

**284.** *Aliphatic Nitro-compounds. Part V. Preparation of 2-Nitroalkanesulphonic Acids by Interaction of  $\alpha$ -Nitro-olefins and Sodium Hydrogen Sulphite.\**

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$\alpha$ -Nitro-olefins react with sodium hydrogen sulphite giving sodium 2-nitroalkanesulphonates, which on catalytic reduction afford the corresponding 2-aminoalkanesulphonic acids in good yield. In the absence of oxygen, sulphurous acid can be used in place of sodium hydrogen sulphite, and in many cases esters of 2-nitro-alcohols (2-nitroethyl nitrate, 2-nitropropyl acetate) behave in the same way as the nitro-olefins.

\* Cf. E.P. 571,157.

THE interaction of  $\alpha$ -nitro-olefins and sodium hydrogen sulphite was studied during a general investigation of the addition reactions of nitro-olefins, as a potentially interesting route to certain 2-aminoalkanesulphonic acids required as intermediates in the synthesis of dyestuffs.

The addition of alkali hydrogen sulphites to unsaturated substances has been studied by many workers. Rosenthal (*Annalen*, 1886, **233**, 38) prepared the diammonium salt of  $\beta$ -sulphopropionic acid by the interaction of ammonium hydrogen sulphite and ammonium acrylate. The addition of potassium hydrogen sulphite to allyl alcohol was demonstrated by Müller (*Ber.*, 1873, **6**, 1442), and the product was established by Marckwald (*ibid.*, 1898, **31**, 1864) as potassium 1-hydroxypropane-3-sulphonate. Kolker and Lapworth (*J.*, 1925, 307) added ammonium hydrogen sulphite to a large number of olefins, whilst the reaction of sodium hydrogen sulphite and styrene, first reported by Ashworth and Burkhardt (*J.*, 1928, 1791), has been shown by Kharasch, Schenck, and Mayo (*J. Amer. Chem. Soc.*, 1939, **61**, 3092) to yield 2-phenylethane-1-sulphonic acid, 2-phenylethylene-1-sulphonic acid, 2-phenylethane-1:1-disulphonic acid, and 2-hydroxy-2-phenylethane-1-sulphonic acid. Knoevenagel and Morrise (*Ber.*, 1904, **37**, 4038) successfully added alkali hydrogen sulphites to  $\alpha\beta$ -unsaturated aldehydes and ketones, and the addition to unsaturated furyl compounds has been described by the American Cyanamide Co. (*Brit. Appl.* 14357/43).

The formation of alkali 2-nitroalkanesulphonates by interaction of an  $\alpha$ -nitro-olefin and alkali hydrogen sulphite was found to be a general reaction; in the absence of oxygen, sulphurous acid behaved in a similar way, giving the free nitro-sulphonic acid, but in the presence of oxygen sulphamic acid was formed, probably by (a) oxidation of sulphurous to sulphuric acid, (b) formation of hydroxylamine from sulphuric acid and the nitro-olefin, and (c) interaction of hydroxylamine and sulphur dioxide. The use of sodium sulphite in this reaction resulted in the formation of the sodium salt of the sodium *aci*-nitroalkanesulphonate, and in two cases esters of 2-nitro-alcohols have been shown to react in the same way as the nitro-olefins.

Accurate analyses of the nitroalkanesulphonates were not easy to obtain owing to the difficulty of freeing them from the last traces of moisture, and many were characterised as their *p*-toluidine salts (see Chambers and Watt, *J. Org. Chem.*, 1941, **6**, 376). All attempts to characterise them by salts of *S*-benzylisothiourrea were unsuccessful.

The *sulphonates* which have been prepared are listed in the Table.

