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6-CYCLOPROPYL- AND 6-(1-METHYLCYCLOPROPYL)-1,4-BENZODIOXANES: SYNTHESIS AND NITRATION. REARRANGEMENT OF NITRO SUBSTITUTED 6-CYCLOPROPYL-1,4-BENZODIOXANES USING CONCENTRATED SULFURIC ACID

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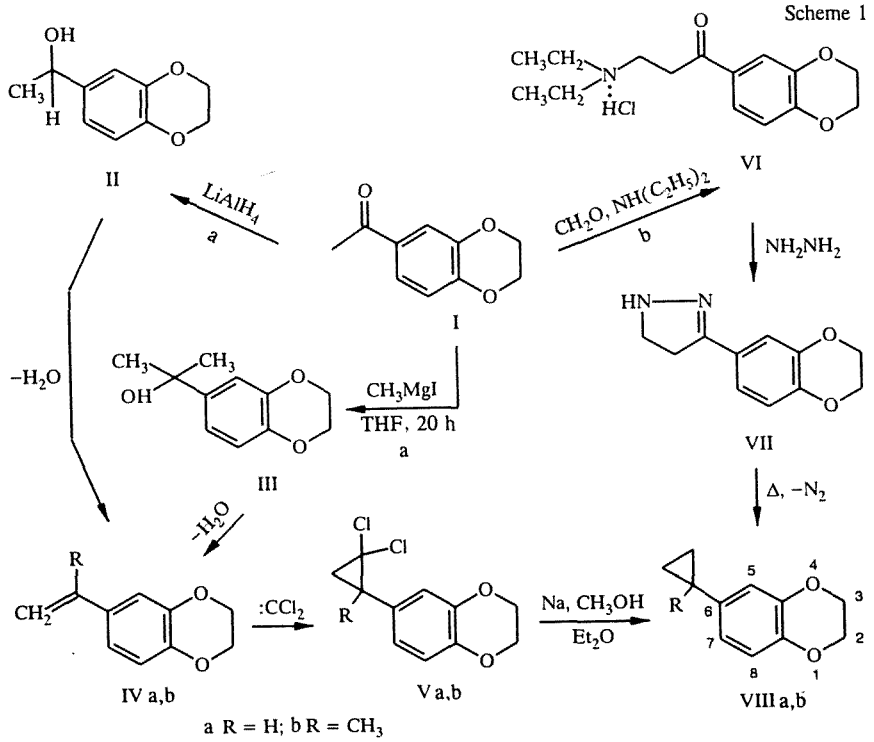
6-Cyclopropyl- and 6-(1-methylcyclopropyl)-1,4-benzodioxanes have been synthesized as the first cyclopropyl-1,4-benzodioxanes. It was shown that nitration of these compounds occurs with retention of the three carbon ring. In contrast to simple ethers of cyclopropylphenols, nitration leads only to 7-nitro-6-cyclopropyl- and 7-nitro-6-(1-methylcyclopropyl)-1,4-benzodioxanes, respectively. Nitration of 7-nitro-6-cyclopropyl-1,4-benzodioxane occurs regioselectively to give exclusively the 7,8-dinitro product. The structure of the nitration products was confirmed by their rearrangement to the corresponding nitrosopropionyl-1,4-benzodioxanes.

Amongst a wide circle of cyclopropanes containing an aromatic fragment directly bonded to the three carbon ring, cyclopropyl 1,4-benzodioxanes have not been reported at this time. It was, therefore, of considerable theoretical and practical interest to synthesize and study the reactivities of cyclopropyl 1,4-benzodioxanes in light of the reactions of electrophilic reagents [1, 2] with phenylcyclopropanes, (in particular o-substituted analogs [3, 4]).

Scheme 1



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In this work we have synthesized 6-cyclopropyl- and 6-(1-methylcyclopropyl)-1,4-benzodioxanes and studied their nitration and the behavior of the corresponding nitro derivatives under the conditions used for the sulfuric acid isomerization of o-nitrophenylcyclopropanes.

