DECEMBER 15, 1938 PHYSICAL REVIEW VOLUME 54

The Absorption of X-Rays of Wave-Length $1.5 \leq \lambda \leq 8.3$A

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(Received September 27, 1938)

The mass absorption coefficients for Be (4), C (6), Al (13), Cu (29), Ag (47) and Au (79) have been determined in the wave-length region $1.5 \leq \lambda \leq 8.3$A. Only characteristic line radiation obtained with a vacuum spectrometer was used. Because of the presence of about 1 percent of heavier impurities in the Be (4), the apparent absorption coefficients for Be (4) may be as much as 100 percent higher than for the pure element. This large effect of a small amount of impurity on the apparent absorption coefficient of Be (4) leads to a suggested method of microanalysis for heavy impurities in elements of low atomic number. Following Jönsson's procedure, a universal absorption curve is plotted.

INTRODUCTION

THE determination of the laws of absorption of x-rays is complicated by the presence of two types of absorption, fluorescence absorption and absorption by scattering. With increase in wave-length, however, absorption by scattering becomes much smaller than photoelectric absorption. For wave-lengths above 1.5A and absorbing elements heavier than Al 13, absorption by scattering is negligible compared with photoelectric absorption. Above 2.5A absorption caused by scattering by elements heavier than C (6) is negligible. Thus accurate absorption measurements at the longer wave-lengths give important data for the developement of the laws of photoelectric absorption.

In 1928 Jönsson[1] measured absorption coefficients at wave-lengths as high as 10A, using a crystal spectrometer as monochromator and measuring intensity photographically. Woernle[2] made measurements from 2 to 10A on gases of low atomic number employing a vacuum spectrometer for monochromatization. The gases to be studied were placed in a special ionization chamber; an electrometer was used as a null instrument. Recently Biermann,[3] employing the Ross[4] balanced filter method to produce a monochromatic beam and measuring intensities with an ionization chamber and electrometer, covered the same range of wave-lengths, using seven elements and Cellophane as absorbers. His values are higher than Jönsson's above 3A for Al (13) and Ag (47), the spread increasing until at 10A for Ag (47) they differ by 45 percent.

This investigation was undertaken with the view of checking some of Jönsson's measurements by use of a more modern and entirely different technique, and of extending the measurements to more elements. While this work was in progress, Biermann's[3] results were published. Since Jönsson, Biermann and the author each employed different methods and each studied absorption by Al and Ag, a comparison of the results should indicate something of the accuracy of measurements in this wave-length region. A plot of all the absorption data by Jönsson's procedure for a universal absorption curve[1] should indicate whether the curve is constantly decreasing in slope or is made up of a series of broken lines, with the absorption coefficient depending on some constant power of the wave-length within any region not including an absorption discontinuity.

APPARATUS

To eliminate any doubt concerning the purity of the monochromatic radiation, characteristic lines of various elements were obtained in a modified Siegbahn-Thoraeus[5] vacuum spectrometer (see Fig. 1). The target arm and ionization chamber arm were each 105 cm long, with slits of such width as to give a resolving power of about 500, resolving power being defined as the ratio of

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[1] E. Jönsson, Thesis, Uppsala (1928).

[2] B. Woernle, Ann. d. Physik **5**, 475 (1930).

[3] H. Biermann, Ann. d. Physik **26**, 720 (1936).

[4] P. A. Ross, J. Opt. Soc. Am. and Rev. Sci. Inst. **16**, 433 (1928).

[5] M. Siegbahn and R. Thoraeus, J. Opt. Soc. Am. and Rev. Sci. Inst. **13**, 235 (1926).

the wave-length to the observed full width of the line at half-maximum. Since, at the long wave-lengths and correspondingly large Bragg angles employed, characteristic line radiation is far more intense than the continuous radiation, high geometric resolving power is not necessary to secure a highly monochromatic beam. The main purpose in the degree of resolution is to reduce radiation of other lines in that wave-length region. Thus, in using $K\alpha_1$-radiation, most of the $K\alpha_2$-radiaton must be eliminated. Since the absorption coefficients for $K\alpha_1$- and $K\alpha_2$-radiation in this wave-length region differ by only a small amount, a small portion of $K\alpha_2$ may be tolerated in the $K\alpha_1$-radiation.

