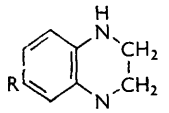
911. *The Synthesis of Some 6-Substituted 1,2,3,4-Tetrahydroquinoxalines.*

By P. Clarke and A. Moorhouse.

Some 6-substituted 1,2,3,4-tetrahydroquinoxalines have been prepared by cyclisation of the corresponding *o*-amino-*N*-2′-chloroethylanilines.

A new route to 1,2,3,4-tetrahydroquinoxaline (I; R = H) was described by Ramage and Trappe.[1] They obtained it and its 2-methyl and 6-nitro- (I; R = NO_2) derivatives by cyclisation of either *o*-amino-*N*-2′-hydroxy- or *o*-amino-*N*-2′-chloro-ethylaniline (II; R^1 = H, R^2 = NH_2, R^3 = OH or Cl). Routes from *o*-chloronitrobenzenes have now been examined in the preparation of 6-acetamido-, 6-bromo-, 6-chloro-, 6-methoxy-, 6-methoxycarbonyl-, and 6-methylthio-1,2,3,4-tetrahydroquinoxalines.

N-2′-Hydroxyethyl-*o*-nitroanilines (II; R^1 = H, R^2 = NO_2, R^3 = OH) gave the corresponding *N*-2′-chloroethyl compounds by treatment with thionyl chloride. Compounds with two deactivating groups in the ring, of type (II; R^1 = Cl; R^2 = NO_2; R^3 = OH) or (III; R^1 = Br, CO_2H, CO_2Me, or NO_2, R^2 = NO_2, R^3 = OH) or (IV; R^1 = NO_2, R^2 = OH), required heating for several hours with thionyl chloride containing a trace of pyridine. Compounds with two activating groups in the ring (II; R^1 = NHAc, OMe, or SMe; R^2 = NO_2; R^3 = OH) rapidly resinified in hot thionyl chloride, and in these cases a solution of the reagent in benzene at room temperature was used.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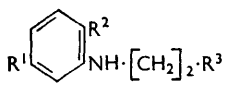
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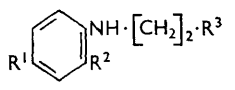
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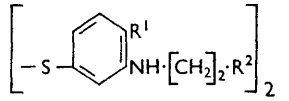
(IV)

The *N*-2′-chloroethyl compounds were reduced catalytically to the *o*-amino-*N*-2′-chloroethylaniline derivatives (II; R^1 = H, R^2 = NH_2, R^3 = Cl) which resinified rapidly in air but, when heated in alcohol under reflux, furnished the tetrahydroquinoxalines (I; R = NHAc, Br, Cl, MeO, CO_2Me, or SMe) in good yield.

Catalytic hydrogenation of the *N*-2′-hydroxyethyl-*o*-nitroanilines gave the corresponding *o*-diamines (II; R^1 = H, R^2 = NH_2, R^3 = OH), cyclisation of which was attempted by heating under reflux with hydrobromic acid, but only very small yields of the 1,2,3,4-tetrahydroquinoxalines (I; R = Br, Cl, or OMe) were obtained. Treatment of the *o*-diamines with thionyl chloride gave the *o*-amino-*N*-2′-chloroethylaniline derivatives (II; R^1 = H, R^2 = NH_2, R^3 = Cl). The crude products were heated under reflux in alcohol, and gave the 1,2,3,4-tetrahydroquinoxalines (I; R = Br, Cl, or OMe) in fair yield.

In the preparation of 6-chloro-1,2,3,4-tetrahydroquinoxaline, the required 5-chloro-*N*-2′-hydroxyethyl-2-nitroaniline [2] (II; R^1 = Cl, R^2 = NO_2, R^3 = OH) yielded a diacetyl derivative by treatment with acetic anhydride containing a trace of sulphuric acid. Use of acetic anhydride, alone or with sodium acetate, gave a monoacetyl derivative (II; R^1 = Cl, R^2 = NO_2, R^3 = OAc) and this yielded an *O*-acetyl-*N*-nitroso-compound by treatment with nitrous acid.