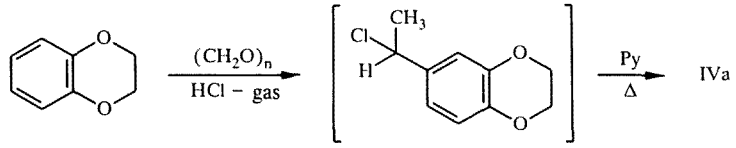
6-Cyclopropyl-1,4-benzodioxane (VIIIa) was prepared by stepwise reaction of 6-acetyl-1,4-benzodioxane (I) using two methods (see Scheme 1).

In the four stage route from I to VIIIa (route *a*) the least efficient and most problematic stage is that of carbinol II dehydration. In all cases the yields of alkene IVa were low. In our hands, the best yield for a number of dehydrating agents (Ac_2O, H_2SO_4, H_3PO_4, TsOH, $K_2S_2O_7$) was achieved with the latter potassium pyrosulfate.

The rather low yield of the 6-vinyl-1,4-benzodioxane (IVa) is apparently linked to the ready polymerization of this alkene. As a relevant example, it should be noted that distilled IVa polymerizes like styrene when left without an inhibitor.

According to our work, 6-vinyl-1,4-benzodioxane has only been reported once, having been synthesized for the first time in 1978 [5] by the following scheme. The yield was also rather low (35%).

Scheme 2



Our many attempts to repeat this route to IVa and to shorten route *a* to compound VIIIa by at least one stage were unsuccessful, the yield of IVa being significantly lower than that reported in [5].

Synthesis of VIIIa by route *b* proved more efficient, despite the modest yields of the intermediates in each of the three stages. It must be stressed that, in this case, the catalytic decomposition of the 6-(pyrazolin-3-yl)-1,4-benzodioxane (VII) occurs without formation of significant amounts of the side products (6-propenyl- or 6-allyl-1,4-benzodioxanes). With phenyl- [6] or furyl-pyrazolines [7], the catalytic conversion of the aryl or hetaryl cyclopropanes is always accompanied by the considerable amounts of the propenyl or allyl substituted benzenes or furans.

Because synthesis of 6-(1-methylcyclopropyl)-1,4-benzodioxane (VIIIb) could not be achieved by route *b* (Scheme 1), it was prepared by route *a* via the homologous 6-isopropenyl-1,4-benzodioxane (IVb). As for VIIIa, the stage which particularly limits the use of method *a* is the carbinol (III) dehydration.

In studying the nitration of cyclopropylbenzodioxanes VIIIa, b (basically ethers of 1,2-dihydroxybenzene), we wanted to compare their behavior with that of o- and p-methoxyphenylcyclopropanes. As has been shown, the latter behave differently when nitrated by concentrated and dilute nitric acids in acetic anhydride. As an example, the reaction of p-cyclopropylanisole (IX, Scheme 3) with conc. HNO_3 occurs with the formation of the nitro derivatives X, XI [8]. By contrast, the reaction of the same substrate IX with 55% nitric acid gives nitrophenol XII as the main product. The nitro derivatives X and XI are also formed but in notably smaller quantities [9]. Specific experiments showed that the hydroxydemethoxylation of IX precedes nitration in the aromatic ring and that it is a consequence of formation of ipso-benzenonium ions of type A and B (Scheme 3) under the reaction conditions.

