containing a catalytic amount (0.1 g) of sodium ethoxide. Evaporation of the reaction mixture and chromatography of the residue on silica using ether as the eluent gave 0.23 g (38%) of **6** as colorless crystals, mp 121–122 °C after recrystallization from cyclohexane.

Anal. Calcd for $C_{11}H_{14}N_2O_2$: C, 64.08; H, 6.80; N, 13.59. Found: C, 64.17; H, 6.93; N, 13.65.

6,7-Trimethylene-5-deazapterin (2-Amino-6,7-trimethylene-4(3*H*)-pyrido[2,3-*d*]pyrimidinone) (2). Method A. A mixture of 0.5 g (0.0024 mol) of the *o*-amino ester **6** and 0.5 g (0.0029 mol) of benzoyl isothiocyanate in 25 mL of acetonitrile was heated under reflux for 2 h. Filtration then gave 0.51 g (56%) of the benzoylthiourea derivative **7**. This compound (0.36 g, 0.001 mol) was added in one portion to a slurry of 0.05 g of 60% sodium hydride/mineral oil in 10 mL of DMF, and the mixture was stirred at room temperature for 1 h. Methyl iodide (0.2 mL) was then added, and the mixture was stirred again for 1 h. The solvent was removed by evaporation under reduced pressure, and the residual S-methylated thiourea derivative **8** was suspended in 20 mL of saturated ethanolic ammonia and heated in a sealed glass tube at 120 °C for 2 h. The contents of the tube were then cooled and filtered to give 0.05 g (15% based upon **6**) of **2**: mp >250 °C; IR (Nujol) 3400–2300, 1710, 1675, 1600 cm^{-1}; NMR (TFA-d_1 + Me_4Si) δ 2.3–2.75 (m, 2 H), 3.3 and 3.5 (2t, 4 H, J = 6 Hz), 8.9 (s, 1 H).

Method B. A mixture of 2.78 g (0.018 mol) of 2-[(dimethylamino)methylene]cyclopentanone[7] and 1.89 g (0.015 mol) of 2,4-diamino-6(1*H*)-pyrimidinone in a mixture of 30 mL of water and 45 mL of acetic acid containing 1 drop of piperidine was heated under reflux for 2 h. The yellow precipitate which had separated was collected by filtration, washed with water, methanol, acetone, and ether, and dried at 100 °C (0.1 mm) for 4 h; yield 2.1 g (69%). This compound was identical in every respect with **2** prepared by method A and also identical with the condensation product of 2,4-diamino-6(1*H*)-pyrimidinone with 2-(aminomethylene)cyclopentanone, prepared as described by Stark and Breitmaier.[2]

Registry No. 1, 49739-08-4; 2, 91159-00-1; 3, 7148-07-4; 4, 91159-01-2; 5, 91159-02-3; 6, 68708-34-9; 7, 91159-03-4; 8, 91159-04-5; ClCH=C(CN)CO_2Et, 78872-04-5; PhCONCS, 532-55-8; 2,4-diamino-6(1*H*)-pyrimidinone, 56-06-4; 2-[(dimethylamino)methylene]cyclopentanone, 62041-55-8; 2-(aminomethylene)cyclopentanone, 42997-61-5.

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Synthesis and Carbon-13 NMR of an Unusual Tricyclic Barbituric Acid Derivative†

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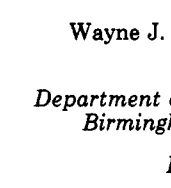
Bicyclic imides of the type illustrated in general structure **1** are of interest because they belong to a class of

