown further after the discovery of the antitumour activity of ellipticine and 9-methoxyellipticine [9]. Since natural sources are not sufficient to satisfy the ever-growing demand of this wonder alkaloid, an explosion of activities toward the construction of the cryptolepine skeleton is obvious. After the first synthesis of *Fichter* and *Boehringer* in 1906 [10], several attempts to synthesize this skeleton were made over the decades [11–15]. All these

methods involve poorly available starting materials and multi-step procedures demanding large amounts of time and energy, often requiring the use of high pressure vessels and drastic conditions sometimes leading to decomposition.

Isatin (**2**) is a versatile molecule, upon which the synthesis of a large number of bioactive heterocycles, spiroheterocycles, and metal complexes is based [16–18]. Ever since the introduction by *Gedye et al.* and *Giguere et al.* [19], microwaves have been extensively utilized to drive organic reactions, thus saving time and energy and providing eco-friendly methods for many reactions [20]. In conjunction with our earlier efforts of simplifying lengthy procedures for synthesis of bioactive heterocycles with the help of microwaves [21–24], and studies on bioactive natural components [25], we report herein a microwave mediated efficient synthesis of cryptolepine starting from readily available materials, anthranilic acid and isatin.

Absorption and fluorescence spectroscopic techniques serve as useful tools for monitoring the different modes of binding of various drugs to protein and DNA [26][27]. The polarity of solvents often modified these spectral parameters [27][28]. Although cryptolepine has been used extensively for studying the intercalative mode of binding [29], its intrinsic fluorescence behaviour has not been reported. Here we studied the fluorescence characteristics of cryptolepine and observed an interesting solvent-polarity dependent two-species spectroscopic behaviour in various common solvents. These observations are helpful in exploring its binding mode toward proteins or DNA and may offer useful insight while tuning its efficacy as a drug.

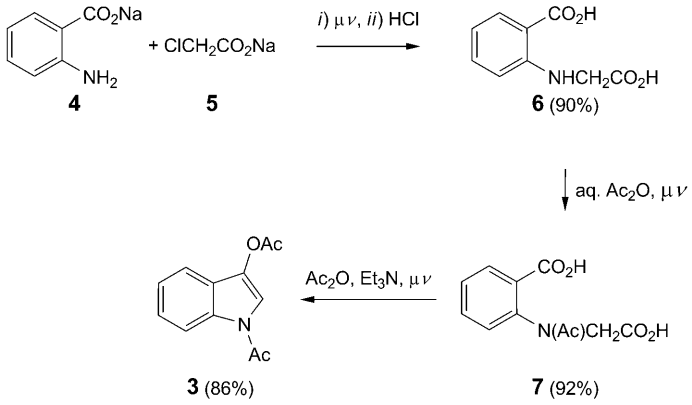
2. Results and Discussion. – 2.1. *Synthesis of Cryptolepine.* All the microwave-promoted steps described in this work were rapid, needing only a few minutes for completion of the reactions that usually take hours, even days to carry out a single step [30]. In most of the cases, conversions were almost complete within the irradiator itself, and pure products were obtained from the reaction mixtures by a simple crystallization step thus further simplifying the purification procedure. The protocol has been divided into two parts: *a*) a simple synthesis of 1-acetyl-1*H*-indol-3-yl acetate (**3**), and *b*) the synthesis of cryptolepine (**1**) through an aqueous-phase microwave-promoted condensation of **3** and **2** (*Schemes 1* and *2*). The synthesis is now so facile that starting from isatin and anthranilic acid, cryptolepine could be obtained in a single day.

In *Scheme 1*, the condensation between the sodium salts of anthranilic acid (**4**), and chloroacetic acid (**5**), in aqueous solution under microwaves is shown. This yielded off-white crystals of 2-[(carboxymethyl)amino]benzoic acid (**6**). *N*-Acetylation of **6** afforded **7** as white solid. Subsequent microwave-induced cyclization afforded 1-acetyl-1*H*-indol-3-yl acetate (**3**) in good yield.