Scheme 3

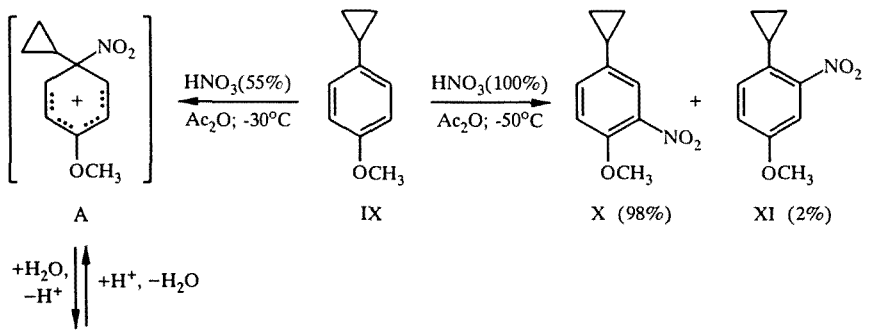
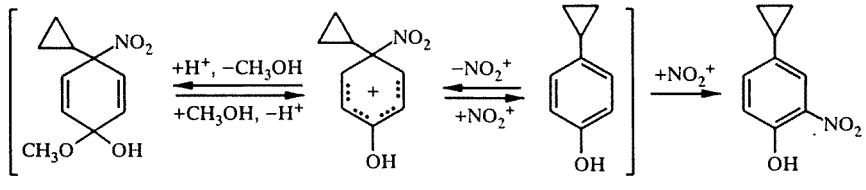


TABLE 1. Constants and PMR Spectral Parameters for Compounds Synthesized

Compound	Empirical formula	mp, °C; bp, °C (mm, Hg)	PMR spectrum, δ, ppm
III	$C_{11}H_{14}O_3$	—	1,28 (6H, s, CH_3), 2,56 (1H, br s, OH), 4,12 (4H, s, $O\underline{CH_2CH_2}O$), 6,78 (3H, m, ArH)
IV a	$C_{10}H_{10}O_2$	110(4)	4,26 (2H, s, $O\underline{CH_2CH_2}O$), 5,10 (1H, dd, $J\alpha,\beta' = -14$, $J\beta,\beta' = 1,8$ Hz), 5,61 (1H, dd, $J\alpha,\beta = 20$, $J\beta,\beta' = 1,8$ Hz), 6,61 (1H, dd, $J\alpha,\beta = 20$, $J\alpha,\beta' = -14$ Hz), double bond protons, 6,67 (3H, m, ArH)
IV b	$C_{11}H_{12}O_2$	183...185(25)	1,99 (3H, s, CH_3), 4,02 (4H, s, $O\underline{CH_2CH_2}O$), 4,82 (1H, m), 5,11 (1H, s), double bond protons, 6,86 (3H, m, ArH)
Va	$C_{11}H_{10}Cl_2O_2$	137...139(2)*	1,81 (2H, m, part of ABM system 2,78 (1H, m, M part of ABM system, cyclopropane protons 4,21 (4H, s, $O\underline{CH_2CH_2}O$), 6,70 (3H, m, ArH)
Vb	$C_{12}H_{12}Cl_2O_2$	181...182 (10)	1,66 (3H, s, CH_3), 1.48 (1H, d, $J_1 = 7,8$ Hz), 1,92 (1H, d., $J_1 = 7,8$ Hz), cyclopropane protons, 4,20 (4H, s, $O\underline{CH_2CH_2}O$), 6,63 (3H, m, ArH)
VI	$C_{15}H_{22}NClO_3$	163	1,31 (6H, dt, $CH_2\underline{CH_3}$), 3,40 (8H, m, $\underline{CH_2}CH_3$, $\underline{CH_2}CH_2$), 4,21 (4H, s, OCH_2CH_2O), 6,81 (3H, m, ArH), 11,86 (1H, br s, NH)
VIIIa	$C_{11}H_{12}O_2$	105...106 (3)*	0,65 (4H, m), 1,74(1H, m), cyclopropane protons, 4,11 (4H, s, $O\underline{CH_2CH_2}O$), 6,62 (3H, m, ArH)
VIIIb	$C_{12}H_{14}O_2$	168...169 (12)*	0,66 (4H, m), cyclopropane protons, 1,31 (3H, s, CH_3), 4,15 (4H, s, $O\underline{CH_2CH_2}O$), 6,61 (3H, m, ArH)
XIIIa	$C_{11}H_{11}NO_4$	82	0,78 (4H, m), 2,34 (1H, m), cyclopropane-protons, 4,30 (4H, s, $O\underline{CH_2CH_2}O$), 6,51 (1H, s, Ar, 5-H), 7,52 (1H, s, 8-H)
XIIIb	$C_{12}H_{13}NO_4$	96...97	0,62 (4H, m), cyclopropane protons 1,56 (3H, s, CH_3), 4,34 (4H, s, $O\underline{CH_2CH_2}O$), 6,82 (1H, s, 5-H), 7,34 (1H, s, 8-H)
XIV	$C_{11}H_{11}NO_4$	119...120	1,14(3H, t, $CH_2\underline{CH_3}$), 2,99 (2H, q, $\underline{CH_2}CH_3$), 4,16 (4H, s, $O\underline{CH_2CH_2}O$), 6,38 (1H, s, 8-H), 7,18 (1H, s, 5-H)
XV	$C_{11}H_{11}NO_3$	65...66	1,38 (3H, t, $CH_2\underline{CH_3}$), 3,07 (2H, q, $\underline{CH_2}CH_3$), 4,21 (4H, s, $O\underline{CH_2CH_2}O$), 6,60 (1H, s), 6,70 (1H, s), ArH
XVIII	$C_{11}H_{10}N_2O_6$	131...132	0,79 (4H, m), 2,12 (1H, м), cyclopropane - protons, 4,23 (4H, s, $O\underline{CH_2CH_2}O$), 6,78 (1H, s, 5-H)
XIX	$C_{11}H_{10}N_2O_6$	113...115	1,14 (3H, t, $CH_2\underline{CH_3}$), 2,61 (2H, q, $\underline{CH_2}CH_3$), 4,47 (4H, s, $O\underline{CH_2CH_2}O$), 6,87 (1H, s, 5-H)