6-Bromo-1,2,3,4-tetrahydroquinoxaline was derived from 4-bromo-*N*-2′-hydroxyethyl-2-nitroaniline (III; R^1 = Br, R^2 = NO_2, R^3 = OH) which was obtained by bromination of *N*-2′-hydroxyethyl-*o*-nitroaniline—a more convenient procedure than the condensation of 1,4-dibromo-2-nitrobenzene with 2-aminoethanol.[3]

[1] Ramage and Trappe, *J.*, 1952, 4406.
[2] Kremer and Meltsner, *J. Amer. Chem. Soc.*, 1942, **64**, 1285.
[3] Waldkötter, *Rec. Trav. chim.*, 1938, **57**, 1294; Feitelson, Mamalis, Moualim, Petrow, Stephenson, and Sturgeon, *J.*, 1952, 2389.

In the preparation of 6-acetamido-1,2,3,4-tetrahydroquinoxaline (I; R = NHAc) the required 3-chloro-4-nitroacetanilide was obtained by nitration of *m*-chloroacetanilide. This nitro-compound was conveniently separated from the 5,2-isomer by crystallisation; earlier methods [4] involved hydrolysis, steam-distillation, and re-acetylation.

Aqueous alcoholic sodium disulphide reacted under mild conditions with 5-chloro-*N*-2′-hydroxyethyl-2-nitroaniline to give 3,3′-di-(2-hydroxyethylamino)-4,4′-dinitrodiphenyl disulphide (IV; $R^1 = NO_2$, $R^2 = OH$). With some other nucleophilic reagents (aqueous potassium hydroxide, methanolic potassium methoxide, aqueous ammonia with cuprous oxide, and aniline with copper or cuprous iodide) there was no reaction under mild conditions but more severe conditions caused extensive decomposition.

N-2′-Chloroethyl-5-methylthio-2-nitroaniline (II; $R^1 = SMe$, $R^2 = NO_2$, $R^3 = Cl$) was available either by methylation of the thiol (II; $R^1 = SH$, $R^2 = NO_2$, $R^3 = Cl$) obtained by reduction of 3,3′-di-(2-chloroethylamino)-4,4′-dinitrodiphenyl disulphide (IV; $R^1 = NO_2$, $R^2 = Cl$), or by the action of thionyl chloride on *N*-2′-hydroxyethyl-5-methylthio-2-nitroaniline (II; $R^1 = SMe$, $R^2 = NO_2$, $R^3 = OH$).

The 6-acetamido-derivative (I; R = NHAc) could not be purified because of its rapid darkening in air and so it was characterised as the diacetyl derivative, which proved identical with the compound obtained by acetylation of the reduction product from either 1,2,3,4-tetrahydro-6-nitroquinoxaline (I; $R = NO_2$) or its monoacetyl derivative.

Experimental

N-2′-*Hydroxyethyl*-o-*nitroanilines*.—(*a*) A mixture of 2,4-dichloro-1-nitrobenzene (0·1 mole), 2-aminoethanol (0·2 mol.), and butanol (40 ml.) was heated under reflux for 12 hr. The solvent was then evaporated under reduced pressure and the residue, after being diluted with water, gave 5-chloro-*N*-2′-hydroxyethyl-2-nitroaniline, orange plates (67%), m. p. 119° (from benzene) (Found: C, 44·3; H, 4·3. Calc. for $C_8H_9ClN_2O_3$: C, 44·4; H, 4·2%).[2] Acetylation (i) by heating under reflux with acetic anhydride gave N-2′-*acetoxyethyl*-5-*chloro*-2-*nitroaniline*, yellow-green prisms, m. p. 114° (from light petroleum) (Found: C, 45·8; H, 4·5. $C_{10}H_{11}ClN_2O_4$ requires C, 46·4; H, 4·3%), (ii) by warming to 60° for 15 min. with acetic anhydride and a trace of sulphuric acid gave the diacetyl derivative [3] as yellow-green prisms, m. p. 93° (from light petroleum) (Found: C, 47·8; H, 4·4. Calc. for $C_{12}H_{13}ClN_2O_5$: C, 47·9; H, 4·4%). By procedure (*a*) above, 3-chloro-4-nitroacetanilide yielded 5-*acetamido*-N-2′-*hydroxyethyl*-2-*nitroaniline* (56%), orange plates, m. p. 180° (from water) (Found: C, 50·1; H, 5·6. $C_{10}H_{13}N_3O_4$ requires C, 50·2; H, 5·5%). 3-Chloro-4-nitroanisole [5] likewise gave N-2′-*hydroxyethyl*-5-*methoxy*-2-*nitroaniline* (69%), orange plates, m. p. 109° (from ethanol) (Found: C, 51·1; H, 5·9. $C_9H_{12}N_2O_4$ requires C, 50·9; H, 5·7%). Methyl 4-chloro-3-nitrobenzoate, in methanol instead of butanol, gave *methyl* 4-2′-*hydroxyethylamino*-3-*nitrobenzoate* (77%), orange needles, m. p. 104° (from methanol) (Found: C, 49·8; H, 5·1; N, 12·1. $C_{10}H_{12}N_2O_5$ requires C, 50·0; H, 5·0; N, 11·7%).

