

molecular weight (10^5 - 10^6) isotactic polystyrene. The spectrum shown was obtained from isotactic polystyrene in deuterated chloroform using a 'Varian V-4300' 40-00-Mc./s. spectrometer. A similar spectrum was obtained in thionyl chloride. Like Bovey *et al.*, we observed no resolution of chain protons for commercial atactic polystyrene. Neither was resolution observed for high molecular weight (10^5) polystyrene prepared using *n*-butyl-lithium initiator nor for benzene-insoluble polystyrene obtained via Alfin sodium alkyl catalysis^{2,3}.

R. J. KERN
J. V. PUSTINGER

Monsanto Chemical Co.,
Dayton 7, Ohio.

¹ Bovey, F. A., Tiers, G. V., and Filipovich, G., *J. Polymer Sci.*, **38**, 73 (1959).

² Morton, A. A., and Grovenstein, E., *J. Amer. Chem. Soc.*, **74**, 5453 (1952).

³ Williams, J. R., Vandenberg, J., Dunham, K. R., and Dulmage, W. J., *J. Amer. Chem. Soc.*, **79**, 1716 (1957).

Dissolution of Chrysotile Asbestos in Water, Acid and Alkali

THE dissolution of the asbestos mineral, chrysotile, $(\text{OH})_6\text{Mg}_3\text{Si}_4\text{O}_{11}$, has been studied as a part of investigations into the nature of the industrial lung disease, asbestosis¹. Because the results may be of value to industrial chemists and to geologists, and they explain apparently anomalous results which have been published^{2,3}, they are outlined here although the details and a discussion on their relevance to the problem of pneumoconiosis will appear elsewhere.

At 85-90° C. chrysotile gives an extract containing appreciable amounts of monosilicic acid and magnesium, for example, 0.5 gm. chrysotile in 100 ml. water gives 0.4 mgm. magnesium and 0.2 mgm. *orthosilicic acid* per 100 ml. solution after 15 min. When extracted with water at 25° C. for some days, chrysotile gives a very low concentration of silicic acid in solution but more than ten times that concentration of magnesium. If the asbestos is removed and the extract is heated to 85-90° C., the concentration of the dissolved silicic acid increases more than ten-fold although the magnesium concentration is unaffected. As an example, after 5 days, 1 gm. chrysotile in 200 ml. water at 35° gave an extract containing 1.1 mgm. magnesium and 0.1 mgm. *orthosilicic acid* per 100 ml. After heating for 3 hr. at 90° C. the extract contained 1.2 mgm. magnesium and 1.4 mgm. *orthosilicic acid* per 100 ml. It is thought that magnesium ions are leached from the chrysotile giving fragments of the two-dimensional sheets of the silicon-oxygen lattice which are resistant to water except at elevated temperatures, when they break down to give *orthosilicic acid*.

Hydrochloric acid (0.1 *N*) attacks chrysotile giving a solution containing *orthosilicic acid* and magnesium ions. Sodium hydroxide (0.1 *N*) scarcely attacks chrysotile, presumably because of a protective coating of hydrated magnesium ions on the surface of the fibres. If acid-washed chrysotile is extracted with sodium hydroxide, silica passes rapidly into solution but very little magnesium; the acid removes the protective layer, allowing the disintegration of the next layer of the silicon-oxygen lattice by the alkali. Evidently the behaviour of chrysotile depends on its history, and it is particularly vulnerable to alternating acid and alkaline conditions.

This communication describes a part of a programme of research which is proceeding under the auspices of the Asbestosis Research Council.

S. G. CLARK
P. F. HOLT

The University,
Reading.

¹ Holt, P. F., "Pneumoconiosis", 142 (Arnold, London, 1957).

² Briscoe, H. V. A., Matthews, J. W., Holt, P. F., and Sanderson, P. M., *Trans. Inst. Min. and Metall.*, **44**, 291 (1937).

³ King, E. J., and McGeorge, M., *Biochem. J.*, **32**, 417 (1938).

Clathrate Compounds in Chromatography

PARTITION and adsorption chromatography can be successfully applied for separation of mixtures of substances which differ in type and number of functional groups, or for separation of members of a homologous series. Less success was attained in separation of isomers. We therefore sought for a process which would be selective as regards the shape and size rather than the chemical properties of molecules.

The discovery of clathrate compounds suggested that inside their structure molecules of definite shape and size might be incorporated^{1,2}. The first inorganic compound of that group was monoamminonickel cyanide^{3,4}, but many other complexes⁵ of nickel, cobalt, manganese and iron with four basic nitrogen molecules and some anions around the metal ion followed. These compounds are said to be highly selective towards clathrate formation with organic molecules; and, more important, their selectivity depends rather on the shape of incorporated molecules than on their volume.

The selectivity, especially towards aromatic molecules, the small solubility of some complexes of that type, and other properties suggested the use of the formation of clathrate compounds for the separation of substances on the chromatographic column.

In preliminary experiments we filled the column with water-insoluble tetra-(4-methylpyridino)-nickel dithiocyanate prepared by the method of Schaeffer *et al.*⁵. Later, we found that another complex, prepared by using for precipitation a mixture of β - and γ -picoline and 2,6-lutidine instead of pure γ -picoline (so-called 3rd coal tar base fraction), has better properties.

One of the most important problems was to find an appropriate solvent composition for use as a mobile phase. We have used the mixture of γ -picoline or 3rd fraction with water containing some potassium thiocyanate.

To obtain high precision of results, we have used the chromatopolarographic method⁶. The compounds separated and incorporated were aromatic and aliphatic nitro-compounds.

In these conditions we have investigated the separation of isomeric nitrophenols, nitroanilines, chloronitrobenzenes and nitrotoluenes. In all instances quantitative separation of *ortho* from other isomers was obtained. The *ortho* isomers are found in the first fractions of eluate and can be very accurately determined polarographically. Using more concentrated solutions of potassium thiocyanate and 3rd fraction, *meta*- and *para*-isomers can be eluted. In some instances the mobile phase composition can be so chosen that it is possible to separate completely all three isomers in one process. We have used 2.5 *M* potassium thiocyanate in 10 per cent 3rd fraction