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Gas-liquid chromatography of imidazoline salts

SIR,—A gas-liquid chromatographic method for analysis of imidazolines in pharmaceutical preparations has recently been published by Boon & Sudds (1967). Their treatment included both antazoline and naphazoline in the group of five heterocyclics investigated. These two compounds may be conveniently analyzed by a procedure which offers the advantages of reduced tailing and inclusion of an internal standard.

A dual column flame ionization chromatograph was operated under the following conditions: flash heater at 250°, column temperature at 239°, isothermal; detector temperature at 265°; helium carrier gas at 40 p.s.i.g. and 70 cc/min; hydrogen at 60 cc/min; air at 300 cc/min; and a recorder range of 10 with attenuation of 32 for carbazole and naphazoline and 64 for antazoline. These recorder adjustments are stated for use of manual quantization. Results were obtained using an integrator. The columns consist of 4 ft of 6 mm O.D. U-shaped glass tubing containing Chrom-Q, 100–120 mesh, treated with 1·0% KOH in methanol and coated with 5·0% of Apiezon-L using methylene chloride as the solvent.

Procedure. Add an aliquot of sample containing approximately 4–40 mg each of naphazoline salt and antazoline salt to a separatory funnel. Make the solution basic with sodium hydroxide and extract immediately with chloroform. Combine the chloroform extracts and add a portion of the internal standard solution containing approximately 4 mg of carbazole in chloroform. Evaporate to approximately 2 ml and inject 0·5 to 1·2 μl of the solution. Calibrate the sample by comparing the ratio of the peak areas of each imidazoline to the peak area of the internal standard.

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