

added. The mixture was heated for fifteen minutes on a steam-bath and then diluted with 300 ml. of water. The precipitation and purification of the base was as described in method A.

SAINT AUGUSTINE'S COLLEGE
RALEIGH, NORTH CAROLINA RECEIVED AUGUST 20, 1946

NEW COMPOUNDS

3,4-Dimethoxy-10-methyl-2,4,7-naphthitatrien-1-one¹

Methylation of the sodium derivative from 1 g. of 2-methoxy-5-methyl-2,7-naphthitadiene-1,4-dione² (0.13 g. of sodium in liquid ammonia) in benzene using 2 g. of methyl iodide, which was added in three portions during twenty-six hours, gave 657 mg. of crude crystals. One recrystallization from benzene followed by two from hexane gave nearly colorless needles, m. p. 136–137° (cor.).

*Anal.*³ Calcd. for C₁₃H₁₆O₃: C, 70.9; H, 7.3. Found: C, 70.5; H, 7.1.

In a second experiment 5 g. of the monomethoxydione gave 4.3 g. of crude dimethyl ether; once crystallized from hexane, it melted at 129–132°. A few mg. on a steam-bath with three drops of concentrated hydrochloric acid for twenty minutes gave 5-methyl-7-naphthitene-1,2,4-trione enol, m. p. 165–169°; mixed with authentic material⁴ m. p. 166–170°.

(1) This work was supported by an allotment from the Special Research Fund (Bankhead-Jones Act of June 29, 1935). Not subject to copyright.

(2) Orchin and Butz, *J. Org. Chem.*, **8**, 509 (1943).

(3) By Dr. T. S. Ma, University of Chicago.

(4) Butz and Butz, *J. Org. Chem.*, **8**, 497 (1943).

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Salicylobutylamide and *o*-Nitroacetophenone 2,4-Dinitrophenylhydrazone¹

Salicylobutylamide.—Methyl salicylate (108 g.) and *n*-butylamine (60 g.) were refluxed together for seven hours. Volatile material was then expelled up to 160° (30 mm.) and the 125 g. of sirupy residue was distilled at 153–156° at 3 mm. The salicylobutylamide was a colorless, viscous sirup.

Anal. (by R. Pivan) Calcd. for C₁₁H₁₅NO₂: N, 7.35. Found: N, 7.43.

***o*-Nitroacetophenone 2,4-Dinitrophenylhydrazone.**—A mixture of 2 g. of 2,4-dinitrophenylhydrazine, 1.5 g. of *o*-nitroacetophenone and 100 ml. of alcohol was heated to boiling, 2 ml. of concd. hydrochloric acid was added, and the mixture was boiled under a reflux condenser for five minutes. Copious separation of crystals occurred on cooling. Recrystallization from alcohol gave a pure product, m. p. 152–154° cor.

Anal. (by W. Brandt) Calcd. for C₁₄H₁₁O₆N₃: N, 20.29. Found: N, 20.18.

(1) This work was done under contracts, recommended by the National Defense Research Committee and the Committee on Medical Research, between the Office of Scientific Research and Development and Northwestern University.

No success attended efforts to synthesize *o*-nitroacetophenone oxime by direct reaction of the ketone with hydroxylamine, although apparently such a compound can be made² by reaction of a nitrite on *o*-nitroethylbenzene.

(2) Hochster Farbw., German Patent 109,663; *Chem. Zentr.*, **71**, II, 458 (1900).

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RECEIVED MAY 16, 1946

A New Acetylene Silver Nitrate Complex

The customary precipitate formed by interaction of acetylene with aqueous silver nitrate solutions has been considered to have the formula Ag₂C₂·AgNO₃, having a crystal form of fine needles and crosses. We find this conclusion to be true for only dilute solutions. As the silver nitrate concentration of the solution is raised above about 10%, there is a tendency for rhombohedral crystals to form and above about 25% silver nitrate concentration the rhombs form to the exclusion of the needles. We have found these rhombs to have the formula of Ag₂C₂·6AgNO₃.

When 80 ml. of acetylene is bubbled through 25 ml. of 30% aqueous silver nitrate at 25° and atmospheric pressure, the acetylene is at first completely absorbed to form a clear solution, followed toward the end of the absorption by the appearance of the white rhombohedral crystals. The mixture is then filtered by suction through a fritted glass filter, washed with acetone and dried at first by suction and then in a vacuum desiccator over sulfuric acid. These rhombs were analyzed by us for silver carbide and total silver gravimetrically and for water-soluble silver volumetrically. We previously found that an acidimetric titration of the filtrate from the rhombs showed free acid equivalent to that customarily released by the acetylene present.

Anal. Calcd. for Ag₂C₂·6AgNO₃: Ag₂C₂, 19.19; AgNO₃, 80.81. Found: Ag₂C₂, 19.13; AgNO₃, 81.30 (molar ratio 1 to 5.97).

This substance crystallizes in the rhombohedral system; it has a characteristic X-ray diffraction pattern, differing from that of AgNO₃ or Ag₂C₂·AgNO₃. It has a great tendency to form supersaturated solutions. Upon heating, it first melts and then suddenly but feebly decomposes with the evolution of red vapors of nitrogen oxides above 308° and below 327°, the respective melting points of sodium nitrate and metallic lead. This datum is to be contrasted with a detonating point of about 212° which we have found for Ag₂C₂·AgNO₃.

Water decomposes the rhombs to Ag₂C₂·AgNO₃ and silver nitrate. Likewise, any reagent that acts to reduce the silver nitrate concentration of the solution in which the rhombs may be existing tends to destroy them in favor of the precipitation of Ag₂C₂·AgNO₃. An excess of acetylene added to the solution will act in this manner, as acetylene removes about fifty times its own weight of silver nitrate from solution under these conditions. Conversely, if rhombs and needles exist in the same solution the needles can be completely removed by increasing the silver nitrate concentration, the normal silver carbide being very soluble in strong silver nitrate.

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