

is recovered in amounts equivalent to the cationic exchange capacity. Cobalt exists as probably cobalt hydroxide since it is in acid solution only (2 N) that it may be extracted. These facts are also supported by X-ray diffraction data: $\text{Co}(\text{NH}_3)_6^{3+}$ saturated montmorillonite expands as usual to 16 Å with glycerol while it does not expand when the complex has been destroyed.

J. CHAUSSIDON
R. CALVET

Centre National de la Recherche Agronomique,
Versailles, Seine-et-Oise, France.

J. HELSEN
J. J. FRIPIAT

Laboratoire des colloïdes et de chimie minérale,
Institut Agronomique, Université de Louvain,
Héverlé, Belgium.

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Detection of Nanogram Quantities of Sulphur Hexafluoride by Electron Capture Methods

As sulphur hexafluoride is now used as a gaseous insulating and arc-quenching medium in electrical equipment, there is some interest in methods for its estimation in mixtures with other gases. The accompanying gas chromatogram (Fig. 1) demonstrates that electron capture methods can be applied to this end.

The detector used was as previously described^{1,2} and was operated with a constant applied potential of 24 V with the carrier gas, nitrogen, flowing from a 4 mm × 70 cm chromatograph column packed with 80-100 mesh silica gel with subsequent dilution making the total gas flow-rate 500 c.c./min.

The peak illustrated was obtained with a 1-μl. load of 1/2,500 by vol. of SF_6 in air, that is, with approximately 2×10^{-2} g of SF_6 , giving a maximum concentration of SF_6 in the detector of the order of 5×10^{-5} (molar). The ionization efficiency in the detector under these deliberately non-optimum conditions was approximately 2 per cent.

A proportion, 1/60 by vol., of CCl_2F_2 was included in the sample for comparison purposes. The ionization efficiency for the three peaks shown are in the sequence $\text{SF}_6 > \text{CCl}_2\text{F}_2 > \text{O}_2$, in accord with the known arc-quenching properties of these gases.

The detector efficiency decreases as the applied voltage is increased, but does so less rapidly for SF_6 than for most other gases, implying that the negative ion formed is relatively heavy. Thus the only peak occurring in the chromatogram when the applied voltage is 42 V is that due to SF_6 . It is suggested, therefore, that it may prove possible to apply electron capture detectors to the routine estimation of the SF_6 content of mixtures of known gases, without preliminary chromatographic separation.

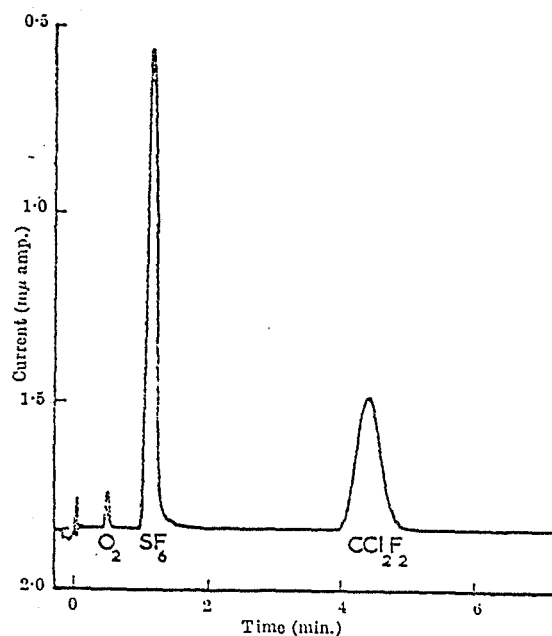


Fig. 1

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N. L. GREGORY

Medical Research Council Laboratories,
Holly Hill, London, N.W.3.

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CRYSTALLOGRAPHY

X-ray Line Broadening in the Alkali Halides

DURING the past few years many investigations have been made of the effects of cold work on the X-ray line profiles of polycrystalline metals. Analysis of the profile broadening has been used to determine particle size, stacking fault probability and strain relationships. Using these same analytical methods, work is now in progress here on the investigation of broadening produced by cold work on face-centred cubic compounds of the alkali halides. This communication covers three of the compounds which have been investigated, namely, sodium chloride, potassium bromide and sodium fluoride.

Initially, the method of cold work was that which had been used in the examination of metal—the filing of block specimens. However, since the broadening produced in the alkali halides is relatively small, and it was therefore desirable to produce the maximum effect, it was found that crushing the specimens in an agate ball mill produced the better results. Similarly, annealing conditions have been varied to achieve maximum resolution in each compound studied, optimum conditions being related to melting point on one hand and solubility in water at room temperature on the other. Thus the broadening of a potassium bromide specimen decreases rapidly at room temperature under normal atmospheric moisture conditions and maximum resolution was achieved under these conditions after seven days. Sodium fluoride and sodium chloride, on the other