THE EFFECT OF GENERAL RADIATION IN THE DIFFRACTION OF X-RAYS BY LIQUIDS

By W. C. Pierce
Department of Chemistry, University of Chicago

(Received May 20, 1931)

Abstract

Previous work has shown that the amount of general radiation transmitted by a single filter may cause peaks in the diffraction pattern of liquids. Balanced strontium and zirconium filters are used to study the magnitude of this effect in x-rays from a molybdenum tube operating at 35 kv. Zirconium alone does not give sufficient filtration under these conditions, but by use of the two filters the effects of the general and characteristic radiation may be completely separated.

IT IS well known that the elimination of hard general radiation is more important in scattering experiments with liquids than with crystalline substances. Thibaud and Trillat,[1] Clark and Stillwell[2] and Stewart and Morrow[3] have independently shown that the presence of hard general radiation in the x-ray beam causes an "inner ring" in the diffraction pattern. Meyer[4] compared the scattering of a monochromatic beam, obtained by crystal reflection, with that of a filtered direct beam. The latter gave a diffraction pattern having much broader peaks and in some cases additional peaks not found for the monochromatic beam.

His method of obtaining a monochromatic beam is not generally applicable for use with an ionization spectrometer because of the low intensity, but the effect of a fairly monochromatic part of the beam may be obtained by the use of balanced filters as suggested by Ross.[5] The present work was undertaken with the objects (1) of testing the balanced filter method and (2) of quantitatively determining the effect due to the hard general radiation.

Apparatus

An ionization spectrometer was used in conjunction with a molybdenum target Coolidge x-ray tube operating at a peak voltage of 35 kv. The entire high tension equipment and the x-ray tube were oil-immersed as described elsewhere.[6] The ionization chamber was 20 cm in length. It was constructed from pure copper, in order to avoid the presence of alpha particles, and was fitted with thin aluminum windows at front and rear. Bakelite insulation was used between the chamber and guard ring, and amber between the guard ring and the collecting rod. A Compton electrometer was used for measuring the

[1] Thibaud and Trillat, C. R. **189**, 751, 907 (1929).
[2] Clark and Stillwell, Radiology **15**, 86 (1930).
[3] Stewart and Morrow, Phys. Rev. **30**, 232 (1927).
[4] Meyer, Ann. d. Physik. (5) **5**, 701 (1930).
[5] Ross, Phys. Rev. **28**, 425 (1926).
[6] Bennett, Gingrich and Pierce, Rev. Sci. Inst. **2**, 226 (1931).

ionization current. It had a scale sensitivity of 25,000 mm per volt with the scale at a distance of 3 m. The capacity of the chamber-electrometer system was estimated at about 50 e.s.u.

Argon was used in the chamber in most of the experiments. Although absorption is not complete in 20 cm of this gas it has the advantage of freedom from clean-up by reaction with the material of the chamber, and gave sufficient sensitivity of detection.

Soller[7] slits were used as recommended by Stewart.[3] Various lengths were employed but all of the results are for slits 15 cm in length and spaced 0.08

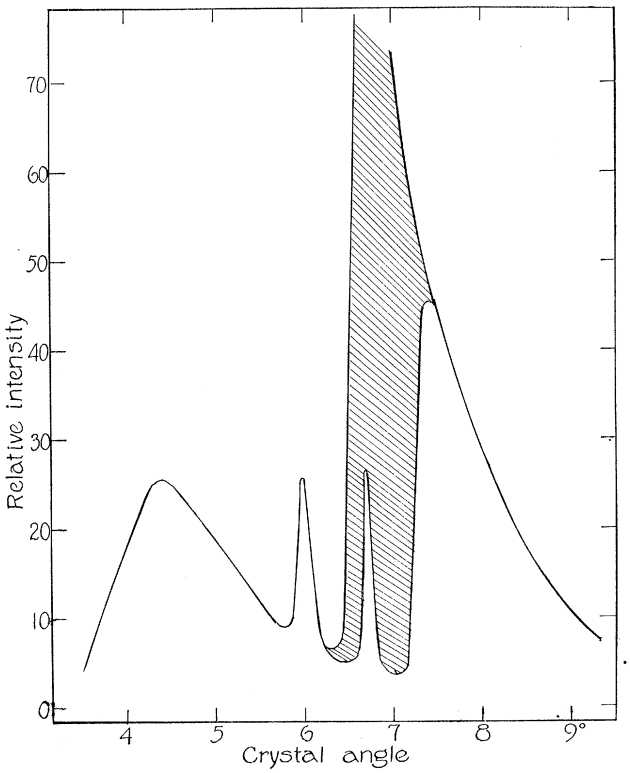

Fig. 1. Transmission of zirconium and strontium filters. Shaded portion represents radiation absorbed by strontium but transmitted by zirconium. Calcite crystals, $d=3.03$A.

cm apart. Intensity may be gained without the sacrifice of too much resolution by using shorter slits; but in the present work the intensity was sufficiently large.

The liquid sample was held in a container mounted on the center of the spectrometer table. In selecting a container for the experiments two objects were held in mind: (1) the container should be so constructed that its scattering of the x-rays would be small in comparison to the scattering of the volume of liquid used, and (2) the thickness of the sample should be less than the

[7] Soller, Phys. Rev. **24**, 139 (1924).

optimum value of $1/\mu$.[8] Flat metal containers with plane cellophane windows gave very little scattering, but no satisfactory glue was found for attaching the windows when used with good solvents.[9] A glass container was finally selected for use with such liquids. It was a very thin walled Pyrex tube, 6 mm in diameter. The beam was narrowed so that only the central portion of the tube was exposed. The scattering of the empty container (and of the air in the axis of the spectrometer) was first determined and this value, corrected for the absorption of the sample, subtracted from the observed scattering of the liquids investigated.

