

About half of the alkaloids are converted to the dextrorotatory form by this procedure.

When attempting to adapt Berg's system to a simpler apparatus, ergonovine and methylegonovine were well separated; but spots were very diffuse and the developing solvent failed to ascend the paper evenly. More satisfactory chromatograms were obtained by diluting the developing solvents with *n*-butyl acetate. Less volatile chlorinated solvents would not separate ergonovine and methylegonovine. Of the volatile chlorinated solvents, only methylene chloride or ethylene chloride would separate the homologs and then only when they constituted half, or more, of the developing solvent.

By simultaneously increasing the acidity of the impregnating solution and the basicity of the mobile solvent as compared to conditions prescribed by Berg, less diffuse spots were obtained while maintaining about the same R_f values. In analyzing alkaloid samples by the procedure described above, unsatisfactory chromatograms were occasionally obtained where the alkaloids had migrated excessively or insufficiently during development. The mobility of the alkaloids could be increased by raising the pH of the mobile solvent or decreased by lowering the pH. The R_f values reported are typical of those obtained. When desired for special cases, greater than usual separation of ergonovine and methylegonovine, or greater separation of a pair of diastereoisomers, can be achieved by such a pH adjustment.

Several techniques were tried for conditioning the paper prior to development. Among these is the commonly used technique whereby papers are impregnated, dried, spotted, moistened by equilibration, and developed. Most satisfactory results were obtained by the "moist-paper" technique described above. Grossly diffuse spots are obtained if the papers are too wet when placed in the tank and immobility of the spots will result if the paper is too dry.

A methylegonovine maleate tablet mix and several injection solutions, of both ergonovine maleate and methylegonovine maleate, were prepared to simulate market products. These were then analyzed by the procedure described in this paper. The results indicated that there is no deterioration of alkaloid during analysis and that the excipients do not interfere. The procedure described in this paper was successfully applied to 14 commercial samples. Neither of the four samples of alkaloid salts, nor any of the seven tablet samples analyzed, contained more than trace quantities (about 1%) of the respective diastereoisomer. Only one of three injection solutions tested contained any dextrorotatory isomer and that, only after a year of storage under refrigeration. The chromatogram for another one of these injection solutions exhibited faint spots representing material not containing the lysergic acid moiety. These may have represented oxidized alkaloids.

REFERENCES

- (1) "United States Pharmacopoeia," 16th rev., Mack Publishing Co., Easton, Pa., 1960, p. 266.
- (2) "The National Formulary," 11th ed., J. B. Lippincott Co., Philadelphia, Pa., 1960, p. 228.
- (3) Roster, G. E., Macdonald, J., and Jones, T. S. G., *J. Pharm. and Pharmacol.*, **1**, 802(1949).
- (4) Macek, K., *Pharmazie*, **9**, 752(1954).
- (5) Voigt, R., and Weiss, F., *ibid.*, **13**, 212(1958).
- (6) Alexander, T. G., and Banes, D., *THIS JOURNAL*, **50**, 201(1961).
- (7) Stoll, A., and Rüeegger, A., *Helv. Chim. Acta*, **37**, 1725(1954).
- (8) Pöhm, M., and Fuchs, L., *Naturwissenschaften*, **41**, 63(1954).
- (9) Horak, P., and Kudrnač, S., *Ceskoslov. Farm.*, **5**, 595(1956); through *Anal. Abstr.*, **4**, 4103(1957), and *Mfg. Chemist*, **29**, 160(1958).
- (10) Berg, A. M., "Chromatographische Scheidung der Möderkoornalkaloiden en een Practische Toepassing Daarvan," Uitgeverij Excelsior 's-Gravenhage.
- (11) Mitchell, L. C., *J. Assoc. Offic. Agr. Chemists*, **40**, 999(1957).
- (12) Levine, J., and Fischbach, H., *THIS JOURNAL*, **44**, 543(1955).
- (13) "United States Pharmacopoeia," 16th rev., Mack Publishing Co., Easton, Pa., 1960, p. 929.
- (14) Hellberg, H., *Acta Chem. Scand.*, **11**, 219(1957).
- (15) Kleiderer, E. C., *J. Am. Chem. Soc.*, **57**, 2007(1935).

ERRATUM

In the paper titled "*In Vitro* Testing of Timed Release Tablets and Capsules" (1), the address of the manufacturer under Fig. 1 should read Ernest D. Menold, 5th and Powhatan Ave., Lester, Pa.

(1) Krueger, E. O., and Vliet, E. B., *THIS JOURNAL* **51**, 181(1962).

ERRATUM

In the paper titled "Study of the Boric Acid-Glycerin Complex II" (1), the formulas on page 238 should read

$$\frac{d \ln S}{dT} = \frac{\Delta H}{RT^2}$$

which when integrated becomes

$$\log S = \frac{-\Delta H}{2.303 RT} + C$$

(1) Sciarra, J. J., and Monte Bovi, A. J., *THIS JOURNAL*, **51**, 238(1962).