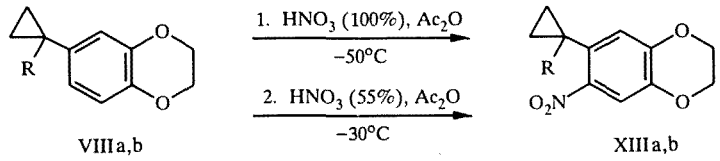
*For Va, n_D^{20} 1.5770; for VIIIa, n_D^{20} 1.5619; for VIIIb, n_D^{20} 1.5643.



B XII (71%)

6-Cyclopropyl-1,4-benzodioxanes VIIIa, b undergo nitration in both concentrated and dilute nitric acid in acetic anhydride in high yields to give only the nitroaromatic products. The entering nitro group occupies the position corresponding to the orientation of the substituent with less steric hindrance (Scheme 4).

Scheme 4

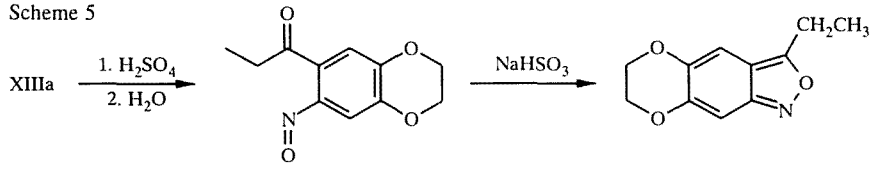


VIIIa,b XIIIa,b

In the PMR spectra of both nitro compounds XIIIa, b there were signals for the protons of the ethylenedioxy and cyclopropyl groups and, in each, two one proton singlet signals at low field for 5- and 8-H (see Table 1).

Additional evidence for the entering nitro group being next to the cyclopropyl group came from the observation that XIIIa could undergo a rearrangement typical of o-nitro substituted arylcyclopropanes [3], i.e., conversion to the corresponding nitrosopropionyl-1,4-benzodioxane (XIV, Scheme 5).

