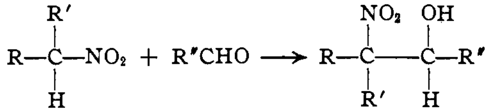
[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, POLYTECHNIC INSTITUTE OF BROOKLYN]

Preparation and Catalytic Reduction of 3-Nitrobutyl-2 *p*-Nitrobenzoate[1]

BY JULIAN R. REASENBERG AND G. B. L. SMITH

The commercial production of the nitroparaffins has greatly stimulated interest in the derivatives of these compounds.[2] Of these derivatives, the nitroalcohols and the corresponding amino alcohols are of prime importance because of their similarity in structure to compounds exhibiting physiological activity.

The reaction between nitroparaffins and aliphatic aldehydes to produce nitroalcohols was first described by Henry,[3] and since then has been studied extensively. A glance at the structure



$$\mathrm{R{-}\overset{R'}{\underset{H}{C}}{-}NO_2 + R''CHO \longrightarrow R{-}\overset{NO_2}{\underset{R'}{C}}{-}\overset{OH}{\underset{H}{C}}{-}R''}$$

of a nitroalcohol formed from a nitroparaffin higher than nitromethane and an aldehyde higher than formaldehyde, will indicate that the compound contains two asymmetric carbon atoms and should exist in diastereoisomeric forms. To date, only one case of the separation of the *erythro* and *threo* forms of an aliphatic nitroalcohol has been reported.[4]

The compound prepared from nitroethane and acetaldehyde, 3-nitrobutanol-2, fulfills these conditions and should exist as two chemical entities. However, careful fractionation of this compound by the authors as well as by Vanderbilt and Hass[4] has failed to show the presence of the diastereoisomerides.

Some of the fatty acid esters of the nitroalcohols have been described. Vanderbilt and Hass[4] have prepared the acetates of a wide variety of nitroalcohols by allowing them to react with acetic anhydride. Tyndall[5] has prepared the propionates, butyrates, and isobutyrates of these compounds. The nitroalcohols and corresponding esters in which the nitro group is linked to a secondary carbon atom are relatively unstable. Boiling water or 0.1 *N* sodium bicarbonate decomposed the esters of 3-nitrobutanol-2 into the acid and 3-nitrobutene-2.[4,5]

3-Nitrobutanol-2 was prepared by a method similar to that described by Vanderbilt and Hass[4] although the method was developed independently before the publication of the method of Vanderbilt and Hass. The product could be purified by vacuum distillation without decomposition although it has been reported to decompose explosively at times upon low pressure distillation.[6] On standing for several months in a sealed container it decomposed somewhat with the formation of nitrobutene. Attempts to prepare the nitrobenzoate by the usual chemical methods resulted in failure until a low temperature acylation in the presence of pyridine was performed. The product so obtained was a white solid with a wide melting point range, 80–85°. After eight to ten recrystallizations from alcohol the product melted sharply at 107–108°. This purification could also be effected with greater ease by a partial and stepwise treatment of the alcoholic solution with sodium hydroxide. Two successive treatments with 10% of the theoretical amount of alkali each time usually sufficed to give a product which, after one recrystallization from alcohol, melted at 107–108°.

The "crude" ester and the purified ester gave the same saponification equivalent 134, and both products contained essentially the same percentage of carbon, hydrogen, and nitrogen. This would indicate that the "crude" ester is a mixture of the diastereoisomerides previously postulated. The lachrymator 3-nitrobutene-2 was always present in the mother liquors from the recrystallization of the impure product but was not present in the liquor from the recrystallization of the pure product. Also the nitro-olefin was present in large quantities in the liquor resulting from the alkali treatment.

All of these facts seem to indicate that the 3-nitrobutanol-2 is a mixture of the two isomers and on esterification, the ester formed is "crude" because of the presence of both isomers. Also, one of the isomers is preferentially hydrolyzed and(or) is decomposed in the presence of alkali to 3-nitrobutene-2. Dilute solutions of ammonium hydroxide and sodium bicarbonate were also effective in this process. Several attempts were made to isolate the unstable isomer by fractional crystallization of the mother liquors but these were unsuccessful because of the thermal instability of the compound,[4,5] and only the decomposition products, *p*-nitrobenzoic acid and 3-nitrobutene-2, were isolated.

