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Note

Separation and identification of some hydroxyxanthones by thin-layer chromatography

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As a part of our programme for studying the effect of chelation on the infrared carbonyl-stretching frequency of some groups of polyphenols[1,2], it became necessary to prepare very pure samples of simple hydroxyxanthones. The methods of Nencki or Grover *et al.* gave mixtures of isomeric hydroxyxanthones, polymeric products and unchanged starting materials[3,4]. Attempts to separate the hydroxyxanthones by previous methods[5] were only partially successful. In the present study, we have utilized our findings[6,7] on the effect of π-complex formation on R_F values and have achieved the separation and identification of some hydroxyxanthones.

EXPERIMENTAL

Glass plates (15 × 20 × 0.3 cm) coated with silica gel G (E. Merck, Darmstadt, G.F.R.) were allowed to stand for 14 h in a rectangular chromatographic tank containing a 2% solution of nitrobenzene in benzene. The plates were dried for 3 h in a desiccating cabinet before use. Methanolic solutions (0.1 ml) of the individual hydroxyxanthones[3,4] (Table I) and their mixture (1 mg/ml) were applied at the baseline with a microsyringe, dried and developed with benzene–xylene–ethyl formate–

TABLE I

R_F VALUES AND SPOT COLOURS OF HYDROXYXANTHONES ON SILICA GEL G PLATES IN METHANOL AT 25°

Compound	R_F	*Detection reagent*		
		Iodine vapour	*Alcoholic iron(III) chloride**	*15% Sulphuric acid*
Xanthone	0.75	Brown	Yellow-green	Pale yellow
1-Hydroxyxanthone	0.84	Brown	Yellow-green	Yellow
1,3-Dihydroxyxanthone	0.64	Brown	Green	Pale yellow
1,3,7-Trihydroxyxanthone	0.50	Brown	Green	Brown
1,8-Dihydroxy-2,6-dimethylxanthone	0.55	Pale brown	Dark green	Pale yellow
3,8-Dihydroxy-2,6-dimethylxanthone	0.69	Pale brown	Green	Yellow
1-Hydroxy-6-methoxy-3,8-dimethylxanthone	0.88	Brown	Yellow-green	Yellow-brown

* Colours observed under UV light.

formic acid (15:35:40:10) in a tank which had been previously saturated with the solvent vapour for 30 min. The spots were detected using iodine vapour, alcoholic iron(III) chloride and 15% sulphuric acid.

RESULTS AND DISCUSSION

The chromatographic results are shown in Table I. Each R_F value represents the mean of three determinations which only showed a slight difference in the last digit. Several other solvent systems were studied but these failed to separate one or more of the compounds. The degree of separation in this system is probably dependent on the formation of a loose complex between the adsorbed nitrobenzene and the substrate. We have previously separated isomeric flavonoid-C-glycosides, which are otherwise very difficult to separate, by using the above technique[6].

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