Gas Chromatography of Sympathomimetic Amines

Sir:

Methods for the separation and identification of microquantities of sympathomimetic amines are of great importance for work in pharmacy, pharmacology, and toxicology. Although the literature in this field is extensive, there is still a need for a simple and rapid method that permits positive identification and lends itself to quantitative analysis.

This communication reports the separation and identification of 11 sympathomimetic amines by gas-liquid chromatography at temperatures ranging from 104° for the nonphenolic to 135° for the phenolic amines. A Barber Colman model 15 gas chromatograph equipped with an argon ionization detector has been used for this work. A suitable column is prepared from Gas-Chrom P, 100 to 140-mesh, coated with about 1% of silicone rubber SE-30. A typical sample is 1.0 μl. of a solution containing 0.5 to 1.0% of each amine. A mixture of six commonly used nonphenolic amines dissolved in chloroform gives five distinct peaks, ephedrine and pseudoephedrine being eluted together (Fig. 1). However, the two latter amines can be separated by using acetone as the solvent. Pseudoephedrine reacts relatively rapidly with acetone to produce an addition product, whereas the reaction between ephedrine and acetone is very slow.

Several sympathomimetic amines react with ketones. The reaction products produce sharp, symmetrical peaks, suitable for quantitative estimation. The compounds are readily identified on the basis of the relative retention values for the free amines and their ketone derivatives.

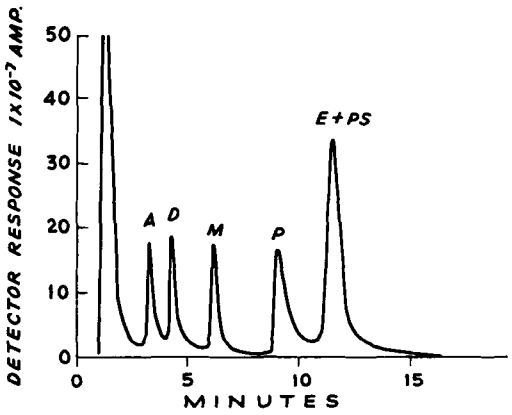

Fig. 1.—Gas chromatogram of six sympathomimetic amines on silicone rubber SE-30, 1.15%. Temp., 104°; column, 8 feet long, 3 mm. i.d.; pressure, 20 p.s.i.; flow rate, 22.5 ml./min. A, amphetamine; D, methamphetamine; M, mephentermine; P, phenylpropanolamine; E, ephedrine; PS, pseudoephedrine.

A complete report will be published shortly.

E. Brochmann-Hanssen
A. Baerheim Svendsen

University of California
School of Pharmacy
San Francisco 22, Calif.

Received January 19, 1962.
Accepted for publication January 30, 1962.
This work was supported by a research grant (M-3487) from the National Institutes of Health, Bethesda, Md.

Occurrence of Psilocin in *Psilocybe baeocystis*

Sir:

Psilocybe baeocystis Singer and Smith is a comparatively small, nondescript agaric belonging to the stirps cyanescens of the section Caerulescentes of the genus *Psilocybe* (1). As such, it might be expected to produce cerebral mycetism following ingestion; indeed, three recent cases of poisoning have been reliably attributed to this species (2). The nature of the active principle(s) in this mushroom has not previously been determined, probably due to its restricted habitat which appears to be limited to western Oregon.

Recently, several dried carpophores of *P. baeocystis*, collected on October 30, 1960 from a lawn in Portland by C. Goetz, were made available to us through the courtesy of Mrs. H. J. Oswald. The identity of the specimens was verified by Prof. D. E. Stuntz, Department of Botany, University of Washington.

Samples of the dried carpophores were extracted with cold 70% methanol and the extract

examined chromatographically, essentially as described by Tyler (3). Solvent systems employed included water-saturated *n*-butanol, *n*-butanol:acetic acid:water (4:1:5), and *n*-propanol:1 *N* ammonia (5:1). In all three of these systems, a compound was detected by spraying with 2% *p*-dimethylaminobenzaldehyde in 1 *N* hydrochloric acid (blue-violet color) and with Pauly's reagent (orange-red color). It had an R_f value identical with reference psilocin and did not separate from that compound when spotted in admixture. Tryptophan was also identified on the chromatograms by its characteristic R_f value and the color formed with the *p*-dimethylaminobenzaldehyde reagent, but psilocybin could not be detected.

