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Note

Analysis of thianaphthene in commercial naphthalene

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Commercial grades of naphthalene contain low levels of various impurities such as thianaphthene, tetralin and methyl naphthalenes¹. Gas chromatography is commonly used to measure low levels of impurities in naphthalene^{2,3}. However, we have found that accurate estimation of thianaphthene at levels below 2% may be difficult because of incomplete resolution of the naphthalene and thianaphthene peaks (Fig. 1). While recognizing that improved resolution probably can be obtained on other columns or by temperature programming, we describe in this note the advantageous use of high-performance liquid chromatography (HPLC) to analyze low levels of thianaphthene in coal tar naphthalene.

EXPERIMENTAL

A Hewlett-Packard Model 5880 gas chromatograph, equipped with a flame ionization detector and a 15 m × 0.25 mm DB-1 column (Chromatographic Specialties Ltd.) was used for gas chromatography. The column temperature was 70°C. A Hewlett-Packard Model 1084 liquid chromatograph equipped with a variable-wavelength detector set at 235 nm was used for liquid chromatography. Isocratic elution was performed with acetonitrile-water (60:40), on a RP-8 (Hewlett-Packard) 5- μ m particle size, 20 cm × 4.0 mm column, at a flow-rate of 0.75 ml/min. Samples of pure naphthalene, thianaphthene and commercial naphthalene were dissolved in acetone for gas chromatography, and acetonitrile for liquid chromatography.

RESULTS AND DISCUSSION

Baseline separation of naphthalene containing 0.1-5% thianaphthene was achieved on RP-8. In addition, detection at 235 nm favored the analysis of thianaphthene while minimizing the response to naphthalene. Detector response to thianaphthene was linear in the range 0.1-5%, the range normally required for coal tar or non-desulphurized naphthalene. Other methods would be required for analysis below 0.1%, the lower practical detection limit, however this is well below the level usually found in coal tar naphthalene (Fig. 1).

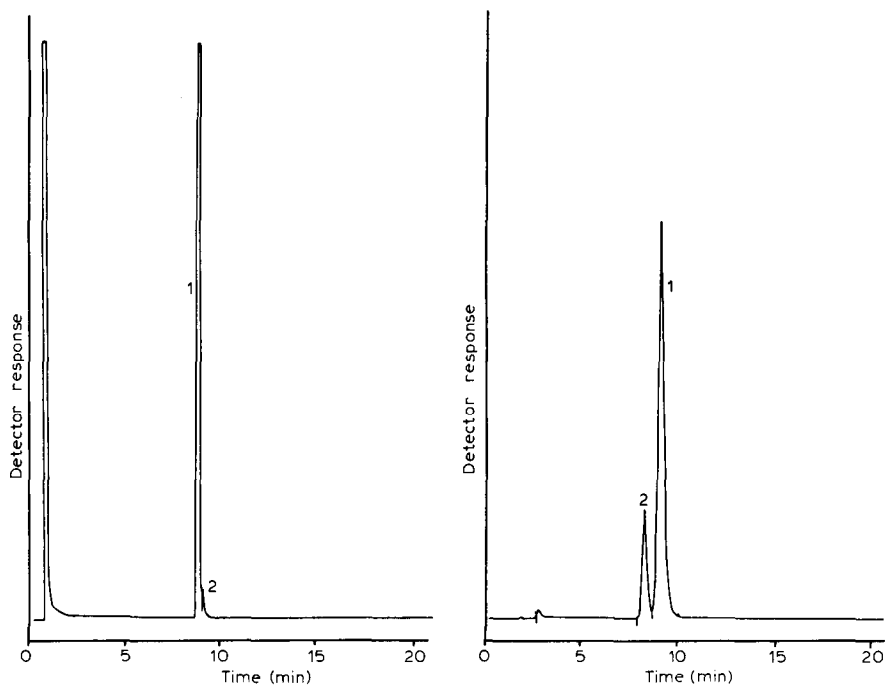


Fig. 1. Gas (left) and liquid (right) chromatograms of a coal tar naphthalene containing 2.2% thianaphthene. Conditions as described in Experimental. Peaks: 1 = naphthalene; 2 = thianaphthene.

REFERENCES

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