B. From Isopropylidenepyridoxol.—Isopropylidenepyridoxol (2.85 g.), oxidized with potassium permanganate (3.0 g.) under reaction conditions similar to those in A, gave 1.35 g. (44%) of the acid, m.p. 220–221° dec.

α^4,3-O-Isopropylidene-5-pyridoxic Acid Methyl Ester (VI).—Isopropylidene-5-pyridoxic acid (5.0 g., 2.24 mmoles) in tetrahydrofuran (500 ml.) was treated with an excess of diazomethane in ether. After standing for 4 hr., the solvent was evaporated *in vacuo*, and the residue was extracted three times with ether. The combined ether extracts were evaporated and yielded 4.63 g. (87%) of the methyl ester, m.p. 85–87°. The analytical sample was crystallized from ethanol and had m.p. 86.5–87°; λ_{max}^{EtOH} 296 mμ (ε 6.3 × 10³). $\lambda_{max}^{0.1\ N\ HCl}$ 301 mμ (ε 8.5 × 10³); after heating for 3 hr. on steam bath (85°), λ_{max} 291 mμ (ε 8.0 × 10³) and 252 (4.4 × 10³).

Anal. Calcd. for $C_{12}H_{15}O_4N$: C, 60.75; H, 6.37; N, 5.90; OCH_3, 13.0. Found: C, 61.06; H, 6.57; N, 5.89; OCH_3, 12.96.

α^4,3-O-Isopropylidene-5-pyridoxamide (VII).—Isopropylidene-5-pyridoxic acid methyl ester (1.0 g., 4.2 mmoles) was suspended in aqueous ammonia (10 ml.), which was saturated at 0° with additional NH_3 and kept in a stoppered tube at room temperature for 7 days. After evaporation *in vacuo* and crystallization from ethanol, 0.85 g. (92%) of the amide, m.p. 174–175°, was obtained; λ_{max}^{EtOH} 290 mμ (ε 5.1 × 10³); $\lambda_{max}^{0.1\ N\ NaOH}$ 288 mμ (ε 5.2 × 10³). $\lambda_{max}^{0.1\ N\ HCl}$ 295 mμ (ε 7.4 × 10³); after heating for 2 hr. on the water bath, the spectrum was identical to that of 5-pyridoxic acid lactone.

Anal. Calcd. for $C_{11}H_{14}O_3N_2$: C, 59.45; H, 6.35; N, 12.60. Found: C, 59.83; H, 6.65; N, 12.39.

α^4,3-*O*-Isopropylidene-5-pyridoxic Acid Hydrazide (VIII).—The methyl ester (1.15 g., 4.88 mmoles) was dissolved in 10 ml. of 50% aqueous methanol, hydrazine (10 g.) was added, and the reaction mixture was heated on a steam bath for 2 hr. Evaporation of the volatiles *in vacuo* yielded a solid, which was crystallized from ethanol and yielded 0.87 g. (75%) of the hydrazide, m.p. 175° dec.; λ_{max}^{EtOH} 289 mμ (ε 6.6 × 10³); $\lambda_{max}^{0.1\ N\ NaOH}$ 298 mμ (ε 8.6 × 10³).

Anal. Calcd. for $C_{11}H_{15}O_3N_3$: C, 55.68; H, 6.37; N, 17.71. Found: C, 55.81; H, 6.74; N, 17.21.

Lactone of 5-Pyridoxic Acid (IX).—Isopropylidene-5-pyridoxic acid (1.01 g., 4.52 mmoles) was dissolved in 15% aqueous acetic acid (150 ml.) and heated on a steam bath for 3 hr. After evaporation and crystallization from ethanol, 0.68 g. (91%) of the lactone, m.p. 265–270° dec., was obtained. After recrystallization from ethanol, m.p. 275–278° dec. (lit.[10] m.p. 272–273°, 268–269° dec.).

Anal. Calcd. for $C_8H_7O_3N$: C, 58.18; H, 4.27; N, 8.48. Found: C, 58.36; H, 4.53; N, 8.30.

