

## SOME EFFECTS OF TEMPERATURE ON X-RAY ABSORPTION

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## ABSTRACT

**Effect of temperature on the average atomic absorption coefficients of Ag and Ni for x-rays.**—The previous results of the author showing a change with temperature of the average atomic absorption coefficients of various metals for x-rays have been confirmed in the case of Ag and Ni. The complete radiation from an x-ray tube operated at 50,000 volts was used. Heating silver to 830°C decreased its transmission about 1.3 percent. Heating Ni to from 700° to 1000°C decreased its transmission about 1.7 percent.

**Effect of temperature on the monochromatic atomic absorption coefficient of Ag between  $\lambda.275$  and  $.870A$ .**—Spectroscopic resolution of the x-rays passed by a silver absorber alternately heated to 830°C and cooled to room temperature demonstrated a marked variation with wave-length of the effect of temperature on the atomic absorption coefficient. The variation was most marked near the K absorption limit of Ag. On the short wave-length side heating the silver to 830°C increased the atomic transmission about 1 percent whereas on the long wave-length side it was decreased about 1.5 percent. If this change be interpreted as due to an increase in the wave-length of the K-limit, calculation shows the increase to be about 0.21XU.

WHILE testing for evidence to substantiate the statements found in the literature that x-ray absorption is independent of the temperature of the absorber, a surprising result was found.<sup>2</sup> Absorption increased slightly with the temperature. In the former work the total x-radiation from a Coolidge tungsten tube was used but the study has now been continued by measuring the temperature effect for the different wave-lengths and in particular on the short wave-length side of the K-limit of Ni, on both sides of the K-limit of Ag, and on the long wave-length side of the K-limit for Pt. Temperature affected the x-ray absorption quite differently at the different wave-lengths.

## APPARATUS AND METHOD

The effect of temperature was large enough to measure easily but the effect of thermal expansion also was large and the real change in absorption was a small difference between the measured change,  $K_m$ , and the calculated change,  $K_c$ , due to expansion only. Since the change in absorption was of the order of one percent consistent results were difficult to obtain. For all experiments one or two assistants accurately adjusted the high voltage and the current supplied to the tungsten Coolidge x-ray

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<sup>2</sup> H. S. Read, Phys. Rev. 27, 373 (1926).

tube.<sup>3</sup> It was possible to hold the high voltage constant to one part in 3000 and the current constant to one part in 20,000, and to measure the x-rays in the ionization chamber to one part in 3000. However, for unknown reasons, the measured x-ray intensities frequently varied by over 1/10 percent.

The apparatus may be described as follows. A Coolidge, tungsten-target, x-ray tube was placed in a lead box in such a manner that the focal spot was a vertical line source of x-rays through a vertical slit in the box. The relay that timed all exposures moved a lead block in front of or away from this first slit. Next came the thin absorber. It was 10 cm long, 2.5 cm wide, and from 1 to  $2 \times 10^{-3}$  cm thick. Two small V-shaped crooks bent into the absorber allowed it to heat up and expand nicely without any detected warping. The absorber was placed horizontally, on edge, and was clamped near the ends into two heavy brass uprights. The clamps were provided with jaws of nickel to prevent corrosion. The uprights were parts of a rigid frame that was moved up and down on guides so that the absorber was either just below the x-ray beam or squarely across and more than covering the beam. The absorber was heated to incandescence by a large 60 cycle alternating current from a step-down transformer and cooled to room temperature by an electric fan. The higher temperatures were measured with a Siemens and Halske optical pyrometer and these temperatures did not vary measurably over an area twenty times larger than the x-ray beam. Two .001 cm Al screens, 8 cm square, were placed on the two sides of and 1 cm from the absorber to keep heat away from the lead slits, spectrometer, and crystal.

The unabsorbed part of the x-ray beam passed on to a second slit only 1.0 mm wide, thence to a calcite crystal on a spectrometer table, on to a third slit, 4.0 mm wide and equally distant from the crystal, and into a large ionization chamber to be absorbed there in methyl-bromide, in the usual manner.

A second ionization chamber with equal and opposite voltage on it was used to neutralize the effects of a very small ionization current due to some natural penetrating radiation. No x-rays entered it.