| Nitro-compound.   | Sulphite.                       | Nitro-sulphonate.   |
|---|---------------------------------|---|
| Nitroethylene .....   | NaHSO <sub>3</sub>              | Na 2-nitroethanesulphonate  |
| 2-Nitroethyl nitrate .....                                  | NaHSO <sub>3</sub>              | Na 2-nitroethanesulphonate  |
| 1-Nitroprop-1-ene .....                                     | NaHSO <sub>3</sub>              | Na 1-nitropropane-2-sulphonate  |
| 1-Nitroprop-1-ene .....                                     | H <sub>2</sub> SO <sub>3</sub>  | 1-Nitropropane-2-sulphonic acid                                       |
| 1-Nitroprop-1-ene .....                                     | Na <sub>2</sub> SO <sub>3</sub> | Na <sub>2</sub> 1- <i>aci</i> -nitropropane-2-sulphonate              |
| 2-Nitroprop-1-ene .....                                     | NaHSO <sub>3</sub>              | Na 2-nitropropane-1-sulphonate  |
| 2-Nitropropyl acetate .....                                 | NaHSO <sub>3</sub>              | Na 2-nitropropane-1-sulphonate  |
| 1-Nitro-2-methylprop-1-ene .....                            | NaHSO <sub>3</sub>              | Na 1-nitro-2-methylpropane-2-sulphonate                               |
| 1-Nitro-2-methylprop-1-ene .....                            | H <sub>2</sub> SO <sub>3</sub>  | 1-Nitro-2-methylpropane-2-sulphonic acid                              |
| 2-Nitrobut-2-ene .....                                      | NaHSO <sub>3</sub>              | Na 2-nitrobutane-3-sulphonate and (?) Na 2-oximinobutane-3-sulphonate |
| 2-Nitrostyrene .....  | NaHSO <sub>3</sub>              | Na 1-nitro-2-phenylethane-2-sulphonate                                |
| 2-Nitrostyrene .....  | H <sub>2</sub> SO <sub>3</sub>  | 1-Nitro-2-phenylethane-2-sulphonic acid                               |
| 1-Nitro-2-(2-furyl)ethylene .....                           | NaHSO <sub>3</sub>              | Na 1-nitro-2-(2-furyl)ethane-2-sulphonate                             |
| Ethyl $\alpha$ -nitro- $\beta\beta$ -dimethylacrylate ..... | NaHSO <sub>3</sub>              | Na 1-nitro-1-carbethoxy-2-methylpropane-2-sulphonate                  |

A few deviations from the normal reaction course were noted. 1-Nitrocyclohexane and 2-nitro-1-phenylprop-1-ene with sodium hydrogen sulphite gave viscous syrups which could not be characterised and on reduction gave nitrogen-free products. 2-Nitrobut-2-ene, condensed with sodium hydrogen sulphite in an atmosphere of nitrogen, gave a syrupy and a crystalline condensation product both of which on reduction yielded amino-sulphonic acids which, whilst agreeing in analysis with the expected 2-aminobutanesulphonic acid, were not identical. The original crystalline product may be the oximino-sulphonic acid, but the structures of the amino-sulphonic acids have not been determined.

The interaction of ethyl  $\alpha$ -nitro- $\beta\beta$ -dimethylacrylate and sodium hydrogen sulphite was described by Bouveault and Wahl (*Bull. Soc. chim.*, 1901, **25**, 910) who made no attempt to isolate or characterise the reaction product. This has been repeated and the product shown to be that of normal addition.

Reduction of the nitro-sulphonates with Raney nickel and hydrogen gave good yields of the corresponding amino-sulphonic acids: in most cases the reduction was sufficiently facile to

proceed at ordinary temperature and pressure and in only a few was it necessary to use hydrogen under pressure.

#### EXPERIMENTAL.

Analyses are by Mr. E. S. Morton. All m. ps. are uncorrected.

**Sodium 2-Nitroethanesulphonate.**—(a) *From nitroethylene.* Nitroethylene (27.5 g.; this series, Part I) was added dropwise to a vigorously stirred solution of sodium hydrogen sulphite (39.2 g.) in water (70 c.c.) at  $-5^{\circ}$  to  $0^{\circ}$  and stirring continued at this temperature for 2 hours and finally at  $20^{\circ}$  for 4 hours. The mixture was evaporated under reduced pressure at  $40^{\circ}$  and the residue extracted with absolute alcohol, giving sodium 2-nitroethanesulphonate in 75% yield. The salt was recrystallised from alcohol (Found: N, 7.9.  $C_3H_5O_5NSNa$  requires N, 7.9%). The *p*-toluidine salt, prepared by addition of aqueous *p*-toluidine hydrochloride to the aqueous sodium salt, formed needles from absolute alcohol, m. p.  $179^{\circ}$  (decomp.) (Found: C, 40.6; H, 5.5; N, 10.6.  $C_2H_5O_5NS, C_7H_9N$  requires C, 41.2; H, 5.4; N, 10.7%).

