

15 cm von der Auftragsstelle. Bei mehrmaligem Entwickeln der gleichen Platte in gleicher Richtung stieg das Lösungsmittel im ersten Lauf 5 cm, im zweiten 10 cm und erst im dritten 15 cm. Bei diesen Versuchen wurden die Lösungsmittel jedesmal neu angesetzt.

Die zu untersuchenden Gerbstoffextrakte wurden entweder in 50%igem Aceton oder in Methanol gelöst und vom Rückstand abdekantiert. Die Methanol-lösung unterschied sich im chromatographischen Bild nicht von der Acetonlösung.

Die Dünnschichtplatten wurden im langwelligen U.V.-Licht (365 m μ) ausgewertet, die Ergebnisse durch Farbaufnahmen festgehalten. Dafür wurde der Agfa C 18 Film verwendet mit einer Sperrfilterkombination 2 mm GG4 und 2 mm GG13 von Schott. Bei Blende 8 bringt eine Belichtungszeit von 15 Sek. bei Verwendung von zwei Hanau-U.V.-Lampen (PL 327) in einem Abstand von etwa 40 cm die beste Wiedergabe.

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Eingegangen den 25. Juni 1964

J. Chromatog., 17 (1965) 587-591

Thin-layer chromatography of tetracyclic triterpenes on silica impregnated with silver nitrate

The rapid, reversible coordination of silver ions with unsaturated compounds, studied extensively by LUCAS and coworkers^{1,2}, can be expected (NICHOLS³) to be useful in the separation of saturated and unsaturated compounds, such as fatty acid esters. Indeed, this principle has been applied by DUTTON, SCHOLFIELD AND JONES⁴ to mixtures of isomeric fatty acid esters, by DE VRIES⁵ to lipids, by BARRETT, DALLAS AND PADLEY⁶ to glycerides, by MORRIS⁷ to cholesteryl esters (see also AVIGAN, DE

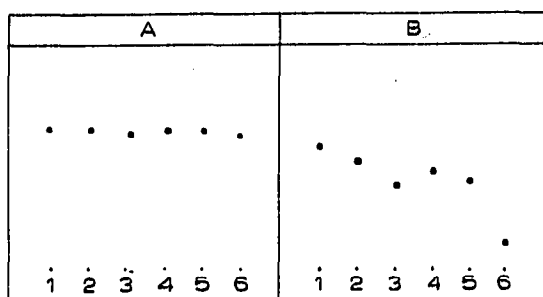
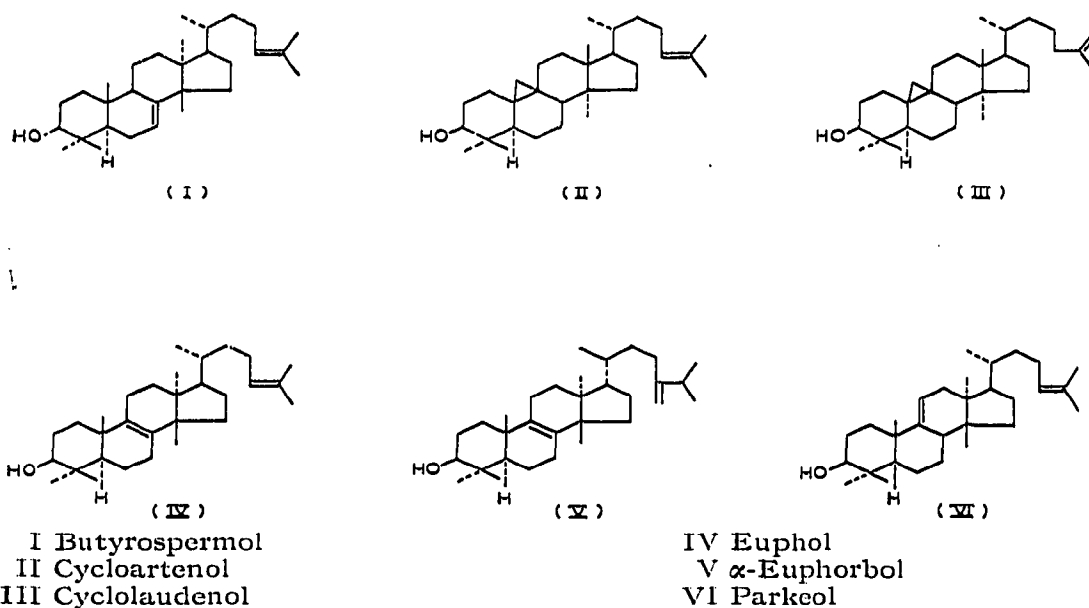