A study was made of the reduction and reduction products of 3-nitrobutyl-2 *p*-nitrobenzoate. Figure 1 illustrates the products formed under varying conditions of reduction with both Raney nickel and Adams platinum oxide catalysts. In all of the reductions employing Raney nickel the catalyst was promoted with platinic chloride

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(2) Hass, *et al.*, *Ind. Eng. Chem.*, **28**, 339 (1936); **30**, 67 (1938); **31**, 118, 648 (1939); **32**, 427 (1940); *Chem. Rev.*, **32**, 373 (1943).

(3) Henry, *Compt. rend.*, **120**, 1265 (1895).

(4) Vanderbilt and Hass, *Ind. Eng. Chem.*, **32**, 34 (1940).

(5) Tyndall, *ibid.*, **33**, 65 (1941).

(6) Private communication, The Commercial Solvents Corp.

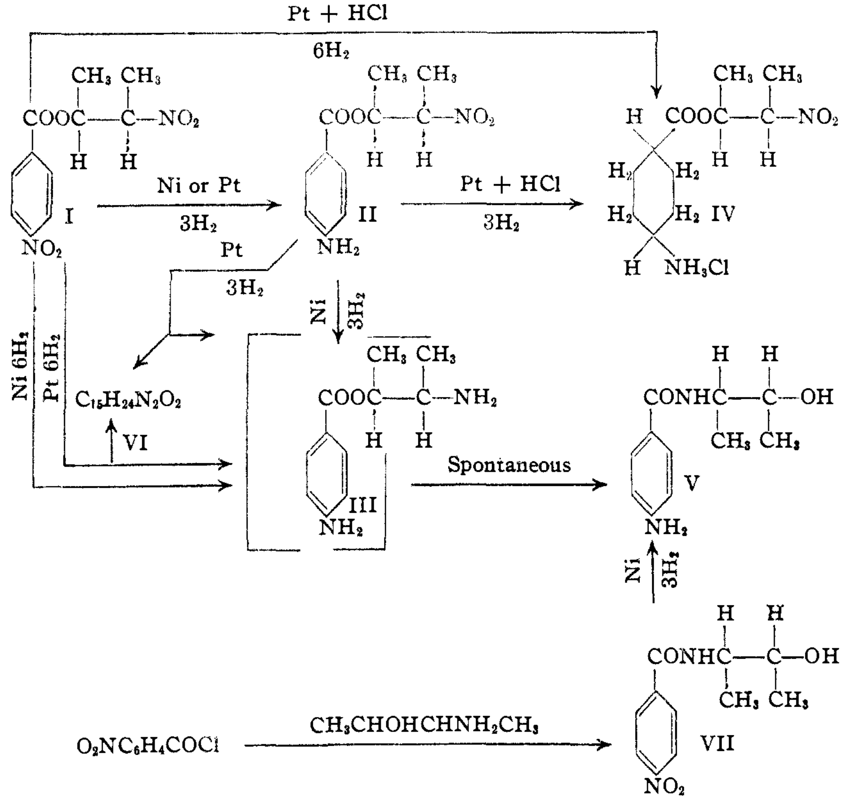

Fig. 1.

as previously described.[7] The qualitative and quantitative results did not vary when similar experiments were performed with the promoted and unpromoted nickel but the reduction proceeded at a much more rapid rate when the platinic chloride was added to the nickel. Also the results did not vary whether the hydrogenation was performed at atmospheric pressure or at 3–4 atmospheres. The apparatus has been described previously.[7]

