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Note

A spray reagent for the detection of terpene derivatives on thin-layer plates

J. C. KOHLI

Department of Chemistry and Biochemistry, Punjab Agricultural University, Ludhiana (India)

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A number of spray reagents for the detection of terpene derivatives on silica gel G thin-layer plates are known[1]. Anisaldehyde–sulphuric acid detects terpenes in addition to sugars and steroids. Very sensitive but non-specific detection is possible using the molybdophosphoric acid reagent. After spraying and heating, amounts as small as 0.05–1 μg can be detected as blue spots on silica gel layers. Antimony tri- and pentachloride reagents are less sensitive and give grey to violet colours. When they are used, the chromatogram should be examined under natural light and long-wave UV light both before and after the heat treatment. Limited use has been made of antimony trichloride as a spray reagent for the identification of isoprenoids[2]. Other spray reagents, such as tin tetrachloride, chlorosulphonic acid–glacial acetic acid and acetic anhydride–sulphuric acid, have been used for detecting triterpenes. Benzidine detects only terpene aldehydes.

It was, therefore, considered necessary to find a sensitive spray reagent that was suitable for the detection of various types of terpene derivatives. This is achieved by spraying with a 1:1 (w/v) solution of arsenic trichloride in glacial acetic acid. This reagent is capable of detecting different types of terpene derivatives as listed in Table I. The colours reported are produced at the 3-μg level, although the reagent appears to be sensitive at concentrations down to 0.5 μg for many terpene derivatives. Grey to light green spots were observed against a light blue background after heating the developed plates for 5 min at 100°.

The reagent gives the observed colour reaction for detecting terpenoid deriva-

TABLE I

DETECTION OF TERPENE DERIVATIVES (3-μg LEVEL) BY SPRAYING WITH A SOLUTION OF ARSENIC TRICHLORIDE IN ACETIC ACID

Compound	*Colour observed*
Khusinol	Grey
Khusinodiol	Yellowish brown
Khusilal	Yellowish green
Khusilic acid	Light green
Khusilic acid methyl ester	Light green
Citronellol	Light brown
Nerol	Dark brown
Citral	Dark brown

tives due to the complex-formation ability of arsenic, which can be further enhanced due to activation by the conveniently located neighbouring group. Interestingly, an activation effect was observed with khusinol, in which a methylenic double bond is present in the position nearest to the secondary hydroxyl group so that the colour reaction occurs immediately.

Kohli *et al.*[3] have already confirmed the neighbouring group participation by secondary hydroxyl groups using antimony trichloride as a Lewis acid. However, antimony has a lower tendency than arsenic to undergo complex formation. A deactivation effect was noticed in the absence of neighbouring group participation. Thus, khusilal, khusilic acid and khusilic acid methyl ester did not produce a colour immediately. Owing to the lack of authentic samples, it has not been possible to test for reactions with diterpene or triterpene derivatives, but these terpenes, containing activated groups, are likely to give characteristic colour reactions.

REFERENCES

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3 J. C. Kohli, M. S. Wadia and P. S. Kalsi, *Indian J. Chem.*, 10 (1972) 1130.