The targets were wedged or soldered to a water-cooled holder. Targets of Cu, Ni, Fe, Ag, Pd, Mo and Al were made from sheet metal. Cr, Va and Ti targets were made by evaporation onto an Ag backing; Si by evaporation onto Al. For Sc, Ca, and Cl targets, Sc_2O_3, CaO, and KCl respectively were pressed into sheets of Ag.

$K\alpha_1$-radiation was used from Cu, Ni, Fe, Cr, Va, Ti, Sc and Ca; the whole $K\alpha$-radiation from Cl, Si and Al; $L\alpha_1$-radiation from Ag, Mo and Zr; $L\beta_1$ from Ag and Mo.

The absorbing foils were mounted on disk *A* Fig. 1, which could be turned by a ground cone *G* so as to bring any desired foil into the path of the beam. Since the maximum Bragg angle with this instrument is about 25°, crystals of calcite, selenite and mica were employed to cover the desired wave-length range.

X-ray intensities were measured with an ionization chamber. A Cellophane window separated the argon in the ionization chamber from the evacuated spectrometer. An FP-54 Pliotron tube amplified the ionization current. Control grid shunts as high as 5×10^{10} ohms were used. To balance out the effects of small changes in the Pliotron filament current, a Brown and DuBridge[6] circuit was employed. Tests at regular intervals showed the amplification to be always linear in the range used.

PREPARATION OF ABSORBERS

In this long wave-length region, a major portion of the problem was to produce sufficiently thin absorbers of known and uniform thickness.

The Be (4) absorbers for use at the shorter wave-lengths and the C (6) absorbers were granular cakes. To form the Be (4) cakes, the smallest grains were sifted from the supply of flakes and pressed together in a mold. The C (6) absorbers were made by turning out a disk from a stick of regraphitized graphite and scraping it down with an apparatus designed for scraping parallel surfaces.

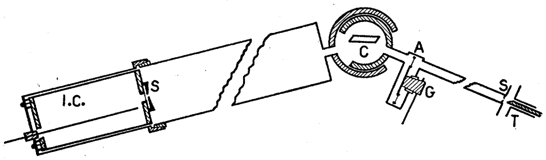

FIG. 1. Diagram of vacuum spectrometer.

All other absorbers used in this work were produced by evaporating the metal in high vacuum from a tungsten spiral basket onto a glass plate. The foils were removed mechanically with a razor blade. Fortunately the thinnest foils rolled into a tight roll as they were peeled from the glass plate. The smallest rolls, or "pipes," thus formed were just large enough to permit a small needle to be inserted. Such foils were more convenient to weigh and mount than those produced by rolling. Moreover, evaporation provided a means of producing foils of hard brittle metals, like Be (4), which could not be rolled. To mount the foils, one edge of the roll was stuck to the holder and the holder inclined until the weight of the needle unrolled the foil.

The absorbers were weighed on microbalances designed to weigh to a thousandth of a milligram. The weights were calibrated by the National Bureau of Standards.

To detect variations from point to point in the thickness of an absorber, the absorber was moved across the x-ray beam. Not one of the metal foils or carbon disks showed a variation of more than one percent in the intensity of the beam transmitted. The Be (4) cakes, however, showed a variation in transmitted intensity as high as 3 percent. To eliminate error caused by variation in thickness of an absorber, the absorbers were moved across the beam during measurements of intensity.

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[6] L. A. DuBridge and H. Brown, Rev. Sci. Inst. **4**, 532 (1933).