Failure to detect psilocybin is remarkable in that it is more stable than psilocin and has been found in every hallucinogenic *Psilocybe* species which has been investigated for its presence, although it is not always accompanied by detectable amounts of psilocin (4). Since psilocin is known to possess psychotropic activity following ingestion, *Psilocybe baeocystis* must be added to the ever increasing list of hallucinogenic mushrooms (5). It is the second such species whose reported distribution is restricted to the northwestern United States (3).

(1) Singer, R., and Smith, A. H., *Mycologia*, **50**, 262(1958).
(2) McKenny, M., Olympia, Wash., personal communication, March 31, 1961.
(3) Tyler, V. E., Jr., *Lloydia*, **24**, 71(1961).
(4) Heim, R., and Hofmann, A., *Compt. rend.*, **247**, 557(1958).
(5) Wasson, R. G., *Botan. Museum Leaflets, Harvard Univ.*, **19**, 137(1961).

R. G. Benedict
L. R. Brady
V. E. Tyler, Jr.

College of Pharmacy
University of Washington
Seattle 5, Wash.

Received February 2, 1962.
Accepted for publication February 16, 1962.
This work was supported by research grant No. RG-7515 from the National Institutes of Health, Bethesda, Md.

Book Notices

Précis de Microbiologie. Vol. 1, Technique Microbiologique Microbiologie Générale. By S. Lambin and A. German. Masson & Cie., 120, boulevard Saint-Germain, Paris VI^e, 1961. viii + 458 pp. 16 × 21 cm. Price 39 NF.

Volume one of this series (in French) is divided into two parts: microbiological technique and general microbiology. It is intended for use as a textbook for pharmacy students. A general index is appended.

Microtechniques of Clinical Chemistry. 2nd ed. By Samuel Matelson. Charles C Thomas, 301–327 East Lawrence Ave., Springfield, Ill., 1961. xv + 578 pp. 15 × 23 cm. Price $14.75.

New sections in this edition cover emission spectroscopy, automation, gas chromatography, and the theory and technique of the urea and creatinine clearance tests. The revised and expanded text is intended to make the book more suitable for use in teaching analytical clinical chemistry. Author and subject indexes are included.

Medicinal Chemistry. Vol. 5. Edited by Walter H. Hartung. John Wiley & Sons, Inc., 440 Park Ave. South, New York 16, N. Y., 1961. vi + 432 pp. 14.5 × 23 cm. Price $18.

Volume five of this monograph series covers Anticonvulsant drugs and Bis(4-aminophenyl) sulfone and related compounds in tuberculosis and leprosy. The style and format of the earlier volumes are continued, and the objective to include in each chapter or monograph references to all the compounds that have been tested for a particular type of pharmacological activity still maintains. The extent of the work with anticonvulsants is indicated by the 1,113 references to publications and 16 pages of patents grouped according to chemical types. A general index is appended.

Structural Forms of Anesthetic Compounds. By Hugh S. Mathewson. Charles C Thomas, 301–327 East Lawrence Ave., Springfield, Ill., 1961. xvi + 223 pp. 15 × 22.5 cm. Price $6.75

Intended as an introductory text, this book is based upon seminars on anesthetic drugs. The relation of structure-activity principles to the pharmacologic properties of drugs in clinical use by anesthesiologists is the frame upon which the subject is developed. An index is appended.

Progesterone and the Defence Mechanism of Pregnancy. Edited by G. E. W. Wolstenholme and Margaret P. Cameron. Little, Brown and Co., 34 Beacon St., Boston 6, Mass., 1961. viii + 108 pp. 12 × 18.5 cm.

A compilation of the reports by the Ciba Foundation Study Group No. 9 in February 1961. A general index is included.

Biological Activity of the Leucocyte. Edited by G. E. W. Wolstenholme and Maeve O'Connor. Little, Brown and Co., 34 Beacon St., Boston 6, Mass., 1961. viii × 120 pp. 12 × 18.5 cm.

A compilation of the reports by the Ciba Foundation Study Group No. 10 in March 1961. A general index is included.