

Argon *K* Absorption Edge by the Counter Response-Method*

HERBERT W. SCHNOPPER

*Department of Physics and Laboratory of Atomic and Solid State Physics,
Cornell University, Ithaca, New York*

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The fine structure of the argon *K* absorption edge has been observed by measuring the response of an argon-filled proportional counter as a function of the incident x-ray energy. It is shown that the response of a *thin* counter is proportional to μ_K , the *K*-shell contribution to the linear absorption coefficient. The fine structure observed in this method is then shown to be the same as is recorded in the conventional absorption method.

THE first experimenter to record the argon *K* edge-absorption region with a two-crystal spectrometer was Parratt.¹ His data, obtained with very high instrumental resolving power, showed very clearly the discrete absorption lines. Brogren² explored the argon *K* edge by an entirely new method. A bent-crystal spectrometer was used with a Geiger counter. The absorption spectrum was obtained by recording the Geiger counter response as a function of wavelength rather than by the usual technique¹ of measuring the transmission through a sample of gas. This gave a result which showed some structure in the edge, but could not be directly related to absorption coefficients because of unknown background effects. A new measurement which is a modification of Brogren's method is discussed here.

In the present study³ the response of a proportional counter, rather than of a Geiger counter, was measured because the pulse-height distribution from the proportional counter can be used to great advantage in eliminating overlapping higher order reflections and spurious background. A conventional absorption curve was also recorded and the results were compared.

A detailed discussion of the two-crystal spectrometer and experimental procedures is found elsewhere,³ and only pertinent details are given here. A platinum-plated copper anode produced a reasonably intense continuous spectrum when operated at 14.3 kV and 54 mA. The incident intensity I_0 was approximately 500 counts/sec. Calcite crystals of good quality (parallel position width 10.1 sec for 1.54-Å radiation) were used as dispersers. Intensity measurements were taken at intervals of 5 sec of arc (0.046 eV) by rotating the first crystal. A preset time of 200 sec/point was used.

The side window (0.001-in. Be) proportional counter was made from $\frac{3}{4}$ - \times $\frac{3}{4}$ -in. brass tubing 5 in. long. The constant absorption path length was 1.94 cm. A filling

of 90% argon-10% methane (*P*-10) was used, and the "thickness" of the counter was adjusted by varying the pressure. A gas absorber cell 1.00 in. long with 0.001-in. Be windows and filled with research-grade argon at various pressures was used for the absorption measurements.

The incident spectrum I_0 was recorded first with a krypton-filled counter. Then the absorber cell was positioned and the transmitted intensity I_T was measured. The absorber cell was then removed and the counter filled with the argon mixture for recording the counter-efficiency intensity I_C .

COUNTER-RESPONSE METHOD

Because a window-type linear amplifier is used, only those x rays which deposit their full energy in the counter are considered. The fraction of incident x rays absorbed in the counter gas is

$$I_A/I_0 = 1 - e^{-\mu_t x}, \quad (1)$$

where μ_t is the total linear absorption coefficient. Of these, a fraction, μ_K/μ_t , is absorbed in the argon *K* shell. The fraction of *K* absorptions for which the full incident x-ray energy appears in the counter pulse is

$$1 - \omega_K + \omega_K(1 - e^{-\mu_1 x_e}) = 1 - \omega_K e^{-\mu_1 x_e}, \quad (2)$$

where ω_K is the *K* shell fluorescence yield,⁴ μ_1 is the linear absorption coefficient for argon *K* α radiation, and x_e is some effective path length in the counter. Therefore, the counting rate corresponding to *K*-shell absorptions where the full x-ray energy is deposited in the counter is

$$I_K = I_0(\mu_K/\mu_t)(1 - e^{-\mu_t x})(1 - \omega_K e^{-\mu_1 x_e}) \quad (3)$$

which, for $\mu_t x \ll 1$ and $\mu_1 x_e \ll 1$ (a thin counter), reduces to

$$I_K \approx I_0 \mu_K x (1 - \omega_K + \omega_K \mu_1 x_e). \quad (4)$$

By a similar analysis it can be shown that the intensity corresponding to *L*, *M*, etc., absorption is (assuming no escape losses)

$$I_{LM} \approx I_0 \mu_{LM} x \quad (5)$$

⁴ For argon, ω_K is approximately 0.14. See T. Watanabe, H. W. Schnopper, and F. N. Cirillo, Phys. Rev. **127**, 2055 (1962).

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¹ L. G. Parratt, Phys. Rev. **56**, 295 (1939).

