the mercury levels in the still-head and filling tube *before* starting the still. This latter method has the added advantage that any slight pressure in the still is automatically compensated for.

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Adaptation of the Diphenylcarbazide Test for Mercury to the Scheme of Qualitative Analysis.—The diphenylcarbazide test for mercury is very delicate, detecting 0.0005 mg. per 2 cc., but its use so far has been restricted to practically neutral solutions.[1]

However, in the confirmation of mercury, both in Group I and in Group II, the solutions are decidedly acid. It has been found that by adding an excess of solid sodium carbonate to such solutions, the presence of considerably less than 0.1 mg. of mercury per cc. can be detected very readily. The procedure outlined below has been used for the past two years with marked success in qualitative analysis courses in this University.

The mercury precipitate in Group I is dissolved in aqua regia, that of Group II is dissolved in hydrochloric acid and sodium chlorate. In either case the solution is evaporated to a volume of about 1 cc., placed in a test-tube and diluted with 5 to 6 cc. of water. Four to eight drops of a saturated alcoholic solution of diphenylcarbazide is added and a large excess of solid sodium carbonate is gradually dropped into the solution. When mercury is present, the foam produced on neutralization assumes a blue tinge, and after the addition of an excess of carbonate the entire solution turns blue.

At the conclusion of the experiment the solution should either be blue or orange to pink in color. If the solution remains colorless, it indicates that the diphenylcarbazide solution has deteriorated.

Freshly prepared diphenylcarbazide solution is colorless, but soon develops a pink to red color. This color does not seem to affect its use for the test. However, on standing for several weeks it finally fails to give a blue color in the presence of mercuric salts. Aqua regia does not interfere provided the test is completed soon after the addition of the diphenylcarbazide solution. If the test solution containing aqua regia is allowed to stand for some hours after the addition of diphenylcarbazide, it will fail to develop any color when an excess of sodium carbonate is added.

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[1] Kolthoff, *Chem. Weekblad,* **21**, 20 (1924); Stock and Pohland, *Z. angew. Chem.,* **39**, 791 (1926).

Neither sodium acetate nor sodium bicarbonate can be successfully substituted for the sodium carbonate in this test.

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THE MERCURATION OF HEMIMELLITIC ACID

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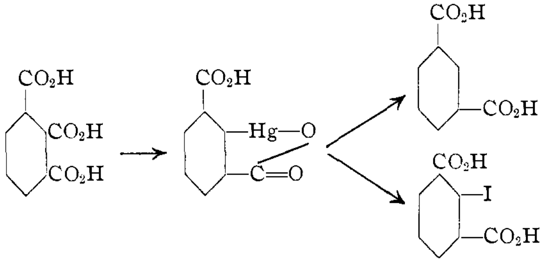
Since the treatment of 3-nitrophthalic acid with mercuric acetate[1] gives over 90% of the calculated amount of the product in which the 2-carboxyl group is replaced by mercury, it seemed desirable to study the effect of other groups on the replacement of one of two ortho carboxyl groups. The simplest case of this kind is presented by hemimellitic acid (benzene-1,2,3-tricarboxylic acid). It was found that the 2-carboxyl is replaced by mercury. No evidence of any replacement of the 1- or 3-carboxyl was found. The structure of the mercury compound was determined by replacing the mercury by hydrogen to give isophthalic acid and by iodine to give 2-iodo-isophthalic acid.



The best method of obtaining the 2-halogen isophthalic acids is undoubtedly by this procedure, starting with hemimellitic acid prepared from acenaphthene.

Experimental

Hemimellitic acid was prepared by a modification of the method of Graebe and Leonhardt.[2] From 100 g. of naphthalic anhydride was obtained 46 g. of hemimellitic acid (44% yield), m. p. 201–203° with decomposition to form the anhydride.

A method is being developed in this Laboratory for the preparation of hemimellitic acid by the direct oxidation of acenaphthene.

A solution of 40 g. (0.2 mole) of hemimellitic anhydride in 110 cc. of 6 *N* sodium

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[1] Whitmore and Culhane, THIS JOURNAL, **51**, 602 (1929).

[2] Graebe and Leonhardt, *Ann.*, **290**, 218 (1896).