(*b*) A mixture of 4-chloro-3-nitrobenzoic acid (0·1 mole), 2-aminoethanol (0·2 mole), and water (100 ml.) was heated under reflux for 6 hr. and then cooled and acidified. The resulting 4-2′-*hydroxyethylamino*-3-*nitrobenzoic acid* (66%) formed yellow needles, m. p. 209° (from water) (Found: C, 47·8; H, 4·5; N, 11·8. $C_9H_{10}N_2O_5$ requires C, 47·8; H, 4·5; N, 12·4%), which with hot acetic anhydride, gave 4-2′-*acetoxyethylamino*-3-*nitrobenzoic acid*, m. p. 183° (Found: C, 49·3; H, 4·3; N, 9·9. $C_{11}H_{12}N_2O_6$ requires C, 49·3; H, 4·5; N, 10·4%). This compound was also obtained on mixing 4-2′-chloroethylamino-3-nitrobenzoic acid with acetic anhydride containing a trace of sulphuric acid.

(*c*) 2-Benzylaminoethanol (0·1 mole) in ethanol (10 ml.) was added slowly to a solution of 1-chloro-2,4-dinitrobenzene (0·05 mole) in ethanol (15 ml.) at 60°. The mixture was set aside for 12 hr., heated under reflux for 2 hr., and diluted with water to give N-*benzyl*-N-2′-*hydroxyethyl*-2,4-*dinitroaniline* (82%), yellow needles, m. p. 112° (from benzene) (Found: C, 56·6; H, 4·8. $C_{15}H_{15}N_3O_5$ requires C, 56·8; H, 4·8%).

[4] Mayes and Turner, *J.*, 1928, 691; Hodgson and Kershaw, *J.*, 1929, 2917.
[5] Hodgson and Handley, *J.*, 1926, 542.

4-Bromo-N-2′-hydroxyethyl-2-nitroaniline.—*N*-2′-Hydroxyethyl-*o*-nitroaniline [1] (1·8 g.) in ethanol (2 ml.) was treated with a slight excess of 3% bromine water at 20°. The resulting bromo-compound (III; $R^1 = Br$, $R^2 = NO_2$, $R^3 = OH$) (84%) formed yellow needles, m. p. 106° (from benzene) (Found: C, 36·8; H, 3·6. Calc. for $C_8H_9BrN_2O_3$: C, 36·8; H, 3·5%).

3,3-Di-(2-hydroxyethylamino)-4,4′-dinitrodiphenyl Disulphide.—5-Chloro-*N*-2′-hydroxyethyl-2-nitroaniline (4·3 g.) in ethanol (20 ml.) was heated under reflux whilst a solution of sulphur (0·35 g.) and sodium sulphide (2·5 g.) in water (17 ml.) was added during 2 hr., and the mixture was then heated for another 2 hr. The resulting *disulphide* (3·9 g.) crystallised from butanol as yellow prisms (3·6 g.), m. p. 215° (Found: C, 44·9; H, 4·1. $C_{16}H_{18}N_4O_6S_2$ requires C, 45·1; H, 4·3%).

N-2′-Hydroxyethyl-5-methylthio-2-nitroaniline.—A mixture of the foregoing disulphide derivative (4·3 g.), ethanol (20 ml.), sodium sulphide (1·9 g.), sodium hydroxide (4·4 g.), and water (20 ml.) was heated on the steam-bath for ½ hr. and the resulting crimson solution was diluted with water (70 ml.). The filtrate was mixed with dimethyl sulphate (5 ml.) and warmed (60°) for 2 hr. The *product* (4·2 g.) formed orange needles, m. p. 106° (from benzene) (Found: C, 47·1; H, 5·0; S, 13·9. $C_9H_{12}N_2O_3S$ requires C, 47·4; H, 5·3; S, 14·0%).