TABLE I. *Purity of the absorbers.*

ELEMENT	PURITY	IMPURITIES	REMARKS
Au	99.975%		Purchased from Baker and Company.
Ag	99.97%	Au 0.00034% Cu 0.0036% O 0.028%	Donated to F. K. Richtmyer by the Raritan Copper Works.
Cu	99.962%	Ag 0.00028% Pb 0.0009% Fe 0.0014% S 0.0025% O 0.033%	Purchased from Am. Brass Company.
Al	99.975%	Si 0.009% Fe 0.008% Cu 0.008%	Donated to F. K. Richtmyer by the Aluminum Company of America and tested in the Aluminum Research Laboratory, New Kensington, Pa.
C	99.9%	Ca	Regraphitized graphite from the Achison Graphite Company. Spectral analysis made by the Cornell University Chemistry Department.
Be (Granular)		Fe 0.4%	Purchased from and tested by Siemens & Halske.
Be (Evaporated foil)		Cu Fe Si W	Tested by spectral analysis by Cornell University Chemistry Department.

The tested purity of each metal, the amount of impurity, and the company responsible for the test are given in Table I.

METHOD OF TAKING DATA

During measurements of intensity, small fluctuations in tube current and tube voltage were controlled manually.

A contour of each characteristic line was constructed to see if there was any background of scattered radiation from the walls of the spectrometer. In no case was a background of half of one percent detected as the chamber was turned to either side of the line in use.

The intensity readings for the measurement of absorption were taken in the order: zero, absorber-out, absorber-in, absorber-out, and zero. The absorber-in reading consisted of a set of ten readings for ten uniformly spaced positions of the absorber as it was moved across the beam.

For each absorber and wave-length, about twenty such determinations of the absorption were made under different conditions of tube voltage and tube current.

RESULTS

In Table II are given the mass absorption coefficients for Au (79), Ag (47), Cu (29), Al (13), C (6), and the apparent mass absorption coefficients for flakes and foils of Be (4). The results for Au (79), Ag (47), Cu (29) and C (6) are plotted in Fig. 2, the results for Al (13) and Be (4) foils in Fig. 3.

Measurements of absorption by Au (79) were extended out beyond the five M absorption discontinuities to the N_1 absorption region. The only other measurements ever made beyond the M limits are those by Jönsson[1] on Pt (78). The plot of log μ/ρ against log λ in Fig. 2 reveals a decrease in slope as the plot is extended to the long wave-length side of the M absorption limits. The slopes computed from the data are 2.537 below the M limits and 2.225 above.

Likewise a plot for Ag (47) shows a decrease in slope from the short to the long wave-length side of the L absorption discontinuities. The computed slopes are 2.655 and 2.478.

The measurements for Cu (29) are entirely within the L_1 absorption region. The computed slope is 2.744. It is to be noted that the slopes decrease with increase in the atomic number of the absorber.

The measurements for Al (13) are within the K absorption region except for one at 8.3A just beyond the K discontinuity for Al. In Fig. 3 a straight line is drawn through the points of data although the data actually lie along a curve of constantly decreasing slope. This curve for Al is the only one in this work extending over a sufficiently long wave-length region to give definite evidence of a decreasing slope within a region not containing an absorption discontinuity. The computed slope, assuming a linear relation between log μ/ρ and log λ is 2.741.

The measurements for C (6) are entirely within the *K* absorption region. The computed slope is 2.834.

For the absorbers Au (79), Ag (47), Cu (29) and Al (13) and for wave-lengths below 3A, the estimated experimental error is less than 2 percent. For wave-lengths above 3A, the estimated experimental error is less than 3 percent. As the C (6) absorber was moved across the beam, no measurable variation of transmitted intensity was exhibited. However, because of the granular nature of graphite, there may have been a variation of transmitted intensity from point to point of the absorber. The experimental error for C (6) is therefore estimated as not more than 4 percent. However, since all the data, except that at 0.7078A, were taken with the same absorber, the slope for C (6) is as accurately determined as that for any of the metal foil absorbers.

