Notes

CHROM. 3975

The gas chromatography of ion-pair complexes

The use of ion-pair formation as a means of extraction and determination of bases is now an established technique[1–3]. We wish to record an extension of the method of potential value in the purification and determination of bases in complex systems.

Ion-pair complexes of the base–sulphonic acid type have been found to decompose under gas chromatographic conditions to give the base. This has been observed on a column (5 ft.) of OV-17 (5 %) and on a column (4 ft.) of Carbowax 20M (1 %) + potassium hydroxide (2 %) at temperatures above 150°C.

This observation suggests a means of isolation and purification based on the sequence:

1 Adjustment of the sample to a suitable pH in the acid range.
2 Extraction with an immiscible solvent and rejection of the extract.
3 Addition of a suitable ion-pairing compound.
4 Extraction of the ion-pair complex with the same immiscible solvent.
5 Gas chromatography of the extract under suitable conditions.

The superior performance of alkali-modified packings for the gas chromatography of bases led to their selection for further work[4,5].

Aqueous solutions (0.1 %) of salts of five basic drugs were adjusted to pH 5 with 0.1 *N* sulphuric acid and treated with a bromothymol blue solution. The solutions were then extracted with chloroform. Standards were prepared by extraction of basified aqueous solutions. Sample and standards were then compared gas chromatographically under the following conditions:

Pye Argon chromatograph with a 4 ft. glass column packed with Carbowax 20M (1 %) + potassium hydroxide (2 %) on acid- and alkali-washed Gas-Chrom P (100–140 mesh), with an argon flow rate of 40 ml/min.

The results obtained by application of the method to five basic drugs are given in Table I. Recoveries are essentially quantitative.

TABLE I

ION PAIR GAS–LIQUID CHROMATOGRAPHY OF BASIC DRUGS

	Column temperature (°C)	R_T *(min)*	*Recovery (%)*
Tripelennamine	200	6	101
Dimethindene	200	8	98
Chlorpromazine	200	14	93
Pethidine	150	5.5	94
Methylphenidate	150	7	96

The principle is also applicable to the extraction and determination of acidic substances. Ion-pair complexes formed by the addition of a basic dye to an acid have been shown to decompose under gas chromatographic conditions. Thus phenobarbitone is recovered by (i) extraction at pH 10 in the presence of methylene blue, and (ii) gas chromatography on a column (5 ft.) of OV-17 (5 %) at 270°.

Analytical Department, CIBA Laboratories Ltd., P. F. G. BOON
Horsham, Sussex (Great Britain) A. W. MACE

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