with 3 drops of glacial HOAc to retard disproportionation. The solvent was removed at 10–15 °C on a rotary evaporator and the residue dissolved in 4 mL of dioxane. Water (1.5 mL) was added followed by drops of 2 N NaOH until the reaction mixture became yellow. The mixture was stirred for 1 h, concentrated under reduced pressure, treated with water, and extracted as previously described. Sublimation of the crude product (110 °C, 0.5 mm) gave 25 mg (30%) of pure **4**.

1-Methylthio[2.2](2,6)pyridinophane (8a). To 120 mg (0.50 mmol) of **6a** in 3 mL of dry CH_2Cl_2 was added over 1 min 120 mg (0.58 mmol) of solid trimethyloxonium hexafluorophosphate. The initially homogeneous mixture became bright yellow after approximately 5 min and then colorless followed by the separation of sulfonium salt **6d**. The reaction was monitored by TLC (alumina, CH_2Cl_2) and upon disappearance of all of the sulfide (approximately 0.5 h) the mixture was evaporated to dryness and the residue suspended in 3 mL of dry THF. To this stirred slurry was added an excess (approximately 75 mg) of NaH likewise slurried in a small amount of THF. The mixture immediately became orange in color and H_2 was evolved. After the mixture was stirred for 3 h water was cautiously added to distroy excess NaH and the mixture was evaporated to dryness. The residue was extracted with CH_2Cl_2 and the extracts were washed with water, dried over Na_2SO_4, and concentrated. The concentrate was passed through a short plug (4 cm) of Woelm neutral alumina (activity grade II), giving 45 mg (35%) of **8a** which was pure by TLC. An analytical sample was recrystallized from benzene–pentane: mp 124–126 °C; IR (KBr) 3080, 3060, 2960, 2908, 2865, 1587, 1569, 1452, 1306, 1148, 1080, 996 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.16 (s, 3, CH_3), 3.0–4.0 (m, 7, CH_2, CHS), 7.0–8.0 (m, 6, PyH).

Anal. Calcd for $C_{15}H_{16}N_2S$: C, 70.28; H, 6.29; N, 10.93. Found: C, 70.18; H, 6.38; N, 10.77.

1-(Methanesulfinyl)[2.2](2,6)pyridinophane (8b). A solution containing 32 mg (0.125 mmol) of **8a** in 0.5 mL of THF was treated with 0.5 mL of water and 3 drops of 30% H_2O_2. Several additional drops of 30% H_2O_2 were added after 0.5 h and stirring was continued all of the sulfide had been consumed as evidenced by TLC analysis (alumina, CH_2Cl_2). Excess H_2O_2 was destroyed by the addition of Na_2SO_3 solution and the mixture was concentrated to near dryness. The residue was extracted with CH_2Cl_2 and the extracts were washed with brine, dried over Na_2SO_4, and concentrated. Preparative layer chromatography (alumina, 15:1 EtOAc–MeOH) gave 25 mg (74%) of **8b** as an oil which crystallized from benzene–pentane: mp 160–164 °C; IR (KBr) 3060, 2995, 2950, 2920, 2850, 1580, 1562, 1449, 1080, 1040 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.56, 2.68 (2 br s, 3, diastereomeric CH_3), 3.0–3.48 (br m, 6, CH_2), 4.12 (br d, 1, CHSO), 7.0–8.0 (m, 6, PyH).

Anal. Calcd for $C_{15}H_{16}N_2OS$: C, 66.15; H, 5.92; N, 10.28. Found: C, 66.02; H, 5.92; N, 10.22.

Pyrolysis of 8b. A solution containing 25 mg (0.9 mmol) of **8b** in 1.0 mL of xylene was heated at reflux for 30 h. The mixture was concentrated on a steam bath and chromatographed (preparative layer chromatography, alumina, CH_2Cl_2), giving 8 mg (42%) of **4**.

Registry No. 4, 76467-41-9; **5**, 76467-42-0; **6a**, 76467-43-1; **6b**, 76467-44-2; **6c**, 76467-45-3; **6d**·PF_6^-, 76467-47-5; **7a**, 76467-48-6; **7b**, 76467-49-7; **8a**, 76467-50-0; **8b**, 76467-51-1.