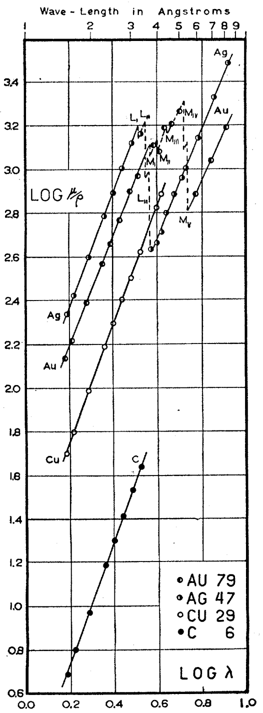

FIG. 2. Mass absorption coefficients for Au, Ag, Cu and C.

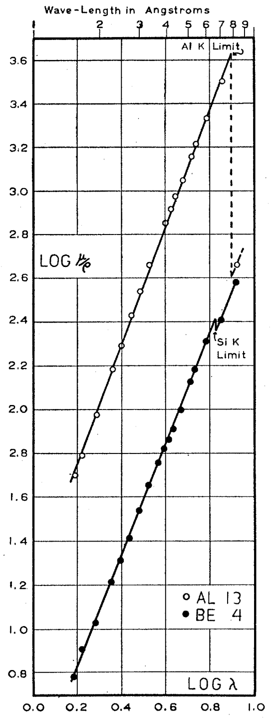

FIG. 3. Mass absorption coefficients for Al and Be.

A comparison of the results of Jönsson,[1] Biermann,[3] and the author in Fig. 4 may give some idea of the precision of absorption measurements in this wave-length region. Each observer used distinctly different methods. The close agreement between the results of the author and those of Biermann, and the large variation from those by Jönsson is significant of the modern improvements in the methods of measuring x-ray intensities. As Biermann has suggested, much of the discrepancy is no doubt caused by scattering from Jönsson's crystal, but also to the inherent difficulties of making precise intensity measurements by photographic means

Since the Be (4) contained nearly 1 percent of higher atomic-number impurities, the values

TABLE II. *The mass absorption coefficients*, μ/ρ, *in* cm^2 g^{-1}.

λ	Au (79)	Ag (47)	Cu (29)	Al (31)	C (6)	Be (4)* FLAKES	Be (4)* FOILS
0.7078A		27.1		5.08	0.594		
1.537	216	217	50.0	49.7	4.87	4.87	6.0
1.655	259	264	63.0	61.3	6.18	6.04	8.15
1.753						5.04	
1.932	387	394	96.7	93.5	9.38	6.55	10.5
2.285	585	612	155	153	15.5	10.1	16.3
2.498	720	779	198	194	20.0		20.4
2.743	925	1007	256	268	26.1		25.8
3.025	1252	1320	319	346	34.0		34.7
3.352	1470	1450	439	459	43.8		45.1
3.694		431					57.2
3.927	2050	459	673	710			66.0
4.146	1910	517	773	822			73.2
4.395	2450	629		941			90.8
4.718	2550	764		1108			99.0
5.167	2910	905		1430			134
5.395		1012		1630			152
6.057	1210	1380		2130			203
7.111	1730	2145		3170			257
8.321	2450	3070		459			381

* No corrections have been made for the impurities in the Be (4). Siemens and Halske found the flakes to contain 0.4 percent of Fe (27). Spectral analysis of the foils revealed traces of Fe (27), Cu (29), Si (14) and W (74).

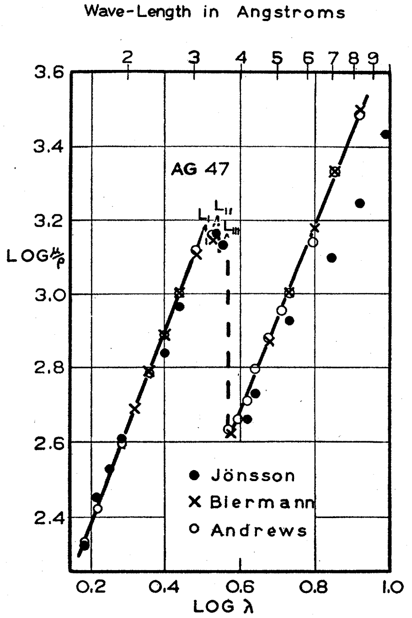

FIG. 4. Comparison of mass absorption measurements of Jönsson, Biermann and the author.

obtained for the mass absorption coefficients are possibly as much as 100 percent higher than they would be for pure Be (4). However, the results are given because some of the experimental difficulties may be pointed out.