N-2′-Chloroethyl-o-nitroanilines.—(*a*) 5-Chloro-*N*-2′-hydroxyethyl-2-nitroaniline (0·01 mole) was heated under reflux with distilled thionyl chloride (10 ml.) for 6 hr., and the excess of the reagent was removed under reduced pressure. The residue was mixed with water and gave *5-chloro-N-2′-chloroethyl-2-nitroaniline* (80%), yellow plates, m. p. 83° (from light petroleum) (Found: C, 40·8; H, 3·6. $C_8H_8Cl_2N_2O_2$ requires C, 40·9; H, 3·4%). Similarly, the following compounds were obtained from the corresponding *N*-2′-hydroxyethyl-*o*-nitroanilines: *4-Bromo-N-2′-chloroethyl-2-nitroaniline* (82%), orange needles, m. p. 86° (from light petroleum) (Found: C, 34·8; H, 3·0. $C_8H_8BrClN_2O_2$ requires C, 34·4; H, 2·9%). *Methyl 4-2′-chloroethylamino-3-nitrobenzoate* (73%), yellow needles, m. p. 83° (from methanol) (Found: C, 46·7; H, 4·2; N, 10·5. $C_{10}H_{11}ClN_2O_4$ requires C, 46·4; H, 4·3; N, 10·8%). This compound was also available (87%) from 4-2′-hydroxyethylamino-3-nitrobenzoic acid by heating with methanol the residue which was left after removal of the excess of thionyl chloride. This same residue was heated under reflux with water for 2 hr. and gave *4-2′-chloroethylamino-3-nitrobenzoic acid* (72%), yellow needles, m. p. 210° (from acetic acid) (Found: C, 44·5; H, 3·9; N, 10·9. $C_9H_9ClN_2O_4$ requires C, 44·2; H, 3·7; N, 11·5%).

(*b*) *N*-2′-Hydroxyethyl-5-methoxy-2-nitroaniline (0·01 mole) was mixed with benzene (20 ml.), thionyl chloride (2 ml.), and pyridine (1 drop) and set aside for 24 hr. The product was isolated as in (*a*) and gave *N-2′-chloroethyl-5-methoxy-2-nitroaniline* (86%) as yellow prisms, m. p. 94° (from ethanol) (Found: C, 46·7; H, 5·0. $C_9H_{11}ClN_2O_3$ requires C, 46·9; H, 4·8%). Similarly, the corresponding hydroxy-compounds gave *N-2′-chloroethyl-5-methylthio-2-nitroaniline* (83%), orange needles, m. p. 95° (from benzene) (Found: C, 44·0; H, 4·5. $C_9H_{11}ClN_2O_2S$ requires C, 43·8; H, 4·5%), and *5-acetamido-N-2′-chloroethyl-2-nitroaniline* (60%), yellow prisms, m. p. 154° (from benzene) (Found: C, 46·7; H, 4·8. $C_{10}H_{12}ClN_3O_3$ requires C, 46·6; H, 4·7%).

(*c*) 3,3′-Di-(2-hydroxyethylamino)-4,4′-dinitrodiphenyl disulphide (4·3 g.) was heated under reflux for 2 hr. with the reagent solution (50 ml.) as used in (*b*) and gave *3,3′-di-(2-chloroethylamino)-4,4′-dinitrodiphenyl disulphide* (84%), yellow prisms, m. p. 187° (from acetic acid) (Found: C, 41·7; H, 3·7. $C_{16}H_{16}Cl_2N_4O_4S_2$ requires C, 41·5; H, 3·5%). This compound (4·6 g.) was added to a solution of sodium sulphide (2 g.), sodium hydroxide (4·6 g.), ethanol (20 ml.), and water (20 ml.). The mixture was kept at 60° for 5 min., cooled, and diluted with water (90 ml.). The filtrate was shaken with dimethyl sulphate (6 ml.) and yielded a solid which crystallised from benzene as orange needles (68%), m. p. 95° alone and mixed with the foregoing *N*-2′-chloroethyl-5-methylthio-2-nitroaniline.

