stereochemistry and indicating the cis configuration (*cis*-stilbene, τ 3.45; *trans*-stilbene, τ 2.90).[12]

The reaction between **4** and **5** was also carried out with lithium ethoxide in dimethylformamide and ethanol (8:1) under more concentrated conditions than used. before. This experiment led to *trans,trans*-**2** in 5.5% yield, although other products were not investigated in this case.

The finding that the reaction between **4** and **5** leads to both *cis,cis*-**2** and *trans,trans*-**2** partly explains the different results reported by Bergmann, *et al.*,[5,10] and by us.[8] However, we have not been able to confirm Bergmann's isolation of *cis,trans*-**2** from this reaction, despite the fact that quite small quantities would have been detected by the typical nmr spectrum.

Experimental Section

Melting points were determined on a Kofler micro hot stage apparatus and are uncorrected. Uv spectra were measured on a Unicam SP 800 and ir spectra on a Unicam SP 200 spectrophotometer. Nmr spectra were determined on a Varian T-60 spectrometer, tetramethylsilane being used as an internal standard. Mass spectra were obtained on an AEI MS-9 spectrometer operating at 70 eV.

Reaction between 2,2′-Bis(triphenylphosphoniomethyl)biphenyl Dibromide (4) and 2,2′-Biphenyldicarboxaldehyde (5). A. With Lithium Methoxide in Methanol.—A solution of **4**[6] (21.0 g, 24.3 mmol) and **5**[7] (5.1 g, 24.3 mmol) in dry methanol (500 ml) was added through one dropping funnel at the same rate as a solution of lithium (700 mg, 100 mmol) in dry methanol (500 ml) was added through a second funnel to dry methanol (4 l.) stirred under nitrogen in a 10-l. three-neck flask, over the course of 12.5 hr. The resulting pale yellow solution was stirred at room temperature under nitrogen for a further 12 hr, and the solvent was then removed under reduced pressure (<30°). Ether (1.5 l.) was added, and the mixture was washed well with dilute hydrochloric acid and then with water. The dried solution was evaporated, and the residue was chromatographed on silica gel (500 g).

Pentane eluted 2,2′-dimethylbiphenyl (650 mg, 15%), the ir spectrum of which was identical with the published one.[13] Pentane–benzene (95:5) then eluted **2** (~500 mg), the nmr spectrum of which showed that it consisted of the cis,cis and trans,trans isomers in a ratio of ~1:4. Further elution with pentane–benzene (95:5 to 90:10) led to **6** (99 mg, 1.5% based on **4**), as colorless crystals from petroleum ether (bp 40–60°): mp 181–182°; λ_{max} (cyclohexane) 290 nm (ϵ 22,900); ir (KBr), only weak band (at 955 cm^{-1}) in 950–1000-cm^{-1} region; nmr (CCl_4) τ 2.4–3.2 (m, 24 H, benzenoid), 3.88 (s, 4 H, olefinic), 7.88 (s, 6 H, methyl); mass spectrum *m/e* 538.267 (calcd 538.266).

Anal. Calcd for $C_{42}H_{34}$: C, 93.63; H, 6.37. Found: C, 93.59; H, 6.45.

Later fractions contained a mixture of stereoisomers of **6** (as determined by the nmr, ir, and mass spectra), but these were not investigated further.

Fractional crystallization of the isomers of **2** from ethyl acetate led to the pure cis,cis isomer (95 mg, 1.1%) and trans,trans isomer (365 mg, 4.2%). *cis,cis*-**2** formed colorless crystals: mp 296–297°; λ_{max} (cyclohexane) 240 nm (ϵ 31,900); ir (KBr) only weak bands (960, 980 cm^{-1}) in 950–1000-cm^{-1} region; nmr (CCl_4) τ 2.35–3.15 (m, 16 H, benzenoid), 4.18 (s, 4 H, olefinic); mass spectrum *m/e* 356.155 (calcd 356.156). The melting point was undepressed on admixture with a sample (mp 295–296°) obtained from *cis*-**1**, and the uv, ir, and nmr spectra were essentially identical. *trans,trans*-**2** formed colorless crystals: mp 302–303° (depressed on admixture with *cis,cis*-**2**); λ_{max} (cyclohexane) 226 nm (ϵ 32,200), 248 sh (24,100), 266 (34,100); ir (KBr) 955 cm^{-1} (s); nmr (CCl_4) τ 2.6–3.0 (m, 16 H, benzenoid), 3.82 (s, 4 H, olefinic); mass spectrum *m/e* 356.155 (calcd 356.156).

