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Thin-layer chromatography of furanocoumarins

Thin-layer chromatography (TLC) of furanocoumarins has been described in a series of papers[1-3], but no satisfactory separation has been achieved for the eight compounds listed in Table I. While studying coumarin derivatives present in the roots of *Heracleum mantegazzianum* Somm. & Lev.[4], these simple furanocoumarins became of interest. Thus the development of TLC conditions necessary to separate these compounds was important.

TABLE I

R_F VALUES AND SPOT COLOURS OF FURANOCOUMARINS

No.	*Furanocoumarin*	*R_F values × 100*[a]		*Colour (fluorescence 254 nm)*
		Solvent 1	*Solvent 2*	
1	Psoralen	51	46	Blue
2	Bergapten	43	51	Yellow
3	Xanthotoxin	33	40	Yellow
4	Isopimpinellin	20	42	Brownish yellow
5	Angelicin	60	56	Blue
6	Isobergapten	58	60	Yellow
7	Sphondin	47	33	Blue
8	Pimpinellin	54	54	Brown

[a] The R_F values are the average values of ten chromatograms.

BEYRICH[1] studied the chromatographic separation of furanocoumarins on silica gel and reported R_F values in several solvent systems. However, when all eight furanocoumarins listed in Table I were present in a mixture, neither solvent system described gave clearly separated spots corresponding to the eight compounds. Formamide-impregnated silica layers[1] also cause considerable variation in the R_F values recorded.

BAERHEIM SVENDSEN described the use of aluminium oxide as an adsorbent in column chromatography of furanocoumarins[5-7]. We extended this work to TLC on aluminium oxide layers and wish to report the successful separation by two-dimensional TLC of the compounds listed in Table I.

We adopted the following method: Fifty grams of Aluminiumoxid G (Merck) were mixed with 60 ml of water, and the slurry was applied to 20 × 20 cm glass plates with a Desaga spreading device. The thickness of the layers was 0.25 mm. The coated plates were air-dried for 20 min at room temperature and were activated in a drying oven for 6 h at 110°. The samples were applied in diethyl ether solutions. The chromatograms were first developed in purified chloroform (Solvent 1), were air-dried and were then developed in dibutyl ether–ethyl acetate (88:12) (Solvent 2). After drying, the spots were detected by fluorescence in U.V. light (254 nm). The fluorescence of angelicin was first evident after spraying with 0.5 % KOH in methanol. The fluorescence colours vary from brown-red to blue and facilitate identification of the separated compounds on the chromatogram.

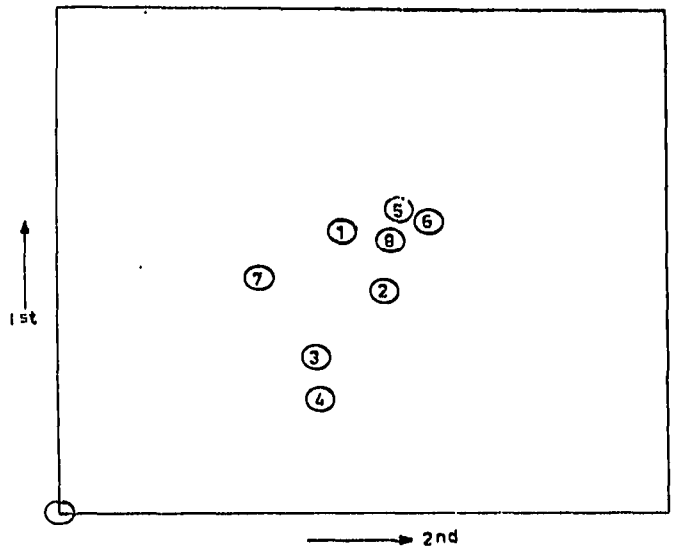

Fig. 1. Two-dimensional chromatogram of eight furanocoumarins. 1 = psoralen; 2 = bergapten; 3 = xanthotoxin; 4 = isopimpinellin; 5 = angelicin; 6 = isobergapten; 7 = sphondin; 8 = pimpinellin.

The R_F values and spot colours of the eight furanocoumarins are shown in Table I. Fig. 1 shows the same compounds separated on a two-dimensional thin layer chromatogram.

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