3-Nitrobutyl-2 *p*-nitrobenzoate I, contains two nitro groups and the reduction could be effected either stepwise or completely by allowing either 3 or 6 moles of hydrogen to be absorbed. With either catalyst the only product formed when 3 moles of hydrogen was absorbed was the corresponding amino compound II. This compound was then reduced under varying conditions to give a number of products. The hydrochloride of II was quite acidic and Raney nickel could not be used to effect its reduction. With Adams catalyst however, the hydrogenation proceeded slowly but smoothly with the formation of the cyclohexane derivative 3-nitrobutyl-2 *p*-aminohexahydrobenzoate hydrochloride IV. The formation of this product is probably due to the fact that acid inhibits the reduction of an aliphatic nitro group by means of platinum and promotes the reduction of the benzene ring by means of platinum.[8] On acid hydrolysis, 3-nitrobutene-2 and *p*-aminohexahydrobenzoic acid, m. p. 285°,[9] were formed.

In neutral solution with Raney nickel, II is reduced slowly to give V, N-(*p*-amino benzoyl)-2,3-butanolamine. The formation of this amide alcohol presumably occurs through the rearrangement of compound III, 3-aminobutyl-2 *p*-aminobenzoate. This rearrangement of esters of β-primary amino alcohols to the corresponding β-hydroxy amide in neutral or alkaline solution has been described for a wide variety of compounds.[10,11,12,13] The structure of compound V was proved by its synthesis from N-(*p*-nitrobenzoyl)-2,3-butanolamine by catalytic reduction.

With Adams platinum oxide in neutral solution, 3-nitrobutyl-2 *p*-aminobenzoate II is reduced to give two products, V and VI. Compound VI is an oily base which is insoluble in water but is soluble in most organic solvents and in dilute acid. The numbing effect of a local anesthetic is produced when a drop of the oil is placed on the tongue and mucous membranes of the mouth. All attempts to produce a solid salt with the common inorganic and organic acids failed. On hydrolysis, *p*-aminobenzoic acid was isolated. A diacetyl derivative was formed which could be obtained in pure crystalline form. The empirical formula of the oil is tentatively set at $C_{15}H_{24}N_2O_2$ and that of the diacetyl derivative at $C_{19}H_{28}N_2O_4$. The picrate was also prepared and analyzed. Its formula is postulated as $C_{21}H_{27}N_5O_9$.

In addition to compounds V and VI there was also isolated from the reduction of II, *p*-amino benzoic acid and ammonia. The difference between the postulated formula of VI and the formula of III is C_4H_8, and this together with the fact that ammonia was one of the products of reduction would indicate that VI might be formed by the reaction of III with butanolamine with the elimination of ammonia and water.

The complete reduction of 3-nitrobutyl-2 *p*-nitrobenzoate I with the absorption of 6 moles of

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[7] Lieber and Smith, THIS JOURNAL, **58**, 1417 (1936); Reasenberg, Lieber and Smith, *ibid.*, **61**, 384 (1939); Scholnik, Reasenberg, Lieber and Smith, *ibid.*, **63**, 1192 (1941).

[8] Hiers and Adams, *Ber.*, **59**, 162 (1926).

[9] Greenstein and Wyman, THIS JOURNAL, **60**, 2341 (1938).

[10] Gabriel and Heymann, *Ber.*, **23**, 2492 (1890).

[11] Gabriel, *Ann.*, **409**, 326 (1915).

[12] Jacobs and Heidelberger, *J. Biol. Chem.*, **21**, 421 (1915).

[13] Bergmann and Brand, *Ber.*, **56B**, 1280 (1923).

hydrogen produces the same results and products as the sum of the two stepwise hydrogenations involved. Thus I with Pt and HCl forms IV; I with Pt in neutral solution gives V and VI; and I with Raney nickel forms V.