(b) *From 2-nitroethyl nitrate.* 2-Nitroethyl nitrate (13.6 g.; Levy, Scaife, and Wilder-Smith, *J.*, 1946, 1096) was added dropwise to a well-stirred solution of sodium hydrogen sulphite (20.8 g.) in water (40 c.c.) at  $-5^{\circ}$  to  $0^{\circ}$  and stirring was continued for 24 hours at  $20^{\circ}$ . 2-Nitroethyl nitrate (3.2 g.) was recovered from the solution by ether extraction, and the aqueous layer, after evaporation and extraction as described in (a) above, gave the nitro-sulphonic acid, characterised as its *p*-toluidine salt, in 50% yield.

**2-Aminoethanesulphonic Acid.**—The nitro-sulphonic acid (above) (1.77 g.) in water (30 c.c.) was shaken with hydrogen and Raney nickel at ordinary temperature and pressure. The filtered solution was evaporated to dryness, treated with concentrated hydrochloric acid, filtered, and again evaporated. The residue on crystallisation from aqueous alcohol afforded 2-aminoethanesulphonic acid (taurine), m. p.  $317^{\circ}$  (decomp.), in 65% yield. Cortese (*J. Amer. Chem. Soc.*, 1936, **58**, 191) gives m. p. 300–305° (decomp.) (Found: C, 19.2; H, 5.6; N, 10.9. Calc. for  $C_2H_7O_3NS$ : C, 19.2; H, 5.6; N, 11.2%).

**Sodium 1-Nitropropane-2-sulphonate.**—(a) *From sodium hydrogen sulphite.* The salt was prepared from 1-nitroprop-1-ene (17.4 g.; this series, Part I) and sodium hydrogen sulphite (20.8 g.) in water (50 c.c.) as described above for sodium 2-nitroethane sulphonate. Sodium 1-nitropropane-2-sulphonate monohydrate separated from alcohol in 78% yield (Found: C, 17.4; H, 4.1; N, 7.0.  $C_3H_7O_5NSNa, H_2O$  requires C, 17.2; H, 3.8; N, 6.7%). The *p*-toluidine salt had m. p.  $164$ – $166^{\circ}$  (Found: C, 43.8; H, 5.8; N, 10.2.  $C_3H_7O_5NS, C_7H_9N$  requires C, 43.5; H, 5.8; N, 10.2%).

(b) *From sulphurous acid.* 1-Nitroprop-1-ene (10 g.) was shaken in a stoppered bottle at room temperature for 2 days with an aqueous solution of sulphurous acid (saturated at  $0^{\circ}$ ). The solution was neutralised with sodium carbonate, evaporated to dryness at  $40^{\circ}$  under reduced pressure, and the residue extracted with hot absolute alcohol to give sodium 1-nitropropane-2-sulphonate, characterised as the *p*-toluidine salt, m. p.  $164$ – $166^{\circ}$ .

(c) *From sodium sulphite.* 1-Nitroprop-1-ene (8.7 g.) was gradually added to a vigorously-stirred solution of sodium sulphite heptahydrate (25.2 g.) in water (25 c.c.) at  $0$ – $5^{\circ}$  and stirring continued for a further 3 hours at  $0$ – $5^{\circ}$  and at  $20^{\circ}$  for 16 hours. The solution was evaporated to dryness and the residue extracted with absolute alcohol to give disodium 1-aci-nitropropane-2-sulphonate trihydrate, which separated from aqueous alcohol in white crystals (Found: C, 13.6; H, 4.0; N, 5.3.  $C_3H_5O_5NSNa_2, 3H_2O$  requires C, 13.6; H, 4.1; N, 5.2%). It was characterised as its *p*-toluidine salt, m. p.  $164$ – $166^{\circ}$  alone and in admixture with the sample prepared under (a) above.

**1-Aminopropane-2-sulphonic Acid.**—The nitro-sulphonic acid (above) was reduced with hydrogen and Raney nickel as described for 2-aminoethanesulphonic acid. The amino-acid, obtained in 79% yield, had m. p.  $290$ – $295^{\circ}$  (decomp.) (Gabriel and Colman, *Ber.*, 1906, **39**, 2891, give m. p.  $290$ – $295^{\circ}$ ) (Found: C, 25.7; H, 6.5; N, 10.6. Calc. for  $C_3H_7O_3NS$ : C, 25.9; H, 6.5; N, 10.1%).