5-Pyridoxamide (X).—The lactone (0.57 g., 3.45 mmoles) was treated with a large excess of liquid ammonia at room temperature for 30 hr. The excess ammonia was allowed to evaporate, and the solid residue was crystallized from methanol, yielding 0.46 g. (73%) of the amide, m.p. 273–275° dec.; λ_{max}^{EtOH} 290 mμ (ε 5.8 × 10³); $\lambda_{max}^{0.1\ N\ NaOH}$ 314 mμ (ε 6.6 × 10³) and 250 (sh) (5.1 × 10³); $\lambda_{max}^{0.1\ N\ HCl}$ 293 mμ (ε 9.2 × 10³).

Anal. Calcd. for $C_8H_{10}O_3N_2$: C, 52.74; H, 5.53; N, 15.38. Found: C, 52.97; H, 5.67; N, 15.32.

5-Pyridoxic Acid Hydrazide (XI).—5-Pyridoxic acid lactone (0.254 g., 1.54 mmoles) was suspended in water (20 ml.), and hydrazine (1.0 g.) was added. The resulting clear solution was kept on a steam bath for 1 hr. and was evaporated to dryness. The solid residue was redissolved in an ethanol–ether–benzene mixture and was repeatedly evaporated to remove traces of hydrazine. Finally, the solid was suspended in ethanol, filtered, and washed with ether. The yield was 0.231 g. (76%) of the hydrazide, m.p. 252–255° dec. An analytical sample was obtained by crystallization from a dimethylformamide–ethanol mixture.

Anal. Calcd. for $C_8H_{11}O_3N_3$: C, 48.72; H, 5.62; N, 21.31. Found: C, 48.84; H, 5.73; N, 21.39.

Tri-O-acetylpyridoxol Hydrobromide.—To a solution of pyridoxol hydrochloride (40 g.) in dry pyridine (150 ml.) 70 ml. of acetic anhydride was added, and the mixture was shaken until a clear solution was obtained. After standing overnight at room temperature, excess solvent was evaporated under reduced pressure. Water was added and the mixture was allowed to stand at 0° for 4 hr. to hydrolyze acetic anhydride. The solution was now flash evaporated; the process was repeated several times to remove most of the acetic acid. The residue was made alkaline with sodium carbonate and extracted three times with ether. Removal of the solvent gave a viscous residue which was dissolved in methanol and to which aqueous hydrogen bromide was added, yielding the hydrobromide (79.0 g., 90%) in colorless needles, m.p. 155°.

Anal. Calcd. for $C_{14}H_{15}NO_6 \cdot HBr$: C, 44.93; H, 4.31; N, 3.74; Br, 21.40. Found: C, 44.72; H, 4.52; Br, 21.61.

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Quinoxaline Studies. XII. Stereodirective Syntheses of *cis*- and *trans*-Decahydroquinoxalines and *cis*- and *trans*-Decahydroquinoxalones-2[1,2]

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5,6,7,8-Tetrahydroquinoxalone-2 (I) was prepared in 64% yield by condensing 1,2-cyclohexanedione with glycinamide in aqueous methanol–sodium hydroxide solution. I was catalytically reduced with platinum to give 100% yield of *cis*-decahydroquinoxalone-2, chemically reduced with sodium–amyl alcohol to give 58% yield of only *trans*-decahydroquinoxaline, and electrolytically reduced in aqueous sulfuric acid at a lead cathode to give 8% yield of *trans*-decahydroquinoxaline plus 13% yield of *trans*-decahydroquinoxalone-2. *cis*-Decahydroquinoxaline was prepared in 12% yield by the catalytic reduction over platinum of an aqueous solution of 1,2-cyclohexanedione and ethylenediamine, as was *trans*-decahydroquinoxaline in 36% yield by a similar reduction of an ethanol solution of *trans*-1,2-cyclohexanediamine and glyoxal. Also reported is a study of the ratios of *cis*- and *trans*-decahydroquinoxalones-2 and *cis*- and *trans*-decahydroquinoxalines obtained in certain syntheses.

Relatively large quantities of the two geometrical isomers of decahydroquinoxaline were needed for complexation studies. The unequivocal syntheses of *cis*- and *trans*-decahydroquinoxalines, the corresponding decahydroquinoxalones-2, and decahydroquinoxaline-diones-2,3 were described in the preceding paper.[2] The purpose of this paper is to report syntheses of *cis*- and *trans*-decahydroquinoxalines, *via* other synthetic routes more efficacious for the preparation of large amounts of materials.

