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# CXVI. *The Interpretation of X-Ray Absorption Spectra of Solids.*

By Y. Cauchois [*] and N. F. Mott [†].

[Received September 27, 1949.]

[Plates XXIV. & XXV.]

## Abstract.

A discussion is given of some recent experimental results on the fine structure of the X-ray absorption edges of solids. Recent work has shown the existence for many solids of a pronounced maximum (raie blanche) at the absorption edge. For K-absorption this may be due to one of two causes: (i) a high density of normally unoccupied states, with *p* symmetry in the neighbourhood of the absorbing atom; this is for example the case for nickel in oxides; and (ii) the formation of exciton levels. Exciton levels are defined as energy levels which are not formed until the electron is removed from the X-ray level, and are caused by the field round the point where the charge is missing. They are to be expected in insulators but not in metals. The experimental evidence is discussed in the light of this theoretical model, with special reference to the semi-metals gallium and arsenic.

## § 1.

The absorption spectrum of a free atom, for X-rays as for visible light, consists of a series of lines leading up to a series limit ; in the case of X-rays the lines are considerably broadened on account of the short life-time, due to Auger effect, of the ionized state of an X-ray level (Richtmyer, Barnes and Ramberg 1934). An example is the K absorption of argon measured by Parratt (1939). It seems fairly certain from the theoretical point of view that the X-ray absorption spectra of solids which do not show metallic conduction ought to consist of a similar series of lines leading to a limit. This is for the following reason : the series limit corresponds to a transition in which the electron from the X-ray (*e. g.* the K) level is ejected directly into the conduction band (transition (*a*) of fig. 1). But as soon as the electron has left the K level, it leaves a residual positive charge on the K shell ; and owing to this there are formed, below the bottom of the conduction band, the series of so-called

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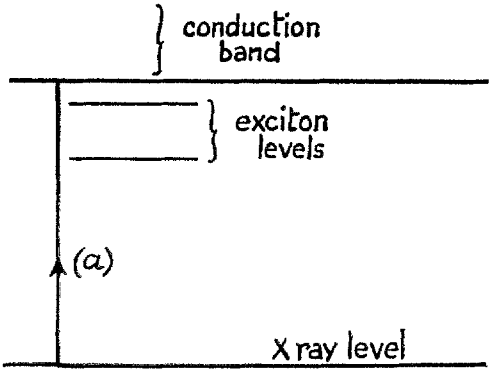
exciton levels into which the electron may make a transition. The existence of these levels has its origin in the fact that the field round the positive charge may, at large distances, be taken to be of the form $e/\kappa r^2$, so that the additional potential energy of an electron moving in it is

$$V(r) = -e^2/\kappa r.$$

$\kappa$ is the dielectric constant at high frequencies (square of the refractive index). The Schrödinger equation for an electron moving in this field, even when superimposed on the periodic field of the lattice, must have a series of quantized values of the energy leading to a series limit.

In general these levels should be much closer together than for the isolated atoms. Consider, for example, the case of solid argon, where, after absorption of X-rays by a K-electron, the electron will be in a $4p$ orbit. The $4p$ orbit has a much greater radius than that of the *neutral* atom, and thus than the interatomic distance in the solid. In the solid, therefore, the potential in which the electron moves, in addition to the periodic field of the lattice, is as already stated $-e^2/\kappa r$. In the formula for the interval between the energy levels, $\kappa$ will enter in the denominator squared; one has only to remember the Bohr formula $2\pi^2 me^4/h^2$ to see this. Therefore one expects that in general these exciton levels will be separated from the series limit by an interval smaller by a factor $\kappa^2$ than for the isolated atom.

Fig. 1.



Energy levels of a non-metal.