An Unusually Stable α-Chloro Epoxide

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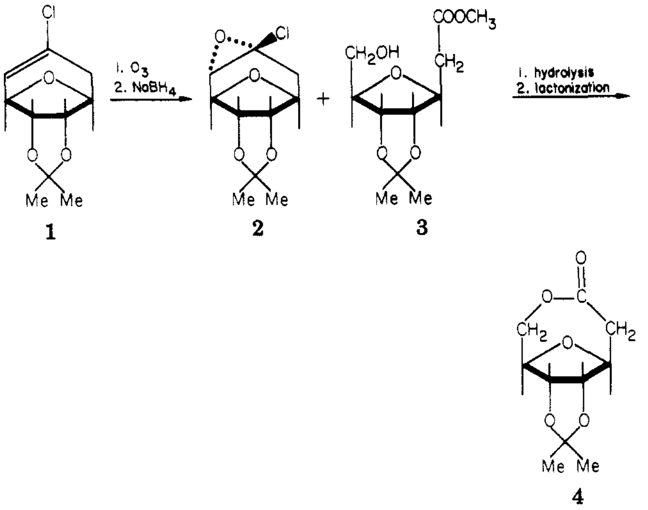
A key step in our synthesis of a homo-C-nucleoside[1] calls for cleavage of the vinyl chloride double bond in compound **1** with ozone. Although slow, ozonolysis of this kind of olefin is well recognized.[2] In our case, the ozonolysis proceeded smoothly at 0 °C, with methanol as solvent in the presence of a cation exchange resin. The oxidative cleavage was followed directly by reduction with sodium borohydride to form the methyl ester **3**, then hydrolysis of the crude reaction mixture, and finally azeotropic dehydration. The goal was to reach intermediate lactone **4**, which we initially took to be the crystalline product (mp 120 °C) isolated in low yield. But continued investigation made this untenable. Thus, we found that the crystalline product was already present in the borohydride product mixture, in which the main product as expected (72%) was the oily methyl ester **3**. Also, after authentic lactone **4** (mp 140–141 °C) had been prepared,[1] it became clear that the crystalline material was different from the lactone. The present note discusses this compound, to which we have now assigned the chloro epoxide structure **2**.[3]



The composition and molecular weight corresponded to $C_{10}H_{13}ClO_4$, as required by **2**. No molecular mass peak was evident in the high-resolution mass fragmentation pattern. However, the most intense peak corresponded to $M^+ - CH_3$, a fragment generated by loss of one of the two isopropylidene methyl groups to form a carbonium ion stabilized by the two neighboring oxygen atoms.[4] The infrared absorption spectrum showed no maxima in the carbonyl region at 1700–1800 cm^{-1}. A maximum at 870 cm^{-1} is present, one that can be ascribed to an asymmetrical stretch vibration of the three-membered ring; also the observed maximum at 1270 cm^{-1} corresponds to the symmetrical "breathing" vibration.[5] The proton magnetic resonance spectrum was consistent with that expected for structure **2**. The carbon-13 NMR results proved to be particularly informative. Thus a doublet at 57 ppm, ascribed to the methine carbon of the epoxide function, corresponded well to the relatively high-field signals reported before for related epoxides.[6] Even more significant

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(1) W. J. Gensler, Sum Chan, and D. B. Ball, *J. Am. Chem. Soc.*, **97**, 436 (1975).

(2) P. S. Bailey, "Ozonation in Organic Chemistry", Vol. I, Academic Press, New York, 1978.

(3) Our work offers no support to the report that *both* carbons of the double bond emerge at the carboxyl state of oxidation. See K. Griesbaum and H. Keul, *Agnew. Chem., Int. Ed. Engl.*, **14**, 716 (1975).

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(5) R. M. Silverstein, G. C. Bassler, and T. C. Morrill, "Spectrometric Identification of Organic Compounds", 3rd ed., 1974, Wiley, New York, p 95.

(6) S. G. Davies and G. H. Whitham, *J. Chem. Soc., Perkin Trans. 2*, 861 (1975); D. R. Paulsen, et al., *J. Org. Chem.*, **40**, 184 (1975); R. B. Bates and D. J. Eckert, *J. Am. Chem. Soc.*, **94**, 8258 (1972). Also cf. L. F. Johnson and W. C. Jankowski, "Carbon-13 NMR Spectra", Wiley-Interscience, 1973, curve no. 337.