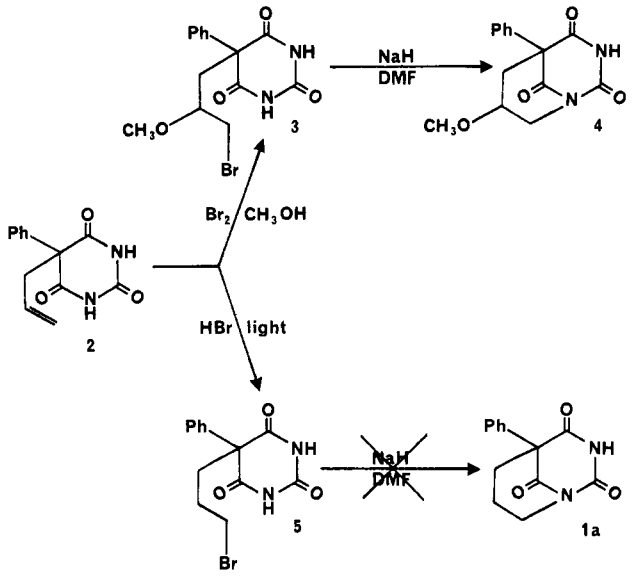


	X	
1a	CONH	barbituric acid
1b	NH	hydantoin
1c	O	2,4-oxazolidinedione

1

anti-Bredt bridgehead nitrogen amides.[1] These compounds were first proposed as potential stereoselective anticonvulsants by E. E. Smissman, whose synthetic efforts[2-11] revealed that structures with n = 1 were very resistant to formation. For example, despite numerous attempts by Smissman to prepare **1a** (n = 1) or analogues,[6-8] the only reported success[6] was for the preparation of methoxy-substituted bicyclic barbiturate **4** using the procedure illustrated in Scheme I. Surprisingly, similar synthetic approaches involving intermediates without the methoxy group, such as **5** in Scheme I, did not yield **1a**. Our interest[2] in these systems prompted us to repeat Smissman's synthesis of **4**. Here we report that the structural assignments for **4** and the immediate synthetic precursor **3** are incorrect, and we provide NMR evidence which shows that the correct structures are novel cyclized barbituric acid derivatives containing the amide acetal functionality.

Scheme I



Results and Discussion

5-Allyl-5-phenylbarbituric acid (**2**, Scheme I) was prepared from 5-phenylbarbituric acid according to the procedure of Vorm and Siegfried.[12] The ^1H and ^{13}C NMR assignments for **2** are presented in Table I and are consistent with the assigned structure. The reaction of **2** with Br_2 and CH_3OH according to the procedure of Smissman[6] produced a crystalline product that was identical in every respect (mp, IR, and ^1H NMR in CF_3CO_2H-d_1) to that reported as **3**. Furthermore, the electron-impact mass spectrum gave the anticipated molecular ion at m/e 356 and confirmed the presence of one bromine. However, the ^1H NMR spectrum of **3** run in Me_2SO-d_6 (shown as compound **6** in Table I) displayed two types of N–H resonances

† Dedicated to the memory of Prof. E. E. Smissman.

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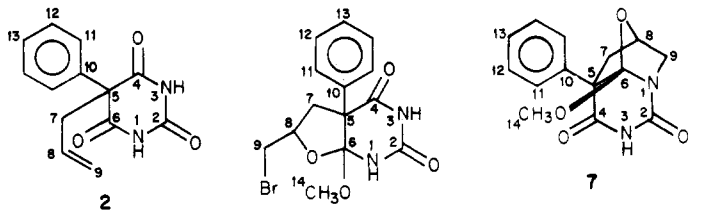
Table I. Summary of NMR Spectral Data for Compounds 2, 6, and 7[a]



2 6 7

	^{1}H NMR, δ			^{13}C NMR					
				2		6		7[b]	
atom no.[c]	2	6	7	δ	J_{CH}[d]	δ	J_{CH}	δ	J_{CH}
1	11.70 (s)[e]	9.29 (s)							
2				149.6		151.1		157.1	B[f]
3	11.70 (s)	10.73 (s)	10.83 (s)						
4				171.1		172.3	B	173.2	B
5				58.9		57.8		57.8	
6				171.1		108.9	B	114.1	B
7	3.03 (d)	2.73 (dd)	2.36 (dd)	*g*		36.4	137.4	*h*	
		2.95 (dd)	2.96 (dd)						
8	5.14 (d)	4.22 (m)	4.91 (m)	131.9	153.1	73.2	154.9	74.5	166.7
9	5.58–5.74 (m)	3.40–3.49 (m)	3.63 (m)	120.5	156.4	35.1	153.1	60.3	148.5
10				137.8	B	135.9	B	136.4	B
11				129.0[i]	161.9	128.4[i]	159.2	129.6[i]	159.6
12	{7.29–7.44 (m)}	{7.27–7.44 (m)}	{7.23–7.33 (m)}	126.2[i]	159.1	127.3[i]	161.0	126.8[i]	160.3
13				128.3	156.4	127.7	161.1	126.7	160.3
14		3.33 (s)	3.30 (s)			49.2	144.3	52.1	145.7