Scheme 5

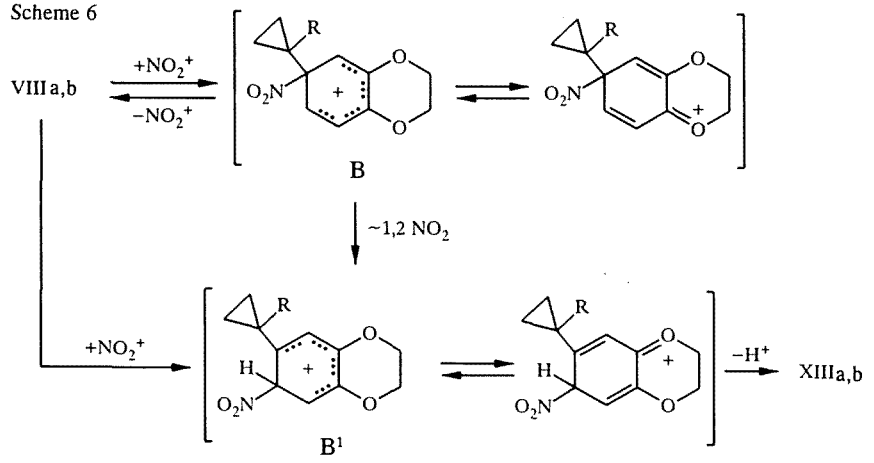


XIV XV

The structure of XIV was confirmed by conversion to 5,6-ethylenedioxybenz[c]isoxazole XV.

Attention should be turned to the fact that nitration of the cyclopropylbenzodioxanes VIIIa, b with dilute nitric acid formed no traces of those reaction products which might indirectly confirm ipso attack by the nitronium ion on the carbon atom in the starting substrate to which the cyclopropyl radical is bonded. This is somewhat surprising since it is known that cyclopropyl- or 1-methylcyclopropylanisole readily forms ipso benzenonium ions (Type A, Scheme 3) when treated with 55% nitric acid. However, the absence of products confirming ipso attack does not show that it does not occur. It seems, therefore, that the degree of ipso attack is significantly lowered in this case because of a high incidence of nitronium ion attack at the relevant position (7- and 6- in cyclopropylbenzodioxanes VIIIa and VIIIb). Alternatively, the ipso benzenonium ions formed (Type B, see Scheme 6) are extremely labile and readily undergo 1,2-migration of the nitro group converting to the σ-complexes (B^1) and leading to nitro compounds XIIIa, b.

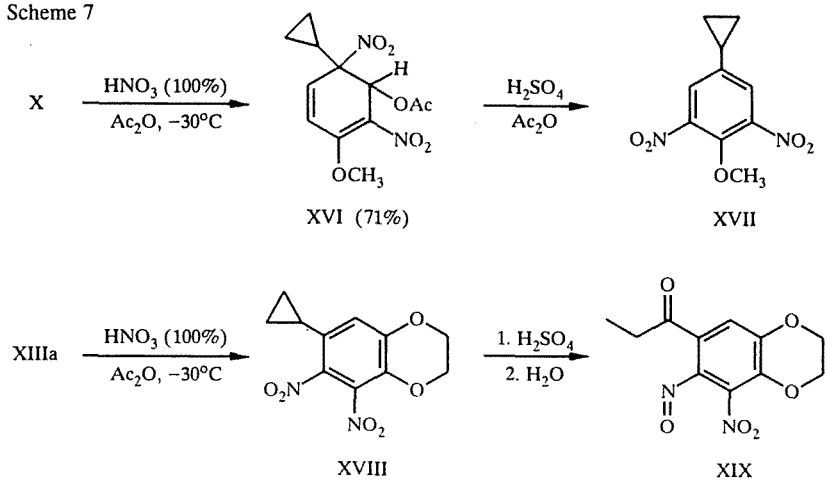
Scheme 6



It was previously found that ipso adducts, formed from nitrocyclopropylanisoles by nitration and occurring as intermediates en route to the indicated arylcyclopropane products (Scheme 7, [10]), are significantly more stable than similar ipso adducts formed during nitration of non-nitro containing cyclopropanes [9]. In this connection, we have proposed that mono nitro cyclopropylbenzodioxanes XIIIa, b could indeed prove to be models which show ipso attack by nitronium ion in the nitration of cyclopropylbenzodioxanes. However, it was found that under the conditions in which 2-nitro-4-cyclopropylanisole (X) forms

the ipso adduct XVI (Scheme 7) in high yield, 7-nitro-6-cyclopropyl-1,4-benzodioxane (XIIIa) gives almost exclusively the aromatic dinitro compound.

