

the reduction products an approximate idea of the rate of decomposition may be obtained.

When a solution of the oxide obtained by partial reduction of the acid was refluxed and the decomposition followed as outlined above by removing test portions, it was found that the decomposition progresses very rapidly at first so that 50% of the oxide has been decomposed after 40 minutes. At the end of 3 hours the reaction was 75% complete and had become very slow.

The arsenic determinations were made according to Lehmann's method.

Summary.

1. The indirect reduction of 3-amino-4-hydroxy-phenyl-arsenic acid to arsphenamine has been investigated and a method developed for the preparation of a relatively nontoxic arsphenamine which is readily soluble in water.

2. Like the direct reduction of 3-amino-4-hydroxy-phenyl-arsenic acid this procedure avoids the isolation of arsphenamine base and has the additional advantage of producing the substance in a readily soluble condition.

3. The rate of decomposition of 3-amino-4-hydroxy-phenyl-arsenic oxide in boiling acid solution has been followed; the final product identified as *o*-aminophenol and 3,3'-diamino-4,4'-dihydroxy-diphenyl-arsenic oxide was found to be an intermediate.

4. The formation of arsphenamine polyarsenide has been investigated and some of the properties of this compound have been recorded.

The writer wishes to express his appreciation to Dr. Reid Hunt for testing the products biologically and to Mr. Lewis I. Nurenberg of the Arsphenamine Laboratory of the Massachusetts Department of Health, for preparing several lots of arsphenamine by this method.

BOSTON, MASS.

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5-NITRO-4-HYDROXY-3-METHYL-BENZOIC ACID.

BY K. PFISTER.

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In the course of an investigation the chance presented itself of correcting an error in the older chemical literature which appears to have passed unnoticed; at least no criticism regarding it could be found in the more recent literature.

In 1882 R. W. Mahon nitrated at a high temperature the so-called *p*-hydroxy-*m*-toluic acid (4-hydroxy-3-methyl-benzoic acid) and obtained another acid which he regarded as a mononitro derivative.¹ That such

¹ Mahon, *Am. Chem. J.*, 4, 186 (1882).

a constitution of Mahon's acid is not in accord with our present knowledge of organic compounds is evident from Mahon's own description of its properties, such as its low melting point, its volatility with water vapor, its marked tinctorial power and so forth. These properties are, however, identical with those of dinitro-*o*-cresol (2-methyl-4,6-dinitro-phenol) which substance evidently Mahon had in hand. It was formed from the starting material by the introduction of one nitro group and the displacement of the carboxylic group by a second nitro group as a result of the violent method employed.

The true nitro-*p*-hydroxy-*m*-toluic acid (5-nitro-4-hydroxy-3-methylbenzoic acid) is easily obtained in a nearly theoretical yield by nitrating the *p*-hydroxy-*m*-toluic acid with mixed acid under the usual precautions.

It crystallizes from strong acetic acid or benzene in small, pale lemon-yellow needles, m. p. 240°; it is nearly insoluble in cold and only slightly soluble in hot water; it further proved to be somewhat soluble in hot benzene and considerably more so in ether, alcohol, and glacial acetic acid.

Subs., 0.5546: (Kjeldahl-Yodlbauer) 14.3 cc., 0.2 *N* HCl. Calc. for C₈H₇O₅N: N, 7.1. Found: 7.2.¹

The methyl ester of this nitro-acid forms crystals of a more pronounced lemon-yellow color than the nitro-acid itself and melts at 103°; on reduction of the methyl ester the corresponding amido compound is obtained, which melts at 141°.

Summary.

It has been discovered that the compound described by Mahon² as nitro-*p*-hydroxy-*m*-toluic acid was in reality dinitro-*o*-cresol.

The genuine acid of the former constitution as well as a few of its derivatives have been prepared.

Therefore, the place in organic chemical literature which has been assigned to Mahon's acid³ belongs to the acid briefly described in this paper.

ROSEBANK, STATEN ISLAND, NEW YORK.

¹ The analysis was made by Mr. V. Vesce.

² *Loc. cit.*

³ Beilstein, Vol. II, 1549, 1896, under the name of "5-Nitro-oxytoluyl-saeure" and in Richter's "Lexikon," Vol. I 1910, 911, No. 18, under the name of "5-Nitro-6-Oxy-1-Methyl-benzol-3-Carbonsaeure."