Experimental

3-Nitrobutanol-2.[14]—A solution of 40 g. of acetaldehyde (0.91 mole) and 71 g. of nitroethane (0.90 mole) dissolved in 60 ml. of 95% ethanol was treated with 0.8 g. sodium hydroxide dissolved in 2 ml. of water. The solution was allowed to stand for five days at room temperature at which time the color had deepened to yellow. The alkali was neutralized with concd. hydrochloric acid, the color changing to blue-green, and the precipitated sodium chloride was allowed to settle and was separated by decantation. The solvent was removed by distillation under diminished pressure and the residue was vacuum distilled. After a small fore-run of 3-nitrobutene-2 (strong lachrymator) the main fraction of 3-nitrobutanol-2 came over as a light yellow liquid with a slight acrid odor weighing 99 g. The product had the following characteristics: b. p. 90° (11 mm.), b. p. 99° (17 mm.), $d^{20}{}_{4}$ 1.1260, $n^{22}{}_{D}$ 1.4425, mol. ref. calcd. 28.0, found 28.0.[15]

2,3-Butanolamine.[16,17,18,19]—A solution of 47.6 g. of 3-nitrobutanol-2 dissolved in 120 ml. of ethanol was reduced at 3–4 atm. with 3–4 g. of Raney nickel. The reduction was complete in two hours. The catalyst was removed by filtration and the filtrate was distilled at atmospheric pressure. The aminoalcohol, 7.8 g., boiled at 159°; $d^{20}{}_{20}$ 0.9414, $d^{20}{}_{4}$ 0.9396, $n^{24}{}_{D}$ 1.4520, mol. ref. calcd. 25.5, found 25.6. Primary oxalate $C_4H_{11}ON{\cdot}H_2C_2O_4$ from ethanol m. p. 164° dec., secondary oxalate $(C_4H_{11}ON)_2{\cdot}H_2C_2O_4$ from ethanol m. p. 206° dec.[20]

3-Nitrobutyl-2 *p*-Nitrobenzoate (I).—Freshly distilled *p*-nitrobenzoyl chloride (53 g. 0.29 mole) was dissolved in 350 ml. of benzene and 34 g. of 3-nitrobutanol-2 (0.29 mole) was added. The beaker and contents were cooled with an ice-bath and, to the well-stirred, yellow solution, was slowly added 26 g. of pyridine (excess), the temperature being kept below 25°. During the addition the color changed to blue-green. The pyridine hydrochloride was removed by filtration and was washed with 50 ml. of benzene. The filtrates were combined and washed with cold dilute solution of sodium bicarbonate which changed the blue-green solution to yellow. The liquid was then washed with water, dried over anhyd. sodium sulfate, and evaporated under diminished pressure to a volume of 100 ml. The temperature was adjusted to 50° and the solution was diluted with 100 ml. of petroleum ether and a precipitate formed. When cold the solution was filtered and the product washed with a little petroleum ether and dried; 70 g. of crude ester m. p. 80–85° was obtained. This was recrystallized once from hot ethanol to give a product melting at 85–90°.

Anal. Calcd. for $C_{11}H_{12}N_2O_6$: C, 49.27; H, 4.51; N, 10.45; sap. eq., 134.1. Found: C, 49.76; H, 4.68; N, 10.17; sap. eq., 134.0.

Separation of Isomers.—The "crude" nitro nitro ester described just above was purified as follows: a hot solution of 450 g. of "crude" ester dissolved in 2500 ml. of ethanol and 500 ml. of water was treated with a solution of 8 g. of sodium hydroxide in 50 ml. of water. The solution darkened to an orange-yellow color and the strong odor of the lachrymous nitrobutene was evolved. The solution was allowed to cool and a white crystalline product started to form. When the temperature reached 40° another 8 g. of sodium hydroxide was added slowly with stirring over a period of one hour. The solution was allowed to stand overnight and the precipitate was then removed by filtration and washed with two 50-ml. portions of cold ethanol and gave on drying 190 g. of ester m. p. 100–104°. This was recrystallized from 2 liters of hot ethanol to give 166 g. of product m. p. 106–108°. One subsequent recrystallization raised the m. p. to 107–108°. The product was a white crystalline compound with a very slight yellowish cast.

Anal. Calcd. for $C_{11}H_{12}N_2O_6$: C, 49, 27; H, 4.51; N, 10.45; sap. eq., 134.1. Found: C, 49.17; H, 4.55; N, 10.39; sap. eq., 134.5.