0022-3263/81/1946-1750$01.25/0

was the unusually large ^{13}C–H coupling value (184 Hz) for this carbon, a consequence of the increased s character in the external bonds of the three-membered ethylene oxide ring.[7] Both observations confirm the presence of an epoxide ring in compound **2**.

Although epoxides have been obtained as products from ethylenes, including chloroethylenes, and reasonable mechanisms for their formation have been proposed,[2] compound **2** showed properties that were hard to reconcile with those expected. For example, it was stable at 120–130 °C and at least for 5 min survived a temperature of 205–250 °C. It failed to solvolyze in methanol and was resistant to reduction with borohydride. It did not deteriorate when allowed to stand in the open and survived unchanged on treatment with boiling aqueous sodium hydroxide, with boiling pyridine, or with 8% acetic acid (65 °C) for 24 h.

Comparison of these properties with those of other α-chloro epoxides[8] shows that compound **2** does not fit well with this class which, in general, has far greater reactivity. Nevertheless, we believe that the assignment is correct. A model shows that the atoms crowding into both the endo and the exo sides of the rigid cage system in **2** interfere with the approach of nucleophiles from either direction and therefore would hinder S_N2 displacement of chloride ion as well as of ring oxygen. S_N1 ionization of the carbon-to-chlorine bond is disfavored because the resulting carbonium ion cannot assume a planar sp^2 hybridization. However, ionization of the epoxide C to O bonds should not be subject to this limitation, and we believe that the failure to solvolyze at oxygen was simply the consequence of employing conditions that were too mild. Accordingly, much of the relative inertness of what generally is a reactive system is a consequence of the ring structure of **2**.

The proton NMR spectrum of our chloro epoxide fits that predicted for the *exo*-epoxide assignment as in **2** better than the endo. Also, the exo assignment is consistent with the easier approach of ozone to the more open exo side of the chloroethylene grouping in starting material **1**.[9]

Experimental Section

General Methods. Analyses for elements and the determinations of molecular weight by vapor-pressure measurements were performed by Galbraith Laboratories, Inc., and by Chemalitics, Inc. Thin-layer chromatograms were obtained on precoated silica gel plates from Brinkman Instruments, Inc.; the spots were brought out with iodine vapor. Proton nuclear resonance spectra were taken on a JEOL 60 MHz instrument as well as on a Hitachi Perkin-Elmer spectrometer, also at 60 MHz.

Ozonolysis of 3-Chloro-*exo*-*cis*-6,7-(isopropylidenedioxy)-8-oxabicyclo[3.2.1]-2-octene (1). An analytical grade of polyacrylic acid cation-exchange resin (sodium form) was washed with three 100-mL portions of absolute methanol, then dried in vacuo in a rotating evaporator, and finally warmed at 50–60 °C (0.01 mm) for 4 h. In this way, 50 g of Bio-Rad Laboratories' Bio Rex 70 (200–400 mesh, with 60–70% water) gave 15 g of treated resin, which was stored in a desiccator.

Ozone generated from oxygen was bubbled through a stirred, heterogeneous mixture of chloroethylene 1 (6.00 g, 27.7 mmol), dry resin (6 g), and absolute methanol (80 mL) at 0 °C at a rate of 6.7 mmol of ozone/min. At intervals, 0.1-mL aliquots were removed and filtered. The filtrates were scanned over the 1600–1700-cm^{-1} region, and ozonolysis was discontinued when the absorption maximum at 1645 cm^{-1} (C=CCl) was no longer evident.

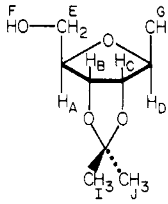
The clear solution obtained when the ozonolysis mixture was filtered through Filter-aid was held at 0 °C and treated dropwise with sodium borohydride (1.1 g, 28 mmol) dissolved in absolute methanol (10 mL). Gas evolution was noted, and the temperature tended to rise. The heterogeneous mixture was stirred at 25 °C for $^3/_4$ h.