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(2) Paper XI of this series: E. Brill and H. P. Schultz, *J. Org. Chem.*, **28**, 1135 (1963).

TABLE I

HYDROGENATION STUDIES OF 5,6,7,8-TETRAHYDROQUINOXALONE-2[a] TO DECAHYDROQUINOXALONE-2

Catalyst	Solvent	Total % yield of isomers obtained: *cis*	*trans*
Adams PtO_2	Ethanol, absolute	100	0
Adams PtO_2	Ethanol–HCl, *ca.* 5%	80	20
Adams PtO_2	Acetic acid, glacial	100	0
Pt black	Ethanol, absolute	No reduction	
Pt black	Ethanol–HCl, *ca.* 5%	No reduction	
Raney Ni, W-2	Ethanol–water, 50%; *ca.* 1 g. of NaOH	No reduction	
Raney Ni, W-2	Ethanol, absolute, *ca.* 3 g. of NaOEt (from 1 g. of Na)	No reduction	
Raney Ni, W-2	Water, *ca.* 1 g. of NaOH	No reduction	
Raney Ni, W-2	Acetic acid, glacial	No reduction	
Raney Ni, W-2	Ethanol, absolute	94.5	0
Raney Ni, W-2	Ethanol, absolute; *ca.* 1 g. of NaOH	80	20
Pd–charcoal, 5%	Ethanol–HCl, *ca.* 5%	No reduction	
Pd–charcoal, 5%	Ethanol, absolute	91	0

[a] All reductions: 4–14 g. of 5,6,7,8-tetrahydroquinoxalone-2 in 50–100 ml. of solvent at 50–70°, 50–80 p.s.i.; all platinum catalysts, 500 mg.; all nickel catalysts, *ca.* 1 g.; all palladium catalysts, 500 mg.

5,6,7,8-Tetrahydroquinoxalone-2 was prepared by condensing 1,2-cyclohexanedione with glycinamide. The stability of the product, as well as its ultraviolet absorption spectra, indicated its aromatic character. In spite of the observation that 5,6,7,8-tetrahydroquinoxalone-2 was soluble in 10% sodium hydroxide and concentrated ammonium hydroxide solutions (as well as in 10% hydrochloric acid solution), the absence of a bathochromic shift in the ultraviolet absorption spectrum in sodium hydroxide solution and the absence of a ferric chloride phenol test indicated no phenolic hydroxyl group in the molecule. A similar conclusion was reached from similar data by Ewing and Steck[3] in their studies of the structure of 2-quinolone and related compounds.

The catalytic reduction of 5,6,7,8-tetrahydroquinoxalone-2 under appropriate conditions gave *cis*-decahydroquinoxalone-2 quantitatively. The various reduction conditions investigated are given in Table I. As earlier reported,[2] both geometrical isomers of decahydroquinoxalone-2 have been reduced to the corresponding decahydroquinoxalines with lithium aluminum hydride.

Chemical reduction of 5,6,7,8-tetrahydroquinoxalone-2 was successful with sodium and amyl alcohol; only *trans-dl*-decahydroquinoxaline was obtained. Sodium and ethanol failed to reduce 5,6,7,8-tetrahydroquinoxalone-2.

The electrochemical reduction of 5,6,7,8-tetrahydroquinoxalone-2 was studied, with special emphasis on the stereochemical path of reduction—virtually a virgin field of study.[4] Only high purity lead cathodes in sulfuric acid solution effected electrolytic reduction of 5,6,7,8-tetrahydroquinoxalone-2. Passage of the theoretical amount of current through the reduction cell gave a mixture of *trans*-decahydroquinoxalone-2 and *trans*-decahydroquinoxaline: yields varied considerably, never exceeding a total of 20%. When twice the theoretical amount of current was used, a 10% yield of only the *trans*-decahydroquinoxaline resulted. Large amounts of tars formed in all electrolytic reductions. It is significant that no *cis* geometrical isomer was isolated, although chromatographic procedures were used in the purification of the reduction product.