***p*-Nitrobenzoic Anhydride.**—The pyridonium chloride obtained in the acylation described above was dissolved in water and the insoluble fraction was filtered, washed with water and dried; 8 g. of product m. p. 180–185° was obtained. This was recrystallized from ethyl acetate and benzene; soft, slightly yellow, nacreous plates m. p. 191–192° were formed. This product is *p*-nitrobenzoic anhydride[21] and was also synthesized by the method of Holleman,[22] m. p. 191–192°, and a mixed m. p. gave the same value.

Anal. Calcd. for $C_{14}H_8N_2O_7$: N, 8.86; eq. wt., 158. Found: N, 8.98; eq. wt., 158.

3-Nitrobutyl-2 *p*-Aminobenzoate (II).—A solution of 26.8 g. of nitro ester (I) (0.10 mole) dissolved in 100 ml. of dioxane was treated with 4.5 g. of Raney nickel and 0.30 ml. of 10% platinum tetrachloride solution as promoter. The hydrogenation was performed at atmospheric pressure and the hydrogen was absorbed at the rate of 100 ml. per min. When 7200 ml. of hydrogen (0.3 mole) had been absorbed, the rate of hydrogenation diminished considerably and the reduction stopped. The reductate was separated from the catalyst by filtration and the yellow filtrate was treated with 16.7 ml. of 6 *N* hydrochloric acid dissolved in isopropanol (0.10 mole). The copious white precipitate which formed was filtered, washed twice with 30 ml. of dioxane and twice with 50 ml. of ether and dried; 21 g. of white product resulted. This was recrystallized from isopropanol and white platelets of 3-nitrobutyl-2 *p*-aminobenzoate hydrochloride (II HCl) m. p. (rapid heating) 182–183° (dec.) resulted. This compound could be diazotized and coupled with beta-naphthol and gave a red dye. Acid hydrolysis of the amino nitro ester hydrochloride yielded *p*-aminobenzoic acid and 3-nitrobutene-2.

Anal. Calcd. for $C_{11}H_{15}N_2O_4Cl$: C, 48.09; H, 5.50; N, 10.20; Cl, 12.91; sap. eq., 91.5. Found: C, 47.90; H, 5.62; N, 10.23; Cl, 12.99; sap. eq., 91.8.

The free base was prepared by dissolving 65 g. of (II HCl) in 600 ml. of hot ethanol and pouring the resulting solution with stirring into 1 liter of ice and water, containing an excess of ammonium hydroxide. The precipitate was allowed to stand for one hour and was filtered, washed with water and dried; 54 g. of product m. p. 100–102° was obtained. This was recrystallized from 350 ml. of isopropanol after treatment with Darco and 48 g. of white crystalline 3-nitrobutyl-2 *p*-aminobenzoate (II) resulted, m. p. 103°, acetyl derivative m. p. 110°, nitrate m. p. (fast heating) 167–169° (dec.).

Anal. Calcd. for $C_{11}H_{14}N_2O_4$: N, 11.76; sap. eq., 119.0. Found: N, 11.52; sap. eq., 119.0.

3-Nitrobutyl-2 *p*-Aminohexahydrobenzoate Hydrochloride (IV).—A solution of 9.15 g. of 3-nitrobutyl-2 *p*-aminobenzoate hydrochloride (II HCl) (0.033 mole) dissolved in 100 ml. of ethanol was treated with 0.40 g. of platinum oxide. The reduction was performed at atmospheric pressure and room temperature. The hydrogenation proceeded very slowly, 200 ml. of hydrogen being absorbed in seventy-five minutes. After about 800 ml. of hydrogen had been absorbed another 0.20 g. of platinum oxide was added until a total of 2670 ml. of hydrogen had been added and a total of 1.0 g. of platinum oxide had been used.

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(14) Henry, *Compt. rend.*, **121**, 210 (1895).

(15) Ref. 6, b. p. 92° (10 mm.), $d^{25}{}_{4}$ 1.1296, $n^{20}{}_{D}$ 1.4420.

(16) Henry, *Ber.*, **33**, 3169 (1900).