Scheme 2 shows the reaction *via* a nucleophilic attack by 1-acetyl-1*H*-indol-3-yl acetate (**3**), at position C(2) of isatin (**2**), resulting in the opening of the indole ring, followed by an intramolecular *exo-trig* cyclization to give quindoline-11-carboxylic acid (**8**), which was decarboxylated by heating in diphenylether to yield quindoline (**9**). Methylation was carried out with iodomethane in tetrahydrothiophene 1,1-dioxide to afford orange-brown crystals of **10**.

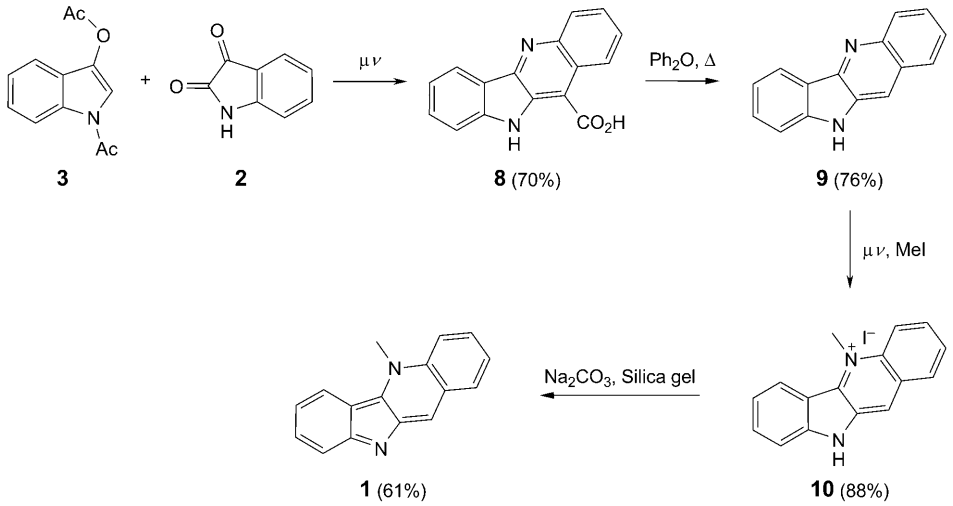
Conversion of the hydriodide **10** to cryptolepine (**1**) was carried out by the method of *Bierer et al.* [15]. Compound **1** crystallized from $CHCl_3$/MeOH as purple needles. The physical characteristics of **10** as well as the NOESY correlations of the MeN group

Scheme 1. *Synthesis of 1-Acetyl-1*H*-indol-3-yl Acetate*



4 **5** **6** (90%) **3** (86%) **7** (92%)

Scheme 2. *Synthesis of Cryptolepine*



3 **2** **8** (70%) **9** (76%) **10** (88%) **1** (61%)

at δ(H) 5.00 (*s*) with H–C(4) at δ(H) 8.73 (*d*, $J = 9$) and H–C(6) at δ(H) 8.75 (*d*, $J =$ 8) in the two-dimensional NMR spectra of **10** unambiguously established the identity of the final product as cryptolepine, thereby discarding the possibility of formation of its structural isomer cryptotackiene (**11**; *Fig. 1*), which would obviously show a different set of NOESY correlations around the MeN group.

2.2. *Absorption and Fluorescence Measurements.* In non-polar solvents like *p*-dioxane and tetrahydrofuran, the lowest energy structured absorption band of cryptolepine appeared in the range 330–430 nm, while in polar solvents (MeCN, EtOH, and MeOH) an additional broad absorption band appeared in the wavelength range 400–500 nm. The structured band was due to the excitation of the normal molecule, while the broad, structureless band was due to the excitation of the charge-

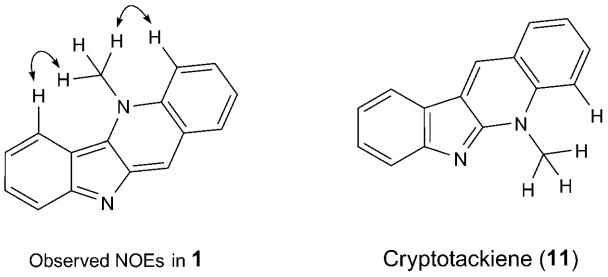


Fig. 1. *Observed NOEs in* **1** *and structure of cryptotackiene*

transfer (CT) species present in polar media. *Fig. 2* shows the representative absorption spectra of cryptolepine in 1,4-dioxane and EtOH. The figure reflects an appreciable blue shift for the structured absorption band in polar solvents. This has been ascribed to the stabilization of the highest occupied molecular orbital (HOMO) in the polar environment.