Anal. Calcd for $C_{28}H_{20}$: C, 94.34; H, 5.65. Found: C, 94.14; H, 5.55.

(12) "High Resolution NMR Spectra Catalog," Varian Associates, Vol. 1, 1962, Spectra 305, 306.

(13) G. Kortüm and H. Maier, *Z. Phys. Chem.*, **7**, 207 (1956).

B. With Lithium Ethoxide in Dimethylformamide and Ethanol.—A solution of lithium (500 mg, 72 mmol) in dry ethanol (50 ml) was added during 4.5 hr to a stirred solution of **4** (8.65 g, 10 mmol) and **5** (2.1 g, 10 mmol) in dry dimethylformamide (400 ml), at room temperature under nitrogen. The dark brown solution was stirred for 16 hr, and was then poured into ice containing concentrated hydrochloric acid. The organic material was extracted with ether, and the ether extract was washed with water, dried, and evaporated. The residue was chromatographed on a column of silica gel (75 × 3.5 cm). Elution with petroleum ether–benzene (80:20) and crystallization from ethyl acetate gave *trans,trans*-**2** (197 mg, 5.5%), mp 303–304°. The melting point was undepressed on admixture with the previously obtained sample, and the uv, ir, and nmr spectra were identical.

Registry No.—*cis,cis*-**2**, 37445-16-2; *trans,trans*-**2**, 37445-17-3; **4**, 37439-54-6; **5**, 1210-05-5; **6**, 37445-18-4.

Acknowledgment.—We are grateful to Professor G. Wittig for sending us a manuscript of the paper by Wittig and Skipka (ref 5).

Preparation of 11-Substituted 5,6-Dihydro-11*H*-6-oxodibenzo[*b*,*e*]azepines (Morphanthridines) and Their *N*-Dimethylaminoethyl Derivatives[1]

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Enhanced stability of extensively delocalized carbanions, arising from the dispersion interaction[3] with dipolar aprotic solvents, favors the formation of Jackson–Meisenheimer complexes[4] (**1**) by the conjugate addition to 9-nitroanthracene of various nucleophiles.[5] Quenching and acidification of the reaction mixtures permit isolation of the adducts (**2**); the stereochemistry, spectral characteristics, properties, and some chemical transformations of these adducts have been reported.[5] Addition of benzyl halide to solutions of the Jackson–Meisenheimer complexes prior to work-up results in the formation of oximes (**3**)[5] and benzaldehyde, the products of the Hass–Bender reaction.[6]

Beckmann rearrangement of these oximes provides 11-substituted 5,6-dihydro-11*H*-6-oxodibenzo[*b*,*e*]azepines (**5**). Many tricyclic compounds of structures similar to **5** with a dialkylaminoalkyl moiety bonded to an atom of the central ring have pharmacological, and in particular, pyschotropic activity.[7] Most notable among these are the tricyclic antidepressants imipramine and amitriptylene; the structure of dibenzepin,[8]

(1) Grateful acknowledgment is made to the U. S. Army Research Office for partial support of this work (Grants DA-ARO(D)-G679 and G857).

(2) NSF Predoctoral Fellow, 1967–1971.

(3) E. Grunwald and E. Price, *J. Amer. Chem. Soc.*, **86**, 4517 (1964).

(4) M. J. Strauss, *Chem. Rev.*, **70**, 677 (1970); J. A. Orvik and J. F. Bunnett, *J. Amer. Chem. Soc.*, **92**, 2417 (1970).

