

Notes

CHROM. 5897

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.

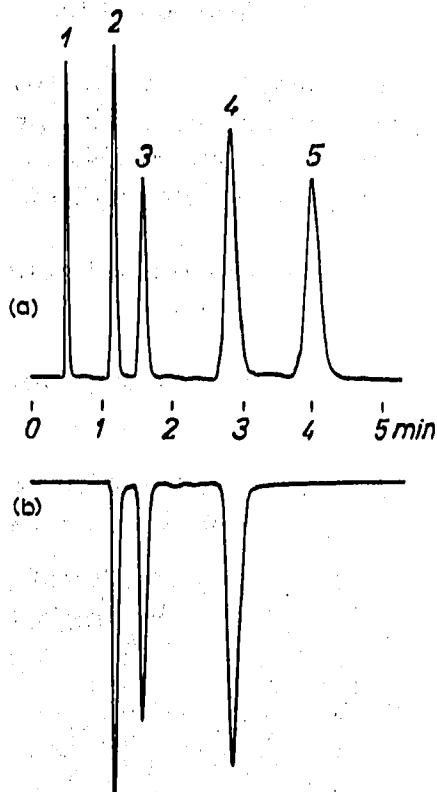


Fig. 1. Chromatogram of a synthetic mixture of different types of tar compounds on (a) a column without H_3PO_4 and (b) a column with an H_3PO_4 precolumn (lower part). 1 = Pyridine; 2 = benzonitrile; 3 = hydrindene; 4 = 2,4-dimethylphenol; 5 = 2,3-dimethylaniline.

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.

*Research Institute of Coal Tar Chemistry,
Urxovy závody N.E., Valašské Meziříčí (Czechoslovakia)*

J. FRYČKA
J. POSPÍŠIL

- 1 M. BEROZA AND M. N. INSCOE, in L. S. ETTRE AND W. H. MCFADDEN (Editors), *Ancillary Techniques of Gas Chromatography*, Wiley-Interscience, New York, 1969, p. 89.
- 2 V. G. BEREZKIN, *Analytical Reaction Gas Chromatography* (translated from Russian), Plenum Press, New York, 1968.
- 3 B. A. BIERL, M. BEROZA AND W. T. ASHTON, *Mikrochim. Acta*, (1969) 637.
- 4 Y. G. OSOKIN, V. S. FELDBLUM AND S. I. KRYUKOV, *Neftekhimiya*, 6 (1966) 333.

Received December 13th, 1971

J. Chromatogr., 67 (1972) 366-367