Aqueous hydrochloric acid (37%) was added slowly until the pH was 4–5. After neutralization to pH 7–8 with 10% aqueous sodium bicarbonate, the mixture was filtered and stripped of solvent. The residual slurry was stirred for 10 min with acetone (100 mL) plus anhydrous magnesium sulfate (1 g) and filtered again. The yellow oily residue remaining after removal of the acetone was distilled through a short-path still to give 6.15 g of product, bp 108–110 °C (0.01 mm). High-pressure liquid chromatography using porous silica on glass (Corasil II) with chloroform as solvent showed six peaks. Gas–liquid chromatography through a 205 °C column of 15% neopentylglycol succinate supported by Anachrom 70/80 also showed six components, one of which corresponded to the methyl ester **3** (91%) and a second to epoxide **2** (7%).

Part of the pale yellow product (3.0 g) was chromatographed through a 30-cm column of silica gel 60 (40 g) with chloroform–acetone (9:1 by volume) as solvent. Almost pure epoxide **2** was obtained as a solid (0.3 g) from the 60–100-mL fraction of eluate; the main product, ester **3** (2.5 g), emerged in the 120–220-mL fraction. Distillation of the ester in a short-path still afforded homogeneous product (2.4 g; 80% calculated from the first distillate or 72% from chloroethylene 1) as a colorless oil, bp 108–110 °C (0.01 mm).

Anal. Calcd for $C_{11}H_{18}O_6$: C, 53.60; H, 7.31. Found: C, 53.42; H, 7.23.

This methyl ester showed a single spot on TLC (4:1 chloroform–acetone on a silica gel plate, R_f 0.41) and a single peak on GLC: IR (CCl_4) 3500 (OH), 1730 cm^{-1} (C=O), etc.; NMR ($CDCl_3$) δ 4.80–4.00 (m, 4, A, B, C, D), 3.80 (broadened s, 5, E + H), 3.10 (t, J = 6 Hz, 1, F), 2.74–2.65 (s and d, 2, G), 1.53 (s, 3, I), 1.35 (s, 3, J). Addition of 1 drop of deuterated water eliminated the δ 3.10 signal.



Control experiments showed that losses of the ester product **3** on silica gel chromatography were appreciable, ranging up to 20%. Ozonolysis in the absence of an acid-absorbing material led to very low yields.[10] Ozonolysis at −78 °C failed altogether.

The epoxide (0.3 g) obtained by chromatography of the ozonolysis product was washed three times with ether before sublimation at 0.01 mm in a 120–130 °C bath. Pure epoxide **2** (0.25 g, 4% from the starting material 1) was collected as white crystals (mp 125–126 °C) showing a single spot on TLC (R_f 0.67 with 4:1 chloroform–acetone on silica gel) and a single peak on a 205 °C GLC column.

Anal. Calcd for $C_{10}H_{13}ClO_4$: C, 51.61; H, 5.63; Cl, 15.26; mol wt 232.7. Found: C, 51.79; H, 5.66; Cl, 15.20; mol wt (vapor-pressure measurements in $CHCl_3$ solvent) 226.

A high-resolution mass spectrum obtained with a CEC 110 B spectrometer operating at 70-eV showed mass peaks that had the following *m/e* values, given in the order of their intensities:

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[7] F. A. Bovey, "Nuclear Magnetic Resonance Spectroscopy", Academic Press, New York, 1969, pp 232–234.

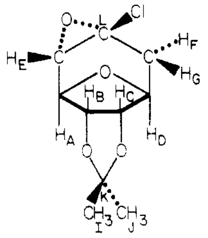
[8] For pertinent examples, see, inter alia: M. Mousseron and R. Jacquier, *Bull. Soc. Chim. Fr.*, 698 (1950); A. Kirrmann, P. Duhamel, and R. Nouri-Bimorghi, *ibid.*, 3264 (1964); *Justus Liebigs Ann. Chem.*, **691**, 33 (1966); G. Büchi, D. Minster, and J. C. F. Young, *J. Am. Chem. Soc.*, **93**, 4319 (1971); M. Zief and C. H. Schramm, *Chem. Ind (London)*, 660 (1964); A. Kirrmann and R. Nouri-Bimorghi, *Bull. Soc. Chim. Fr.*, 3213 (1968); K. Griesbaum, R. Kibar, and B. Pfeffer, *Justus Liebigs Ann. Chem.*, 214 (1975); K. Griesbaum, H. Keul, R. Kibar, B. Pfeffer, and M. Spraul, submitted for publication in *Justus Liebigs Ann. Chem.*

[9] The stereochemical arguments suggesting that epoxides originate by initial attack of ozone on the *more* hindered side of the double bond are hard to accept. See ref 2, p 210.