Broadbent, *et al.*,[5] condensed *trans*-1,2-cyclohexanediamine with glyoxal, and, without isolating the intermediate *trans*-hexahydroquinoxaline, reduced the reaction mixture over rhodium–alumina catalyst at 200° and 2000 p.s.i. to give a 20% yield of *trans*-decahydroquinoxaline. Our studies of this method of preparation of *trans*-decahydroquinoxaline utilized a more active catalyst, with the resultant lower temperature and pressure decreasing the possibility of isomerization. The yield of *trans*-decahydroquinoxaline doubled when platinum oxide was used as a reduction catalyst at 70° and 80 p.s.i. Purification of the reduction product by chromatographic means, rather than vacuum sublimation, yielded the above plus a 1% yield of *trans*-decahydroquinoxalone-2—the presence of which can be most easily attributed to air oxidation of the intermediate hexahydroquinoxaline prior to its reduction.

Attempts to prepare *cis*-decahydroquinoxaline by condensation of *cis*-1,2-cyclohexanediamine with glyoxal followed by reduction using conditions outlined in the preceding paragraph, resulted in failure; the reaction solution turned black at once and no hydrogen was absorbed. Only black, irresolvable tar was obtained, indicating that extensive polymerization and decomposition had occurred.

The above preparative objective was attained, however, when 1,2-cyclohexanedione was condensed with ethylenediamine, followed by immediate reduction of the reaction mixture without isolating the intermediate hexahydroquinoxaline, giving a 12% yield of *cis*-decahydroquinoxaline. Much tar was formed. No *trans*-decahydroquinoxaline was isolated, although the reduction product was purified by chromatographic separation.

Experimental

The infrared spectra were determined on a Baird Atomic Model KM I spectrophotometer at concentrations of 30 mg./ml. of chloroform. The ultraviolet absorption spectra were determined with both a Beckman quartz spectrophotometer Model DU and a Bausch and Lomb Spectronic 505 spectrophotometer at concentrations of 10 mg./l. of solvent.

5,6,7,8-Tetrahydroquinoxalone-2.—A solution of 25.4 g. of 1,2-cyclohexanedione[6] in 40 ml. of methanol was added dropwise

(3) G. W. Ewing and E. A. Steck, *J. Am. Chem. Soc.*, **68**, 2181 (1946).
(4) F. D. Popp and H. P. Schultz, *Chem. Rev.*, **62**, 19 (1962).
(5) H. S. Broadbent, E. L. Allred, L. Pendleton, and C. W. Whittle, *J. Am. Chem. Soc.*, **82**, 189 (1960).
(6) R. T. Arnold, *Org. Syn.*, **32**, 35 (1952).

to a cold (−10°) solution of 25 g. of glycinamide hydrochloride[7] in 40 ml. of water in a three-neck, round-bottom flask equipped with a stirrer. Ten milliliters of 12.5 *N* sodium hydroxide–water solution was then added dropwise. (It was observed that, if the temperature rose above 0° during these additions, yield of product was drastically reduced.) The reaction mixture was stored at −10° for 12 hr., after which hydrochloric acid, Sp. Gr. 1.18, was added to the solution in 5-ml. portions until the solution was slightly acid to litmus paper. The precipitate was filtered with suction and rinsed with ethyl ether to give 25.4 g. of yellow crystalline product, m.p. 270–278°. Three recrystallizations from absolute ethanol (40 ml./g.) afforded 22.1 g. (64%) of white 5,6,7,8-tetrahydroquinoxalone-2, m.p. 275–276°; $\lambda_{max}^{95\%\ EtOH}$ 228.5 mμ (ε 6450), 337 (6890); $\lambda_{max}^{10\%\ NaOH}$ 233 mμ (ε 8900), 331 (8000); $\lambda_{max}^{10\%\ HCl}$ 228 mμ (ε 11,580), 368 (9760). The compound was insoluble in 10% aqueous sodium bicarbonate and sodium carbonate, but soluble in 10% sodium hydroxide, concentrated ammonium hydroxide, and hydrochloric acid solutions.