(17) Batalin and Ugryumov, *C. A.*, **30**, 6701 (1936).

(18) Wieland and Bossert, *Ann.*, **509**, 1 (1934).

(19) Leffler and Adams, THIS JOURNAL, **59**, 2252 (1937).

(20) Ref. 18, acid oxalate, m. p. 164–165° dec., normal oxalate, m. p. 208° dec.

(21) Thiele. *Ann.*, **314**, 305 (1901).

(22) Holleman, *Rec. trav. chim.*, **15**, 362 (1896).

This corresponds to 3 equivalents of hydrogen plus that required for the reduction of the platinum oxide. The reductate was filtered and the filtrate was evaporated under diminished pressure until all of the solvent had been removed and 30 ml. of benzene and 50 ml. of *n*-butanol were added. A white precipitate formed which was filtered, washed with butanol and ether and dried; 2.50 g. of 3-nitrobutyl-2 *p*-aminohexahydrobenzoate hydrochloride (IV) was obtained. This was recrystallized from isopropanol and a fine white crystalline product resulted which decomposed at 177° with slow heating and at 184° with rapid heating. The compound formed a neutral aqueous solution which could not be diazotized and coupled with β-naphthol. Upon alkaline hydrolysis the lachrymous nitrobutene was evolved. The chloroplatinate was formed in water solution from which it crystallized as orange cubes with 2 moles of water of crystallization which could be removed by heating at 110° leaving a hygroscopic product.

Anal. Calcd. for $C_{11}H_{21}N_2O_4Cl$: C, 47.06; H, 7.54; Cl, 12.63. Found: C, 46.86; H, 7.70; Cl, 12.71. Calcd. for $(C_{11}H_{21}N_2O_4Cl)_2 \cdot PtCl_4 \cdot 2H_2O$: C, 28.27; H, 4.96; Pt, 20.89. Found: C, 28.47; H, 4.73; Pt, 20.78.

p-Aminohexahydrobenzoic Acid.—One hundred ml. of 10% sulfuric acid was added to 1.92 g. of IV and the solution was refluxed for twenty-four hours. 3-Nitrobutene-2 was evolved during the initial stages of the hydrolysis. The resulting clear colorless solution was treated with an excess of silver sulfate and the silver chloride removed by filtration. The solution was treated with hydrogen sulfide, the precipitate, silver sulfide, was separated by filtration, and the excess hydrogen sulfide was expelled by boiling. The solution was then treated with barium hydroxide until alkaline to phenolphthalein and the slight excess of barium hydroxide was carefully neutralized with 0.1 *N* sulfuric acid. The barium sulfate was removed and the clear colorless solution was evaporated to a volume of 20 ml. On dilution with isopropanol a precipitate formed which was separated by filtration, washed with alcohol and ether and dried; 0.6 g. of white product was obtained. This was recrystallized from aqueous acetone and *p*-aminohexahydrobenzoic acid, m. p. 285°,[9] resulted.

Anal. Calcd. for $C_7H_{13}NO_2$: C, 58.63; H, 9.14. Found: C, 58.45; H, 9.04.

N-(*p*-Aminobenzoyl)-2,3-butanolamine V.—A solution of 7.63 g. of II in 100 ml. of alcohol was treated with 4.5 g. of promoted Raney nickel and reduced at 3–4 atm. The equivalent of 3 moles of hydrogen was absorbed in two and one-half hours and on further shaking for two hours no more was absorbed. The solution was filtered and the filtrate was evaporated *in vacuo*. The oily residue was dissolved in 75 ml. of dioxane and saturated with hydrogen chloride. The insoluble oil which formed was separated from the dioxane and dissolved in 50 ml. of isopropanol, 35 ml. of benzene was added and the solution was evaporated to a volume of 35 ml. when, on cooling,. a precipitate formed. This was filtered, washed with a few ml. of isopropanol and dried: 3.56 g. of N-(*p*-aminobenzoyl)-2,3-butanolamine hydrochloride was obtained.