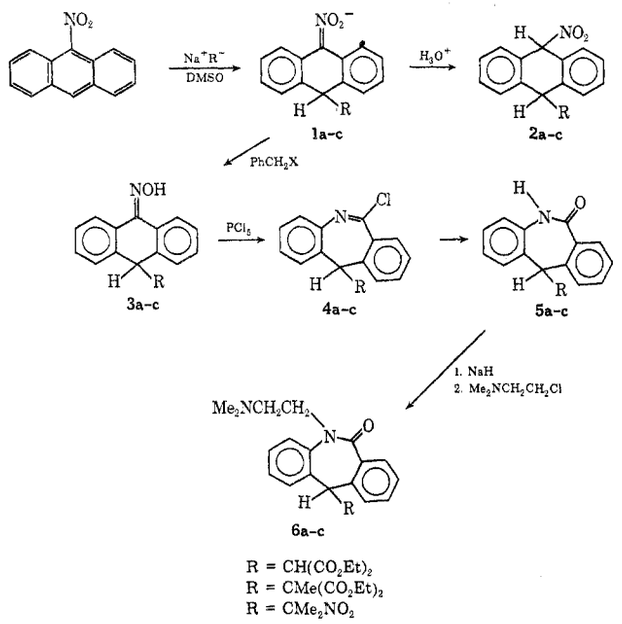
(5) R. H. Williams and H. R. Snyder, *J. Org. Chem.*, **36**, 2327 (1971); R. H. Williams, Ph.D. Thesis, University of Illinois, Urbana, Ill., 1971.

(6) H. B. Hass and M. L. Bender, *J. Amer. Chem. Soc.*, **71**, 1767, 3482 (1949); N. Kornblum and R. A. Brown, *ibid.*, **86**, 2681 (1964).

(7) G. N. Walker, D. Alkalay, A. R. Engle, and R. J. Kempton, *J. Org. Chem.*, **36**, 466 (1971), and references cited therein.

(8) F. Hunziker, H. Lauener, and J. Schmutz, *Arzneim. Forsch.*, **13**, 324 (1963); F. Hunziker, F. Kunzle, and J. Schmutz, *Helv. Chim. Acta*, **49**, 1433 (1966).

another antidepressant, may be obtained by replacing the CHR bridge of **6** with NMe. For this reason, the *N*-(2-dimethylaminoethyl) lactams (**6**) are of interest for pharmacological evaluation.



Beckmann rearrangement of oxime tosylates frequently occurs under very mildly acidic conditions, if not spontaneously;[9] the tosylate of **3c** was recovered unchanged after exposure for 12 hr to boiling glacial acetic acid. In many instances polyphosphoric acid (PPA) has been the reagent of choice for effecting the Beckmann rearrangement;[9] attempts to induce rearrangement of oximes **3a** and **3c** by brief heating with PPA resulted in the formation of complex mixtures of products.[10] Treatment of each of the oximes with a twofold excess of PCl_5 in refluxing CCl_4 and subsequent work-up afforded the corresponding lactam **5**. A sample of the intermediate imino chloride **4c** was isolated and purified for evaluation as a potential precursor of **6c**.

Attempted preparation of **6c** by treatment of **4c** with sodium 2-dimethylaminoethoxide produced a complex mixture of products.[12] A more facile synthesis of **6c** (and of **6a**) entails alkylation of the anion of **5**[13] with 2-dimethylaminoethyl chloride (which, conveniently, may be liberated from the commercially available hydrochloride salt *in situ*).

Difficulties were encountered in the purification of **6a**, as the free base or as the hydrobromide, and of the base **6c**. Walker and Alkalay[14] evidently encountered similar difficulties with an analogous *N*-dimethylaminoethyl compound as the free base, the picrate, or the methiodide. The assignments of structures **6a** and **6c** to the products of alkylation of the anions of **5a** and **5c** are fully supported by the nmr, ir,[15] and mass spectra.

(9) L. G. Donaruma and W. Z. Heldt, *Org. React.*, **11**, 1 (1960).

(10) In the case of PPA treatment of **3a**, products other than **5a** may have arisen *via* ester–amide interchange;[11] the number of products formed by similar treatment of **3c** cannot be explained in this manner.

(11) H. A. Lloyd and E. C. Horning, *J. Amer. Chem. Soc.*, **76**, 3654 (1954).