**Sodium 2-Nitropropane-1-sulphonate.**—(a) *From 2-nitroprop-1-ene.* This was prepared from 2-nitroprop-1-ene (this series, Part I) and sodium hydrogen sulphite as described for sodium 1-nitropropane-2-sulphonate. The salt was crystallised from absolute alcohol, giving sodium 2-nitropropane-1-sulphonate in 88% yield (Found: C, 18.7; H, 4.1; N, 7.0.  $C_3H_6O_5NSNa$  requires C, 18.8; H, 3.1; N, 7.8%).

Catalytic reduction of the salt in the usual way afforded a 60% yield of 2-aminopropane-1-sulphonic acid, which separated from aqueous alcohol, m. p.  $325$ – $335^{\circ}$  (decomp.); Gabriel and Ohle (*Ber.*, 1906, **39**, 2891) give m. p.  $323^{\circ}$  (decomp.) (Found: C, 25.9; H, 6.7; N, 10.0. Calc. for  $C_3H_7O_3NS$ : C, 25.9; H, 6.5; N, 10.1%).

(b) *From 2-nitropropyl acetate.* A mixture of 2-nitropropyl acetate (7.35 g.; Blomquist, Tapp, and Johnson, *J. Amer. Chem. Soc.*, 1945, **67**, 1519), sodium hydrogen sulphite (20.8 g.), and water (50 c.c.) was stirred vigorously at  $35^{\circ}$  for 16 hours. The homogeneous solution was evaporated to dryness and extracted with absolute alcohol. Evaporation of the alcohol gave a mixture of sodium acetate and sodium 2-nitropropane-1-sulphonate, which was reduced with hydrogen and nickel in the usual way, giving 2-aminopropane-1-sulphonic acid, m. p.  $325$ – $328^{\circ}$  (decomp.).

**Sodium 1-Nitro-2-methylpropane-2-sulphonate.**—(a) *Using sodium hydrogen sulphite.* This reaction was carried out in the normal way using 1-nitro-2-methylprop-1-ene (Levy and Scaife, in the press) in place of the nitropropenes as described above. Recrystallisation from alcohol gave sodium 1-nitro-2-methylpropane-2-sulphonate monohydrate in 95% yield (Found: C, 22.3; H, 4.2; N, 6.1.  $C_4H_8O_5NSNa, H_2O$  requires C, 21.5; H, 4.5; N, 6.3%).

(b) *Using sulphurous acid.* The nitro-olefin (10.1 g.) was shaken for 5 days at room temperature in a stoppered bottle with aqueous sulphurous acid (100 c.c. saturated at  $0^{\circ}$ ) in an atmosphere of nitrogen. The solution was neutralised with sodium carbonate and worked up in the normal manner, giving sodium 1-nitro-2-methylpropane-2-sulphonate in 55% yield. In a parallel experiment in which the sulphurous acid solution had been prepared in the presence of air, the only isolated product was sulphamic acid, m. p.  $212^{\circ}$  (decomp.), obtained in 31% yield, identical with an authentic specimen (Found: H, 3.0; N, 14.3; S, 32.6. Calc. for  $H_2O_3NS$ : H, 3.1; N, 14.4; S, 33.0%).

**1-Amino-2-methylpropane-2-sulphonic Acid.**—The nitrosulphonic acid (above) (5 g.) in water (50 c.c.) was reduced with hydrogen and Raney nickel, and the product isolated as described for 2-aminoethane-sulphonic acid. **2-Amino-2-methylpropane-2-sulphonic acid** separated from aqueous alcohol as colourless crystals, m. p. 350—360° (decomp.), in 75% yield (Found : C, 31.0; H, 7.2; N, 8.8.  $C_4H_{11}O_3NS$  requires C, 31.3; H, 7.2; N, 9.1%).