During an exposure of the order of 30 seconds timed to 1/300 second the unabsorbed x-rays were reflected from the calcite crystal and ionized the gas in one chamber. As the chamber rod and string electrometer wire charged up, the electrometer showed a deflection. The deflection was counteracted by changing the voltage on the second ionization chamber just sufficiently to bind all the collected charge onto the rod there. The

<sup>3</sup> The high voltage circuit used was a development of that first described by D. L. Webster, Proc. Nat. Acad. Sc. 6, 28, 269 (1920).

x-ray intensity was recorded in terms of voltage change necessary to keep the electrometer deflection at zero. This method had the advantage that the voltage on the working ionization chamber never changed and any slight lack of saturation voltage there caused no effect. The counteracting voltage was measured accurately by use of a calibrated galvanometer with a 4000 mm scale at 5 meters distance. The usual galvanometer deflection was from 2000 to 3000 mm which was equivalent to an electrometer deflection of 200 to 300 divisions. Thus although the electrometer never moved appreciably it was reset to zero to within one part in 2000 to 3000, i.e., to a tenth of one division. The electrometer had a sensitivity of 200 divisions per volt on the string but only 50 divisions per volt on the second ionization chamber.

The measurements of x-ray intensity with the absorber below the beam and across the beam gave data used to calculate the absorption coefficient in the usual way. While the absorber was held in place across the beam alternate measurements of x-ray intensity for the absorber cool and hot indicated the percent increase of transmission due to heating,  $K_m$ .

The expected percent change,  $K_c$ , due to the thermal expansion of part of the absorber out of the beam, was calculated. In the well known transmission equation

$$I = I_0 e^{-anx}$$

we need only remember that  $an$ , which includes a density factor, changes inversely as the cube of the dimension and that  $x$  changes directly as the first power of the dimension. The linear dimension of the absorber changes with temperature in a manner represented by the equation

$$L_t = L_0(1 + \alpha T + \beta T^2) = L_0(1 + g)$$

Let  $W_1$  be the total transmitted intensity for a cool absorber and  $W_2$  for it when heated. The percent increase in transmission due to heating may be defined as  $(W_2 - W_1)/W_1$ . Using the approximations

$$(1 + g)^{-2} = 1 - 2g, \quad e^{2ganx} = 1 + 2ganx$$

we get approximately, assuming the change in transmission to be due solely to thermal expansion,

$$K_c = 2ganx$$

The *measured* percent change of transmission,  $K_m$ , does not seem to equal  $K_c$  and this unaccounted for difference ( $K_m - K_c$ ) is here reported as due to an intrinsic change with temperature of the atomic absorption coefficient of the absorber. The values of  $K_m - K_c$  are shown in tables and curves.

## PRECAUTIONS

Several precautions were needed to insure and to test the accuracy of the method, and to expose experimental errors. Heat from the absorber was kept away from all other parts by the two thin Al screens. Successive runs with the absorber heated were made without the alternate cooling to learn whether the leakage of heat to the absorber holders expanded them enough to move the absorber as a whole or not, but no effect was found. A test was made to learn if the magnetic effect of the absorber heating currents and the heat lost from the absorber to the air, Al screens, and to the supports had any effect. For this test the absorber was lowered to a position just below the x-ray beam and while there alternate readings were taken with the absorber cool and hot. The averages of the two sets of readings were equal to within one part in 3000. This indicates that the above mentioned causes produced only negligible effects.

An attempt was made to learn whether or not the change of absorption which included scattering was due to a change in scattering. The absorber was placed across the x-ray beam as usual and the unabsorbed x-rays reflected. After a plot was made of the distribution of the x-ray beam at the ionization chamber with the crystal fixed, the chamber was set just off the beam where it received scattered and perhaps a small amount of reflected x-rays. These scattered x-rays had 0.04 of the intensity of the main x-ray beam. Alternate readings with the absorber hot and cold were nearly equal. If there is any change of scattering with temperature it was not one-tenth large enough to explain the change of absorption. However, this test does not prove that there is no temperature effect on scattering.

Second order reflections were not present except in a few cases not reported here and then the change of transmission due to heating was some average value for the two wave-lengths.

## RESULTS

1. *Change of absorption.* The results are best shown in the form of tables and curves. Table I shows the coefficients of expansion that were used, copied from Landolt and Bornstein, 5th edition, p. 1228. It is quite possible that the metals used here, because of their state of purity or because of rolling, had slightly different coefficients of expansion. The metals were purchased from Eimer and Amend. Any error in calculated expansion would change the result but could not account for the variation in the change of absorption with wave-length.

