LETTERS TO THE EDITORS

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Trace Analysis by X-Ray Emission Spectrography

We have used X-ray emission spectrography[1] (X-ray fluorescence) for rapid and accurate direct analysis of small quantities of material by a technique resembling that of the familiar spot test[2]. Our results on zinc (Fig. 1) show the simplicity and speed of the method.

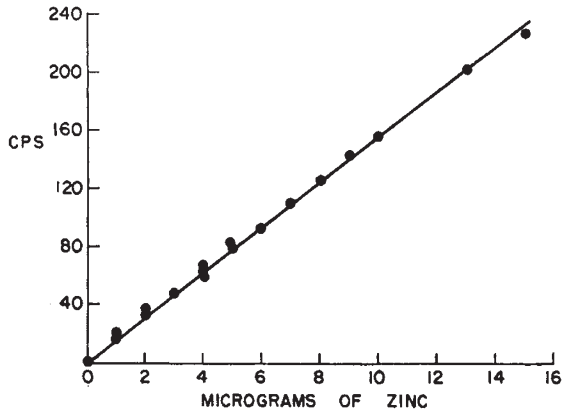

Fig. 1. Counting-rate of zinc $K\alpha$ line for various amounts of zinc on Schleicher and Schuell No. 740*E* ½-inch disks. Samples prepared by placing measured amounts of zinc sulphate solution on the paper and drying to remove the water

The samples containing zinc were dissolved and the solutions dispersed over a well-defined area that could be placed completely in the X-ray beam. As a carrier for the dispersion, Schleicher and Schuell bioassay paper (No. 740*E*) or Whatman's No. 50 filter-paper worked well. The thinness and rigidity of the paper simplified the problem of keeping the sample at the proper place in the X-ray system; other carriers, however, can be used. The intensity of the zinc characteristic $K\alpha$ radiation above the surrounding background gave the direct quantitative measure of the zinc content. Typically, 15·6 counts/sec. of zinc $K\alpha$ radiation per microgram were observed above a background of 49·7 counts/sec. using the bioassay paper. With the filter-paper the background was only 15 counts/sec. The reduced background decreased the time necessary for the detection of 1/30 microgram of zinc from 7 to 2 min. counting time. Different samples of paper gave varying backgrounds, with a standard deviation of 3 counts/sec., which made it possible to determine the larger zinc concentrations without a separate background determination.

Fig. 1 shows the accuracy of the method for numerous samples; the linear range extends to 100 μgm. before absorption effects introduce curvature. The same technique was used for the determination of small amounts of vanadium, copper, iron, silver, bromine, gold, and lead with standard deviations of well under 1 microgram for a determination taking 10 min. or less.

We have increased the sensitivity by counting for longer times. Care is necessary, however, because long-time fluctuation in the X-ray equipment can cause difficulty. Therefore, we have used a commutation method in which the sample and background are counted alternately until the desired number of counts is accumulated.

The samples we used are thin—a microgram of zinc spread over 1 sq. cm. is only 3 atoms or about 10 A. thick; radiation produced in such a thin layer will be essentially unaffected by the presence of other atoms in like quantities. It should therefore be possible to analyse mixtures without concern for the interferences that occur in the X-ray emission spectrographic analysis of bulk materials.

Except for the rather rare overlapping of X-ray lines, the components of mixtures will respond independently, and no previous chemical processing beyond dispersing the sample is required.

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[1] Von Hevesy, G., "Chemical Analysis by X-Ray and its Applications" (McGraw-Hill, 1932).
[2] Feigl, F., "Qualitative Analysis by Spot Tests" (New York, Elsevier Pub. Co., 1946). Winslow, E. H., and Liebhafsky, H. A., *Anal. Chem.*, **21**, 1338 (1949).

Visibility of X-Ray Quantum Fluctuations on Image Intensifier Radiographs

The gain in brightness with an X-ray image intensifier is accompanied by a more or less disturbing effect, namely, the visibility of a kind of visual 'noise', especially when the X-ray intensity is low, the intensification factor of the intensifier is high (greater than 1,000 times) and the optical magnification of the final image large.

In intensifier radiography, in certain circumstances a coarse mottling is observed on the photographic image of the viewing screen. In Fig. 1, four prints are shown of intensifier radiographs (original size 5 mm. diameter) on Agfa Fluorapid film. An object