Anal. Calcd. for $C_8H_{10}N_2O$: C, 64.0; H, 6.68; N, 18.7. Found: C, 64.0; H, 6.71; N, 18.6.

cis-**Decahydroquinoxalone-2. A. By Catalytic Reduction of 5,6,7,8-Tetrahydroquinoxalone-2 over Platinum Oxide Catalyst.**—Fourteen grams of 5,6,7,8-tetrahydroquinoxalone-2, 100 ml. of absolute ethanol, and 0.5 g. of Adams platinum oxide catalyst[8] were reduced in a Parr hydrogenation apparatus for 4 hr. at 50° and 80 p.s.i. After removal of the catalyst by filtration, the solvent was evaporated to give 14.4 g. (100%) of *cis*-decahydroquinoxalone-2, m.p. 132–136°. Recrystallization from benzene gave 13.6 g. (94.6%) of product, m.p. 135–136° (lit.[2] m.p. 135–136°). Mixture melting points and infrared spectra demonstrated that this product was identical with material produced by the reaction of *cis*-1,2-cyclohexanediamine with chloroacetic acid, as described in the previous paper.[2]

B. By Catalytic Reduction of 5,6,7,8-Tetrahydroquinoxalone-2 over W-2 Raney Nickel Catalyst.—Four grams of 5,6,7,8-tetrahydroquinoxalone-2, 50 ml. of absolute ethanol, and 1 g. of W-2 Raney nickel catalyst[9] were reduced in a Parr hydrogenation apparatus at 50° and 80 p.s.i. The product was 3.9 g. (94.5%) of *cis*-decahydroquinoxalone-2, m.p. 134–136°. Its identity was demonstrated as described above.

cis-**Decahydroquinoxaline. A. By Chemical Reduction of *cis*-Decahydroquinoxalone-2.**—This material was prepared by the reduction of *cis*-decahydroquinoxalone-2 with lithium aluminum hydride according to the procedure described in the previous paper.[2]

B. By Catalytic Reduction of 2,3,5,6,7,8-Hexahydroquinoxaline.—A 500-ml. Parr reduction bottle was charged with 11.2 g. of 1,2-cyclohexanedione[5] and a solution of 6.0 g. of ethylenediamine in 14 ml. of water.[6] After 5 min., 60 ml. of absolute ethanol and 0.6 g. of Adams platinum oxide catalyst were added. The mixture was hydrogenated for 6 hr. at 75° and 80 p.s.i.; 62.5% of the theoretical amount of hydrogen was absorbed by the reaction solution. The filtered reaction solution was evaporated to dryness, and the residue was triturated with 20% sodium hydroxide solution. Insoluble tars were removed by filtration. Organic material was extracted from the basic filtrates with five 20-ml. portions of ethyl ether. After drying the ether solution of amine with anhydrous sodium sulfate, the ether was removed and the residue was distilled to give 1.7 g. (12.1%) of white crystalline material, b.p. 85–88° (1 mm.), m.p. 54–56° (lit.[5] b.p. 85–87° (0.25 mm.), m.p. 56–58°). Mixture melting points, derivative melting points, and infrared spectra demonstrated that this product was identical with material obtained by the lithium aluminum hydride reduction of *cis*-decahydroquinoxaline-2,[2] as well as to that produced by Broadbent[5] by the rhodium–alumina reduction of quinoxaline.

trans-**Decahydroquinoxalone-2 and *trans*-Decahydroquinoxaline by Electrolytic Reduction of 5,6,7,8-Tetrahydroquinoxalone-2.**—A square sheet of 1/16-in. lead, 100 cm.², of 99.5% purity was cleaned in 1:1 nitric acid and prepared as a cathode by the modified Tafel procedure.[10] The lead sheet was rolled into a cylinder and inserted in a porous porcelain cup, 5 × 10 cm., which was in turn placed in a 600-ml. beaker. The anode was a cylindrical sheet of lead which surrounded the porcelain catholyte cup. The catholyte solution was prepared by dissolving 2 g. of 5,6,7,8-tetrahydroquinoxalone-2 in 100 ml. of 20% sulfuric acid; the anolyte solution was 20% sulfuric acid. This electrolytic cell, equipped with a magnetic stirrer, was assembled in the usual manner,[10] with an applied line potential of 3 v. delivered from a rectifying transformer power supply.