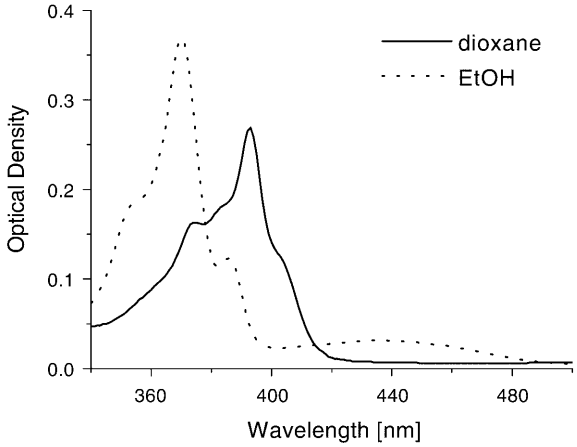


Fig. 2. *Absorption spectra of cryptolepine in 1,4-dioxane and EtOH*

In non-polar solvents the fluorescence spectrum of cryptolepine appeared as a structured band in the range 400–550 nm. The excitation spectrum corresponding to this emission resembled the absorption spectrum. Further, the excitation and fluorescence spectra showed mirror image relationship. This confirmed that the fluorescence originated directly from the lowest excited state (S_1) of the molecule. In polar solvents, the fluorescence spectrum of cryptolepine got modified drastically. In *Fig. 3*, the fluorescence emission and excitation spectra of the molecule in 1,4-dioxane and EtOH solvents have been shown. The normal fluorescence band observed in the non-polar media decreased remarkably and a lower energy, broad and structureless emission band (450–700 nm) characteristic of a charge-transfer spectrum appeared.

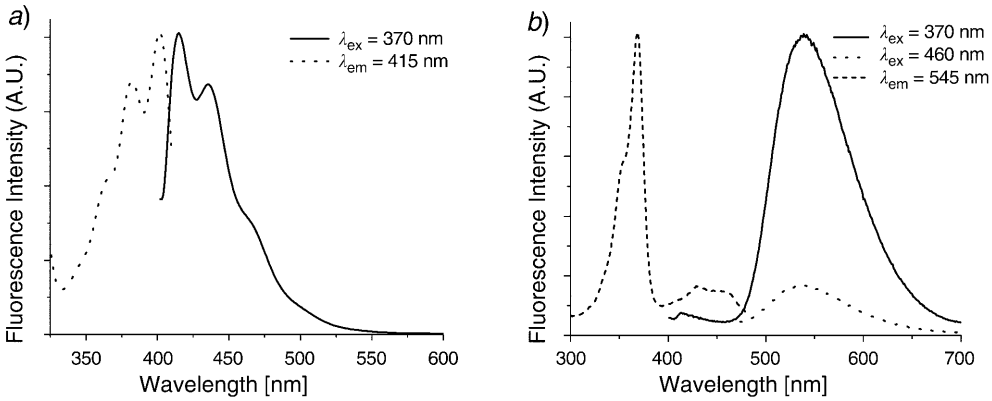


Fig. 3. *Fluorescence emission and excitation spectra of* **1** *in* a) *1,4-dioxane and* b) *EtOH* (excitation and monitoring wavelengths are indicated in the insets)

This low energy emission showed a mirror relationship with the broad absorption (400–500 nm) observed in polar solvents. Irrespective of the wavelength of excitation the CT emission dominated in polar solvents. This indicated that in the photo-excited state (S_1) the equilibrium shifted more towards the CT species in polar media. The photophysical parameters of cryptolepine in some selected solvents have been described in the *Table*.