1,2,3,4-Tetrahydroquinoxalines from N-2′-Chloroethyl-o-nitroanilines.—5-Chloro-*N*-2′-chloroethyl-2-nitroaniline (0·01 mole) was shaken under hydrogen with Raney nickel (2 ml. of settled suspension) in ethanol (100 ml.). The reduced product was filtered, diluted with ethanol (200 ml.), and heated under reflux for 30 hr. Solvent was then evaporated and the residue, after being treated with saturated aqueous potassium carbonate, gave an oil which was extracted with hot water. The cooled extracts deposited 6-chloro-1,2,3,4-tetrahydroquinoxaline [6] which crystallised from aqueous solution (charcoal) as plates (52%), m. p. 114°, b. p. 160°/0·5 mm. The same procedure with the corresponding nitro-compounds (II; R^1 = OMe, SMe, or NHAc;

$R^2 = NO_2$, $R^3 = Cl$) or (III; R^1 = Br or CO_2Me, $R^2 = NO_2$, $R^3 = Cl$) gave: (a) 1,2,3,4-tetrahydro-6-methoxyquinoxaline [6] (45%), needles, m. p. 81° (from light petroleum), (b) 1,2,3,4-*tetrahydro-6-methylthioquinoxaline* (60%), needles, m. p. 65° (from light petroleum) (Found: C, 60·2; H, 6·3. $C_9H_{12}N_2S$ requires C, 60·0; H, 6·7%). This base yielded a *picrate*, orange needles, m. p. 158° (decomp.) (from 10% aqueous acetic acid) (Found: C, 44·4; H, 3·7. $C_{15}H_{15}N_5O_7S$ requires C, 44·0; H, 3·7%), and a *hydrochloride*, buff prisms, m. p. 185° (from acetone) (Found: C, 49·0; H, 5·8. $C_9H_{12}N_2S$,HCl requires C, 49·9; H, 6·0%), (c) the acetamido-compound (I; R = NHAc) (45%), which resinified rapidly in air but, with acetic anhydride, gave 6-*acetamido*-1,4-*diacetyl*-1,2,3,4-*tetrahydroquinoxaline*, needles, m. p. 225° (from water) (Found: C, 57·5; H, 6·9. $C_{14}H_{17}N_3O_3,H_2O$ requires C, 57·3; H, 6·5%), (d) by use of methanol instead of ethanol, 1,2,3,4-*tetrahydro*-6-*methoxycarbonylquinoxaline* which was isolated as the *hydrochloride* (75%), needles, m. p. 215° (decomp.) (from aqueous hydrochloric acid) (Found: C, 52·1; H, 5·6; N, 12·2. $C_{10}H_{13}ClN_2O_2$ requires C, 52·5; H, 5·7; N, 12·25%). The free *base* formed yellow prisms, m. p. 88° (from light petroleum) (Found: C, 62·0; H, 6·4; N, 14·0. $C_{10}H_{12}N_2O_2$ requires C, 62·5; H, 6·3; N, 14·6%), (e) 6-*bromo*-1,2,3,4-*tetrahydroquinoxaline*, plates (48%), m. p. 118° (from water) (Found: C, 45·1; H, 3·9; N, 12·8. $C_8H_9BrN_2$ requires C, 45·1; H, 4·3; N, 13·1%).

Acyl Derivatives from 1,2,3,4-*Tetrahydro*-6-*nitroquinoxaline*.—(*a*) The nitro-compound [1] was mixed with acetic anhydride containing a trace of sulphuric acid, and quickly deposited 1-*acetyl*-1,2,3,4-*tetrahydro*-7-*nitroquinoxaline*, orange needles, m. p. 204° (from acetic acid) (Found: C, 54·6; H, 4·9. $C_{10}H_{11}N_3O_3$ requires C, 54·3; H, 5·0%). This acetyl derivative reacted with aqueous nitrous acid to give 4-*acetyl*-1,2,3,4-*tetrahydro*-6-*nitro*-1-*nitrosoquinoxaline*, buff needles, m. p. 160° (from ethanol) (Found: C, 48·6; H, 4·0. $C_{10}H_{10}N_4O_4$ requires C, 48·0; H, 4·0%).

