CHROM. 16,996

Note

New reagent for the detection of sesquiterpene lactones by thin-layer chromatography

A. VILLAR*, J. L. RIOS, S. SIMEÓN and M. C. ZAFRA-POLO

Departamento de Farmacognosia y Farmacodinamia, Facultad de Farmacia, Universidad de Valencia, Avenida Blasco Ibáñez, 13, Valencia-10 (Spain)

(First received May 25th, 1984; revised manuscript received June 19th, 1984)

In an extensive review and experimental study of reagents for thin-layer chromatography (TLC) in order to standardize the study of folk medicinal plants, a great number of spray reagents have been tested. One of them, an ethanolic solution of aluminium chloride (5%), is known as a specific reagent for flavonoids¹⁻⁴, both in their free and glycosidic forms. There is no mention in the literature of this reagent being used for any compounds other than flavonoids.

We have now shown that a 5% solution of aluminium chloride in ethanol can be useful in the detection of sesquiterpene lactones. The reaction (violet or brown colour on the plate, or yellow, brown and green fluorescence under UV light at 366 nm) is evident only 10-15 min after heating at 120°C in an oven, whereas in the case of flavonoids a yellow colour or brown fluorescence appears immediately. The reaction is specific for sesquiterpene lactones. Other terpenes, steroids, anthraquinones, glucosides and alkaloids do not react under the above conditions.

Plants extracts were investigated by TLC with several reagents. They were assayed after chromatography on silica gel 60 Merck sheets (0.2 mm) with chloroform-ethyl acetate (3:7), benzene-chloroform-methanol (45:45:15), hexaneethyl acetate (1:1), chloroform-ethyl acetate (1:1), chloroform-ethyl acetate (7:3) or hexane-ethyl acetate (3:1) as mobile phase.

Finally, we tested aluminium chloride with sesquiterpene lactones and demonstrated a reaction similar to that of hydroxylamine and resorcinol-phosphoric acid, but these reagents are less sensitive than AlCl₃.

In Table I we report the results obtained with pure sesquiterpene lactones after chromatography on silica gel. The MDA (minimal detectable amount), colour on the plate and fluorescence under UV light at 366 nm after heating at 120°C in an oven are given.

Aluminium chloride was shown to be more specific than other lactone reagents. such as resorcinol-phosphoric acid and hydroxylamine⁵. It does not react with other types of lactones such as coumarins, cardenolides, saturated or unsaturated y-lactones.

TABLEI

COLOURS AND MDA (MINIMAL DETECTABLE AMOUNT) OF SESQUITERPENE LACTONES FOR DETECTION WITH 5% AICI3 AFTER THIN-LAYER CHROMATOGRAPHY ON SILICA GEL

	I-β-Hydro. H-eudesm-	I-β-Hydroxy-6β,7α,11β- H-eudesm-4-en-12-oiide	1-0x0-68,7x,118- H-enderm-1 - m. 12-olide	118-	Barrelin		Dihydroxyen-		Artemisiifolin	
			t dufference of	2011-17-01106		21.1	caucstranouac		1732	7477
	Vis	AD	Vis	AA	S S	40	Vis	40	3	
MDA (µg/cm²) Colour	6 Violes	9	100	12	1	9	9	24	100	9
7		r ettow-	Yellow	Green-	Not	Blue	Yellow	Brown	Yellow-	w nic-
		Drown		yellow	visible				brown	yenow

308 NOTES

ACKNOWLEDGEMENT

The authors express their gratitude to Professor G. B. Marini-Bettolo (Universitá Cattolica del Sacro Cuore, Rome) for discussion of these results.

REFERENCES

- 1 K. G. Krebs, D. Heusser and H. Wimmer, in E. Stahl, *Thin Layer Chromatography. A Laboratory Handbook*, Springer, Berlin, 1969, p. 856.
- 2 G. Zweig and J. Sherma, Handbook of Chromatography, Vol. II, CRC Press, Cleveland, 1974, p. 182.
- 3 N. R. Farnsworth, J. Pharm. Sci., 55 (1966) 225.
- 4 G. B. Marini-Bettòlo, M. Nicoletti, M. Patamia, C. Galeffi and I. Messana, J. Chromatogr., 213 (1981) 113.
- 5 B. Drozdz and E. Bloszyk, Planta Med., 33 (1978) 379.