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Short Communications

A Colour-Reaction for Detection of Methylenedioxy Groups

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All methods for detection of methylenedioxy groups are based on splitting off formaldehyde with hot sulphuric acid and detection of this by some specific reaction.

Labat [1], Pictet and Cramers [2], and Leonhart and Fay [3] made use of the green colour produced by gallic acid, whereas Weber and Tollens [4] carried out the reaction with phloroglucinol dissolved in concentrated sulphuric acid. The formaldehyde split off reacts with phloroglucinol forming an insoluble amorphous precipitate while the solution turns red. Clowes and Tollens [5] used the same procedure in a semiquantitative analytical method for determination of methylenedioxy groups.

Eegriwe [6] showed that chromotropic acid (1,8-dihydroxy-3,6-naphthalenedisulphonic acid) produces a purple colour with formaldehyde in strong sulphuric acid, this being a very sensitive and specific reaction. Also compounds yielding formaldehyde under the conditions used in the reaction produce the colour. Consequently the reaction can be used for detection of methylenedioxy groups as well.

Unfortunately many compounds give red colours with sulphuric acid alone. By diluting with water the dark by-products are precipitated while the colour produced by formaldehyde and chromotropic acid remains.

Piperonal, safrole, narcotine, piperine, berberine, methysticin, *o,o'*-di-methoxymethoxy-benzilic acid, *o*-methoxymethoxybenzaldehyde, and methylene-di-*p*-chlorphenylether have been used as test substances. The reaction has been checked on a great variety of compounds not giving off formaldehyde with hot strong sulphuric acid, and no implications have been noted from any of them. Eegriwe [6] found that a red colour was produced with furfural. With this modification in procedure, however, the test with furfural is found to be negative.

Procedure.

1) About 4—5 mg of the compound are dissolved in 4—5 ml of 90 % sulphuric acid. The solution is divided into two equal parts in two test tubes, and 4—5 mg of chromotropic acid are added to one of them.

The test tubes are kept at 70—80° for 20 minutes. After cooling 10 ml of water are added to each test tube. If necessary, the solutions are filtered. If methylenedioxy groups are present, the solution containing chromotropic acid is purple to bluish-purple while the other solution is colourless or faintly yellow.

2) About 0.2 mg of the compound is dissolved in 4—5 drops of 90 % sulphuric acid. The solution is divided into two equal parts in two small centrifuge cones (1 ml), and about 1 mg of chromotropic acid is added to one of them. The cones are kept at 70—80° for 20 minutes. Two drops of water are added to each cone, and the contents are stirred with a glass thread and centrifuged. As to the colours of the supernatant liquids, see procedure 1.

1. Labat, M. A. *Bull. soc. chim. France* (4) **5** (1909) 745.
2. Pictet, A., and Cramers, G. H. *Ber.* **43** (1910) 1334.
3. Leonhart, H., and Fay, K. *Arch. Pharm.* **273** (1935) 58.
4. Weber, K., and Tollens, B. *Ann.* **299** (1898) 318.
5. Clowes, G. H. A., and Tollens, B. *Ber.* **32** (1899) 2841.
6. Eegriwe, E. *Z. anal. Chem.* **110** (1937) 22.

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