(*b*) The nitro-compound was heated under reflux with 50% aqueous formic acid for ½ hr., and the solution was cooled and diluted with water. The resulting 1-*formyl*-1,2,3,4-*tetrahydro*-7-*nitroquinoxaline* formed bright red prisms (87%), m. p. 177° (from water) (Found: C, 52·5; H, 4·3; N, 20·1. $C_9H_9N_3O_3$ requires C, 52·2; H, 4·4; N, 20·3%).

(*c*) The nitro-compound was heated with 98—100% formic acid and the product was evaporated under reduced pressure. The resulting 1,4-*diformyl*-1,2,3,4-*tetrahydro*-6-*nitroquinoxaline* formed buff prisms (80%), m. p. 176° (from acetone) (Found: C, 51·7; H, 4·0; N, 17·8. $C_{10}H_9N_3O_4$ requires C, 51·1; H, 3·9; N, 17·9%). This diformyl derivative gave the foregoing monoformyl derivative when treated as in (*b*).

1,2,3,4-tetrahydro-6-nitroquinoxaline and its monoacetyl derivative were each reduced catalytically in the presence of Raney nickel in ethanol. The filtered products were each evaporated and mixed with acetic anhydride, and each gave 6-acetamido-1,4-diacetyl-1,2,3,4-tetrahydroquinoxaline (*ca.* 70%), m. p. 225° alone or mixed with a sample prepared *via* cyclisation of 5-acetamido-*N*-2′-chloroethyl-2-nitroaniline.

o-*Amino*-N-2′-*hydroxyethylanilines*.—5-Chloro-*N*-2′-hydroxyethyl-2-nitroaniline (0·01 mole) was hydrogenated in the presence of Raney nickel (2 ml. of settled suspension) in ethanol (100 ml.), and the solvent was removed under reduced pressure from the filtered product. The resulting 2-amino-5-chloro-*N*-2′-hydroxyethylaniline (85%) formed needles, m. p. 105° (from benzene) (Found: C, 51·8; H, 6·2. Calc. for $C_8H_{11}ClN_2O$: C, 51·5; H, 5·9%). With hot acetic anhydride it gave 2-*acetamido*-N-2′-*acetoxyethyl*-5-*chloroacetanilide* which formed needles, m. p. 147° (from benzene) (Found: C, 54·0; H, 5·8; N, 9·3. $C_{14}H_{17}ClN_2O_4$ requires C, 53·8; H, 5·5; N, 9·0%). Similarly, the following compounds were prepared from the corresponding *o*-nitroanilines: 5-Acetamido-2-amino-*N*-2′-hydroxyethylaniline, needles (from methanol) which darkened rapidly in air. This base was mixed with acetic anhydride and gave 2,5-*diacetamido*-N-2′-*acetoxyethylacetanilide*, prisms, m. p. 182° (from water) (Found: C, 57·1; H, 6·4. $C_{16}H_{21}N_3O_5$ requires C, 57·3; H, 6·3%). 2-*Amino*-4-*bromo*-N-2′-*hydroxyethylaniline* (79%), formed needles, m. p. 132° (from benzene) (Found: C, 42·2; H, 4·8. $C_8H_{11}BrN_2O$ requires C, 41·6; H, 4·8%). 2-*Amino*-N-2′-*hydroxyethyl*-5-*methoxyaniline* (68%), formed plates, m. p. 137° (from benzene) (Found: C, 59·5; H, 7·9. $C_9H_{14}N_2O_2$ requires C, 59·3; H, 7·7%).

1,2,3,4-*Tetrahydroquinoxalines from* o-*Amino*-N-2′-*hydroxyethylanilines*.—The *o*-diamine (0·01 mole) was warmed with thionyl chloride (10 ml.) at 60° for ½ hr., and the excess of reagent was evaporated under reduced pressure. The residue was treated with saturated aqueous

[6] Cavagnol and Wiselogle, *J. Amer. Chem. Soc.*, 1947, **69**, 795.

potassium carbonate and gave the crude *N*-2′-chloroethylaniline derivative. This was heated in ethanol, and the cyclised product was isolated as described in the preparation from *N*-2′-chloroethyl-*o*-nitroanilines. The procedure gave, from the corresponding *o*-diamines, 6-chloro-, 6-bromo-, and 6-methoxy-1,2,3,4-tetrahydroquinoxalines in yields of 20%, 17%, and 25%, respectively.

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