[10] Cf. P. Deslongchamps and C. Moreau, *Can. J. Chem.*, **49**, 2465 (1971). These authors find that free HCl facilitates overoxidation of peroxidic products and, in addition, in our case, would catalyze solvolysis of the protecting isopropylidene group.

217.02487 (base peak) for $C_9H_{10}O_4Cl$ or $M^+ - CH_3$, 219.02350 for $C_9H_{10}O_4{}^{37}Cl$ or $M^+ - CH_3$, 139.03849 for $C_7H_7O_3$, 71.01330 for $C_3H_3O_2$, 111.04394 for $C_6H_7O_2$, etc.

The epoxide **2** had the following absorption characteristics: IR (CCl_4) 2990, 2930, 1420, 1380, 1330, 1270, 1220, 870 (epoxide ring) cm^{-1}; UV (CH_3OH) λ_{max} 210 nm (ϵ 244), 260 (63); ^1H NMR ($CDCl_3$) δ 4.90 and 4.68 (2 d, $J_{BC} = 5.6$ Hz, $J_{AB} = J_{CD} = 0$, 2, B and C), 4.45 (d, $J_{AE} = 1.5$ Hz, $J_{AB} = 0$, 1, A), 4.15 (d, $J_{DF} = 6.7$ Hz, $J_{DG} = J_{CD} = 0$, 1, D), 3.33 (t, $J_{AE} = J_{EF} \simeq 1.5$, 1, E), 2.88 (ddd, $J_{FD} = 6.7$ Hz, $J_{FE} \simeq 1.2$ Hz, $J_{FG} = 15$, 1, F), 2.10 (d, $J_{FG} = 15$ Hz, $J_{GD} = 0$, 1, G), 1.50 (s, 3, I), 1.35 (s, 3, J). Decoupling irradiation at the 4.45-ppm signal for A changed the 3.33-ppm triplet for E to a rough singlet. Irradiation at 3.33 ppm (E) changed the doublet at 4.45 ppm (A) to a singlet. Irradiation at 4.15 ppm (D) simplified the 2.88 ddd for F to dd.



The neglible vicinal couplings between A–B, C–D, and D–G correspond well to the respective dihedral angles (Dreiding model) of 90°. The A–E dihedral angle of 37°, according to the Karplus relation, should give rise to $J_{AE} = 5.1$ Hz, a value that does not agree with the $J_{AE} = 1.5$ observed. However, attention has been called[11] to the effect of an epoxide ring in decreasing vicinal coupling constants for such systems. Making use of the relevant equation[11] leads to a predicted J_{AE} of 3.5 Hz. The tetrahydrofuran oxygen attached to the carbon bearing the A hydrogen would reduce the A–E vicinal coupling still more and bring the predicted value even closer to that observed. The H_E triplet at 3.33 ppm involves not only vicinal coupling with A but also one other equal coupling with a remote hydrogen, possibly F.

The ^{13}C nuclear magnetic resonance curve was determined at 15 MHz with a JEOL FX 60 Q instrument and with $CDCl_3$ as solvent: δ 113.2 (s, C_K), 84.9 (d), 81.3 (d), 77.7 (d), 76.4 (d), 70.2 (s, C_L), 57.0 (d, $J = 184$, C_E), 36.6 (t, $J \simeq 133$ Hz, C_{FG}), 26.2 (q, $J = 127$, C_I), 25.0 (q, $J = 126$, C_J). The four doublets from 84.9–76.4 ppm were assigned to the tetrahydrofuran ring carbons; each showed an approximate coupling constant of 155 Hz.