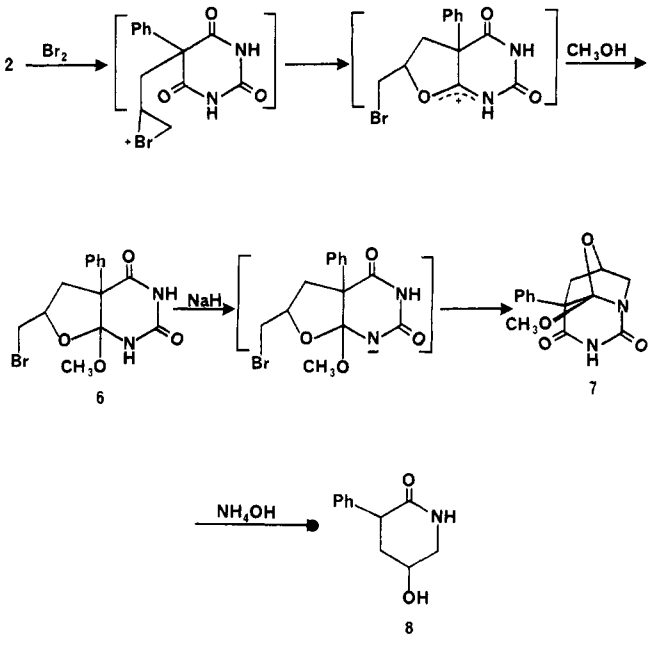
[a] All spectra were recorded in Me_2SO-d_6 at 30 °C unless otherwise noted. [b] Recorded at 50 °C. [c] For purposes of comparison, atom numbering is not in agreement with nomenclature. [d] Coupling constants are reported in hertz. [e] Peak multiplicities are indicated by s = singlet, d = doublet, dd = doublet of doublets, and m = multiplet. [f] B = broadening due to long-range coupling. [g] Masked by solvent peak. [h] Masked by solvent peak but observed in CF_3CO_2H-d_1 (30 °C) at 38.7 ppm (J_{CH} = 136.0 Hz). [i] Assignments for C-11 and C-12 may be reversed.

(not seen in CF_3CO_2H-d_1 due to exchange). That similar to the resonance for barbiturate imide hydrogens[13] (as seen in **2**, Table I) occurred at 10.73 ppm (integrating for one H), but an unexpected N–H resonance also occurred at 9.29 ppm (integrating for one H), typical for an amide, suggesting that this product is not a barbiturate. The remaining ^{1}H NMR data was otherwise consistent with structure **3**.

The ^{13}C NMR spectrum (labeled as **6** in Table I) confirmed that structure **3** is not correct in that it contained resonances for only two carbonyl carbons at 172.3 and 151.1 ppm. While it has been observed that the resonances for the C-4 and C-6 carbonyls near 170 ppm are degenerate in symmetrical barbiturates (as seen in **2**, Table I), chiral substitution at C-5 (as in **3**) results in a chemical shift difference between C-4 and C-6 of about 0.3 ppm.[14] Careful observation of the 172.3-ppm resonance in **3** revealed a line width identical with that for the 151.1-ppm resonance. Furthermore, integration of the decoupled carbon spectrum, obtained under conditions to yield quantitative data (see Experimental Section), provided integrated area ratios of 1:1 for the two carbonyl resonances, confirming the presence of only one carbonyl carbon at 172.3 ppm. An additional resonance for a nonprotonated carbon was observed in the carbon spectrum of **3** at 108.9 ppm, suggesting that the "missing" carbonyl carbon was transformed to a functionality with the above chemical shift. The remainder of the ^{13}C NMR data was otherwise consistent with structure **3**.