So great is the effect of a heavy impurity on the apparent mass absorption coefficient of a light element such as Be (4), that measurements of the absorption coefficients in the neighborhood of an absorption limit of an impurity provide a means of microanalysis for that impurity.

Be (4) flakes were obtained from Siemens and Halske and were estimated by that company to contain 0.4 percent of Fe (27). The mass absorption coefficients given in Table II for a cake of Be (4) flakes were plotted against the cube of the wave-length in Fig. 5 and the Fe K discontinuity revealed 0.44 percent of Fe impurity.[7]

Even if the Be is pure, cakes of flakes are much too granular to permit accurate absorption measurements in this wave-length region. For the first time, Be foils have been produced to permit an attempt at measurement at long wave-lengths. With considerable difficulty, for Be (4) is much more brittle and clings more firmly to the glass plate than do the other metals, thirty-seven foils were produced. This stack of foils was sufficient to absorb half of the intensity at 4A.

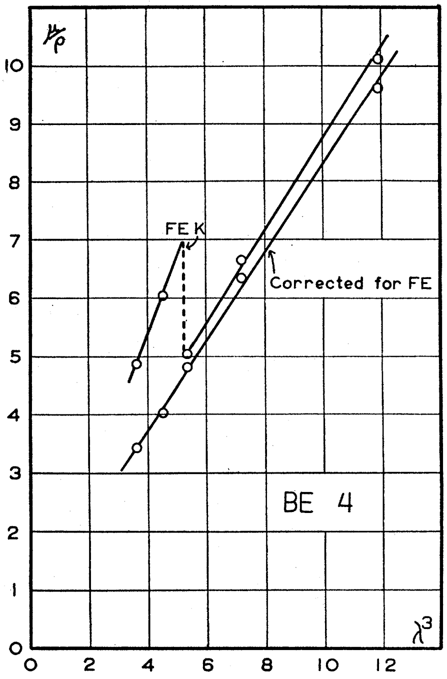

FIG. 5. Plot of mass absorption coefficients of beryllium which shows the Fe K absorption discontinuity caused by 0.44 percent of Fe impurity.

The measurements of the apparent mass absorption coefficients for Be (4) foil are given in Table II and plotted in Fig. 3. This stack of the thickest foils that could be produced without the formation of bubbles behind them, was too thin to permit accurate measurements below 2A. Thus

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[7] If the masses per unit area of the total absorber, the Be, the Fe, and all the other combined impurities are represented respectively by m_T, m_{Be}, m_{Fe}, and m_I and if, at the Fe K absorption limit, the maximum and the minimum absorption coefficients for the total absorber and for Fe are, respectively, $(\mu/\rho)_T$, $(\mu/\rho)_T'$, $(\mu/\rho)_{Fe}$, and $(\mu/\rho)_{Fe}'$ and for Be and the other combined impurities are $(\mu/\rho)_{Be}$ and $(\mu/\rho)_I$, then

$$(\mu/\rho)_T m_T = (\mu/\rho)_{Fe} m_{Fe} + (\mu/\rho)_{Be} m_{Be} + (\mu/\rho)_I m_I \quad (1)$$

and

$$(\mu/\rho)_T' m_T = (\mu/\rho)_{Fe}' m_{Fe} + (\mu/\rho)_{Be} m_{Be} + (\mu/\rho)_I m_I. \quad (2)$$

Subtracting Eq. (2) from Eq. (1) and solving for m_{Fe}/m_T, we obtain, independently of any knowledge of the other impurities or of the absorption coefficient of pure Be, the fraction of Fe present.

$$\frac{m_{Fe}}{m_T} = \frac{(\mu/\rho)_T - (\mu/\rho)_T'}{(\mu/\rho)_{Fe} - (\mu/\rho)_{Fe}'} = \frac{6.80 - 5.00}{460 - 54} = 0.0044.$$

The mass absorption coefficients for Fe were taken from Allen's tables in Compton and Allison's book, *X-rays in Theory and Experiment.*

no attempt was made to analyze for Fe (27) at its K limit.