Table. *Photophysical Parameters of Cryptolepine in Selected Solvents*

Solvent	Absorption maximum [nm]	Fluorescence maximum [nm]	Fluorescence quantum yield	
			Normal emission	CT emission
1,4-Dioxane	374 (*s*), 394, 405 (*s*)	415 (structured)	0.020	–
THF	373 (*s*), 391, 404 (*s*)	415 (structured)	0.021	–
MeCN	350 (*s*), 367, 388 (*s*), 428 (broad)	538 (broad)	–	0.036
EtOH	354 (*s*), 370, 386 (*s*), 433 (broad)	539 (broad)	–	0.031
MeOH	353 (*s*), 369, 434 (broad)	543 (broad)	–	0.024

3. Conclusion. – With the simple expedient of dielectric heating, *i.e.* using a domestic microwave oven, we simplified the synthesis of a major drug molecule, cryptolepine, to such an extent that starting from readily available inexpensive starting materials *viz.* anthranilic acid and isatin, the drug could be obtained within a single day. Use of microwaves and carrying out the key condensation step in aqueous phase offered additional advantage of environment-friendliness. Our protocol simplifies and effectively complements the earlier exhaustive procedures in a relatively 'greener' way. The absorption and fluorescence spectral studies reveal that there is a solvent dependent equilibrium between the normal and the charge transfer species of cryptolepine. In polar solvents, the equilibrium shifted towards the CT species both

in the ground and the photo-excited state. These observations will be helpful in future projects targeted at tuning the efficacy of the skeleton or its simple derivatives as a drug.

Experimental Part

General. The technical-grade anthranilic acid was crystallized from H_2O, containing a little HCl, twice, until the material showed reliable m.p. (144°). All other chemicals employed were either of A. R. grade or purified before use. All the solvents were distilled and dried (wherever necessary) before use. Column chromatography (CC): silica gel SRL (SiO_2; spherical, 60–120 mesh). Microwave irradiations: 1200W *BPL-SANYO* domestic microwave oven, pierced in the roof. Melting points: electrical melting point apparatus (*Sunvic*, U. K.). Absorption and fluorescence spectra: *Shimadzu MPS 2000* spectrophotometer and *Spex Fluorolog II* spectrofluorimeter, respectively. For the spectral measurements, spectroscopic grade solvents from *Aldrich Chemicals* were employed. The spectral studies were performed at ambient temperature. The fluorescence quantum yields were determined against quinine sulfate in 0.1N H_2SO_4 soln. ($\phi = 0.53$) [31]. IR Spectra: *Perkin-Elmer FT/IR-300E* spectrophotometer; in cm^{-1}. ^1H- and ^{13}C-NMR spectra: *Bruker AV 300* (300 MHz) instrument using (D_6)DMSO as solvent with Me_4Si (^1H) and the middle resonances of (D_6)DMSO (octet, 40.0, for ^{13}C) as internal standards. δ in ppm, J in Hz. Two dimensional NMR experiments: *Bruker Biospin DRX 500* instrument. MS: *JEOL JMS 600* instrument with direct inlet; in m/z. CHN analyses: *Perkin-Elmer 2400 Series-II* CHNS/O Analyzer.

2-[(Carboxymethyl)amino]benzoic Acid (**6**). **4** (2.1 g, 15 mmol) in 2.0 ml H_2O was dissolved in a soln. of 0.65 g NaOH in 1.8 ml H_2O, and was mixed cautiously, to avoid excessive foaming, with a soln. of **5** (1.4 g, 17 mmol) and 0.81 g Na_2CO_3 in 2 ml H_2O. The mixture was refluxed carefully under dielectric heating conditions for 14 min at 70% power level. The partially solidified mixture was dissolved in warm 1N NaOH and then acidified with conc. HCl. The resulting off-white solid that promptly turned to snuff color in acid soln. was collected and crystallized from MeOH/H_2O to yield 2.5 g (90%) of **6**. Off-white crystals turning snuff readily. M.p. 220° (dec). IR (KBr): 3374 (NH str.), 3260–2820 (br., peak at 3164, CO_2H), 2369, 1725 (CO str.), 1662, 1221. ^1H-NMR: 2.51 (br. *s*, NH); 3.99 (*s*, ArNHC$H_2$$CO_2H$); 7.80–6.60 (*m*, 4 arom. H); 12.80 (br. *s*, CO_2H). ^{13}C-NMR: 44.7 (*t*, C(2)); 111.0 (*d*, C(3′)); 112.0 (*s*, C(1′)); 115.2 (*d*, C(5′)); 132.1 (*d*, C(6′)); 134.9 (*d*, C(4′)); 150.4 (*s*, C(2′)); 170.1 (*s*, C(7′)); 172.2 (*s*, C(1)). Anal. calc. for $C_9H_9NO_4$: C 55.38, H 4.62, N 7.18, found: C 55.34, H 4.48, N 7.04.

