

Nevertheless quantitative estimations of reasonable to high accuracy may be made in many pathological cases. Almost any conditions of chromatography will permit the detection and estimation of compounds such as homogentisic acid in alkaptonuria, phenyllactic acid in phenylketonuria and *p*-hydroxyphenyllactic acid in gross tyrosyluria. However it is always desirable to check results with a second column and may be essential when only moderately abnormal excretions, such as that of 4-hydroxy-3-methoxymandelic acid in some cases of phaeochromocytoma, are encountered.

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The determination of acetonitrile and other trace impurities in acrylonitrile by gas chromatography

In a recent communication¹, a column packed with 10% Bentone 34-10% PEG on Chromosorb P was used to estimate acetonitrile in acrylonitrile. The method was not suitable at low concentration (<100 p.p.m.) because the acetonitrile was eluted on the tail of the acrylonitrile peak.

We experienced similar difficulties using oxydipropionitrile as stationary phase. With the introduction of Porapak (Waters Associates), however, the analysis became much more satisfactory because acetonitrile was eluted before acrylonitrile.

This packing has been used for over two years for this particular analysis under the gas chromatographic conditions shown below.

Experimental

Gas chromatograph: Perkin Elmer F-11.

Detector: flame ionisation.

Column dimensions: length 2.5 m, diameter 2.5 mm I.D.

Column temperature: 160°.

Injection temperature: nominal 150°.

Carrier gas: helium, flow rate about 70 ml/min.

Sample size: 1 μ l.

The retention data are given in Table I.

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TABLE I

RELATIVE RETENTION VOLUMES OF VARIOUS SOLUTES IN ACRYLONITRILE ON PORAPAK Q

<i>Compound</i>	<i>Relative retention volume</i>
Air/methane	0.00
Water	~ 0.1
Hydrogen cyanide	~ 0.1
Methanol	0.20
Acetaldehyde	0.31
Ethanol	0.50
Acetonitrile	0.67
Acrolein	0.80
Acetone	0.88
Acrylonitrile	1.00
Propionitrile	1.65
Time to acrylonitrile	8 min

As HCN has very little response, it does not interfere with the analysis and does not appear to affect the column, which normally lasts about six months. A concentration of 10 p.p.m. of acetonitrile in acrylonitrile is readily detected.

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