Ozonolysis of the chloroethylene **1** at 0 °C in ethyl acetate saturated with 1% aqueous potassium hydroxide afforded the epoxide in somewhat higher yield. Here the ozonolysis mixture was treated with borohydride in 1:1 water–dioxane at 0 °C, the volatiles were removed, and the residue in 8% aqueous acetic acid was held at 65 °C for 24 h. Again the solvents were removed, and in order to replace isopropylidene groups that the acetic acid might have removed, the product mixture was allowed to stand in dry acetone containing anydrous magnesium sulfate. Continued processing, including a toluene azeotrope with the intent of effecting lactonization to **4**, led to epoxide **2** (31%; mp 119–120 °C) as the only characterized product.

Treatment of Chloro Epoxide 2 with Base. A suspension of the epoxide **2** (0.100 g, 0.43 mmol) in 0.1 N aqueous sodium hydroxide (3 mL) was refluxed for 20 min. After neutralization at room temperature with 1 N hydrochloric acid, the heterogeneous mixture was stripped of all volatile material. Sublimation of the residual white solid at 120 °C (0.01 mm) afforded unchanged epoxide **2** (0.085 g, 85% recovery; mp 125–126 °C) with infrared and NMR spectra identical with those obtained with the starting material.

Boiling 0.100 g (0.43 mmol) of epoxide **2** in pyridine (3 mL) for 25 min, removing the solvent, and subliming the residue gave the epoxide as a white solid (0.090 g, 90% recovery) with unchanged melting point and infrared and ^1H NMR absorption spectra.

Acknowledgment. We acknowledge the sponsorship of the American Cancer Society, which through Grant No. T533 helped to support this work. We also thank Waters Associates, Inc., for their cooperation with several high-pressure liquid chromatography determinations. Professor David A. Forsyth very kindly provided the ^{13}C nuclear magnetic resonance data reported here; Dr. Catherine Costello performed the high-resolution mass spectral determination (Division of Research Resources, NIH Grant No. RR 00317 to Klaus Biemann at Massachusetts Institute of Technology).

Registry No. **1**, 55036-18-5; **2**, 76529-25-4; **3**, 55036-19-6.

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[11] N. S. Bhacca and D. H. Williams, "Applications of NMR Spectroscopy in Organic Chemistry", Holden-Day, San Francisco, 1964, p 100. In this connection, the total absence of gem coupling for the two hydrogen atoms on the ring methylene group of 2-chloro-2-*tert*-butyloxirane is noteworthy. (See the article to appear by Griesbaum et al. cited in ref 8.)

A New Approach to 4-Substituted Indoles

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As bifunctional reagents, 1*H*-4-carboxaldehyde (**4**) and 1*H*-indole-4-acetaldehyde are attractive starting materials for the synthesis of alkaloids related to the clavine family and lysergic acid. Aldehyde **4** has very recently been used in elegant syntheses of chanoclavine I and isochanoclavine I[1,2] and was prepared from 1*H*-indole-4-carboxylic acid methyl ester via oxidation of 1*H*-indole-4-methanol with either potassium permanganate[3] or manganese dioxide, pioneered by Plieninger[4] and recently adopted by Kozikowski.[5] Alternatively, **4** was synthesized by reduction of 1*H*-indole-4-carbonitrile[6] with lithium triethoxyaluminium hydride,[4] by catalytic hydrogenation and trapping of the aldehyde as a hydrazone,[4] or with sodium hypophosphite/Raney nickel.[7]

1-Acetyl-1*H*-indole-4-acetaldehyde has also been prepared before as follows. 1-Aminonaphthalene was reduced with sodium in ethanol to furnish 1-amino-5,8-dihydronaphthalene which was N-acetylated. The resulting *N*-(5,8-dihydro-2-naphthalenyl)acetamide was then ozonized, yielding *N*-[2,3-bis(2-oxoethyl)phenyl]acetamide. Acid-catalyzed cyclization and dehydration of this dialdehyde led to 1-acetyl-1*H*-indole-4-acetaldehyde which was isolated as the bisulfite adduct.[8] 1-(*p*-Toluenesulfonyl)-1*H*-indole-4-acetaldehyde was prepared in similar fashion.[9]

We now describe a short route to aldehyde **4** (Scheme I) based on the regiospecific benzylic oxidation of the inexpensive 2,3-dimethylnitrobenzene (**1**). It is then possible to either prepare **4** in a subsequent step or to elaborate the generated carboxaldehyde function, thus establishing a side chain at C-4 of indole prior to indole-

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