2-[Acetyl(carboxymethyl)amino]benzoic Acid (**7**). To a soln. of 1.70 g anh. Na_2CO_3 in 1.70 ml H_2O, 1.95 g (10 mmol) of **6** was added and, after dissolution by 73 ml acetic anhydride, irradiated under microwave for 4 min. The solid that separated out on acidification with conc. HCl was collected, washed with a little H_2O and dried to afford 2.20 g (92%) of **7** crystallized from MeOH containing a few drops of $CHCl_3$. Off-white crystals. M.p. 208–210° (dec.). IR (KBr): 3434 (NH str.), 3220–2860 (br., peak at 3019, CO_2H), 2365, 1725 (CO str.), 1601, 1391, 1229. ^1H-NMR: 1.69 (*s*, MeCO); 3.63 (*d*, $J = 17.2$, ArN(Ac)CH*H*CO_2H); 4.61 (*d*, $J = 17.2$, ArN(Ac)C*H*HCO_2H); 7.93–7.50 (*m*, 4 arom. H); 12.84 (br. *s*, CO_2H). ^{13}C-NMR: 22.2 (*q*, C(9′)); 51.3 (*t*, C(2)); 129.1 (*d*, C(3′)); 129.9 (*s*, C(1′)); 131.0 (*d*, C(5′)); 131.7 (*d*, C(6′)); 133.7 (*d*, C(4′)); 142.9 (*d*, C(2′)); 167.1 (*s*, C(8′)); 169.5 (*s*, C(7′)); 171.0 (*s*, C(1)). Anal. calc. for $C_{11}H_{11}NO_5$: C 55.70, H 4.64, N 5.91; found: C 55.96, H 4.59, N 6.20.

*1-Acetyl-1*H*-indol-3-yl Acetate* (**3**). In a mixture of 9 ml Ac_2O and 1.7 ml Et_3N, 1.65 g (7 mmol) of powdered **7** was dissolved, and irradiated under microwave at 80% power level for 4 min under strict N_2 atmosphere. Most of the liquid was then evaporated *in vacuo*. The remaining oily residue on shaking with 60 ml H_2O yielded a crude solid, which on purification through CC afforded 1.30 g (86%) of **3**. Snow white crystals. M.p. 85–86° (dec.). IR (KBr): 1745 (CO str.), 1699, 1451, 1380, 1211. ^1H-NMR: 2.39 (*s*, OCOMe); 2.62 (*s*, NCOMe); 7.31 (*t*, $J = 7.5$, H–C(5)); 7.37 (*t*, $J = 7.5$, H–C(6)); 7.51 (*d*, $J = 7.6$, H–C(7)); 7.89 (*s*, H–C(2)); 8.36 (*d*, $J = 7.6$, H–C(4)). ^{13}C-NMR: 21.0 (*q*, C(13)); 24.2 (*q*, C(11)); 116.2 (*d*, C(7)); 116.5 (*d*, C(6)); 118.3 (*d*, C(4)); 124.0 (*d*, C(5)); 124.1 (*s*, C(3)); 126.2 (*d*, C(2)); 133.1 (*s*, C(8)); 134.1 (*s*, C(9)); 168.8 (*s*, C(10)); 169.9 (*s*, C(12)). Anal. calc. for $C_{12}H_{11}NO_3$: C 66.36, H 5.07, N 6.45; found: C 66.52, H 5.07, N 6.88.