The key to the structure assignment appeared to reside in identification of the nonprotonated carbon resonance

Scheme II



at 108.9 ppm. In this region one typically finds some alkenes, aromatic carbons adjacent to O- or N-substituted positions, certain nitriles,[14] ortho esters (triethyl orthoformate exhibits a carbon resonance at 112.5 ppm in $CDCl_3$),[15] and amide acetals (the dimethyl acetal of dimethylformamide exhibits a carbon resonance at 112.8 ppm in Me_2SO-d_6).[16] From a mechanistic point of view,

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(15) Johnson, L. F.; Jankowski, W. C. "Carbon-13 NMR Spectra"; Wiley: New York, 1972; p 279.
(16) Recorded by us at 25 °C for a 20% solution (dimethylformamide dimethyl acetal was purchased from Aldrich).

the most reasonable alternative among those above appears to be an amide acetal functionality. As shown in Scheme II, such a functionality might readily be formed from one of the carbonyls in 2 upon treatment with Br_2/CH_3OH to yield product 6, 6-(bromomethyl)-7a-methoxy-4a-phenyltetrahydrofuro[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione.

The mechanism proposed in Scheme II for the formation of 6 is consistent with a common method for preparing the amide acetal functionality.[17] N,N-Dialkylamides react with good alkylating agents such as dimethyl sulfate to provide a (dialkylamino)alkoxycarbenium salt, which is subsequently reacted with an alkoxide to form the amide acetal. Numerous amide acetals have been prepared by this as well as other procedures, including the 4,4-diethyl acetals of 1,3-diarylbarbituric acids.[18] While amide acetals are typically labile under hydrolytic conditions, some examples display unusual stability, even under acidic conditions.[17]

We next treated 6 with NaH in DMF according to the method reported by Smissman[6] for converting 3 to 4 and again obtained a material that was identical in every respect (mp, IR, ^1H NMR in CF_3CO_2H-d_1, mass spectrum) with the product assigned structure 4. The ^1H NMR spectrum in Me_2SO-d_6 (labeled as 7 in Table I) was also consistent with 4 in that it contained an imide N–H resonance at 10.83 ppm (integrating for one H) and no amide N–H resonance. However, the ^{13}C NMR spectrum (shown as 7 in Table I), obtained under conditions similar to that for 6, confirmed that 4 is not correct in that only two carbonyl carbons were present with resonances at 173.2 and 157.1 ppm. Also, as similarly observed with 6, a resonance for a nonprotonated carbon occurred at 114.1 ppm, which is inconsistent with structure 4 and suggests that the amide acetal functionality is also present in this product. The remaining ^{13}C NMR data were otherwise consistent with 4.

The above results can be explained by an intramolecular N-alkylation of 6 at the amide nitrogen to provide product 7 (Scheme II), 1,6-methano-7a-methoxy-4a-phenyltetrahydrofuro[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-dione. While imide hydrogens are more acidic than amide hydrogens, the preference for amide N-alkylation over imide N-alkylation is consistent with the observation that anti-Bredt bridgehead nitrogen amides (of which 7 is an example) are more easily formed if an atom with a lone electron pair remains in resonance with the carbonyl group.[1]

Strong support for the amide acetal functionality in 7 was also obtained via ^{13}C selective decoupling experiments. As shown in Figure 1, spectrum a, the nonprotonated carbon resonance at 114.1 ppm is significantly broadened by long-range coupling. This broad singlet collapsed to a doublet when the OCH_3 protons were irradiated (Figure 1, spectrum c), establishing a long-range interaction of these spins that is consistent with the $^3J_{CH}$ coupling provided by structure 7. The residual coupling under these conditions established another long-range interaction which is likely the $^3J_{CH}$ coupling between the 114.1-ppm resonance and the methine proton. This was supported by irradiation of the methine proton, resulting in a narrowing of the 114.2-ppm resonance, although resolution was insufficient to observe the quartet expected from OCH_3 coupling.

Finally, a key piece of evidence used by Smissman to support structure 4 was that hydrolysis under basic conditions (NH_4OH) provided 5-hydroxy-3-phenyl-2-piperidinone (8), shown in Scheme II, although cleavage of the methyl ether under these conditions was considered to be anomalous. In view of the report that acyclic amide acetals undergo rapid hydrolysis under basic conditions to exclusively give C–O bond cleavage,[19] it is much more likely that 7 would undergo basic hydrolysis to yield 8.