Scheme 7



XVI (71%) XVII

XVIII XIX

In our view, the preferred place for entry of the nitro group in the aromatic ring of XIIIa should have been position 5 and thus we expected formation of 5,7-dinitro-6-cyclopropyl-1,4-benzodioxane. However, PMR spectra for the dinitro compound point to a 7,8-dinitro-6-cyclopropyl-1,4-benzodioxane structure (XVIII). Thus, the PMR spectrum of the reaction product showed both signals for the protons of the ethylenedioxy group and the preserved cyclopropyl together with a one proton singlet signal whose chemical shift was identical to that of the 5-H proton in the PMR spectrum of XIIIa (see Table 1). This shows that nitration of XIIIa occurs at a position next to the nitro group.

Additional confirmation of the structure of XVIII came from its rearrangement in concentrated sulfuric acid only to one nitro product (XIX, see Scheme 7) and not to two as expected if the nitro compound had the 5,7-disubstituted structure.

It was also of interest that the PMR spectra data for 6-propionyl-7-nitroso-8-nitro-1,4-benzodioxane (XIX) indirectly confirm the dinitro structure XVIII in a further way. It has been found that, in nitrosobenzenes, the chemical shift for the benzene ring proton next to the nitroso group is shifted to high field by ~0.8 to 1.0 ppm [11], due to the nitroso anisotropic effect. In the example of nitroso compound XIX, the chemical shift of the PMR signal for the only aromatic proton has virtually the same value (6.87) as the signal for the aromatic proton in the spectrum of dinitro compound XVIII (6.78 ppm, see Table 1). On this basis, it is virtually certain that the nitroso group in XIX is situated between two substituents and, therefore, the dinitro compound undergoing isomerization has the structure shown.

EXPERIMENTAL

IR Spectra were recorded on a UR-20 instrument in vaseline oil and UV spectra on a Cary-119 using CH_2Cl_2. PMR Spectra were taken on a Varian XL-400 and a Tesla BS-467 instrument (CCl_4, $CDCl_3$, CD_2Cl_2) with TMS internal standard. PMR Spectral parameters and compound constants are given in Table 1.

Elemental analytical data for C and H in the compounds prepared agreed with those calculated.

6-(α-Hydroxyethyl)-1,4-benzodioxane (II). 6-Acetyl-1,4-benzodioxane (38 g, 0.21 mole) was added to a solution of $LiAlH_4$ (3.8 g, 0.1 mole) in absolute ether (250 ml). The reaction mixture was stirred for 4 h at 35°C, cooled to 10°C, and decomposed initially with moist ether and then with gradual addition of water with vigorous stirring. The organic layer was separated and the aqueous extracted with ether. The ether solutions were washed with water and dried with $MgSO_4$. The solvent was evaporated off and the residue distilled in vacuo to give carbinol II (36 g, 95%) with bp 151-152°C (0.5 mm Hg).

2-(1,4-Benzodioxan-6-yl)propan-2-ol (III). 6-Acetyl-1,4-benzodioxane (I, 23.6 g, 0.13 mole) in dry THF (~100 ml) was added to the Grignard reagent prepared from magnesium (4.8 g) and methyl iodide (28.4 g) at 10°C. The product was stirred for 20 h at 40°C, cooled to 10°C, and poured into 50% NH_4Cl solution with ice (30 g). The product was extracted with ether and the ether dried with $MgSO_4$. Evaporation of solvent gave carbinol III (15.4 g, 60%).

