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Removal of interfering substances from vegetable extracts prior to the determination of organochlorine pesticide residues

Attention has been drawn by SISSONS, TELLING AND USHER[1] and by ABBOTT, TATTON AND WOOD[2] to the interferences which can be caused by co-extractives from carrots and onions when these vegetables are examined by gas–liquid (GLC) or thin-layer (TLC) chromatography for the presence of residues of organochlorine pesticides. Extracts from *Cruciferae*, *e.g.* radish, white cabbage, turnip, similarly give GLC traces with an electron-capture detector, which can also interfere with the interpretation of the chromatograms[3]. These interfering compounds are usually carotenes or organic sulphides and they can either mask, or masquerade as, residues of the organochlorine pesticides. In order to obtain satisfactory chromatograms, it is necessary to remove these compounds from the sample extracts.

The following method, which was briefly described in the Report of the Government Chemist 1968[4], is designed for this purpose and has now been fully tested on extracts from vegetables over the past two years.

Method

Dissolve 0.75 g of silver nitrate in 0.7 ml of water. Warm the solution and slowly add 4 ml of acetone. Rapidly mix this solution with 10 g of alumina (containing 7 % moisture)[8], stirring the mixture the whole time. As an aid to the removal of the acetone, place the alumina in a small, open flask and shake this on a Microid shaker until no further odour of acetone can be detected.

Add 1.0 g of this 'prepared' alumina to a glass chromatographic column (150 × 8 mm I.D.), which contains a plug of solvent-washed cotton-wool and 5 ml of *n*-hexane. When the alumina has settled to the bottom of the column, run off the surplus *n*-hexane until its meniscus just enters the surface of the alumina.

Transfer the cleaned-up sample extract (concentrated to 1.0 ml in *n*-hexane) to the top of the alumina. Allow the extract just to pass into the alumina, wash the containing vessel with 1.0 ml of *n*-hexane and add the washings to the alumina column. Elute with 10 ml of *n*-hexane; any carotenes present in the sample extract show up as a narrow, reddish-coloured band, while the sulphur compounds appear as a black band.

The eluate is then suitable for concentration, using a Snyder column[5], for examination of organochlorine pesticide residues by GLC or TLC. All the commonly used organochlorine pesticides pass through the column unchanged, with the exception of Heptachlor which forms a derivative, 4,5,6,7,10,10-hexachloro-4,7,8,9-tetrahydro-4,7-methyleneinden-1-yl nitrate, which has retention times similar to Aldrin on silicone and Apiezon columns[6].

Where primary clean-up of the sample extract has been carried out with dimethylformamide and alumina[7] or by dimethylsulphoxide and Florisil–alumina[8], the 1.0 g of 'prepared' alumina may be more conveniently used at the bottom of either of these two adsorption columns as an integral part of the clean-up procedure.

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