The ultra-violet absorption spectrum of the alkali-halides has been discussed by Mott and Gurney (1940) in terms of this model; the absorption is due to a transition of the outer $p$-electron of the halide ions into an exciton level, supposed to have $s$ symmetry relative to the halide ion, or directly into the conduction band. The separation between the exciton level and the series limit is of the order of 2 electron volts. For substances with higher dielectric constant one would expect it to be still smaller, perhaps a fraction of an electron volt. With separations of this order, for the K absorption of solid substances one would expect the exciton level to be difficult if not impossible to observe. On the other hand, if there are vacant $p$-orbitals in the absorbing atom (*e. g.* in arsenic, solid halogens, etc.,) a K-electron ejected into an exciton level may well remain within its own atom, so that the argument given above about the reduction in the force between the electron and the positive charge no longer applies. In these cases, therefore, the exciton level may well be several electron volts below the conduction band and therefore observable in X-ray absorption spectra.

It is the principal purpose of this paper to examine the experimental material on X-ray absorption and to see what evidence there is for the existence of these exciton levels. This will be done in § 2. We shall first consider some other details of the absorption spectrum of non-metals, and then contrast the expected behaviour of metals.

In a free atom, whether one is dealing with optical or X-ray absorption, the *theoretical* value of the absorption coefficient at the series limit is finite, not zero. On theoretical grounds, also, the same should be true for an insulator. This result, whether for the free atom or for the insulator, seems at first sight surprising, because N(E), the density of states in a band or in a vacuum, tends to zero as $E^{\frac{1}{2}}$ with E, and the intensity of absorption I(E) is proportional to N(E). If $p(E)$ is the transition probability, we may write

$$I(E)=\text{const.}\, p(E)N(E),$$

and it thus follows that $p(E)$ must tend to infinity as $E^{-\frac{1}{2}}$ as $E \rightarrow 0$. That this is so is actually a consequence of the Coulomb field of potential $-e^2/\kappa r$ superimposed on the lattice field in which the electron moves when ejected into the conduction band. The transition probability $p(E)$ is proportional to the square of an integral of the type

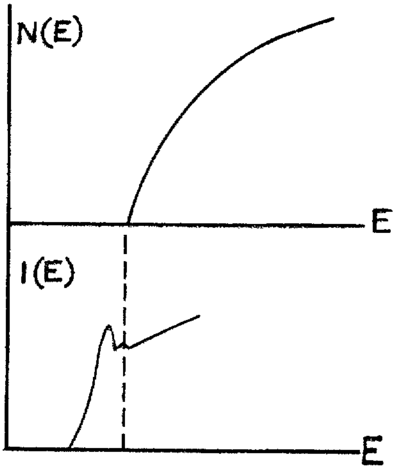
$$\left| \int \psi_f^* \frac{\partial}{\partial x} \psi_K \, d\tau \right|^2,$$

where $\psi_K$ is the wave function in the initial K state and $\psi_f$ in the final state. On account of the attractive Coulomb field, the value of $|\psi_f|^2$ at the origin (and thus in the region occupied by the K shell) tends to infinity as E tends to zero—or rather as the velocity of the electron tends to zero (*cf.* for example Mott and Massey 1949).

All these arguments ought to apply not only to the bottom of the first empty band but to the bottom of all the other bands as well. The velocity (group velocity $\partial E/\partial k$) is zero here, just as at the bottom of the first band. Thus one expects a finite absorption coefficient at the bottom of *any* band, and exciton levels below it. The spectrum should thus be as shown in fig. 2 (for reasons to be given below this applies only to non-metals). Probably experimental resolution would not be sufficiently great to show up the exciton levels, but what one expects is a *sharp* beginning to each absorption minimum in the Kronig structure, as indicated in fig. 3 (curve (*a*)). In figs. 4 and 5 we shall show at least two insulators (Millerite (NiS) and $Ni_2O_3$) where these predictions seem to be borne out by experiment.