(12) Treatment of the imino chloride with alkoxide would initially produce a lactim. Lactim–lactam interconversion favors the lactam, and should proceed with facility, particularly in this case, in view of the stability of the 2-dimethylaminoethyl cation, a species which is invoked in a plausible mechanism for the migration.

(13) The anion is ambident; alkylation may occur at either heteroatom.[12]

(14) G. N. Walker and D. Alkalay, *J. Org. Chem.*, **36**, 461 (1971).

Experimental Section[16]

11-Dicarbethoxymethyl-5,6-dihydro-11*H*-6-oxodibenzo[*b*,*e*]-azepine (5a).—A 2-mmol (734 mg) sample of **3a**[5] was added to a suspension of PCl_5 (4 mmol, 830 mg) in 25 ml of CCl_4, and the mixture was heated to the boiling point and maintained under reflux for 2 hr. Volatile materials were removed *in vacuo*, the resulting oil was dissolved in 50 ml of 50:50 CH_2Cl_2–CCl_4, and the solution was washed with water, dried, and concentrated. The oil was dissolved in a small volume of CH_2Cl_2 and the solution was heated to the boiling point. EtOH was added as the CH_2Cl_2 was boiled off; crystallization occurred upon standing for several hours at −15°, yield 625 mg (85%), mp 196–201°. The analytical sample was obtained by recrystallization from benzene: mp 197–201°; ir (KBr) 1650, 1750, 1380, 1250, 1145, and 1730 cm^{-1}; nmr (DMSO-d_6–CCl_4) δ 1.0 (6 H, t, J = 7 Hz, CH_2CH_3), 3.9 (4 H, q, J = 7 Hz, CH_2CH_3), 4.4 [2 H, AB, J = 11.5 Hz, $\Delta\delta$ 0.46 ppm, $CHCH(CO_2Et)_2$], 7–8 (8 H, m, aromatic), and 10.2 ppm (1 H, s, NH).

Anal. Calcd for $C_{21}H_{21}NO_5$: C 68.65; H, 5.76; N, 3.81. Found: C, 68.84; H, 5.83; N, 3.92.

11-(1,1-Dicarbethoxyethyl)-5,6-dihydro-11*H*-6-oxodibenzo-[*b*,*e*]azepine (5b).—The reaction of **3b**[5] with PCl_5 was conducted as described above; after hydrolysis, the product was crystallized from EtOH, yield 49%, mp 151.5–153°. The analytical sample was obtained by recrystallization from EtOH: mp 153–154.5°; ir (KBr) 1650, 1720, 1250, 1385, 1590, and 1450 cm^{-1}; nmr (acetone-d_6) δ 1.05 and 1.10 (6 H, two triplets, J = 7 Hz, CH_2CH_3),[17] 1.35 [3 H, s, $C(CH_3)(CO_2Et)_2$], 4.0 (4 H, q, J = 7 Hz, CH_2CH_3),[17] 5.2 [1 H, s, $CHCMe(CO_2Et)_2$], 7–8.2 (8 H, m, aromatic), and 10.2 ppm (1 H, s, NH).

Anal. Calcd for $C_{22}H_{23}NO_5$: C, 69.27; H, 6.08; N, 3.67. Found: C, 69.08; H, 6.08; N, 3.71.

11-(2-Nitro-2-propyl)-5,6-dihydro-11*H*-6-oxodibenzo[*b*,*e*]azepine (5c).—The reaction of **3c**[5] with PCl_5, hydrolysis, and work-up were conducted as described for **5a**. The product of crystallization from EtOH melted indistinctly and volatilized at temperatures above 200°; two recrystallizations from THF–CCl_4 afforded a material of mp 225–226° (sublimes), yield 62.5%. The analytical sample was obtained by recrystallization from CH_2Cl_2–EtOH: mp 225–226°; ir (KBr) 1655, 1540, 1375, 1395, 1595, 1575, and 1450 cm^{-1}; nmr (acetone-d_6) δ 1.5 (6 H, s, CH_3), 4.7 (1 H, s, $CHCMe_2NO_2$), 7.1–8.1 (8 H, m, aromatic), and 10.0 ppm (1 H, s, NH).