² G. Brogren, Nova Acta Regiae Soc. Sci. Upsaliensis **14**, No. 4 (1948).

³ H. W. Schnopper, Ph.D. thesis, Cornell University, 1962, (to be published).

and the total intensity I_C is

$$I_c \approx I_0 [\mu_K (1 - \omega_K + \omega_K \mu_1 x_e) + \mu_{LM}] x. \quad (6)$$

Thus, for a sufficiently thin counter, the change of counter efficiency through the region of the K edge, is directly proportional to the change in the absorption coefficient, μ_K , if the Auger coefficient, $1 - \omega_K$, can be assumed to be constant, and if necessary corrections are made for L , M , etc., absorption. Further, from Eq. (6), the value of x_e can be estimated if the Auger coefficient, the absorption coefficients, and I_0 are all known.

DATA

Figures 1 and 2 show the fine structures of the argon K edge as observed by the counter-response method and the absorption method, respectively. The intensity scale in Fig. 1 is proportional to I_C and that for Fig. 2 is proportional to $\ln(I_0/I_T) = \mu_i x$. The angle β is measured in units of the Bragg angle 2θ with $\beta=0$ arbitrarily chosen at the peak of the first resonance line ($2\theta = 79.2^\circ$).

Each curve represents the average of eight complete passes through the wavelength region of interest. The raw data have been corrected for (1) the shape of the incident spectrum, (2) the absorption in the detector window or gas cell windows, and (3) background. The data are further corrected^{3,5} for the finite resolution of the instrument ($\lambda/\Delta\lambda \approx 11\,000$). All the curves were then normalized to unit-integrated intensity.

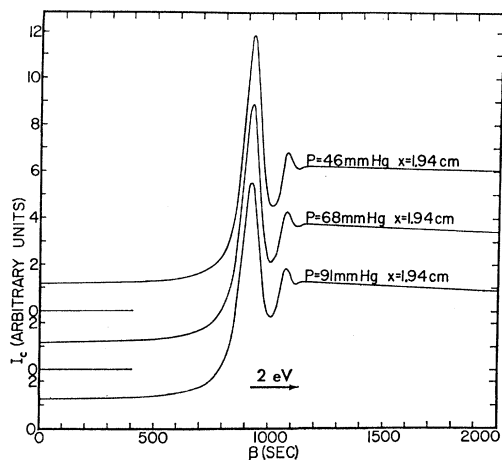


FIG. 1. The data from the counter-response experiment for various pressures in the counter. Only the corrected data are shown. Energy increases to the right.

⁵ For a discussion of the thickness effect see L. G. Parratt, C. F. Hempstead, and E. L. Jossem, Phys. Rev. **105**, 1228 (1957).

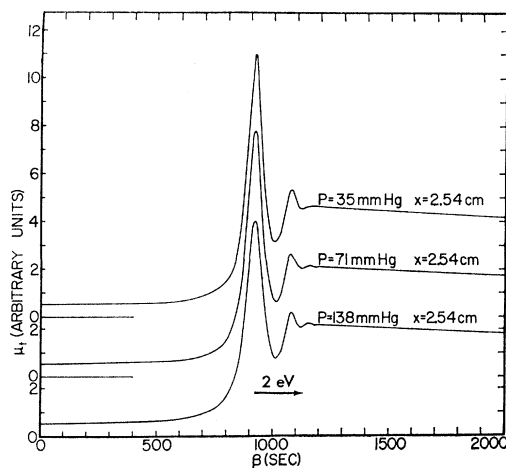


FIG. 2. The absorption curves derived from the transmission experiment for various pressures in the absorber cell. Only the corrected data are shown. Energy increases to the right.

After correction for L , M , etc., absorption, the transmission data can be compared in detail with the counter-response data by the use of Eq. (4). The counter-response curves show the same fine-structure features as the transmission curves.

CONCLUSIONS

It has been shown that the counter-response method leads to essentially the same absorption curve as the transmission method. The counter-response method has certain unique features. The counting rates are high in the region of large absorption contrast where the structure is most interesting. No corrections for x-ray scattering need be made since the sample is also the detector. For a sufficiently thin counter, an absorption curve is given directly. Because of the high-intensity contrast, a complete correction for resolution cannot be made. Also, I_0 must be measured accurately with a different counter filling and, therefore, absorption coefficients are difficult to obtain with this method.

The transmission method is standard for absorption work and for good reason: With the proper choice of sample thickness, the resolution correction can be reasonably complete and more accurate absorption coefficients obtained.

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