6-Vinyl-1,4-benzodioxane (IVa). Carbinol II (22 g, 0.12 mole) and powdered $K_2S_2O_7$ (5 g) were placed in a Claisen flask. The reaction mixture was carefully heated (not exceeding 65°C) for 30 min. The vacuum was lowered and the dehydration product gradually distilled off to give IVa (11 g, 52%) with bp 110°C (4 mm Hg). According to [5], bp = 98-101°C (3 mm Hg).

6-Isopropenyl-1,4-benzodioxane (IVb). Concentrated H_2SO_4 (2 drops) was added to a solution of carbinol III (31 g, 0.16 mole) in dry benzene (90 ml) and an azeotropic mixture of benzene and water distilled off. After distilling 45 ml, a further 45 ml of dry benzene was added and the same amount of azeotropic mixture distilled off. The residue was poured into water, the benzene layer separated and washed with water to neutral reaction and dried with $MgSO_4$. The solvent was evaporated and the residue distilled in vacuo to give IVb (10.0 g, 36%).

1,1-Dichloro-2-(1,4-benzodioxan-6-yl)cyclopropane (Va). To a solution of 6-vinyl-1,4-benzodioxane (IVa, 40.5 g, 0.25 mole) in CH_2Cl_2 (200 ml) and $CHCl_3$ (100 ml) was added benzyltriethylammonium chloride phase transfer catalyst (0.5 g). A solution of NaOH (50%, 39.6 ml) was added over 1.5 h with stirring. The product was stirred for 2 h at 50°C, cooled to 20°C, and the organic layer separated, washed with water to neutral pH and dried over $CaCl_2$. Evaporation of solvent and distillation of the residue in vacuo gave the dichlorocyclopropane IVa (58.8 g, 96%).

1,1-Dichloro-2-methyl-2-(1,4-benzodioxan-6-yl)cyclopropane (Vb) was obtained as for Va from 6-isopropenyl-1,4-benzodioxane (IVb, 10 g) to give 7.0 g of Va (46%).

6-(β-Diethylamino)propionyl-1,4-benzodioxane Hydrochloride (VI). A mixture of 6-acetyl-1,4-benzodioxane (20 g, 0.112 mole), diethylamine hydrochloride (9.1 g), paraformaldehyde (4.1 g), and ethanol (12 ml) was refluxed for 6 h, concentrated HCl (1 ml) was added, and refluxing continued for 6 h. The product was diluted with an equal amount of water and NaOH solution (40%) added until complete separation of the Mannich free base. The product was extracted with ether and the extract washed with water and dried with $MgSO_4$. After drying the filtrate, the ether solution was treated with dry HCl gas and the resultant precipitate was washed with dry ether to give VI (15.5 g, 46%).

3-(1,4-Benzodioxan-6-yl)pyrazoline (VII). A mixture of methanol (16 ml), hydrazine hydrate solution (85%, 56 ml), and KOH (4.8 g) was heated to 60°C and a solution of Mannich base hydrochloride (VI, 36.4 g, 0.12 mole) in methanol added. The product was refluxed for 20 h and excess hydrazine hydrate and methanol removed to give pyrazoline VII* (23.5 g, 95%).

6-Cyclopropyl-1,4-benzodioxane (VIIIa). A. Finely dispersed metallic sodium (33 g) was added to peroxide free ether (200 ml). Dichloride Va (44 g, 0.18 mole) in a mixture of methanol (48 ml) and dry ether (20 ml) was added dropwise with vigorous stirring to the sodium suspension at a rate such that the ether refluxed vigorously in the reaction flask. When the dichloride had been added a further 10 ml of methanol was added dropwise and the mixture stirred until complete solution of sodium (3-5 h). The product was treated with water (~250 ml), adding it initially dropwise. The formed clear ether solution was separated. The aqueous layer was extracted with ether (3 × 50 ml). The ether solution was washed with water (3 × 60 ml) and dried with K_2CO_3. The solvent was evaporated and the residue distilled in vacuo to give 6-cyclopropyl-1,4-benzodioxane (VIIIa, 28.1 g, 84%).