Anal. Calcd for $C_{17}H_{16}N_2O_5$: C, 68.90; H, 5.44; N, 9.45. Found: C, 69.08; H, 5.50; N, 9.52.

(15) The ir band corresponding to lactam C=O stretch is, perhaps, the most indicative probe of structure. It is this band which has the greatest intensity in the ir spectrum of each lactam (**5**) and each substituted lactam (**6a** and **6c**). The ir spectrum of imino chloride **4c** does contain a band at 1645 cm^{-1}; however, the intensity of the absorption is considerably weaker than the 1540-cm^{-1} NO_2 band, in direct contrast to the relative intensities of these bands in spectra of both **5c** and **6c**. Although the intensity of their absorption arising from the C=N stretch is variable, it is weaker than that arising from C=O stretch.

(16) Commercially available reagents were used as supplied. Sodium hydride was obtained by washing the oil-dispersed material with several portions of hexane prior to use. Melting points were determined with a Kofler micro stage apparatus and are uncorrected. The ir spectra were obtained by the use of a Perkin-Elmer 521 spectrophotometer. The peaks listed are the most intense peaks in the spectrum of the particular compound, with the exception of the tosylate of **3c**; for this compound the peaks which have been listed are those which indicate that the rearrangement (with migration of the $ArSO_2$ moiety from O to N) has not occurred.[4] Microanalyses were performed by Mr. J. Nemeth and associates. Routine nmr spectra were recorded on a Varian A-56/60 or A-60A spectrometer.

(17) The two carbethoxy groups are diastereotopic; hence potential magnetic nonequivalence is inherent. The complexity of the signal arising from resonance of the methyl protons is in sharp contradistinction with the simplicity of that arising from resonance of the methylene protons. Examination of the 100- or 220-MHz spectrum confirms the assigned multiplicities.[5] The 100- and 220-MHz spectra were recorded by Mr. Robert L. Thrift and associates on Varian HA-100 and HR-220 spectrometers, respectively. We gratefully acknowledge a grant to the School of Chemical Sciences of the University of Illinois at Urbana—Champaign from the National Science Foundation, which helped make purchase of the HR-220 possible.

11-(2-Nitro-2-propyl)-6-chloro-11*H*-dibenzo[*b*,*e*]azepine (4c).—A 10.0-mmol (2.94 g) sample of **3c**[5] was added to a suspension of 20 mmol (4.16 g) of PCl_5 in CCl_4 (50 ml); the mixture was heated to the boiling point and maintained under reflux for 2 hr. Solvent was removed *in vacuo*, and the residual oil was heated at 65° for 2 hr at 0.5 mm to remove other volatile materials. The oil was dissolved in CH_2Cl_2 (10 ml), the solution was filtered, and the filtrate was diluted with 20 ml of CCl_4, heated to the boiling point, and concentrated to a volume of 20 ml. A 10-ml portion of CCl_4 was added and the solution was reconcentrated to 20 ml; this procedure was repeated one additional time. Crystallization was initiated only with considerable difficulty; after 12 hr at room temperature, 2.1 g of crystals had been deposited. A second crystallization afforded 1.29 g (41%) of analytically pure product: mp 124–129°; ir (KBr) 1540, 760, 1645, 940, 1350, 1375, and 1400 cm^{-1}; nmr (CCl_4) δ 1.45 (6 H, s, CH_3), 4.7 (1 H, s, $CHCMe_2NO_2$), and 7–8 ppm (8 H, m, aromatic).

Anal. Calcd for $C_{17}H_{15}N_2O_2Cl$: C, 64.85; H, 4.81; N, 8.90; Cl, 11.27. Found: C, 64.69; H, 4.78; N, 9.01; Cl, 11.37.