**Sodium 2-Nitrobutane-3-sulphonate.**—2-Nitrobut-2-ene (50.5 g.; this series, Part III) was added dropwise to a cooled solution of sodium hydrogen sulphite (52 g.) in water (120 c.c.). The reaction was carried out in an atmosphere of nitrogen, and stirring was continued for 16 hours. Unreacted 2-nitrobut-2-ene was removed by extraction with ether, the aqueous solution evaporated to dryness under reduced pressure, and the residue extracted with absolute alcohol from which, by evaporation, a pale yellow viscous syrup (80 g.) was obtained. A crystalline solid (10 g.) (see below) separated from this syrup on standing. The syrup, apparently crude sodium 2-nitrobutane-3-sulphonate, gave a *p*-toluidine salt which decomposed below its melting point (Found : C, 45.5; H, 5.8; N, 9.1.  $C_4H_9O_3NS, C_7H_9N$  requires C, 45.5; H, 6.2; N, 9.6%). Reduction of the syrup with Raney nickel and hydrogen, followed by isolation in the usual way, gave **2-aminobutane-3-sulphonic acid**, m. p. 267—270°, in 60% yield (Found : C, 31.3; H, 6.9; N, 9.5.  $C_4H_{11}O_3NS$  requires C, 31.4; H, 7.2; N, 9.2%). The crystalline solid (above) could not be identified with certainty. It may be sodium 2-oximinobutane-3-sulphonate (Found : C, 24.7; H, 4.4; N, 7.4. Calc. for  $C_4H_9O_4NSNa$  : C, 25.4; H, 4.2; N, 7.4%). The *p*-toluidine salt had m. p. 186—188° (Found : C, 47.1; H, 6.6; N, 10.4. Calc. for  $C_4H_9O_4NS, C_7H_9N$  : C, 48.1; H, 6.6; N, 10.2%). On reduction with hydrogen and Raney nickel in the usual way it gave an amino-acid, m. p. 320—324° (decomp.), in 60% yield (Found : C, 30.6; H, 6.8; N, 9.0. Calc. for  $C_4H_{11}O_3NS$  : C, 31.4; H, 7.2; N, 9.2%).

**2-Nitro-4-methylpent-2-ene.**—To a cooled mixture of isobutaldehyde (36 g.) and diethylamine (10 g.), nitroethane (37.5 g.) was added during 30 minutes, the temperature being kept at 20—30° by intermittent cooling. The mixture was kept at 20° for 15 hours, acidified (2*N*-hydrochloric acid), extracted with ether, and the dried extract fractionated, giving **2-nitro-4-methylpent-2-ene** (40.5 g.; 65%), b. p. 67°/10 mm., as a pale yellow lachrymatory oil (Found : C, 56.2; H, 8.1; N, 10.9.  $C_6H_{11}O_2N$  requires C, 55.8; H, 8.5; N, 10.8%).

**2-Amino-4-methylpentane-3-sulphonic Acid.**—2-Nitro-4-methylpent-2-ene (12.9 g.) in methyl alcohol (35 c.c.) was added dropwise with stirring to a solution of sodium hydrogen sulphite (24 g.) in water (100 c.c.) and methyl alcohol (35 c.c.) at 20°. The mixture was maintained at 30° for 16 hours, evaporated, and extracted with hot absolute alcohol. Concentration of this extract yielded crude sodium 2-nitro-4-methylpentane-3-sulphonate as a pale yellow viscous syrup (yield, 99%), which could not be crystallised. Reduction in aqueous solution with Raney nickel and hydrogen at 25° and 100 atms. (initial pressure) gave **2-amino-4-methylpentane-3-sulphonic acid**, white crystals from aqueous alcohol, decomp. > 340°, in 75% yield (Found : C, 40.0; H, 8.3; N, 7.7.  $C_6H_{15}O_3NS$  requires C, 39.7; H, 8.3; N, 7.7%).

**Sodium 1-Nitro-2-phenylethane-2-sulphonate and 1-Amino-2-phenylethane-2-sulphonic Acid.**—(a) *From sodium hydrogen sulphite.* A solution of 2-nitrostyrene (14.9 g.) in dioxan (60 c.c.) was added slowly to a well-stirred solution of sodium hydrogen sulphite (15 g.) in water (90 c.c.) at 20°, and stirring then continued at 30° for 10 hours. Unchanged nitrostyrene was removed with ether, and the aqueous dioxan solution evaporated to dryness. The residue was crystallised from hot absolute alcohol, giving **sodium 1-nitro-2-phenylethane-2-sulphonate** as white crystals, unmolten at 300°, in 80% yield (Found : N, 5.2.  $C_8H_9O_3NSNa$  requires N, 5.5%). Reduction in aqueous solution with Raney nickel and hydrogen at ordinary temperature and pressure, followed by working up in the normal way, afforded **1-amino-2-phenylethane-2-sulphonic acid** in 70% yield, pearly plates from aqueous alcohol, m. p. 358—360° (decomp.) (Found : C, 47.1; H, 5.3; N, 7.0.  $C_8H_{11}O_3NS$  requires C, 47.7; H, 5.5; N, 7.0%).

