Organic Spot Tests. II. Detection of 1,2-Dihydroxy Compounds (Differentiation of Polyhydroxy Compounds)

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The micro detection of acid substances by the color reaction with the mixed solution of sodium sulfanilate, sodium nitrite and α -naphthylamine in aqueous alcohol or dioxan¹⁾ has been developed to a new method of detection of 1, 2-dihydroxy compounds.

Boric acid is so weak that the orange to orange red color begins to appear only in $10\sim15\,\mathrm{min}$. after adding it to the reagent. It has long been known that addition of polyhydroxy compounds, e.g., glycerol, sorbitol and mannitol, to boric acid raises the acidity²⁾ and thus this weak acid can be titrimetrically determined³⁾. This is due to the co-ordinate complex ester (I)⁴⁾ of boric acid with 1, 2-dihydroxy compounds as follows:

It has been found that boric acid behaves as a strong acid in the presence of a 1,2-dihydroxy compound also in the above-mentioned reaction to give immediately an intense coloration and that boric acid reciprocally makes it possible to detect 1,2-dihydroxy compounds by the same color reaction. Thus, ethylene glycol, propylene glycol, glycerol and other various polyhydroxy compounds with adjacent hydroxyl groups gave posi-

tive reactions with the reagent in the presence of boric acid but trimethylene glycol was indifferent. Catechol also gave a positive reaction, while resorcinol and hydroquinone gave no reaction.

peri-Dihydroxy derivatives of naphthalene behave similarly in this new color reaction. Namely, disodium chromotropate (1,8-dihydroxynaphthalene-3,6-disulfonate) gave the same coloration when the above method was applied.

In the case of a polyhydroxybenzoic acid, its small quantity is evaporated to dryness with one drop of ammonia water in a microcrucible on a water bath and then it may be tested. In this way, 2,3-dihydroxybenzoic acid and gallic acid gave an intense red color, but 2,4-, 2,6- and 3,5-dihydroxybenzoic acids did not.

Accordingly the reaction described above gives a new method of detecting the presence of two hydroxyl groups in 1,2-positions.

General Procedure of the Test.—Several drops of the reagent solution are placed in a depression of a spot plate. A small amount of boric acid is added to it and stirred with a glass rod. One drop of the test solution or a pinch of the solid sample is added to it. An immediate orange red color indicates the presence of a compound with two or more adjacent hydroxyl A blank test without addition of the sample, is recommended if only a small amount of a 1,2-dihydroxy compound is suspected. An acid substance which gives a positive reaction with the reagent solution alone (without the addition of boric acid) must be treated with ammonia water as in the case of the polyhydroxybenzoic acids before the present method is applied.

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Y. Nomura, This Bulletin, 32, 536 (1959).

²⁾ L. Vignon, Compt. rend., 78, 148 (1874); G. Bouchardat, ibid., 80, 120 (1875); D. Klein, ibid., 86, 526 (1878); 99, 144 (1884).

³⁾ W. Horsch, Anal. Abstr., 2531 (1957).

⁴⁾ Cf. J. Böeseken, "Advances in Carbohydrate Chemistry", Vol. 4, Academic Press, Inc., New York (1949), p. 193-210.