TABLE I

*Coefficients of expansion used.*

$$L_t = L_0(1 + \alpha T + \beta T^2)$$

	$\alpha$	$\beta$	Range
Nickel <sup>4</sup>	$13.460 \times 10^{-6}$	$35.15 \times 10^{-10}$	500–1000°C
Silver	18.270	47.93	0–875°C
Platinum	8.868	13.24	0–1000°C

Two attempts were made to compare the results of the new system of measurement with those of the former paper.<sup>2</sup> With the ionization chamber directed straight into the slit system at the x-ray tube target and with the calcite crystal removed, all the wave-lengths of the x-ray spectrum were used. Since in the previous work 50 kilovolts a.c. was used on the tube and in the present work 50 kilovolts d.c. the energy spectra are different but as nearly alike as was possible. The new values agree with the old values more nearly than was expected, Table II and Fig. 1.

TABLE II

*Check on previous results.*

All wave-lengths from the x-ray tube at 50,000 volts d.c. were used.

	$T$	$2g$	$anx$	$K_m$	$K_c$	$K_m - K_c$	Note
Ag	20–830	.0359	.989	.0225	.0355	-.0130	new
	20–830	taken from published curve				-.0180	old
Ni	20–764	.0236	1.41	.0182	.0333	-.0151	new
	20–880	.0280	1.42	.0233	.0396	-.0163	new
	20–1000	.0328	1.42	.0262	.0465	-.0203	new
	20–764	taken from published curve				-.0140	old
	20–880	redrawn after omitting the				-.0165	old
	20–1000	880° point.				-.0190	old

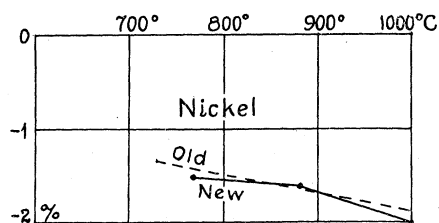


Fig. 1. Decrease due to heating of the total transmission of x-rays by nickel. Dotted curve shows previous work; solid line curve shows present work.

The final measurements are represented in Table III and in the solid line curve in Fig. 2. For some wave-lengths measurements were repeated and the average value is plotted. The dotted curve represents preliminary tests during the development work on the apparatus. It is less reliable and yet shows clearly the same general shape. Experiments at shorter

<sup>4</sup> Also Scientific Papers of the Bureau of Standards 17, 500 (1922), gives these values for nickel.

TABLE III

Change of transmission of silver when heated from 20° to 830°C

$\lambda$	$anx$	$K_m$	$K_c$	$K_m - K_c$
.275A	.540	.0194	.0194	.0000
.330	.860	.0265	.0309	-.0044
.330	.748	.0270	.0246	-.0024
.366	1.07	.0300	.0386	-.0086
.385	1.18	.0538	.0427	.0109
.385	1.18	.0435	.0423	.0012
.403	1.31	.0500	.0472	.0028
.437	1.63	.0689	.0586	.0103
.437	1.68	.0627	.0605	.0022
.457	1.84	.0718	.0664	.0054
.457	1.87	.0735	.0669	.0066
.457	1.82	.0725	.0657	.0068
.457	1.72	.0631	.0620	.0011
.475	1.95	.0725	.0703	.0022
.475	1.87	.0665	.0673	-.0008
.493	.627	.0136	.0225	-.0089
.500	.649	.0192	.0234	-.0042
.510	.447	.0142	.0164	-.0022
.510	.458	.0090	.0165	-.0075
.545	.509	.0223	.0183	.0040
.650	.792	.0236	.0285	-.0049
.755	1.12	.0338	.0402	-.0064
.870	1.35	.0259	.0450	-.0141

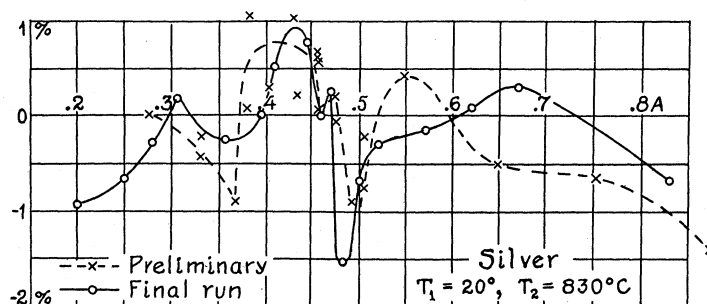


Fig. 2. Variation with wave-length of the increase in transmission of a silver absorber heated to 830°C.

wave-lengths failed because the higher required voltage was very difficult to maintain constant on account of corona loss and leakage. At times the services of two experienced assistants adjusting voltage and current were not sufficient. Experiments at longer wave-lengths failed because the x-ray intensity was too small to measure accurately. Greater resolution could easily have been attained by simply reducing the slit widths. However, the 1.00 mm slit used was a practical minimum since that much x-ray intensity was required to allow intensity measurements to better than 1/10 percent.

