The methylated membranes are brittle when dry, but when moist they have sufficient mechanical strength to render them useful in the form of an assembly pack in the electrodecantation apparatus. Care should be taken not to fold them as they crack easily at the position of the bend[2].

Zusammenfassung. Die Ladungen, eine Folge der Karboxylgruppen an Zellophanmembranen, können durch Methylierung entfernt werden. So behandelte Membranen zeigen eine beträchtliche Verminderung ihrer Elektroendosmosis und sind daher für Multimembranelektrodekantation sehr geeignet.

A. POLSON

C.S.I.R. and U.C.T. Virus Research Unit, University of Cape Town (Republic of South Africa), 3 December 1968.

[2] Acknowledgment. Grateful acknowledgment is made to Dr. B. W. RUSSELL for the electro-endosmotic measurements on the membranes.

Separation of Cardiotonic from Flavanoid Compounds of the Squill, *Urginea maritima* Baker

STOLL et al.[1] have identified most of the cardiotonic glycosides of the Squill; anthocyanins[2] and flavanoid compounds[3] have also been found. The ethyl-acetate extracts consist mostly of the cardiotonic and flavanoid compounds; however, a good method for their separation is not yet available. The method here described is simple and reliable enough to separate both groups of compounds.

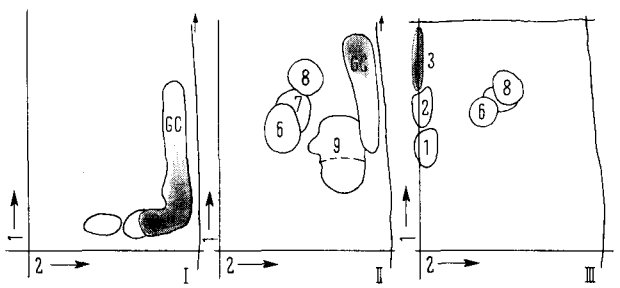
Materials and methods. Squill extracts. The ethyl-acetate extracts of the triploid, tetraploid and hexaploid Spanish bulbs, obtained by the method of STOLL et al.[1], were used.

Chromatography. The fractions of the extracts were checked by paper chromatography[4] for both cardiotonic and flavanoids. Thin layer chromatography (TLC) in polyamide (Woelm) developed with water–ethanol–methylethylketone–acetylacetone (65:1:5:15:5, by vol.)[5] for flavanoids and Silicagel G developed with water saturated methylethylketone for cardiotonics. The chromatograms were sprayed with 10% $SbCl_3$ in chloroform or 10% aminoethylester of diphenylboric acid in methanol[6].

Filtration through polyamide. 10 g of the dry ethyl-acetate extracts suspended in 1 l of distilled water were stirred for 15 min with 100 g of polyamide (Ultramid-Pulver BASF) free of monomers[7]; the slurry was poured into a funnel, with a sintered glass plate, the water was filtered out; then the polyamides were washed with 2 l of 10% aqueous methanol, filtered out and added to the water (I); 10 l of 50% methanol were passed with stirring (II) and finally 10 l of absolute methanol (III). With fraction II, where the separation was not complete, the process was repeated over the same polyamide used; washing with 10% methanol, the remaining cardiotonics were passed and added to fraction I. The polyamide washed with dimethylformamide did not retain any appreciable amount of these compounds (see Figure).

Results and discussion. Fraction I was shown to contain cardiotonics and some flavanopolyglycosides, that were isolated by preparative paper chromatography, after separating most of the cardiotonics by crystallization of the concentrated liquids; fraction II contained most of flavano-3-glycosides, very little dihydroquercetin and polyglycosides; fraction III contained all flavanoids with 3-OH free and some of the 3-glycosides. This sequence is in accordance with their Rf using aqueous solvents on paper. The isolation of the individual flavanoid compounds from these simple fractions was achieved by the ordinary chromatographic techniques and will be published elsewhere.

The precipitation with lead acetate or hydroxyde[1] has to be repeated more than 3 times to achieve any substantial separation, and some alteration occurred mainly on the 3-OH free compounds. We have worked some 25 runs of the extracts by column chromatography on cellulose powder, carboxymethylcellulose, Magnesol, polyamides etc.; the results were not satisfactory; the polyglycosides of both groups behaved on the chromatographic systems in a similar way, quercetin, dihydroquercetin and flavanomonoglycosides are difficult to



Whatman No. 1 chromatograms of the fractions sprayed with $SbCl_3$. Solvents: (1) *n*-butanol–acetic acid–water (4:1:5, by vol. upper layer); (2) 2% acetic acid. GC, cardiotonic glycosides; 8, dihydroquercetin; (3) condensation product; 1,2,3-OH free flavanoids; other spots, flavanoid-3-glycosides.

[1] A. STOLL, E. SUTER, W. KREIS, B. B. BUSSEMAKER and A. HOFMANN, Helv. chim. Acta *16*, 703 (1933).
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[6] R. NEU, Naturwissenschaften *44*, 181 (1957).
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separate from the less glycosidated cardiotonics. With the filtration technique, both types could be separated with less time and solvent volumes and without substantial losses. We hope that other plant extracts, as Digitalis, Oleander etc. could be processed in a similar way.

Resumen. Se describe un método de filtración sencilla a través de poliamidas, con el cual se han conseguido los mejores resultados en la separación de los glucósidos cardiotónicos y flavonoides de las preparaciones de escila.

F. A. VEGA, M. FERNANDEZ,
P. G. CASADO and M. ESTERUELAS

Facultad de Farmacia, Universidad de Navarra, Pamplona (Spain), 21 June 1968.

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