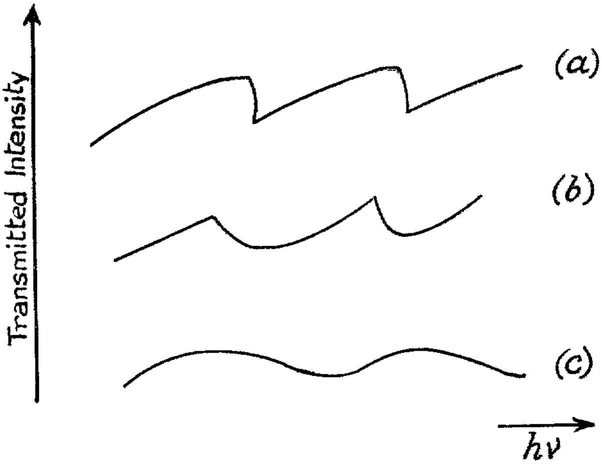
It must be admitted that there are many reasons why this behaviour should be obscured, particularly the very short life-time of the excited states due to collisions between electrons in the conduction band and those in the lower filled bands. No quantitative theoretical estimates have been made of these life-times, and we have no idea how they vary from one compound to another. Also the width of the X-ray levels due to Auger effect may be important. The examples of the Kronig fine structure of $Cu_2O$ and CuO and ZnO shown in figs. 12, 13 and 14 illustrate how, for instance, the sharpness of minima in the absorption can vary from one substance to another.

Fig. 2.



Density of states and absorption coefficient at the beginning of a band in a non-metal.

Fig. 3.



(*a*) Expected type of absorption for non-metal.
(*b*) Metal.
(*c*) Effect of finite breadth of lines.

The position in metals from the theoretical point of view is quite different. It is characteristic that a positive charge in a metal is screened by the surrounding electrons, so that the potential energy of an electron in its field is no longer to be represented by $-e^2/r$ but by

$$V(r) = -e^2/r \exp(-qr), \quad \ldots\ldots \quad (1)$$

where $q^{-1}$ is of the order 1 to 2A. This principle is used by Mott and Jones (1936) to calculate the additional resistance due to additions of Zn etc. to Cu, and other applications will be mentioned below. In considering the X-ray absorption edge of metals, then, the field in which the electron moves after the absorption process consists of the lattice field with in addition the field derived from the potential (1). Now for an electron moving in a field of the type (1), there will be at most a *finite* number of stationary states, and if $q$ is large enough, no bound stationary states at all.

One of us (Mott 1949) has recently given reasons for thinking that, in general, in metals $q$ *is* so large that no stationary states exist. The reason is that it is characteristic of a metal that the electrons are free, and thus that the electrons and positive ions do not combine to form pairs from which the electron is incapable of moving. This being the case, one would not expect exciton lines at the absorption edges of metals; also the transition probability $p(E)$ would not tend to infinity at the bottom of a band. Thus the absorption spectrum of a metal in the Kronig region may be expected to be much less marked at the beginning of each band, following more truly the N(E) curve (*cf.* fig. 3 (curve (b)). Though here probably too, the short life-time of the excited states will make everything much less distinct, so that the curve in fact looks like fig. 3 curve (*c*).

Although one does not in general expect exciton lines for metals, it is probable that arsenic, antimony and bismuth may be exceptions. These (*cf.* Mott and Jones 1936) have a very small number of free electrons and positive holes, which are prevented from forming pairs only because of their very low effective mass (Mott 1949). This exceptional state of affairs will be broken down as soon as a vacancy is formed in the X-ray level. We return to the consideration of this point in § 3.

## § 2.

We shall now discuss the experimental material with a view to seeing what evidence there is for the existence of exciton levels. One of us in a recent note (Cauchois 1949 a), has emphasized the frequent occurrence of a "white line," (French raie blanche, denoted in what follows by R.B.), or maximum of the absorption coefficient, immediately on the high frequency side of the K and L absorption edges of mineral compounds, and its absence in most metals. In this section we shall discuss certain cases in which this white line can probably be interpreted as due to the formation of exciton levels in the field of the positive charge left by the ejected electron.

An R.B. *not* due to an exciton level can only arise if there are electronic states of the required symmetry with a very high density of levels at the absorption edge. The best known case is that of the transition metals, where, owing to the small radius and consequent small overlap of the *d*-orbitals, there is a high density of empty states of predominantly *d* symmetry. Consequently an R.B. appears in the $L_2$ and $L_3$ absorption edges of elements such as W (Veldkamp 1935), and in the $L_3$ of Pt (Cauchois and Manescu 1940, for an explanation of its absence in the $L_2$ edge, *cf.* Mott 1949).

