

beam. As the copper-aluminium interface was traversed, the count-rate increased to reach a constant value when the entire beam was collected on the aluminium section. The length of the plateau was determined by the thickness of the aluminium sheet, and in Table 1 widths calculated from half-height are compared with values measured optically with a travelling microscope. It can be seen that the agreement is good.

Table 1. WIDTH OF ALUMINIUM SHEETS IN TARGETS

Sample	Optical measurement (mm)	Proton scan (mm)
1	1.14	1.13
2	2.52	2.50
3	3.87	3.75

In these experiments, the minimum time required for an irradiation was governed by the long dead-time of the multi-channel analyser (900 μ sec). If a faster analyser had been available, it would have been possible to reduce the irradiation time by using a higher beam-current, and taking advantage of the higher yield of 0.84-MeV γ -rays obtained on increasing the proton-energy.

Aluminium was scanned in this work because it can easily be incorporated in targets as sheets of known thickness, but particle scanning can equally well be applied to the determination of any element which can be measured by a prompt-radiation technique similar to that described for aluminium. By careful choice of bombarding-particle and energy, and with the aid of γ -ray spectrometry, methods are available for most of the light elements.

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¹ Garton, F. W., Campbell, J. T., and Watling, J., *Proc. Soc. Anal. Chem. Conf., Nottingham* (1965).

² Pierce, T. B., and Peck, P. F., *Proc. Soc. Anal. Chem. Conf., Nottingham* (1965).

³ Morgan, I. L., *Proc. Euratom Meet. Practical Aspects of Activation Analysis with Charged Particles, Grenoble* (1965).

⁴ Pierce, T. B., *Proc. Euratom Meet. Practical Aspects of Activation Analysis with Charged Particles, Grenoble* (1965).

Iron Enneacarbonyl

At various times¹ doubt has been expressed as to the authenticity of iron enneacarbonyl, $\text{Fe}_2(\text{CO})_9$, owing to its low solubility in all solvents and apparent lack of volatility. We have therefore carried out an investigation of this carbonyl, which was made by exposing iron pentacarbonyl, sealed under high vacuum in 'Pyrex' vessels, to sunlight; tri-iron dodecacarbonyl, which also formed in small amounts¹, was removed by repeated washings with petrol ether. A series of analyses on different samples of iron enneacarbonyl clearly indicates that the empirical formula is $\text{Fe}_2(\text{CO})_9$. (Found (per cent): carbon, 29.9, 29.7, 30.2, 29.6; oxygen, 39.2, 38.0, 39.4. Calculated (per cent) for $\text{C}_9\text{O}_9\text{Fe}_2$: carbon, 29.7; oxygen 39.6.)

Although it is stated that iron enneacarbonyl is involatile¹, we have observed that under a good vacuum (10^{-5} to 10^{-6} mm) it will sublime at about 70° C to form hexagonal-shaped crystals. The sublimation occurs cleanly if the enneacarbonyl has been dispersed, by crushing and rubbing, against the apparatus walls, but attempts at the sublimation of bulk samples cause rapid decomposition and the formation of an iron mirror; volatility comparisons to the other metal carbonyls, including tri-iron dodecacarbonyl, suggest a molecular weight higher than that corresponding to $\text{Fe}_2(\text{CO})_9$. However, the mass spectrum, obtained under conditions such that the enneacarbonyl was introduced into the electron beam at room temperature to minimize any possible thermal decomposition, definitely

shows the molecular ion $\text{Fe}_2(\text{CO})_9^+$, centred at mass 364 and has no peaks of higher molecular weight; furthermore, none of the ions formed on fragmentation contains more than two iron atoms. An interesting fact which emerges from a study of the mass spectrum is that the peak due to $\text{Fe}_2(\text{CO})_9^+$ is the most intense of the $\text{Fe}_2(\text{CO})_x^+$ ions. An attractive explanation for this is that the three carbon monoxides represent the bridging carbonyl groups in the original iron enneacarbonyl because it seems reasonable to assume these particular carbonyls (which are bonded simultaneously to the two iron atoms) will be less readily lost on fragmentation of the molecule than the terminal carbonyl groups. This work, therefore, substantiates the formulation of iron enneacarbonyl, but in no way explains the unusually low solubility and volatility for a compound of such low molecular weight.

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¹ See, for example Abel, E. W., *Quart. Rev.*, **17**, 133 (1963).

Decomposition of Ferrosic Sulphide to Pyrrhotite in an Electron Microscope

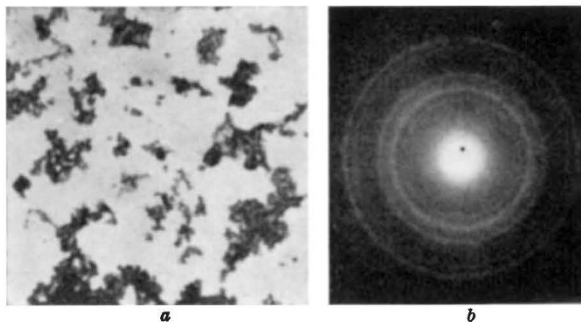
FRAGILE compounds undergo thermal decomposition in an electron microscope in which specimens are exposed to a high vacuum and to electron bombardment¹⁻⁵.

Ferrosic sulphide with a spinel structure has just become recognized as a well-defined compound since Skinner *et al.*⁶ recently discovered mineral greigite. They have observed the thermal breakdown of greigite to pyrrhotite and pyrite at 320° C in a sealed, evacuated silica tube. In contrast with their result, the decomposition of ferrosic sulphide to pyrrhotite and sulphur vapour occurred when the specimen was heated above 300° C in a vacuum with the pump operating.

It was interesting to see if the same experimental condition could be achieved in an electron microscope, enabling one to follow the crystallographic and morphological changes during the decomposition.

In a previous paper⁷, a synthetic method of ferrosic sulphide is described in some detail. The powder particles of the ferrosic sulphide were mounted on a specimen grid covered with a collodion film. The crystals were examined in a Hitachi HS-7 electron microscope operated at 50 kV. The $L\lambda$ values (L is the effective camera length and λ is the electron wave-length) were determined with a gold foil standard.

Electron micrograph and diffraction patterns at the initial stage were recorded, care being taken not to subject the specimen to more than the minimum beam intensity.



Figs. 1a and 1b. Before decomposition