

The detection and separation of the diphenyliodonium ion in the presence of thallium(I), tetraphenylarsonium and tetraphenylphosphonium ions

Earlier studies on the analytical properties of the diphenyliodonium ion have shown that the tetraphenylarsonium, tetraphenylphosphonium and thallium(I) ions seriously interfere with the determination of the diphenyliodonium ion^{1,2}. The present note describes chromatographic methods of separating these ions. The separation is effected on paper impregnated with liquid paraffin using 78 % isopropanol-22 % v/v water as eluent. The compounds are detected by spraying the dried paper with a mixture of dimethylformamide in ethanol and a carbon tetrachloride solution of dithizone. This reagent has the advantage of producing a coloured spot with the diphenyliodonium compound quite distinct from those of the other two onium compounds and also from the thallium(I) ion which in many of its reactions resembles the diphenyliodonium ion³. The colours obtained, the R_F values and the limits of detection are given in Table I. A carbon tetrachloride solution of dithizone without the addition of dimethylformamide gives buff coloured spots with the three onium ions; a red spot is still obtained with the thallium(I) ion.

TABLE I

SEPARATION AND DETECTION OF CERTAIN ONIUM AND THE THALLIUM(I) IONS ON PAPER IMPREGNATED WITH LIQUID PARAFFIN USING 78 % V/V ISOPROPANOL-22 % V/V WATER AS ELUENT

<i>Ion</i>	<i>R_F value</i>	<i>Colour with dithizone-dimethylformamide reagent</i>	<i>Limit of detection (μg)</i>
Tetraphenylarsonium	0.85	Salmon pink	2.0
Tetraphenylphosphonium	0.85	Salmon pink	1.6
Diphenyliodonium	0.79	Yellow	2.0
Thallium(I)	0.07	Red	0.2

An alternative procedure which is not quite so sensitive as that outlined above but which gives somewhat better separation involves the use of commercially prepared thin-layer sheets (MN-Polygram Cel 300) with an isopropanol-ammonia eluent. The results obtained with this system are summarized in Table II.

TABLE II

SEPARATION AND DETECTION OF CERTAIN ONIUM AND THE THALLIUM(I) IONS ON MN-POLYGRAM CEL 300 SHEETS USING ISOPROPANOL-AQUEOUS AMMONIA AS ELUENT

<i>Ion</i>	<i>R_F value</i>	<i>Colour with dithizone-dimethylformamide reagent</i>	<i>Limit of detection (μg)</i>
Tetraphenylarsonium	0.95	Salmon pink	6
Tetraphenylphosphonium	0.95	Salmon pink	6
Diphenyliodonium	0.80	Yellow	6
Thallium(I)	0.09	Red	2

Experimental

Reversed-phase paper chromatography

Impregnating reagent. A 10% v/v solution of liquid paraffin (sp.gr. 0.850-0.865) in cyclohexane or benzene.

Eluent. 78% v/v isopropanol-22% v/v water.

Spray reagent. A mixture of 8 parts of a 0.05% w/v solution of dithizone in carbon tetrachloride and 5 parts of a 40% v/v solution of dimethylformamide in ethanol.

Procedure. Saturate Whatman No. 1 filter paper with the impregnating reagent, blot off the excess reagent and allow the paper to dry at room temperature. Spot aqueous or methanol sample solution onto the paper (10 × 10 cm sheets), dry the spots and elute with the isopropanol-water mixture (1.5-2 h). Dry the paper in a current of warm air and develop the spots with the spray reagent. It is advantageous to spray twice, drying the paper completely after each spraying.

Thin layer chromatography

Thin layer sheets, MN-Polygram Cel 300 manufactured by Macherey, Nagel and Co., Duren, were used.

Eluent. A 9:1:4 mixture by volume of isopropanol, 0.880 ammonia and water.

Spray reagent. As for the reversed phase method.

Procedure. The sample solutions are spotted, dried, eluted, and sprayed as above. Elution times of 1-2 h are required.

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