A similar explanation, due originally to Coster and Kiestra (1948) can account for the R.B. obtained at the metal K edge of many metallic oxides. According to these authors the empty *p*-band (of symmetry *p* in the neighbourhood of the metal ion) becomes narrow and with wave functions less and less hybridized as more oxygen is added and the metal ions move further from each other. This is shown in figs. 12, 13 and 14, which illustrate results obtained in the Paris laboratory for ZnO, CuO and $Cu_2O$. Zinc oxide shows a very marked R.B., as does also CuO ; the far more covalent $Cu_2O$, in which the copper ions are closer together, does not show it. Similar results have been obtained for the oxides of nickel (Cauchois and Manescu 1949, to appear in the *Journ. de Chimie Phys.* ; see also Cauchois 1949 b).

Turning now to cases where the R.B. observed is probably due to an exciton, we have first the K-absorption of arsenic shown in fig. 8 obtained by Hulubei and Cauchois (1940). As already stated, it seems to us unlikely that there should be any particularly high density of *p*-states at the surface of the Fermi distribution in this metal ; and probably exciton formation occurs. For the elements of similar structure, no R.B. has been observed for the K-absorption of Sb, perhaps on account of insufficient resolution, but Sandström (1935) found one for the $L_1$ edge ; bismuth shows one for $L_1$ (Cauchois 1942). These metals are discussed further in § 3.

Gallium shows a very marked R.B. in both the solid and liquid states (Hulubei and Cauchois 1940, M. Vidal 1949) (see also fig. 6). Without attempting a detailed explanation, for the behaviour of the liquid, we imagine that the situation is somewhat similar to that for arsenic (*cf.* § 3).

As expected, there is no R.B. in the K-absorption of metals such as Cr, Mn, Fe, Co, Ni, Cu (*cf.* fig. 9 for Ni) ; the case of zinc is doubtful ; some minima have been observed by one of us (Y.C.) which seem to disappear in zinc of the highest purity. In tin no R.B. has been observed in the K-absorption, though Sandström finds one for $L_1$ ; the difference may be due to insufficient resolving power for the K-absorption.

A very interesting example of an R.B. at an absorption edge which is almost certainly an exciton level is found in the K-absorption of arsenic in NiAs (fig. 10). Here, unlike the case for NiO, there is no R.B. at the metal edge (fig. 11), or, at any rate, no minimum of importance in comparison with the usual fluctuations of intensity. Without giving a detailed explanation, it appears likely enough that the highly covalent nature of this compound results in heavy coupling between the *vacant* *p*-orbitals of Ni, thus preventing a high density of vacant *p* states and so an R.B. at the Ni K-edge. In any case the absence of an R.B. for Ni shows that there is no dense band present *until* the arsenic K-level has lost its electron ; in other words, we have to do here with an exciton level.

It is interesting that if one compares the R.B. for As in NiAs with that for As in the elementary form, one finds that the former is probably very slightly displaced towards low frequencies. For a compound one usually obtains a displacement towards large frequencies. This perhaps suggests that we have to do with excitons in NiAs at any rate. The nickel discontinuity on the other hand is displaced, relative to that of the metal, in the other direction*.

A recent observation by one of us (Y.C.) of the rubidium K-edge of RbCl is illustrated in fig. 7. This shows a very sharp R.B. of the type expected. Finally, we show in fig. 4 the fine structure of the absorption of natural NiS (Millerite). This non-metal shows, in contrast to Ni, an absorption very much of the type anticipated for insulators (fig. 3) with sharp increases in the absorption coefficient and relatively gentle decreases. It does not seem true, however, that this is a general feature of non-metals.

## § 3. THE ABSORPTION SPECTRA OF METALS SUCH AS ARSENIC AND GALLIUM.

These are of particular interest. There is no reason to believe that the strong R.B. observed could be due to any high density of *p* states existing before the K level was ionized. On the other hand it seems impossible that an exciton level of the type described above could be formed, because there is no zone of forbidden energies where it could be placed.