B. Pyrazoline VII (23 g) was placed in a Wurtz flask and finely powdered KOH (0.3 g) added. The product was heated to 200-250°C. At the end of the vigorous evolution of gas, the mixture was cooled and extracted with ether. Passage of the solution through an Al_2O_3 layer, evaporation of the solvent, and distillation in vacuo gave VIIIa (14.7 g, 74% based on the Mannich base).

6-(1-Methylcyclopropyl)-1,4-benzodioxane (VIIIb) was obtained by reduction with metallic sodium in a methanol–ether mixture similarly to that described above for reduction of Va. Dichlorocyclopropane Vb (9 g, 0.035 mole), sodium (6.7 g), and methanol (15 ml) gave VIIIb (5.4 g, 78%).

Nitration of 6-Cyclopropyl-1,4-benzodioxane (VIIIa) by 55% HNO_3 in Acetic Anhydride. A solution of VIIIa (3.5 g, 0.02 mole) in acetic anhydride (15 ml) at −30°C was added to a nitrating mixture prepared at −30°C from HNO_3 (d = 1.34, 2.15 ml) and acetic anhydride (40 ml). The temperature of the mixture was raised to −10°C and stirred for 2.5 h. The product was poured into water and the slight yellow crystals filtered, washed three times on the filter with water, and recrystallized from alcohol to give XIIIa (3.8 g, 88%).

Similarly, VIIIb (1.9 g, 0.01 mole) gave XIIIb (1.6 g, 66%).

*Compound not isolated but underwent immediate catalytic decomposition.

Nitration of 6-Cyclopropyl-1,4-benzodioxane (VIIIa) by 100% HNO_3 in Acetic Anhydride. The reaction was carried out similarly to that for VIIIa with 55% HNO_3, the only differences being the use of 100% HNO_3 (d = 1.51) and reaction at −50°C. VIIIa (5.3 g, 0.03 mole) gave XIIIa (5.4 g, 82%).

Isomerization of 7-Nitro-6-cyclopropyl-1,4-benzodioxane (XIIIa) in Concentrated H_2SO_4. XIIIa (2.2 g, 0.01 mole) was gradually added with stirring to conc. H_2SO_4 (15 ml) at −30°C. The temperature of the mixture was raised to −10°C and stirring continued for 1 h. The product was poured into a mixture of ice (40 g) and water (100 ml). The crystals produced were extracted with chloroform (2 × 30 ml), the chloroform solution washed with water to neutral reaction, and dried ($MgSO_4$). Evaporation of solvent and recrystallization of the residue from alcohol gave XIV (1.41 g, 64%). IR Spectrum: 1685 cm^{-1} (C=O). UV Spectrum: λ_{max} 742 nm (ε = 34, N=O).

3-Ethyl-5,6-ethylenedioxybenz[c]isoxazole (XV). A solution of sodium bisulfite (40%, 15 ml) was added to a solution of XIV (2.2 g, 0.01 mole) in ethanol (40 ml) at 50°C. The product was stirred at this temperature for 1 h, the alcohol distilled off, and the product extracted with CH_2Cl_2. The extract was washed with water, dried with Na_2CO_3, and the solvent evaporated to give XV (1.4 g, 68%).

Nitration of 7-Nitro-6-cyclopropyl-1,4-benzodioxane (XIIIa) by 100% HNO_3 in Acetic Anhydride was carried out as described above but at −30°C, XIIIa (4.4 g, 0.02 mole) gave the dinitro product XVIII (4.7 g, 88%).

Isomerization of 7,8-Dinitro-6-cyclopropyl-1,4-benzodioxane (XVIII) in Concentrated H_2SO_4. The reaction was carried out as described above for XIIIa but at 0-5°C and for 2 h. XVIII (2.65 g, 0.01 mole) gave the nitro/nitroso product XIX (1.82 g, 69%). IR spectrum: 1690 cm^{-1} (C=O), UV spectrum: λ_{max} 744 nm (ε = 32, N=O).

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