

Notes

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Phosphoric acid as a subtracting agent for nitrogen bases in reaction gas chromatography

One technique of reaction gas chromatography is the use of a subtractive precolumn process^{1,2}. In an application of this process to coal tar materials we were faced with the problem of finding a subtracting agent for nitrogen bases.

Knowing that amines as organic bases will form stable salts with mineral acids, we found that orthophosphoric acid was a suitable agent for the quantitative removal of nitrogen bases in a chromatographic precolumn. Phosphoric acid has already been used as a subtracting agent for epoxides^{3,4} in reaction gas chromatography.

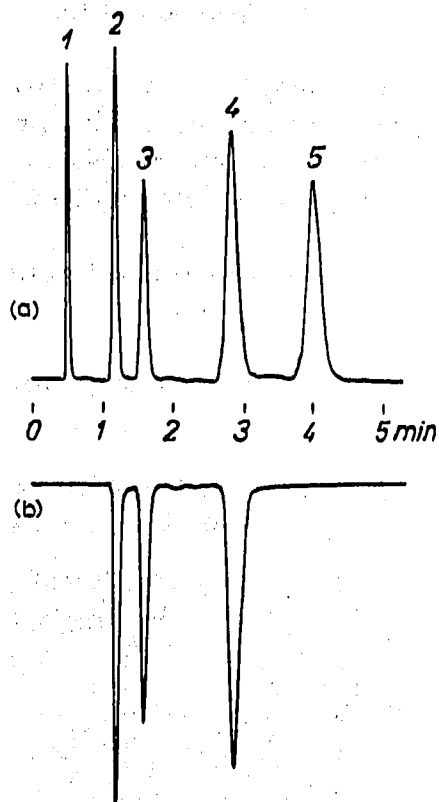


Fig. 1. Chromatogram of a synthetic mixture of different types of tar compounds on (a) a column without H_3PO_4 and (b) a column with an H_3PO_4 precolumn (lower part). 1 = Pyridine; 2 = benzonitrile; 3 = hydrindene; 4 = 2,4-dimethylphenol; 5 = 2,3-dimethylaniline.

The upper part of Fig. 1 shows a chromatogram of a synthetic mixture of tar compounds of different types on a column without H_3PO_4 . The gas chromatograph was a Chrom 2, with an FID (Laboratory Instruments N.E., Prague) and a glass column, 90 cm \times 3 mm, with 10% Apiezon M on Chromosorb W, 80-100 mesh, column temperature 150°, carrier gas (argon) flow rate of 22 ml/min, sample size

0.1 μ l. The lower part of Fig. 1 shows a chromatogram of the same mixture after inclusion of an H_3PO_4 precolumn (a glass tube, 7 cm \times 3 mm, containing Chromosorb W, 80–100 mesh, coated with 10% of 85% H_3PO_4 from a water–methanol solution) in the system. From comparison of the two chromatograms it is evident that the bases (pyridine, 2,3-dimethylaniline) have been removed by the H_3PO_4 precolumn. This fact may be useful in tar analysis.

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