2. *Change in the K-limit.* The observed change in transmission through Ag for wave-lengths very near the K-limit was very marked. If it is due to a change in the K-limit the change can be calculated. F. K. Richtmyer<sup>5</sup> has shown that the K-limit is an abrupt break as shown in Fig. 3. Consider the case in which the slit admits wave-lengths and intensities bounding the areas  $A_1$  and  $A_2$ . The energy transmitted is  $A_1 + A_2 = A_C$  or  $A_H$ , when cool or hot. Since  $I_1$  is approximately  $1/6 I_2$

$$A_C = \frac{1}{2}SI_1 + \frac{1}{2}SI_2 = 3.5 SI_1$$

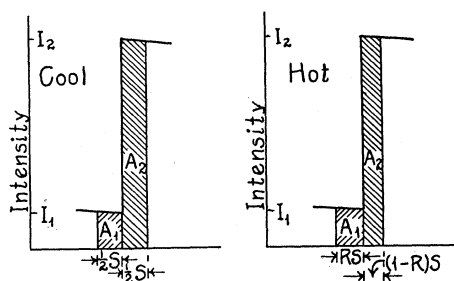


Fig. 3. The K-limit shift is used to account for the observed decrease in transmission near the K-limit of Ag.

When hot the division is not half the half but  $R$  and  $(1-R)$  say.

$$A_H = RSI_1 + (1-R)SI_2 = (6-5R)SI_1$$

The measurements show  $A_C = 1.015 A_H$ . Thus  $R = .510$  and  $R - .500 = .010$ . Heat shifted the limit  $.010S$  toward longer wave-lengths.

The actual curve was not like Fig. 3 but had the usual rounded corners. The difference in crystal angles for the maximum and minimum points was used to calculate the range of wave-lengths covered by the slit. Here the slit covered  $0^\circ 15'$  or  $21XU$ . Thus the shift was  $.010 \times 21XU = 0.21XU$ , a very small amount. Since the limit,  $485XU$ , requires  $25,400$  volts the shift corresponds to  $11$  volts. The heat of molecular agitation at  $830^\circ C$  corresponds to less than one volt.

The above hypothesis suggests that heat aided the quanta in ionizing atoms in some undescribed way, that a smaller quantum was able to ionize a hot atom. If this hypothesis is established a slight shift in the emission spectrum and the short wave-length limit toward shorter wave-lengths should occur when the target of the tube is allowed to heat up to incandescence as is common. Scattered x-rays from a hot scatterer might likewise be of slightly shorter wave-length than the primary x-rays or than the x-rays from a cool scatterer. The writer does not know of any

<sup>5</sup> F. K. Richtmyer, Phys. Rev. **26**, 724 (1925).

other measurements to substantiate the hypothesis but points out that only very accurate measurements would show such a small shift of wave-length.

It appears that a very critical test might be made by comparing the measured transmissions through a hot and cool Ag absorber of a K line from an Ag target tube. If the shift suggested above is real the absorber on being heated up should transmit more of any K line, even more of an increase than thermal expansion would suggest.

D. L. Webster has suggested a photographic method. If a plate is exposed to reflected general or white radiation the plate will be darkened about 6.0 times more on the shorter wave-length side of the K-limit than on the long wave-length side, due to absorption in the emulsion. If a silver absorber of the right thickness is interposed it will neutralize the effect and give a uniformly exposed plate. Now if heating the absorber shifts its K-limit to a longer wave-length a narrow line of less exposure should appear on the plate. The heated absorber should be surrounded by some inert gas or a vacuum and the wall of the chamber carefully kept at constant temperature.

3. *Change in absorption.* In the previous paper<sup>2</sup> it was shown that if  $a'$  and  $a$  are the average atomic absorption coefficients at the temperature  $T$  and at room temperature respectively, the results are fairly well represented by the equation

$$a' = (1+g)a,$$

where

$$1+g = 1 + \alpha T + \beta T^2$$

This was based on measurements at several temperatures and on several metals for which  $2K_m = K_c$ , approximately. The present relation is not so simple. Here

$$K_m = M_\lambda K_c$$

and  $M_\lambda$  is greater than unity for some wave-lengths and less than unity for others. Using as a good approximation of  $K_c$  the expression  $2 \int a_n x$  it can be shown that  $a' = (1+g)a$  should be replaced by the more general

$$a' = [1 - g(2 - M_\lambda)]a$$

This gives at best an inadequate idea of the change of absorption and in fact no explanation of the phenomena is offered.

It does not seem possible to explain the change in terms of a shift of the white spectrum because for each wave-length the voltage was adjusted so that the wave-length used came near the flat topped maximum of white x-radiation.



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