Tosylate of 3c.—A 3.0-mmol (889 mg) sample of **3c**[5] was added to a suspension of 3.34 mmol of oil-free NaH in THF (50 ml), and the mixture was warmed briefly at the boiling point prior to the addition of a solution of 3.0 mmol of *p*-toluenesulfonyl chloride in a small volume of THF. The reaction mixture was maintained at the boiling point for 12 hr prior to solvent removal (*in vacuo*). The residue was dissolved in benzene (50 ml) and the solution was washed with two 100-ml portions of water, dried (Na_2SO_4), and concentrated to a residue. The residue was crystallized from CH_2Cl_2–EtOH: yield 870 mg (64%); mp 184–187° dec; ir (KBr) 1375, 1195, and 1180 ($ArSO_3$–) and 1595 cm^{-1} ($Ar_2C{=}N$–); nmr (acetone-d_6) δ 1.2 [6 H, s, $C(CH_3)_2NO_2$], 2.4 (3 H, s, $ArCH_3$), 4.8 (1 H, s, $CHCMe_2NO_2$), and *ca.* 8.5 ppm (12 H, m, aromatic).

Anal. Calcd for $C_{24}H_{22}N_2O_5S$: C, 63.98; H, 4.92; N, 6.22; S, 7.12. Found: C, 63.78; H, 4.91; N, 6.32; S, 7.03.

5-(2-Dimethylaminoethyl)-11-dicarbethoxymethyl-5,6-dihydro-11*H*-6-oxodibenzo[*b*,*e*]azepine (6a).—A 25.0-mmol (9.19 g) sample of **5a** was added to a suspension of 62.5 mmol of oil-free NaH in 125 ml of DMSO, and the mixture was stirred at room temperature for 1 hr prior to the addition of a suspension of 37.5 mmol (3.6 g) of 2-dimethylaminoethyl chloride hydrochloride in 50 ml of DMSO. The reaction mixture was stirred for 3 hr and poured into 600 ml of water. An orange semisolid separated; the supernatant was rendered strongly alkaline by the addition of 10 ml of 10 *N* NaOH, and was extracted with five 70-ml portions of CCl_4. The semisolid was dissolved in the combined extract and the solution was washed, dried (Na_2SO_4), and concentrated *in vacuo;* the resulting oil was purified by chromatography on silica gel. Elution with 10% EtOH–CH_2Cl_2 afforded (after solvent removal) 1.17 g (12.7%) of recovered **5a**. Elution with 25% EtOH–CH_2Cl_2 provided (after solvent removal *in vacuo*) an oil which could be induced to crystallize from toluene with considerable difficulty, yield 2.94 g, mp 134–154°. Two recrystallizations from PhMe afforded 2.06 g (21.5% conversion) of product: mp 142–146°; ir (KBr) 1640, 1730, 1750, 1370, 1290–1330, 1250, and 1445 cm^{-1}; nmr (acetone-d_6) δ 0.98 and 1.03 (6 H, two triplets, J = 7 Hz, CH_2CH_3),[17] 3.0 [6 H, s, $N(CH_3)_2$], 3.6–5.0 [10 H, m, CH_2CH_3, $CHCH(CO_2Et)_2$, and $CH_2CH_2NMe_2$], and 7.0–8.0 ppm (8 H, m, aromatic).

A 4.7-mmol (2.05 g) sample of **6a** was dissolved in EtOH (15 ml), and hydrobromic acid (4.7 mmol, 0.53 ml) was added. Dilution with an equal volume of Et_2O and cooling at −15° for 12 hr caused the crystallization of the HBr salt: yield 1.90 g (78%); mp 162.5–165°; mass spectrum[18] *m/e* 438.2143 (calcd for $C_{25}H_{31}N_2O_5Br$ − HBr: 438.2154).

Anal. Calcd for $C_{25}H_{31}N_2O_5Br$: C, 57.81; H, 6.02; N, 5.39; Br, 15.38. Found: C, 58.36; H, 6.25; N, 5.43; Br, 15.14.