The free base was prepared from the hydrochloride, which decomposes without melting, by dissolving it in water and making the solution ammoniacal. On standing this solution deposited crystals which were recrystallized from water to give V as white crystals which can be diazotized and coupled with β-naphthol, m. p. 145–146°. The acetyl derivative, formed from acetic anhydride and water solution of V, crystallized from water as white needles, m. p. 194–195.

Anal. Calcd. for $C_{13}H_{18}N_2O_3$: C, 32.38; H, 7.25; N, 11.19. Found: C, 62.31; H, 7.44; N, 11.40.

V was also synthesized directly. The corresponding *p*-nitrobenzamide was prepared by allowing 1 mole of *p*-nitrobenzoyl chloride to react with 2 moles of 2,3-butanolamine in chloroform solution. The resulting solid was recrystallized from water and gave N-(*p*-nitrobenzoyl)-2,3-butanolamine, VIII, m. p. 158°. This was reduced in alcohol with Raney nickel and 3 moles of hydrogen was absorbed. The reductate was filtered, evaporated, and a solid resulted which by recrystallization from water yielded V, m. p. 145–146° (mixed m. p. 145–146°); acetyl derivative, m. p. 194–195° (mixed m. p. 194–195°).

$C_{15}H_{24}N_2O_2$, VI.—A solution of 12.0 g. of amino nitro ester II, 0.7 g. of platinum oxide and 80 ml. of methanol was treated with hydrogen at atmospheric pressure. A total of 3750 ml. of hydrogen (3 mol.) was absorbed in six hours. The reductate was filtered free of catalyst and the filtrate was evaporated on the water-bath under vacuum after the addition of 20 ml. of benzene. The gummy residue was dissolved in dilute hydrochloric acid and treated with Darco. The filtrate was made neutral to litmus with sodium bicarbonate and the small amount of oil which had formed was removed by filtration. The clear solution was made ammoniacal and the oil which formed was extracted with ether. This was dried over anhydrous sodium sulfate and evaporated; 2.50 g. of oil was obtained which was anesthetic to the tongue.

The water solution which remained after the ether extraction was treated with an excess of acetic anhydride; 3.55 g. of *p*-acetaminobenzoic acid m. p. 255 was formed. The filtrate was made ammoniacal once again and on standing deposited 1.74 g. of N-(*p*-acetaminobenzoyl)-butanolamine m. p. 190–193° (acetyl derivative of V).

The anesthetic oil is a yellow viscous liquid insoluble in water but soluble in all the common organic solvents and dilute acids. Attempts to form a solid salt of this base with the common acids failed. On alkaline hydrolysis, *p*-aminobenzoic acid is formed. The acetyl derivative prepared from acetic anhydride and recrystallized from benzene-ligroin solution melts at 151°.

Anal. Calcd. for $C_{19}H_{28}N_2O_4$: C, 65.49; H, 8.10; N, 8.04. Found: C, 65.58; H, 8.35; N, 8.20.

The picrate, a bright yellow solid, was formed in benzene, m. p. 171.5–172.0°.

Anal. Calcd. for $C_{21}H_{27}N_5O_9$: C, 51.11; H, 5.51; N, 14.20. Found: C, 50.99; H, 5.51; N, 15.09.

Summary

1. The existence of diastereoisomers in 3-nitrobutanol-2 has been demonstrated. One of these had been isolated in the form of the *p*-nitrobenzoate.

2. The catalytic reduction of 3-nitrobutyl-2 *p*-aminobenzoate has been studied under a variety of conditions.

3. Reduction of 3-nitrobutyl-2 *p*-aminobenzoate with Raney nickel results in the formation of N-(*p*-aminobenzoyl)-2,3-butanolamine by rearrangement of the expected product O-(*p*-aminobenzoyl)-2,3-butanolamine.

4. Reduction of 3-nitrobutyl-2 *p*-aminobenzoate with platinum in acid solution causes the ring to be reduced without affecting the aliphatic nitro group.

5. Reduction in neutral solution with platinum results in the formation of an anesthetic oil whose structure has not been completely elucidated.

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