We believe that the proper description of the process is somewhat as follows : the metals discussed are those in which the overlap between the full band and the empty band is very small ; they are only just metals. Consequently the wave-length of the conduction electrons is very large, many multiples of the lattice constant. Consider then the wave-function of the *p*-electron that has been ejected from the K-level of an arsenic atom. The wave-function, we may suppose, is not too different from that of atomic arsenic in the same state of ionization. But in the metal the energy of the electron must be considered as at the surface of the Fermi distribution ; therefore the wave-function must join up, at the boundary of the atomic polygon surrounding the arsenic atom considered, with the wave-functions of the conduction electrons. These, as already seen, have very long wave-length. A phenomenon therefore occurs familiar in the theory of scattering of slow neutrons ; a wave of long wave-length has to be fitted to a "potential hole" in which there is a virtual quantized energy level at zero energy. Under these conditions, as is well known, a kind of resonance occurs and the amplitude of the wave-function within the "hole" becomes large compared with that of the

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* One takes here for the metal the *first* of the two discontinuities, $K_1$ of fig. 9, in agreement with the usual interpretation.

waves of long wave-length outside. Details of this will be found in Mott and Massey 1949. This, then, is the mathematical description of the cause of the R.B., since the transition probability $p(\mathrm{E})$ is determined by the amplitude of the wave-function within the "hole." Physically therefore, we may say that when a substance is only just metallic, it is possible for it to behave as though it were a non-metal and give an exciton type of absorption characteristic rather of the free atom than of the solid.

It is hoped to publish a more detailed mathematical investigation of this phenomenon in the near future.

## § 4. DETAILS OF EXPERIMENTAL MATERIAL.

The absorption spectra which have been used to illustrate the theoretical part of this paper were obtained under the following conditions: the small demountable X-ray tube in the laboratory of Physical Chemistry of the University of Paris can be used up to 50 kV. Most of the absorption spectra were, however, obtained at a much lower voltage, less than twice the excitation potential; the reason for this is to avoid exciting radiation with a frequency two or three times that of the edge under consideration, which could be superimposed on the actual spectrum because of reflections of a higher order. This precaution is not absolutely necessary when the second order reflection is very weak (as in the case of the (100) plane of mica).

The spectrograph consists of a curved sheet of mica, and use is made of the $(h0l)$ reflections through the sheet. The radius of curvature is 40 cm. The 100 reflection give the best contrast for the Kronig structures; but the first, second and third order reflections on $(\bar{2}\ 0\ 1)$ allow better resolution and make it possible to effect more exact measurements, especially of the actual edges, with the help of narrow K lines of well known wave-lengths.

The absorbing screen was generally placed between the crystal and the photographic plate in such a way as only to receive the scattered radiation. In these conditions the photochemical action of the X-rays during the exposure remains very weak. If, on the other hand, one places the screen between the window of the tube and the crystal in the intense incident ray, the chemical change may be important. Further, the fluorescent spectrum is superimposed on the absorption spectrum and could be a nuisance.

The materials for which the absorption was measured were for nickel rolled sheet 0·1 mm. in thickness; for other substances, a homogeneous layer of powder spread out on greasy cigarette paper or Scotch tape. The mass per unit area of each element of which the absorption was obtained was, as far as possible, the same for each case of different chemical composition. The photographs were taken on Ilford or Gevaert plates and examined with microphotometers. The exposure times, reduced to a 5 milliamperes run, varied from 20 minutes to some hours.

### *Details of Figures 4 to 13 and of the Table.*

All the microphotometer curves show the K absorption of the element indicated, the frequency increasing from left to right. We have shown in the accompanying table the wave-lengths of some typical absorption curves (Ni, As and Rb). We have added, too, some measurements made on metallic arsenic in 1940 with a resolving power greater than that used in 1949. These measurements give a more complete description of the structure up to 50 eV. from the absorption edge, but the structure for higher energies was not investigated. The strong absorption of arsenic in NiAs made the use of a higher resolving power difficult.

