Elimination of the Water Effect on Argon Ionisation Detectors fitted to Pye Chromatographs

By J. L. MARTIN

(*Glaxo Laboratories Ltd., Greenford, Middlesex*)

To obtain quantitative results for small amounts of alcohols in aqueous solutions from a gas chromatograph fitted with an argon ionisation detector, it is necessary to prevent water passing into the detector. If this is not done the drop in sensitivity, which is proportionate to the amount of water and the temperature of the detector, reduces the peak areas of components subsequently eluted. The back-flushing technique[1] is not conveniently applied at temperatures approaching 100° C if the substance to be measured has a larger retention volume than water (even when the extremely polar diglycerol is used as stationary phase), and the device described was introduced primarily for determining 0·02 to 0·25 per cent. of 2-phenylethanol in aqueous solutions. However, the problem of determining small amounts of relatively non-volatile substances in aqueous solution is one that is frequently met in pharmaceutical analysis, and the modification has proved of general use.

It was thought desirable temporarily to divert the gas flow during the time that it was carrying water vapour, so that the detector would be by-passed. It is difficult to contrive a tap at the lower end of the column that can be manipulated during elution, owing to the particular geometry of the Pye instrument used; the column itself was therefore used as a tap control as described below.

The adaptor consists of as short a length of Pyrex-glass tubing as possible (to reduce dead space to a minimum), fitted with a B7 socket at one end (the top) and a B7 cone at the other (see Fig. 1 (*a*)). The B7 socket is grooved lengthwise internally, and a hole is also drilled in it 180° from the groove. The lower B7 cone of the chromatographic column to be used is modified so that the only exit for gas is a hole drilled in the side of the cone: the hole coincides, when column and adaptor are assembled, with the hole in the adaptor socket.

When the two holes are juxtaposed in use (Fig. 1 (*b*)), the argon, together with any water separated on the standard 4-foot column, is vented to the atmosphere, leaving the detector unaffected. When the argon gas flow has to be diverted again into the ionisation cell, the column is turned through 180° without stopping the flow, and the hole in the column cone then corresponds with one end of the groove leading to the detector (Fig. 1 (*c*)). A preliminary chromatogram must be prepared, to indicate at what time the column should be turned.

Benzyl alcohol, chlorocresol, trichloroethanol and the esters of *p*-hydroxybenzoic acid have been determined by means of the modification; it has also proved useful during the conditioning of chromatographic columns by removal of the volatile impurities from them.

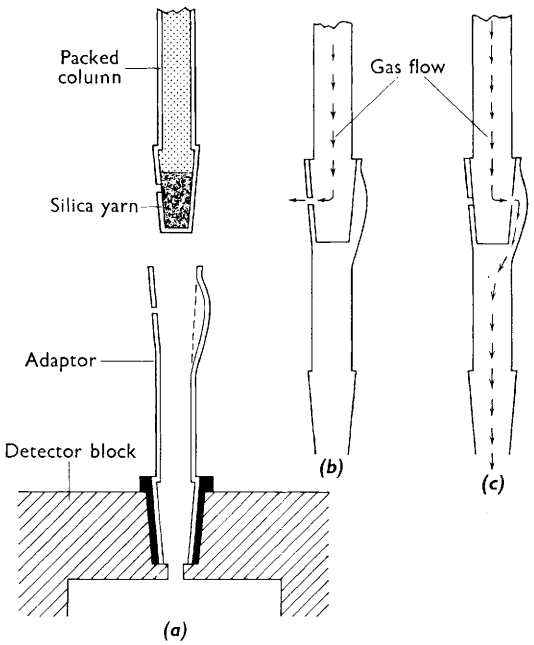

Fig. 1. Diagram of adaptor

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