*10*H-*Indolo[3,2*-b*]quinoline-11-carboxylic Acid* (**8**). Powdered **3** (3.62 g, 15 mmol) was added to a well cooled, N_2-purged soln. of isatin (**2**; 1*H*-indole-2,3-dione; 2.17 g, 15 mmol) in 100 ml 6N KOH. The mixture was refluxed carefully under dielectric heating conditions for 16 min keeping the power level at 70%. H_2O (60 ml) was then added. The mixture was heated to 70°, while drawing air through the soln. for 20 min. The soln. was filtered hot through a *Celite* pad, the bed rinsed with hot H_2O, an equal volume of EtOH was added and then the soln. was brought to pH 2 with 6N HCl and chilled. The resulting very fine yellow precipitate was collected, washed with H_2O, followed by a little EtOH and dried under vacuum to afford 2.7 g (70%) of **8**. Very fine yellow crystals. M.p. 322°. IR (KBr): 3345 (NH str.), 3040–2780 (broad, peak at 2927, CO_2H), 2369, 1742 (CO str.), 1675, 1574, 1437, 1251. ^{1}H-NMR: 7.34 (*t*, *J* = 7.6, H–C(7)); 7.70 (*t*, *J* = 8, H–C(2)); 7.70 (*d*, *J* = 8, H–C(6)); 7.66 (*t*, *J* = 8, H–C(8)); 7.76 (*t*, *J* = 8, H–C(3)); 8.29 (*m*, H–C(9)); 8.37 (*d*, *J* = 8, H–C(4)); 9.08 (*m*, H–C(1)); 11.5 (br. *s*, NH). ^{13}C-NMR: 111.8 (*s*, C(14)); 113.0 (*d*, C(9)); 120.9 (*s*, C(15)); 120.9 (*d*, C(8)); 121.8 (*d*, C(6)); 124.1 (*s*, C(17)); 125.4 (*d*, C(7)); 126.5 (*d*, C(1)); 127.3 (*d*, C(2)); 129.8 (*d*, C(3)); 131.0 (*d*, C(4)); 132.7 (*s*, C(11)); 142.4 (*s*, C(13)); 143.6 (*s*, C(18)); 145.0 (*s*, C(16)); 168.2 (*s*, C(12)). Anal. calc. for $C_{16}H_{10}N_2O_2$: C 73.28, H 3.82, N 10.69; found: C 73.39, H 3.45, N 10.91.

*10*H-*Indolo[3,2*-b*]quinoline* (**9**). 2.6 g (10 mmol) of **8** in 20 ml diphenyl ether heated in an oil bath for 4 h at 250°. The mixture was cooled, then 30 ml petroleum ether (PE) was added and filtered. The residue was washed with PE, dried, and on crystallization from MeOH 1.64 g (76%) of **9** were obtained. Light yellow crystals. M.p. 248–250°. IR (KBr): 3414 (NH str.), 1610, 1564, 1484, 1460, 1394, 1332, 1219. ^{1}H-NMR: 7.28 (*t*, *J* = 7, H–C(7)); 7.56 (*t*, *J* = 7, H–C(8)); 7.58 (*d*, *J* = 7, H–C(9)); 7.64 (*t*, *J* = 8, H–C(2)); 7.66 (*t*, *J* = 8, H–C(3)); 8.11 (*d*, *J* = 7, H–C(6)); 8.20 (*d*, *J* = 8, H–C(1)); 8.29 (*s*, H–C(11)); 8.37 (*d*, *J* = 8, H–C(4)); 11.4 (br. *s*, NH). ^{13}C-NMR: 112.0 (*d*, C(9)); 113.5 (*d*, C(8)); 119.8 (*d*, C(6)); 121.5 (*s*, C(16)); 121.8 (*d*, C(7)); 125.3 (*d*, C(2)); 126.5 (*d*, C(1)); 127.2 (*s*, C(14)); 128.0 (*d*, C(4)); 129.2 (*d*, C(3)); 130.2 (*d*, C(11)); 133.0 (*s*, C(13)); 143.9 (*s*, C(17)); 144.5 (*s*, C(12)); 146.2 (*s*, C(15)). EI-MS: 218 (100, M^+), 190 ($[M - C_2H_4]^+$), 164, 115, 109, 89. Anal. calc. for $C_{15}H_{10}N_2$: C 82.57, H 4.59, N 12.84; found: C 82.82, H 4.45, N 13.10.