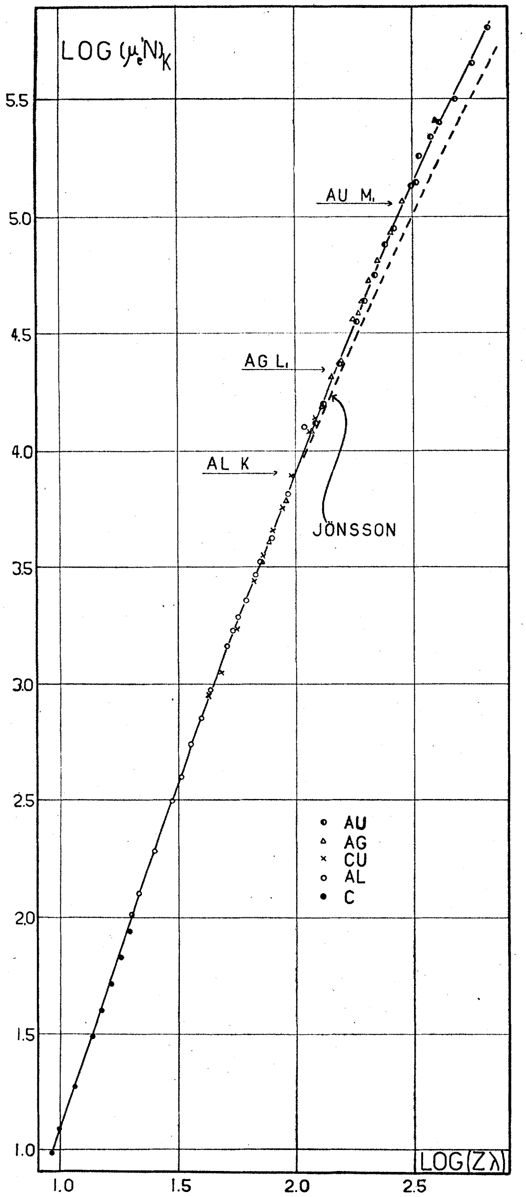

FIG. 6. Plot of electronic absorption coefficients which verifies Jönsson's conclusion that they are simple functions of the product $Z\lambda$.

Such foils of Be (4) could be of considerable value as x-ray windows and as backings on which to evaporate heavier elements for absorption measurements at still longer wave-lengths.

UNIVERSAL ABSORPTION CURVE

The electronic absorption coefficient, μ_e, is defined by the expression

$$\mu_e = (\mu/\rho)(A/ZN), \tag{1}$$

where N is the number of atoms per gram atom, A the atomic weight of the absorber, and Z the atomic number of the absorber. As a means of eliminating the absorption discontinuities, Jönsson[1] has suggested multiplying the absorption coefficient by E_K/E_x. E_K is the energy of the K state of an atom of the absorber and E_x is the energy of that state of an atom of the absorber corresponding to the first absorption discontinuity on the long wave-length side of the wave-length under consideration. $\mu_e N E_K/E_x$ may, without confusion, be expressed by the abbreviation $(\mu_e N)_K$.

In Fig. 6 the author's values of log $(\mu_e N)_K$ are plotted against log $Z\lambda$. Although this curve lies above Jönsson's, most of the data for the five elements lie along a sufficiently smooth curve to confirm, within the limits of experimental error, Jönsson's conclusion that the electronic absorption coefficient is some simple function of the product $Z\lambda$.

ACKNOWLEDGMENTS

The author wishes to express his appreciation of the constant encouragement and advice which he has received from Dean F. K. Richtmyer during the course of this problem, of valuable suggestions from Professor L. G. Parratt, and of help from Dr. S. Kaufman who had previously designed much of the apparatus.