Fig. 1. (A) Silica gel G. (B) Silica gel G, impregnated with silver nitrate (5% w/w).

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GOODMAN AND STEINBERG⁸) and to fatty acid methyl esters⁹, and by SHABTAI, HERLING AND GIL-AV¹⁰ to cyclic olefins. Recently, thin layers impregnated with silver nitrate have also been used for the separation of sesquiterpene hydrocarbons¹¹.

In the present study, this method has been extended to include some tetracyclic triterpenes. The compounds studied had been shown to have practically the same R_F values on thin layers of alumina G¹². However, on silica gel G, impregnated with silver nitrate, the R_F values were sufficiently different to enable the substances to be identified separately (Fig. 1). The values and the colors obtained by spraying the spots with three reagents are summarized in Table I.

TABLE I

Triterpene	R_F on silica gel G		Colors with spray reagent*					
	Treated with $AgNO_3$	Untreated	A		B		C	
			Room temp.	After charring	Room temp.	After charring	Room temp.	After charring
Butyrospermol (I)	0.40	0.42	—	brown	light yellow	blue green	light orange	brown
Cycloartenol (II)	0.33	0.42	—	brown	orange	blue green	orange	brown
Cyclolaudenol (III)	0.26	0.41	—	brown	light yellow	light green	light orange	dark brown
Euphol (IV)	0.30	0.42	—	brown	light yellow	light green	yellow	brown
α -Euphorbol (V)	0.27	0.42	—	brown	light yellow	light green	yellow	brown
Parkeol (VI)	0.11	0.41	—	brown	light pink	light green	light yellow	light brown

* Spray reagents: A = 50% sulfuric acid in water¹³; B = 10% phosphomolybdic acid in ethanol¹⁴; C = a mixture of chlorosulfonic and acetic acids (1:2)¹¹.

For convenience the structural formulae of the triterpenes are drawn.

Although the number of examples is too small to draw significant conclusions as to the correlation between R_F and structure, it is interesting to note the differences in the values for butyrospermol (I), euphol (IV) and parkeol (VI), which differ from each other in the position of the ring double bond. Even the shift of the double bond of the side chain, in passing from cycloartenol (II) to cyclolaudenol (III), has a distinct influence on the migratory tendency.

Experimental

Preparation of plates. The suspension for five plates (20 × 20 cm) was prepared by shaking 30 g of silica gel G and 60 ml of water for 30 sec and applied uniformly to a thickness of 0.25 mm with a Desaga applicator. After 30 min at room temperature, the plates were heated in an oven at 125–130° for 45 min. After cooling they were sprayed with concentrated aqueous-methanolic silver nitrate solution, 5% relative to silica gel, and then activated at 120° for 30 min.

This method permits impregnation of only part of the plate, which can thus be used for comparative chromatography.

The plates were used immediately after cooling.

Development. The samples were dissolved in chloroform (1 mg/1 ml), and applied with micropipettes along a line 2 cm above the edge of the plate. The experiments were performed at room temperature (24–27°). Chloroform was used as mobile phase; it was allowed to ascend to a distance of 15 cm. The plates were removed and dried in air.

Detection. The triterpenes were detected by spraying with the three reagents, followed by heating in an oven at 150° for 15 min. The colors of the spots were recorded before and after charring.

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Received July 2nd, 1964

J. Chromatog., 17 (1965) 591–593