*Synthesis of 5-Methyl-10*H-*indolo[3,2*-b*]quinolin-5-ium Iodide* (**10**). A suspension of **9** (2.2 g, 10 mmol) and MeI (7 ml, 80 mmol) was heated for 4 min under dielectric conditions (with tetrahydrothiophene 1,1-dioxide) at 70% power level in a carefully sealed round bottom flask. The excess MeI was removed *in vacuo* and the brown precipitate was collected, washed with Et_2O, and dried, yielding 3.3 g (88%) of **10**. Crystallization from H_2O containing a few drops of MeOH gave bright yellow crystals. M.p. 280°. IR (KBr): 3436 (NH str.), 2369, 1607, 1498, 1462, 1347, 1250. ^{1}H-NMR: 5.00 (*s*, MeN); 7.48 (*t*, *J* = 8, H–C(7)); 7.79 (*d*, *J* = 8, H–C(9)); 7.89 (*t*, *J* = 8, H–C(8)); 7.91 (*t*, *J* = 9, H–C(3)); 8.13 (*t*, *J* = 9, H–C(2)); 8.55 (*d*, *J* = 8, H–C(1)); 8.73 (*d*, *J* = 9, H–C(4)); 8.75 (*d*, *J* = 8, H–C(6)); 9.25 (*s*, H–C(11)); 12.81 (br. *s*, NH). ^{13}C-NMR: 41.1 (*q*, C(18)); 114.0 (*d*, C(9)); 114.6 (*s*, C(15)); 118.7 (*d*, C(8)); 122.2 (*d*, C(7)); 125.6 (*d*, C(6)); 127.0 (*s*, C(14)); 127.1 (*d*, C(3)); 127.9 (*d*, C(1)); 130.7 (*d*, C(4)); 133.3 (*d*, C(2)); 134.1 (*s*, C(16)); 134.8 (*d*, C(11)); 136.2 (*s*, C(13)); 138.9 (*s*, C(17)); 146.5 (*s*, C(12)). Anal. calc. for $C_{15}H_{10}N_2$: C 82.57, H 4.59, N 12.84; found: C 82.82, H 4.45, N 13.10.

Synthesis of Cryptolepine (=*5-Methyl-5*H-*indolo[3,2*-b*]quinoline*; **1**). Hydroiodide **10** (1.8 g, 5 mmol) was shaken with a 5% soln. of Na_2CO_3 (100 ml) and then extracted with $CHCl_3$ (2 × 200 ml). Na_2CO_3 was added to the soln., the mixture was concentrated and the adsorbed product was loaded onto a short column of basic alumina. Elution with $CHCl_3$ to elute the quindoline impurity followed by further elution with 2% MeOH in $CHCl_3$ gave 0.70 g (61%) of **1** as a purple solid. M.p. 172–174°. IR (KBr): 2373, 1628, 1493, 1457, 1357, 1302, 1252, 1128. ^{1}H-NMR: 4.33 (*s*, MeN); 6.77 (*t*, *J* = 8, H–C(7)); 7.30 (*t*, *J* = 8, H–C(2)); 7.40 (*d*, *J* = 8, H–C(9)); 7.46 (*t*, *J* = 8, H–C(8)); 7.70 (*t*, *J* = 9, H–C(3)); 7.84 (*d*, *J* = 9, H–C(4)); 7.94 (*d*, *J* = 8, H–C(6)); 8.00 (*d*, *J* = 9, H–C(1)); 8.28 (br. *s*, H–C(11)). ^{13}C-NMR: 37.4 (*q*, C(18)); 113.2 (*s*, C(16)); 115.3 (*d*, C(4)); 117.2 (*d*, C(9)); 117.4 (*d*, C(2)); 123.8 (*d*, C(11)); 123.84 (*d*, C(3)); 124.6 (*s*, C(13)); 125.0 (*d*, C(1)); 129.0 (*d*, C(7)); 129.02 (*d*, C(6)); 130.6 (*d*, C(8)); 132.9 (*s*, C(14)); 138.3 (*s*, C(12)); 142.8 (*s*, C(15)); 158.3 (*s*, C(17)). EI-MS: 232 (100, M^+), 217 ($[M - Me]^+$), 204 ($[M - C_2H_4]^+$), 190, 146, 116, 109, 88.

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