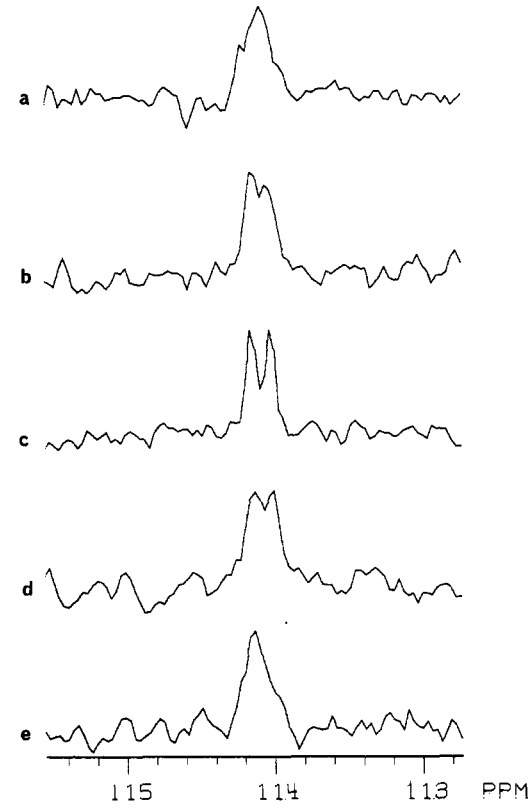

Figure 1. Effect of selective proton decoupling on the C-6 carbon resonance of 7. Spectra a–e were obtained by setting the decoupler frequency at values which differed from the C-14 methyl proton resonance as follows: a = –300 Hz, b = –90 Hz, c = 0 Hz (residual $^3J_{CH}$ = 9.9 Hz), d = 90 Hz, and e = 178 Hz.

In summary, while Smissman's reported syntheses of 3 and 4 are in agreement with the spectral data he obtained, our ^1H and ^{13}C NMR investigations have shown that the structural assignments are incorrect. These spectral results along with mechanistic and degradative evidence have led to the proposal of compounds 6 and 7 as the correct structures, which represent the first examples of cyclized barbituric acid derivatives containing the amide acetal functionality.

Experimental Section

^1H (300.1 MHz) and ^{13}C (75.5 MHz) NMR spectra were obtained on a Nicolet 300 wide-bore system equipped with a 1280 Nicolet computer and 293C pulse programmer. ^1H NMR spectra were run using a standard one-pulse experiment with 90° pulses, 32K points, and sweep widths of 2000 Hz. Repetition rates were about 4 s. Selective homonuclear decoupling was accomplished by using continuous wave irradiation at 30 dB during aquisition. ^{13}C NMR spectra were obtained by gating the ^1H decoupler to provide either decoupled spectra with nuclear Overhauser enhancement (NOE), decoupled spectra without NOE, or coupled spectra with NOE; 50° pulses were used with 16K points and 15 000-Hz sweep widths. Repetition rates were 5 s for nonquantitative spectra. Quantitative ^{13}C NMR spectra were obtained by using 15-s delays with the decoupler gated off. Doubling the length of the experiment had no effect on the integrals. Solutions contained 50 to 100 mg of sample in 3 mL of Me_2SO-d_6 (Aldrich)

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or CF_3CO_2H-d_1 (Merck), and 1K to 2K transients were collected. Chemical shifts from tetramethylsilane were referenced internally to Me_2SO-d_6 (39.5 ppm) and CF_3CO_2H-d_1 (116.6 ppm). Spectra were run at either 30 °C or 50 °C as indicated.

Registry No. 2, 115-43-5; 3, 25860-24-6; 4, 25860-23-5; 6, 91159-05-6; 7, 91159-06-7.

Methyleneindolines, Indolenines, and Indoleniniums. 19.[1] A New Entry into the Hexahydropyrrolidino[2,3-*d*]carbazole System

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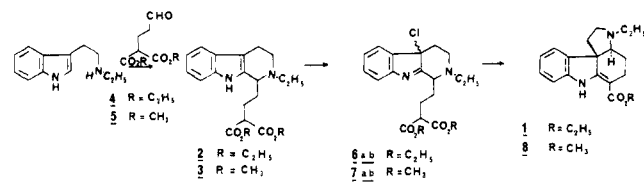
The pyrrolidino[2,3-*d*]carbazole system is part of many important indole alkaloids of the Aspidosperma and Strychnos families inter alia, and it has been the object of several recent synthetic efforts[3]. We report herein a novel approach to this system, exemplified by a three-step synthesis of 1.