(b) *From sulphurous acid.* 2-Nitrostyrene (10 g.) was shaken with saturated aqueous sulphurous acid (100 c.c.) in a stoppered bottle in the absence of oxygen for 6 days at room temperature. The unreacted nitrostyrene (2.0 g.) was removed by filtration and the solution neutralised with solid sodium carbonate. The crude nitro-acid was reduced with Raney nickel and hydrogen, giving the amino-acid, m. p. 358—360° (decomp.), in 56% yield.

**Sodium 1-Nitro-2-(2-furyl)ethane-2-sulphonate.**—1-Nitro-2-(2-furyl)ethylene (13.9 g.; Thiele and Landers, *Annalen*, 1909, **369**, 303) in dioxan (50 c.c.) was slowly added to a stirred solution of sodium hydrogen sulphite (24 g.) in water (100 c.c.) and dioxan (50 c.c.) at 25°, and stirring continued for a further 16 hours. The solution was evaporated to dryness, and the residue, extracted with absolute alcohol, gave **sodium 1-nitro-2-(2-furyl)ethanesulphonate** as a colourless syrup which crystallised slowly on keeping, but could not be recrystallised (Found : C, 26.2; H, 4.2; N, 5.4.  $C_6H_6O_4NSNa, 2H_2O$  requires C, 25.8; H, 3.6; N, 5.0%). Reduction with Raney nickel and hydrogen in methyl alcohol at ordinary temperature and pressure gave **1-amino-2-(2-furyl)ethane-2-sulphonic acid**, white crystals from aqueous dioxan, m. p. 250—252° (decomp.), in 30% yield (Found : N, 7.0.  $C_6H_6O_4NS$  requires N, 7.3%).

**1-Amino-1-carbethoxy-2-methylpropane-2-sulphonic Acid.**—Ethyl  $\alpha$ -nitro- $\beta\beta$ -dimethylacrylate (5 g.; Bouveault and Wahl, *Compt. rend.*, 1900, **131**, 687, 748) was added dropwise to a vigorously stirred solution of sodium hydrogen sulphite (5 g.) in water (20 c.c.) at 30°, and stirring was continued for a further 20 hours. The solution was evaporated under reduced pressure and extracted with hot absolute alcohol to give crude sodium 1-nitro-1-carbethoxy-2-methylpropane-2-sulphonate as a colourless glass in 84% yield. Reduction of the above salt (6.7 g.) in water (60 c.c.) with Raney nickel and hydrogen at ordinary temperature and 90 atms. (initial pressure), followed by evaporation of the filtered solution, afforded **sodium 1-amino-1-carbethoxy-2-methylpropanesulphonate**, white crystals from aqueous alcohol, in 45% yield (Found : N, 5.4.  $C_7H_{14}O_5NSNa$  requires N, 5.7%). Acidification of an aqueous solution of the sodium salt with concentrated hydrochloric acid gave the free **amino-sulphonic acid**, white crystals from aqueous alcohol, m. p. 223°, in 34% yield (Found : N, 6.0.  $C_7H_{14}O_5NS$  requires N, 6.2%).

**Sodium 1-Nitro-2 : 4 : 4-trimethylpentane-2-sulphonate.**—A mixture of 1-nitro-2 : 4 : 4-trimethylpent-1-ene (5 g.; see Levy, Scaife, and Baldock, in the press), dioxan (15 c.c.), and saturated aqueous sodium hydrogen sulphite (50 c.c.) was stirred at 20° for 8 hours. After concentration under reduced

pressure at 20°, the residual white solid was extracted with alcohol, filtered, and the extract treated with dry ether. A white crystalline salt was precipitated, and purified by reprecipitation from alcohol with ether. *Sodium 1-nitro-2:4:4-trimethylpentane-2-sulphonate* was obtained as white crystals, m. p. 189° (decomp.) (Found: N, 5.4; S, 12.8.  $C_8H_{16}O_6NSNa$  requires N, 5.4; S, 12.3%).

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