The white lines (maxima in the absorption) marked with S are due to multiple reflections in the analysing crystal; they thus give a measure of its resolving power.

All the curves reproduced here are obtained with the (100) reflection of a curved mica sheet of radius 40 cm.

In the table, maxima are printed in bold-faced type.

Nickel metal (fig. 9).

| | X.U. | eV. |
|---|---|---|
| $K_1$ .... | $1485\cdot0_{9}$ | $0$ |
| $K_2$ .... | $1483\cdot1_{7}$ | $10\cdot_{8}$ |
| 1st min. | $1482\cdot0_{5}$ | $17\cdot_{6}$ |
| $\alpha$ ...... | $\mathbf{1478\cdot_{9}}$ | $\mathbf{35\cdot_{2}}$ |
| A ..... | $1476\cdot_{0}$ | 51 |
| $\beta$ ...... | $\mathbf{1474\cdot_{6}}$ | **60** |
| | $1473\cdot_{8}$ | 64 |
| | $\mathbf{1472\cdot_{5}}$ | **72** |
| B...... | $1467\cdot8$ | 97 |
| $\gamma$ ...... | $\mathbf{1463\cdot_{1}}$ | **125** |
| | $1461\cdot_{6}$ | 134 |
| | $\mathbf{1459\cdot_{0}}$ | **149** |
| C...... | $1455\cdot_{8}$ | 168 |
| $\delta$ ...... | $\mathbf{1451\cdot_{7}}$ | **191** |
| D .... | $1448\cdot_{0}$ | 213 |
| $\epsilon$ ...... | $\mathbf{1445\cdot_{0}}$ | **230** |
| E .... | $1441\cdot_{3}$ | 252 |
| $\eta$ ...... | $\mathbf{1439\cdot_{0}}$ | **267** |

Nickel in NiAs (fig. 11).

| | X.U. | eV. |
|---|---|---|
| K .... | $1484\cdot0_{2}$ | 0 |
| 1st min. | $1481\cdot_{9}$ | $12\cdot_{2}$ |
| $\alpha$...... | $\mathbf{1479\cdot_{7}}$ | $\mathbf{24\cdot_{4}}$ |
| A ..... | $1476\cdot_{7}$ | 42 |
| $\beta$...... | $\mathbf{1473\cdot_{0}}$ | **62** |
| B .... | $1468\cdot_{5}$ | 88 |
| $\gamma$...... | $\mathbf{1464\cdot_{2}}$ | **114** |
| C .... | $1456\cdot_{4}$ | 158 |
| $\delta$...... | $\mathbf{1451\cdot_{3}}$ | **188** |

Arsenic in NiAs (fig. 10).

| | X.U. | eV. |
|---|---|---|
| K edge ...... | $1043\cdot0_{4}$ | 0 |
| Bottom of R.B. | $1041\cdot_{8}$ | $13\cdot_{5}$ |
| Max. ........ | $\mathbf{1040\cdot_{2}}$ | $\mathbf{31\cdot_{2}}$ |
| Min. ........ | $1039\cdot_{2}$ | $43\cdot_{3}$ |
| $\alpha$ ............ | $\mathbf{1037\cdot_{5}}$ | **62** |
| A............ | $1035\cdot_{3}$ | 88 |
| $\beta$ ............ | $\mathbf{1032\cdot_{9}}$ | **115** |
| | $\mathbf{1031\cdot_{0}}$ | **138** |
| B............ | $1029\cdot_{3}$ | 157 |
| $\gamma$ ............ | $\mathbf{1026\cdot_{9}}$ | **186** |
| C............ | $1023\cdot_{4}$ | 226 |
| $\delta$ ............ | $\mathbf{1021\cdot_{2}}$ | **252** |
| | to $\mathbf{1019\cdot_{3}}$ | **275** |

Arsenic metal (fig. 8).

