Journal of Chromatography, 320 (1985) 455–456
Elsevier Science Publishers B.V., Amsterdam — Printed in The Netherlands

CHROM. 17,352

Note

Improved procedure for the thin-layer chromatography of terpenoids on silver ion–silica gel layers

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(Received October 31st, 1984)

Thin-layer chromatography on silver nitrate–silica gel layers, first introduced[1–3] in the early 1960s, has now become an accepted technique for the separation of terpenoids. However, no significant modification to the original technique has been introduced and the original procedures involving the use of silica gel layers containing silver nitrate and gypsum continue to be employed. Prasad *et al.*[4] found that silver perchlorate is superior to silver nitrate for certain olefin separations. We have now found that silver iodate is superior to silver nitrate for certain terpenoid separations.

EXPERIMENTAL

Materials

Silica gel G was obtained from BDH Labs. Chemical Division (Bombay, India), silver nitrate (laboratory-reagent grade) from Sarabhai Chemicals (Baroda, India) and silver iodate from Wilson Laboratories (Bombay, India).

Preparation of plates

Silver nitrate or silver iodate (15 g) was dissolved in water (23 ml) and the solution slowly diluted with acetone (250 ml) with stirring, avoiding light. To the mixture was added silica gel (100 g) with stirring. After stirring for a further 15 min, the solvent was removed on a water-bath under water pump suction, avoiding exposure to light, to give a free-flowing powder. The products were stored in dark bottles.

The above preparations were slurried with water (2–2.5 parts) by gently rubbing in a pestle and mortar and plates (15 × 3.5 cm) were coated with the slurry with the aids of an applicator, giving 0.5 mm layers. The plates were dried in air for about 4 h at room temperature and finally activated at 110°C for 2.5 h and used after cooling in dry air for 10–15 min.

Thin-layer chromatography of terpenoids on silver ion–silica gel layers

The terpenoids, dissolved in diethyl ether, were applied on a thin-layer chromatographic plate 1 cm from the base. The plates were developed in benzene–ethyl acetate (4:1), then dried, sprayed with methanol–concentrated sulphuric acid (1:1) and heated in an electric oven at 110°C for 5 min to reveal the spots. The R_F values

0021-9673/85/$03.30

TABLE I

R_F VALUES OF TERPENOIDS ON SILVER ION–SILICA GEL PLATES

Solvent: benzene–ethyl acetate (4:1).

Compound	*R_F value*	
	Silver nitrate-impregnated plate	*Silver iodate-impregnated plate*
Khusinol	0.32	0.62
Khusol	0.31	0.40
Isopulegol	0.67	0.74
Cholesterol	0.42	0.56
Methyleugenol	0.19	0.18
Carotol	0.52	0.40
α-Ionone	0.59	0.50
β-Ionone	0.32	0.46
Carvone	0.50	0.56
Zerumbone	0.51	0.56
Santonin	0.26	0.22
Costunolide	0.31	0.48
Dehydrocostus lactone	0.23	0.30
Δ^3-Carene	0.36	0.43

of the terpenoids were noted using both silver nitrate- and silver iodate-impregnated silica gel plates.

RESULTS AND DISCUSSION

It can be seen from Table I that in all instances the R_F values are higher with the silver iodate than with the silver nitrate-impregnated plates. From the degree of separation of terpenoids, it can be shown that weight for weight silver iodate gives a superior resolution in some instances and in others it is at least as good as silver nitrate. Further, silver iodate has a silver content of 38.1% compared with 63.5% for silver nitrate. Although it is known[5] that the stability of silver ion–olefin complexes is dependent on the negative counter ion and that silver iodate is superior to silver nitrate as a complexing agent, it is difficult at present to explain the differences in the behaviour of terpenoids shown in Table I.

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