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Separation of sulphidimines of sulphides on paraffin-treated paper

Several methods have been described for the paper chromatographic separation of the sulphidimines prepared from aliphatic sulphides and Chloramine T. LEAVER AND CHALLENGER[1] used both conventional techniques and reversed phase chromatography. PETRANEK AND VECERA[2] also used reversed phase techniques and the sulphidimine prepared by reacting the monochloroamide of *p*-nitrobenzenesulphonic acid with the sulphide instead of Chloramine T.

In studies on the metabolism of marine algae the volatile compounds produced in the cultures were examined by aspirating, into mercuric cyanide, mercuric chloride and mercuric acetate[3,4]. The volatile sulphides were precipitated as co-ordination compounds with mercuric chloride and converted to the sulphidimine by the method of LEAVER AND CHALLENGER[1]. The separation between the sulphidimines from dimethyl sulphide, ethyl methyl sulphide and diethyl sulphide was not sufficient to permit the distinguishing of sulphidimine-like spots, believed to be due to cyclic sulphides, from the former. Tetrahydrothiophene was found to behave in a similar manner when reacted with Chloramine T[5] and recrystallised from benzene.

The purpose of this communication is to suggest that the method of ASATOOR[6] using paraffin-impregnated paper, may with small modifications be used to give improved separation of the short-chained aliphatic sulphides chromatographed as sulphidimines.

Experimental

A strip of Whatman 3 MM paper (23 × 57 cm) was placed in a shallow tray (24 × 60 cm) containing a 5 % solution of liquid paraffin (sp. gr. 0.83–0.87) in 80–100° petroleum ether. The solution was allowed to flow over both surfaces of the paper by tilting the tray from side to side and by turning the paper. The paper was dried by

pressing between two sheets of absorbent paper and dried at room temperature for an hour. The solvent was prepared by mixing chloroform, water and liquid paraffin 25:25:1 and allowing it to stand for 12 h. The chromatograms were run by the descending technique for 7 h. The lower organic phase was placed in a dish on the floor of the tank. The sulphidimines were located by spraying with acidified potassium iodide (1 % in 0.2 *N* hydrochloric acid) and heated at 80°.

TABLE I

CHROMATOGRAPHY ON PARAFFIN PAPER

Sulphidimine	R_{methyl}
Dimethyl sulphide	1
Methyl ethyl sulphide	0.72
Diethyl sulphide	0.51
Di-*n*-propyl sulphide	0.20
Ethyl isopropyl sulphide	0.31
Ethyl *n*-propyl sulphide	0.29
Tetrahydrothiophene	0.65

Results

Owing to slight variations in impregnation it was found advisable to use the technique of ASATOOR[6] and express the rate of movement of each sulphidimine spot relative to that of the sulphidimine of dimethyl sulphide. The R_{methyl} values for the average of six experiments are set out in Table I.

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A new detection method for aromatic compounds

In paper chromatography, gaseous compounds are particularly suitable for spot indication as they will not distort the spot. In our institute we have employed iodine vapour as a developer[1].

Owing to its higher vapour pressure and reactivity bromine was also thought to be suitable. A number of organic compounds were exposed to the action of bromine vapour. It was found that, except for a few instances, the spots had only a very slight pale yellow hue, or were colourless, and that as soon as the paper strips were removed