| | X.U. | eV. |
|---|---|---|
| K edge ...... | $1042\cdot8_{6}$ | 0 |
| Bottom of R.B. | $1041\cdot_{2}$ | 19· |
| $\alpha$ ............ | $\mathbf{1038\cdot_{8}}$ | **46** |
| A............ | $1035\cdot_{5}$ | 84 |
| $\beta$ ............ | $\mathbf{1033\cdot_{7}}$ | **106** |
| | $\mathbf{1030\cdot_{9}}$ | **138** |
| B............ | $1028\cdot_{1}$ | 171 |
| $\gamma$ ............ | $\mathbf{1026\cdot_{3}}$ | **191** |
| C............ | $1024\cdot_{5}$ | 213 |
| $\delta$ ............ | $\mathbf{1022\cdot_{6}}$ | **234** |
| | to $\mathbf{1018\cdot_{0}}$ | **290** |

Measurements by Hulubei and Cauchois in 1940, with a greater resolving power than that used here, showed a fine structure of the R.B. of arsenic metal, as shown below.

| Arsenic metal | X.U. | eV. |
|---|---|---|
| K edge | $1042{\cdot}8_6$ | 0 |
| R.B. | $1042{\cdot}4_1$ | $5{\cdot}_0$ |
| dark line | **$1041{\cdot}8_7$** | **$11{\cdot}_2$** |
| minimum | $1041{\cdot}_2$ | $18{\cdot}_8$ |
| dark line | **$1040{\cdot}_6$** | **$25{\cdot}_5$** |
| minimum | $1039{\cdot}_7$ | 36 |

NOTE.—The difference between the edges of Ni in NiAs and nickel metal is +6·0 eV.; between those of As in NiAs and arsenic metal is −2·0 eV.

Rubidium in RbCl (fig. 7).

| | X.U. | eV. |
|---|---|---|
| K edge | $813{\cdot}9_4$ | 0 |
| minimum | $813{\cdot}_6$ | $5{\cdot}_4$ |
| maximum | **$813{\cdot}_1$** | **$14{\cdot}_9$** |

## ACKNOWLEDGMENTS.

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# CXVII. *Notices of New Books and Periodicals received.*

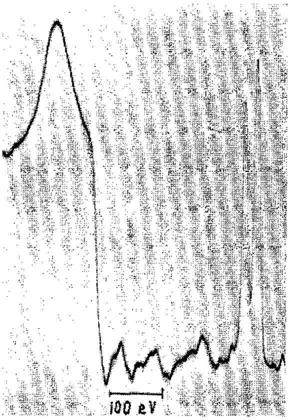
*A Textbook on Heat.* By J. H. AWBERY. [Pp. 298.] (London : Longmans, 1949.) Price 15*s.*

IN this refreshingly original treatment the author sets out to emphasize general principles rather than the detailed behaviour of particular substances. The first nine chapters are an excellent and a clear exposition of the underlying physical principles. The usefulness of the book as a whole, however, is somewhat impaired by a very variable level of treatment, ranging from a chapter on thermal expansion at about Higher School Certificate level to a chapter on heat conduction, introducing complex variable methods.

This lack of balance will not prevent the book becoming a " recommended text," but may cause it to appear in the section for " supplementary reading."

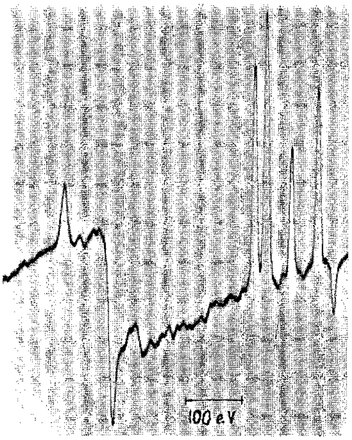
N. T.

FIG. 4.



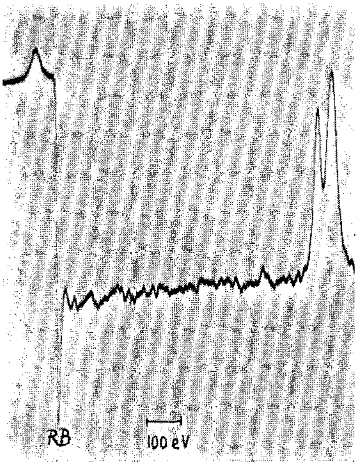
Nickel in NiS (millerite).