Filters. Since the critical absorption limits of strontium and zirconium lie at 0.7693A and 0.6872A respectively they show a great difference in transmission for rays of wave-length between these values. The zirconium filter was one supplied by the General Electric Company and the strontium filter was constructed by suspending strontium oxide in paraffin at a concentration of about 0.03 gm per cm^2. The latter filter was experimentally balanced against the zirconium by carefully shaving down until the two gave the same transmission for rays in the region 0.4–0.6A. After balancing, the transmission of the two filters was determined over the entire wave-length range by analysis of the transmitted beam with a calcite crystal spectrometer. Results of the analysis are shown in Fig. 1, in which the shaded area represents the radiation transmitted by the zirconium and not by the strontium. Argon was used in the chamber for this determination. A similar one with methyl bromide in the ionization chamber gave higher intensity throughout and showed a greater amount of general radiation, but the balance of the two filters agreed as well as when argon was used.

In making a determination of the intensity of scattered radiation successive readings of the intensity were taken with each of the filters. The difference in the ionization current for the two represents the contribution due to the $K\alpha$ lines.

Effect of the General Radiation

N-hexyl alcohol was chosen as the scattering liquid because it gives two well-defined peaks in the intensity curve and because the results obtained could be readily checked by comparison with those of Stewart and Morrow.[3] Separate scattering curves for each filter and for the balanced filter are shown in Fig. 2a and 2b, taken respectively with methyl bromide and argon in the ionization chamber. The strontium filter curves give the results of general radiation, while the zirconium filter curve gives the results of general plus characteristic radiation. A fairly true curve is obtained with the zirconium filter alone when argon is used for the detector, the effect of the general radiation being shown only by a general elevation of the intensity. With methyl bromide, on the other hand, the more complete absorption of the white radiation causes a peak at 6.5°. When higher voltages are employed this peak shifts to smaller angles as found by Thibaud and Trillat.[1] When a

[8] This was done because of the effective filtration of the beam by the sample (see Thibaud and Trillat, Ref. 1).

[9] Ambroid Liquid Cement was the most resistant one tried. For liquids that did not attack celluloid, windows were constructed of this and attached by Duco Household Cement.

photographic method of detection is used the effect of the general radiation is more pronounced because of the greater absorption in a photographic emulsion than in a gas-filled ionization chamber.

The corrected diffraction pattern of n-hexyl alcohol is given by curve 4 of Fig. 2b. This is in very close agreement with the result of Stewart and Morrow[3] for this liquid, and confirms their conclusion that the minor peak at 3° is due to the same radiation as the main peak.[10]

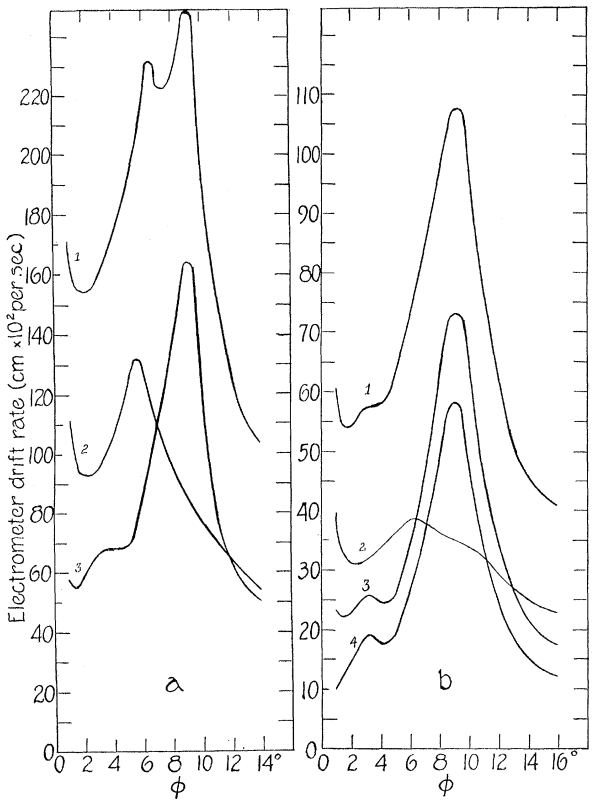

Fig. 2. Intensity-angle curves for n-hexyl alcohol. 2a. Methyl bromide in ionization chamber. (1) Zirconium filter. (2) Strontium filter. (3) Balanced filter- difference between (1) and (2). 2b. Argon in ionization chamber. (1) Zirconium filter (2) Strontium filter (3) Balanced filter-uncorrected (4) Balanced filter-corrected for scattering of container.

Conclusions

(1). The balanced filter is a satisfactory agent for isolating the effects of the general and characteristic radiation. (2). Under the conditions of the experiment the rays transmitted by the zirconium filter contain more general than characteristic radiation. (3). The diffraction pattern of a liquid for general radiation shows a sharp peak at an angle determined by the effective wave-length of x-rays. (4). Argon is a satisfactory detector for molybdenum x-rays when the relative intensity alone is desired. (5). The small angle peak found by Stewart and Morrow for n-primary alcohols is confirmed.

[10] Debye and Menke [Ergebnisse der technischen Röntgenkunde, **2**, 1 (1931)] suggest that perhaps this maximum is due to the general radiation.