5-(2-Dimethylaminoethyl)-11-(2-nitro-2-propyl)-5,6-dihydro-11*H*-6-oxodibenzo[*b*,*e*]azepine (6c).—A solution of **5c** (6.0 mmol, 1.78 g) in DMSO (15 ml) was added to a suspension of 6.4 mmol of oil-free NaH in 10 ml of DMSO. After 1.5 hr a suspension of 9.9 mmol of oil-free NaH in a solution of 2-dimethylaminoethyl chloride hydrochloride (9.0 mmol, 1.30 g) in 10 ml of DMSO was added, and stirring was continued for an additional 2.5 hr. The mixture was poured into 250 ml of water and the suspension was extracted into CH_2Cl_2 (five 30-ml portions). The extract was washed with a 200-ml portion of water, and basic substances were extracted into 0.15 *N* HCl (three 40-ml portions). The aqueous solution was made strongly alkaline by the addition of concentrated NaOH and the free amine was extracted into CCl_4 (five 30-ml portions). Upon concentration of the CCl_4 extract to a small volume and cooling for several hours at −15°, 1.066 g of product crystallized. Recrystallization from CH_2Cl_2–EtOH afforded 977 mg (44%) of **6c**, mp 157–162°. The analytical sample was obtained by repeated recrystallizations from CH_2Cl_2–EtOH, and melted at 160–162°: ir (KBr) 1630, 1530, 1380, 1455, 1323, and 1400 cm^{-1}; nmr ($CDCl_3$) δ 1.6 [6 H, s, $C(CH_3)_2NO_2$], 2.4 [6 H, s, $N(CH_3)_2$], 2.7–4.4 (4 H, m, $CH_2CH_2NMe_2$), 4.4 (1 H, s, $CHCMe_2NO_2$), and 7.0–8.0 ppm (8 H, m, aromatic); mass spectrum[18] *m/e* 367.1901 (calcd for $C_{21}H_{25}N_3O_3$: 367.1896).

Anal. Calcd for $C_{21}H_{25}N_3O_3$: C, 68.64; H, 6.86; N, 11.44. Found: C, 68.14; H, 6.77; N, 11.43.

Registry No.—**3a**, 29925-32-4; **3b**, 29925-33-5; **3c**, 29925-34-6; **3c** tosylate, 37387-62-5; **4c**, 37387-63-6; **5a**, 37387-64-7; **5b**, 37387-65-8; **5c**, 37387-66-9; **6a**, 37387-67-0; **6a**, 37387-67-0; **6a** HBr, 37387-68-1; **6c**, 37387-69-2.

(18) Exact mass measurements were obtained by the peak-matching technique by Mr. J. Carter Cook using a MAT 731 high-resolution mass spectrometer and data-processing equipment provided by NIH grants CA 11388 and GM 16864, from the National Cancer Institute and the National Institute of General Medical Studies, respectively.

4,5,6,7-Tetrafluoroindole

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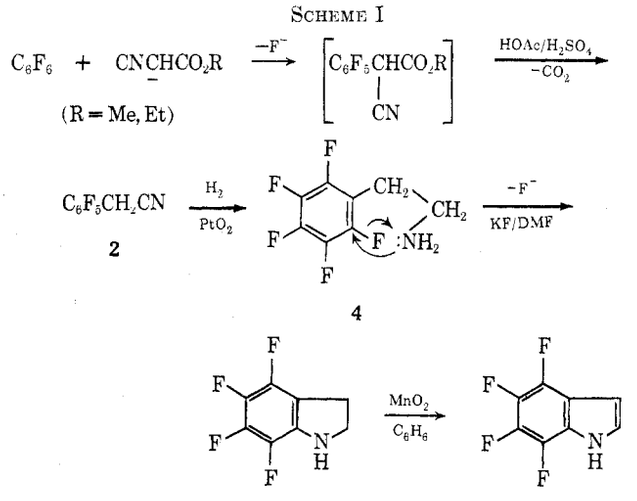
We wish to report a convenient synthesis of 4,5,6,7-tetrafluoroindole (**1**) by a vastly improved five-step sequence starting with hexafluorobenzene (Scheme I).

SCHEME I



Previous routes to **1** have suffered from serious drawbacks, *e.g.*, tedious multistep procedures[3] from readily

(1) Abstracted, in part, from the Ph.D. Thesis of S. M. W., May 1972.
(2) NSF Undergraduate Research Participant, summer 1971.
(3) V. P. Petrov, V. A. Barkhash, G. S. Schegoleva, T. D. Petrova, T. I. Savchenko, and G. G. Yakobson, *Dokl. Akad. Nauk, SSSR*, **178**, 864 (1968).