FIG. 5.



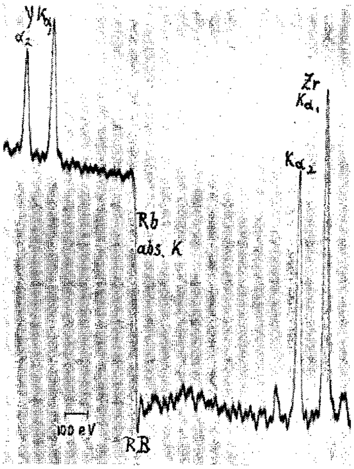
Nickel in $Ni_2$ $O_3$.

FIG. 6.



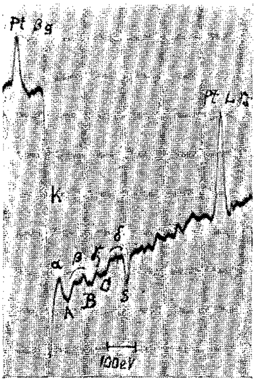
Metallic gallium (solid) at a temperature near melting point.

FIG. 7.



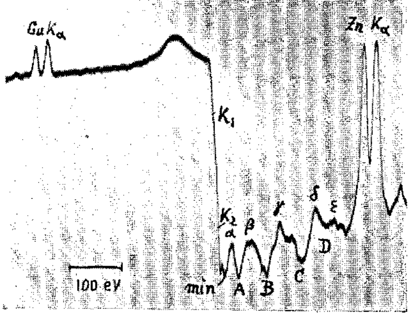
Rb in RbCl.

FIG. 8.



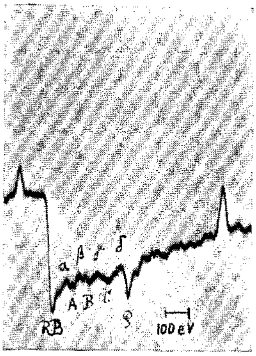
Metallic arsenic.

FIG. 9.



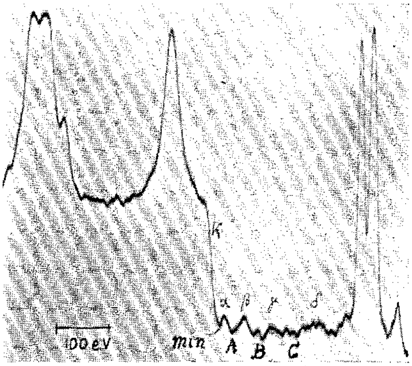
Metallic nickel.

Fig. 10.



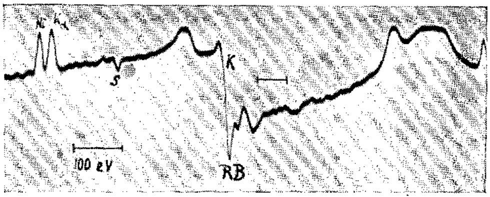
As in NiAs.

Fig. 11.



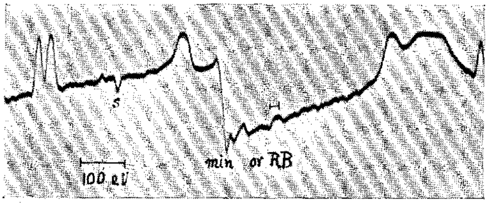
Nickel in NiAs.

Fig. 12.



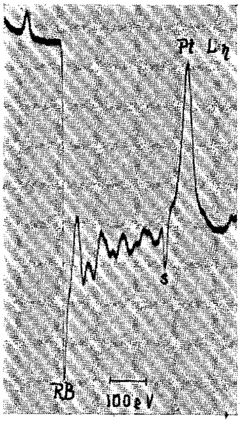
Copper in CuO.

Fig. 13.



Copper in $Cu_2O$.

Fig. 14.



Zinc in ZnO.