The reduction was executed at a current density of 0.02 amp./cm.², and the temperature of the cell was maintained at 30–40° by means of a water bath. After 1.4 hr., the theoretical reduction time, the catholyte was neutralized with solid sodium carbonate, then made strongly basic with 50% sodium hydroxide solution. This basic solution was extracted with four 20-ml. portions of chloroform; the chloroform solution in turn was washed with saturated brine, then dried with anhydrous sodium sulfate. The dry chloroform solution was poured onto a 1 × 20 cm. column of neutral alumina (Woelm, activity grade I), and the column was eluted with alcohol-free chloroform. Twenty 10-ml. fractions of the eluate were collected and evaporated to dryness. Determination of melting points and infrared spectra demonstrated that two products were obtained: 0.14 g. (7.5%) of *trans*-decahydroquinoxaline, m.p. 149–151°, was obtained in the first 15 fractions; 0.26 g. (12.7%) of *trans*-decahydroquinoxalone-2, m.p. 197–200°, was obtained in the last five fractions. The identities of the two products were established by means of mixture melting points and the comparison of infrared spectra with authentic samples.[2]

Twice the electrolytic reduction time at 0.02 amp./cm.² gave a 10% yield of only the *trans*-decahydroquinoxaline.

trans-**Decahydroquinoxaline. A. By Chemical Reduction of 5,6,7,8-Tetrahydroquinoxalone-2.**—A 500-ml., three-neck, round-bottom flask equipped with a stirrer and reflux condenser carrying a drying tube was charged with 300 ml. of freshly distilled isoamyl alcohol and 4 g. of 5,6,7,8-tetrahydroquinoxalone-2. Refluxing was begun, with 0.8 g. of sodium added over a 2-hr. period. Refluxing continued for an additional 6 hr. After cooling, 200 ml. of water was added to the reaction solution, and the layers were separated. The alcohol layer was extracted with four 30-ml. portions of 10% hydrochloric acid solution. The combined acid extracts were neutralized with sodium carbonate, then made strongly basic with 50% sodium hydroxide solution. This basic solution was then extracted with four 20-ml. portions of chloroform; the chloroform solution was washed with brine, then dried with anhydrous sodium sulfate. Removal of the chloroform *in vacuo* gave a white crystalline product still possessing an odor of isoamyl alcohol; no crude yield, therefore, was determined. The crude product was crystallized from ligroin (b.p. 66–75°) to yield 2.2 g. (58%) of *trans*-decahydroquinoxaline, m.p. 150–151° (lit.[5] m.p. 147–148°, lit.[11] m.p. 150–151°, lit.[12] m.p. 152.5–153°). The N,N-dinitroso derivative had m.p. 108–110° (lit.[11] m.p. 108–110°).

B. By Catalytic Reduction of *trans*-Hexahydroquinoxaline.—A 500-ml. Parr reduction bottle was charged with 3.85 g. of *trans*-1,2-cyclohexanediamine[13] and 6.5 g. of 30% aqueous solution of glyoxal. After 5 min., 60 ml. of absolute ethanol and 0.6 g. of Adams platinum oxide catalyst were added, and the solution was reduced at 75° and 80 p.s.i. The theoretical amount of hydrogen was absorbed in 8 hr. The solution was then filtered and evaporated to dryness, and the tarry residue was dissolved in ethyl ether. The resulting solution was poured onto a 1 × 20 cm. column of neutral alumina (Woelm, activity grade I), and the column was eluted with alcohol-free chloroform. Forty 10-ml. fractions of the eluate were collected, evaporated to dryness, and examined. The first 35 fractions consisted of 2 g. (42%) of *trans*-decahydroquinoxaline, m.p. 149–150°; recrystallized from ligroin (b.p. 66–75°), the final yield of product was 1.7 g. (35.6%), m.p. 150–151°.

In addition, the last five fractions yielded 100 mg. of product, m.p. 196–200°, with a strong infrared carbonyl peak. A mixture melting point with authentic *trans*-decahydroquinoxalone-2[2] (m.p. 199–199.5°) was 196–200°.

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