

Für kleinere Laufstrecken benutzt man ein kürzeres (etwa 15 cm langes) Gummiband, das man leicht mit Hilfe von zwei Halteschrauben mit dem ursprünglichen auswechseln kann. Selbstverständlich lässt sich der Verhältnismasstab insgesamt auch in anderer, z.B. kleinerer Grösse anfertigen. Dehnt sich nach längerem Gebrauch das elastische Band oder wird es "müde", dann muss man es durch ein neues ersetzen.

*Deutsche Akademie der Wissenschaften zu Berlin,
Institut für Medizin und Biologie, Berlin-Buch (Deutschland)*

K.-H. SEGEL

¹ L. B. ROCKLAND UND M. S. DUNN, *Science*, 111 (1950) 332.

² D. JERCHEL, W. JACOBS UND W. MÖHLE, *Angew. Chem.*, 66 (1954) 298.

³ R. L. CLEMENTS, *Anal. Chem.*, 30 (1958) 160.

⁴ H. BERBALK, *Monatsh. Chem.*, 89 (1958) 548.

⁵ D. M. P. PHILLIPS, *Nature*, 162 (1948) 29.

⁶ E. MASUCH, *D.B.-Patent* 1002954 vom 1.8.57.

Eingegangen den 17. Oktober 1960

J. Chromatog., 5 (1961) 177-178

Contribution to the paper chromatographic separation of *Withania somnifera* alkaloids

A recent report¹ implied the existence of eight alkaloids in the root of *Withania somnifera*. Our investigations² have established the occurrence of eleven bases. Seven of these have been isolated as white crystalline compounds; one as an amorphous white powder. An admixture of three alkaloids has not been resolved. Qualitative data on the isolated alkaloids will be published; preliminary data on the nature of each base is presented in Table I.

TABLE I

Compound	R_F
I. Tertiary nitrogen alkaloid	0.07
II. Quaternary ammonium base	0.10
III. Tertiary nitrogen alkaloid	0.14
IV. Quaternary ammonium base	0.20
V. Tertiary nitrogen alkaloid	0.23
VI. Quaternary ammonium base	0.35
VII. Unqualified alkaloid	0.43
VIII. Unqualified alkaloid	0.53
IX. Unqualified alkaloid	0.69
X. Neutral alkaloid	0.84
XI. Neutral alkaloid	0.92

All alkaloids in a semi-pure extract of the root are revealed on one chromatogram although the quaternary ammonium bases are minor components of the admixture. The average R_F values of an alkaloid mixture, as a root extract, chromatographed

on Whatman No. 1 paper impregnated with 0.5 M KCl solution, dried, and developed 25–35 cm with *n*-butanol–HCl (98:2 v/v), water-saturated, are reported in Table I. Revelation was achieved by spraying the dried paper with modified Dragendorff reagent.

The major alkaloids, in the several root samples investigated, are III and V; the major quaternary base is VI. Compound II is choline. None of these alkaloids is nicotine and this compound has not been observed in root and leaf samples investigated.

This work was supported in part by contract with the United States Army Chemical Corps.

University of Connecticut School of Pharmacy, Storrs,
Conn. (U.S.A.)

A. ROTHER
C. K. ATAL
D. GOLD
A. E. SCHWARTING

¹ M. COVELLO AND G. CIAMPA, *J. Chromatog.*, 3 (1960) 591.

² C. K. ATAL, *Ph. D. Dissertation*, University of Connecticut, 1958, and unpublished data.

Received September 29th, 1960

J. Chromatog., 5 (1961) 178–179

Gas chromatographic analysis in fractional distillation of multi-component systems

The course of fractional distillation of a multi-component system is commonly followed by noting the changes in boiling points and/or refractive indices. These techniques are not completely adequate in those cases in which the boiling points are very close and the differences in refractive indices are very small or non-existent. Gas chromatography offers a very useful and indispensable tool for following fractional distillations of this type. Gas chromatographic analysis of each fraction as the distillation proceeds permits one to make a more judicious choice in effecting the best separation of the components and therefore obtaining a maximum amount of each component in high purity.

In this note, the utilization of gas chromatography in the purification of 2,2,4,6,6-pentamethylheptane (PMH) by fractional distillation is described.

Experimental

PMH was prepared by catalytic hydrogenation of commercially available triisobutylene¹. The crude PMH, after washing and drying, was charged to a total condensation, intermittent take-off-type distillation column. The packed section, 1 in. by 4 ft., was packed with Podbielniak Heli-Pak No. 2117 stainless steel packing. The column and take-off valve were designed so that the distillate did not come in contact