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Chromatography of sulphur: separation from hydrocarbons*

In an investigation of components of sewage sludges, we have found elemental sulphur in hydrocarbon fractions eluted from liquid adsorption columns. Free sulphur is commonly found in hydrocarbon sources such as petroleum and lake sediments, and is to be expected in any organic matter that has undergone successive reducing and oxidising conditions. However, there is little available information on the chromatographic behaviour of sulphur and its separation from hydrocarbons with which it is found.

Sulphur has been separated from polycyclic aromatic components of coal tar pitch by chromatography on alumina[1] and from the compounds S_7NH, $S_6N_2H_2$, and S_4N_4 on columns of alumina or silica gel[2–4], but no retention of sulphur relative to the solvent was reported.

This report shows that elemental sulphur can be separated from very weakly adsorbed classes of hydrocarbons, but not from slightly more strongly adsorbed classes, by chromatography on the usual adsorbents. Most of the experiments were carried out using alumina but similar results were obtained with silica gel and Florisil.

Experimental

Alumina (Aluminum oxide, neutral, Brockmann activity, grade 1; J. T. Baker Chemical Co.) was activated at 170° overnight. Columns were prepared in 1 cm diameter tubes by the slurry technique using *n*-heptane (*n*-heptane, pure grade, Phillips Petroleum Company) that had been distilled before use. Packed column length was 20 cm.

The hydrocarbon (100 mg) to be chromatographed was dissolved in 3 ml of *n*-heptane that had been kept over powdered sulphur. The solution was added to the column and the components were eluted with *n*-heptane. The eluate was collected in fractions (*ca.* 2 ml) from which the solvent was removed by evaporation in a stream of nitrogen.

Visual inspection or determination of the IR absorption spectrum identified the residue.

Results

On columns of alumina under the described conditions, eicosane (*n*-$C_{20}H_{42}$) and squalane (2,6,10,15,19,23-hexamethyltetracosane) were eluted before sulphur and were completely separated from it. Tetradecahydrophenanthrene was eluted ahead of sulphur but the separation was not quite complete. 1-Phenyldodecane was eluted slightly ahead of sulphur but the resolution was far from complete. Biphenyl was eluted after sulphur and was completely separated from it.

For comparison purposes mixtures of the hydrocarbons were chromatographed in a similar manner. Under these conditions eicosane and squalane were not appreciably separated from each other, but they were eluted before tetradecahydrophenanthrene and were largely resolved from it. All three saturated hydrocarbons were eluted before 1-phenyldodecane and were completely resolved from it.

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Conclusions

Elemental sulphur will pass through liquid chromatographic procedures along with hydrocarbons, and can be expected to contaminate the polycyclic naphthenic and monocyclic aromatic fractions of hydrocarbons purified or fractionated by this technique.

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