Tetrahydro-β-carbolines **2** and **3** are obtained in high yield (Scheme I) through Pictet–Spengler condensation of N_b-ethyltryptamine[4] with aldehydes **4** and **5**, the Michael addition products of acrolein with ethyl and methyl malonate, respectively.[5] Treatment of **2** with *t*-BuOCl[6] gives a quantitative yield of the chloroindolenines **6a,b** in a 1:1 ratio. When treated with NaH in THF, **6a,b** is transformed into a mixture containing the rearranged **1** (48%) and unaffected **6a** (18%). Similar treatment of **3** affords two chloroindolenines **7a,b**, which are separated before being subjected to the rearrangement conditions. While the less polar isomer **7a** is recovered unchanged, **7b** is cleanly transformed by NaH into the α-methyleneindoline **8** (75%).

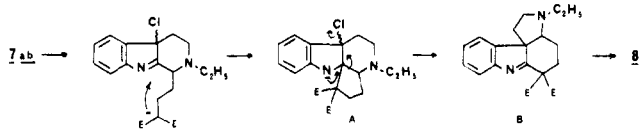
Compounds **1** and **8** display the typical UV spectra of β-anilinoacrylate esters (λ_{max}^{MeOH} 226, 298, 328 nm) and give intense blue TLC spots upon spraying with Ce(IV). Their mass spectra are dominated by the retro-Diels–Alder fragmentation of ring C, accompanied by the rupture of the tryptamine chain α and β to N_b.[7] Final structural proof for **1** and **8** was obtained by an independent synthesis according to a literature procedure.[3d]

Although several pathways may explain the transformation **6** → **1**, we favor a mechanism in which the initially formed malonate anion intramolecularly attacks C-2 of the

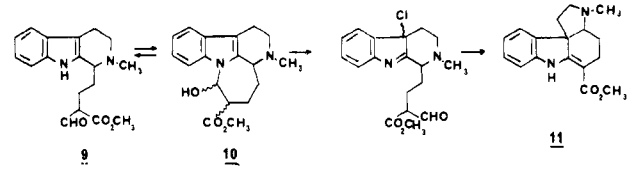
Scheme I



Scheme II



Scheme III



indole (Scheme II). C-ring contraction with chloride expulsion is followed by Krapcho-like decarbalkoxylation of intermediate B, which occurs under extremely mild conditions because of the triactivated nature of the ester-bearing carbon.[8] Repetition of the experiment with enantiomerically enriched **3** ($[\alpha]_D$ + 18° (*c* 0.8, EtOH)) leads to optically active **8** ($[\alpha]_D$ + 72° (*c* 0.5, EtOH)); this rules out mechanisms in which achiral intermediates are produced. The unreactivity of chloroindolenines **6a** and **7a** is probably due to the wrong relative stereochemistry of the side chain and the chlorine atom. Fortunately, the unreacted chloroindolenine can be reduced back to the parent indoles **2** and **3** with thiophenol.[9]

Additions of carbon nucleophiles at C-2 of chloroindolenines are rare, and one of the few examples reported is Kuehne's addition of diethyl thalliomalonate to the chloroindolenine of tetrahydrocarbazole.[10] Use of the thallium counterion was found to be essential; sodiomalonate led only to reduction to tetrahydrocarbazole, probably by attack at chlorine rather than at carbon. In our case, the length of the chain between the indole nucleus and the nucleophile does not favor attack at chlorine.

Brief exploration of the scope of the rearrangement led us to examine the behavior of the more reactive aldehydo ester **9** (which exists mainly in the carbinolamine form **10**). Surprisingly, its treatment with *t*-BuOCl (Scheme III) leads directly to α-methyleneindoline **11**. Rearrangement of the (undetected) intermediate chloroindolenine is probably catalyzed by triethylamine.

The availability of synthons such as **9** by direct formylation[11] of esters renders this approach attractive, and application of these rearrangements to the synthesis of pentacyclic natural alkaloids is currently being explored. The production of diversely substituted indolo[2,3-*d*]-indoles opens the way to the synthesis of the corresponding vindoline adducts[3d] and benzofurans.[12]

(1) Part 18 in this series: Hugel, G; Lévy, J. *J. Org. Chem.*, in press.
(2) Taken in part from J. Vercauteren's Thesis, Reims, May 24, 1983 (no. 2, 1983).
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