NOTES ON EXPERIMENTAL TECHNIQUE AND APPARATUS

A simple injection port and column holder for use in gas chromatography

J. SCI. INSTRUM., 1965, VOL. 42

A simple injection port and column holder for use in gas chromatography

A. J. ARNOLD and K. A. LORD

Department of Insecticides and Fungicides, Rothamsted Experimental Station, Harpenden, Herts.

MS. received 27th July 1964, *in revised form* 21*st September* 1964

Abstract. An easily constructed injection port fits directly into a commercial oven, and supports both column and detector within the oven. This leads to improved performance.

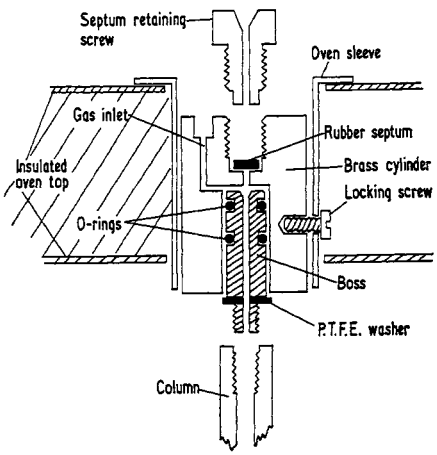
Difficulties in gas chromatography of pesticides caused by temperature differences within the apparatus have been described (de Faubert Maunder *et al.* 1964, Egan *et al.* 1964). We have avoided these difficulties by fitting the injection port, column and detector inside a commercially available stirred air oven. (We used the Griffin & George precision air thermostat oven which has been made especially for gas chromatography.)

The uniform temperature of all components ensures that substances injected volatilize readily, that there is no decomposition through local heating and that substances do not condense and contaminate the detector.

An O-ring seal makes an easily assembled gas-tight joint between the injection port and column; this makes rapid changing of hot columns possible. The bracket supporting the column prevents the separation of this joint when high pressures are used. Our system has several other advantages. Background noise is decreased and detector stability improved because the temperature of the detector is not affected by draughts and the detector is screened from electrical interference by complete enclosure in the oven. Normally the sample is injected directly into the glass fibre packing at the top of the column, and a glass lining has not been found necessary. If the sample is discharged into the injection port it is not diluted with a large volume of carrier gas, because the dead space is kept small. There is little or no restriction on the siting of the detector, so that straight columns a few inches long can be used as well as longer, bent or coiled columns. The use of very short columns facilitates the rapid analysis of substances which have low volatility and are thermally unstable.

The injection port and gas inlet (see figure), made from a brass cylinder 1·625 in. long, fits into a 1·25 in. diameter sleeve through the top of the oven, and is locked in place by a single screw. A 0·030 in. hole is drilled through the axis of the cylinder. The upper end is then enlarged to a depth of 0·5 in. and seated to take a silicone rubber septum, which was held in place by a 0·125 in. BSP screw with a 0·030 in. hole through its axis. The lower end of the cylinder is bored to a depth of 1 in. to receive a brass O-ring boss which is used to connect the injection port and column. Two O-rings are used to make the joint rigid. The carrier gas is introduced just below the septum through holes drilled in the injection port.

A polytetrafluoroethylene boss, similar to that used on the Shandon universal gas chromatograph analyser unit, connects column and detector. The boss screws directly into the end of the column and is a tapered fit into the detector. The column and detector are supported by a bracket slotted to slide over two existing studs in the oven so that columns



Injection port and column holder.

of different lengths may be used. Electrical connections between the detector and recorder pass through holes already in the back of the oven. They are insulated with polytetrafluoroethylene sleeves inside the oven.

The equipment is now being used for routine measurements of chlorinated and organophosphorus insecticide residues using an electron capture detector (de Faubert Maunder *et al.* 1964) and either a Shandon 'Universal' gas chromatograph or a Pye–Argon chromatograph recording unit.

References

EGAN, H., HAMMOND, E. W., and THOMPSON, J., 1964, *Analyst*, **89**, 175.

DE FAUBERT MAUNDER, M. J., EGAN, H., and ROBURN, J., 1964, *Analyst*, **89**, 157.