



Composite three-dimensional electron density map of di-*p*-xylylene

Mann *et al.*². Attempts to prepare di-*p*-xylylene by standard organic chemical methods have not been successful, and it is possible that the strained system may only be formed under drastic pyrolysis conditions and low pressures.

A fuller description of the properties and structure determination of this new hydrocarbon will be submitted for publication in the *Journal of the Chemical Society*. We are indebted to Dr. R. S. Cahn, of the Chemical Society, for consultation about nomenclature.

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¹ Szwarc, M., *J. Chem. Phys.*, **16**, 128 (1948).

² Baker, W., Banks, R., Lyon, D. R., and Mann, F. G., *J. Chem. Soc.*, **27** (1945).

Spectrographic Examination of Chromatographic Columns

IN the determination of metallic traces, insufficient attention has been paid either to the advantages of direct spectrography of chromatographic columns, or to the chromatographic behaviour of organic metal complexes, although in the latter sphere the use of oxine¹ and of violuric acid² has been reported.

Working with alumina as adsorbent and using dithizone complexes, we have established the position of a number of metals in the undeveloped column by cutting it into a series of short sections and examining these sections spectrographically. We have concentrated initially on the estimation of very small traces of cadmium, and the procedure outlined below for this element illustrates the general technique.

The sample solution (normally acid) is made ammoniacal in the presence of ammonium citrate,

and is then completely extracted with a solution of dithizone in carbon tetrachloride. A column of activated alumina (Spences 'H' 100-200 mesh) 5 cm. long and 4 mm. in diameter is prepared, and the extract after washing and filtration is poured through, using suction. The first centimetre, which normally weighs between 100 and 130 mgm., is cut out and retained for the estimation of cadmium.

Spectrographic examination is carried out using a carrier-distillation technique similar to that described by Scribner and Mullin³, but employing silver chloride⁴ as carrier and arsenic as internal standard. The anode cup-charge of 40 mgm. is subjected to a direct-current arc of 10 amp., and duplicated spectra are photographed on a Kodak B10 plate, using a Hilger large quartz spectrograph. The ratio of strengths of cadmium 2288.0 Å. and arsenic 2288.1 Å. is determined and compared with that of standard samples photographed on the same plate. Quantities of cadmium between 0.1 and 0.5 µgm. can conveniently be determined by the method with a standard deviation of 10 per cent. This represents concentrations of between 0.02 and 0.1 parts per million on a 5-gm. sample; this sample weight can, of course, be varied to enable a wider range of concentrations to be covered. With special care, a quantity of cadmium as small as one-fiftieth of a microgram can be detected qualitatively.

It is planned to extend the procedure to include other elements amenable to chromatographic treatment, using dithizone or other reagents.

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¹ Erlenmeyer and Dahn, *Helv. Chim. Acta*, **22**, 1369 (1939).

² Erlenmeyer and Schoenauer, *Helv. Chim. Acta*, **24**, 878 (1941).

³ Scribner and Mullin, *J. Res. Nat. Bur. Stand.*, **37** (December 1946).

⁴ Harrison and Kent, U.S. Atomic Energy Research Report *MDDC* 1581 (declassified).

Diffusion in Ion-Exchange Media

DURING an investigation into the behaviour of solutions of direct cotton dyes of the aromatic azo sodium sulphonate type on ion-exchange materials, it was found that selective penetration of the resin structure occurs.

Solutions containing the derived sulphonic acids of a number of dyes of relatively high molecular weight were obtained (together with the inorganic acids derived from the salts present in the commercial products) by treatment with the cation-exchange 'Zeokarb 215' operating on the hydrogen cycle. These solutions, when passed through the acid-absorbing, anion-exchange resin 'De-Acidite B', gave an effluent containing the pure dye acid with, apparently, little absorption of the dye on the basic resin. This was unexpected as the dye acids were thought to be highly dissociated in solution, and this was afterwards confirmed by electrometric titration.

Cernescu¹ has shown that the effective capacity of the natural zeolite, chabazite, for the substituted ammonium ion decreases with increasing ionic radii. Kunin and Winters² have also shown a similar de-