The L Radiation from Sulphur

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The L-emission band from a sulphur target has been studied spectrophotometrically, and the intensity contour of the band has been deduced. The essential features consist of a strong peak at 149 ev, less pronounced structures at about 158 ev, and a broad peak or "tail" around 136 ev, in substantial agreement with previous studies. In contrast with the corresponding bands from the metallic elements of the second row of the periodic table, the drop in intensity on the high energy side of the band occurs gradually. Because of this fact, and perhaps also because of insufficient resolving power, the L_{II} and L_{III} edges have not been separated. A tentative interpretation has been given for the main observed features. No trace of the $L_I - L_{III}$ transition has been found.

THE L spectrum has been investigated by Prins and Takens[1] and by Siegbahn and Magnussen[2] who have carried out wave-length determinations of the characteristic features of the radiation which lies in the spectral range from about 75A to about 85A. Skinner,[3] in his report on the emission bands from certain solids, has published a curve showing the L band of sulphur. The present investigation constitutes a more refined photometric determination of the intensity distribution of the radiation which is emitted when vacancies occur in the L_{II} and L_{III} levels. Information of this type is of value in the study of the level system of the valence electrons in solids, and particular interest attaches to the present study since sulphur is a typical insulator.

EXPERIMENTAL

The general description of the experimental features dealing with the x-ray tube, vacuum spectrograph, and the photometric method have been described previously;[4] only additional techniques and observations related to the study of sulphur will be given here. Because of the high chemical affinity of sulphur towards nickel and copper under conditions of operation of the x-ray tube, the target of the tube, as well as filament leads, were made of stainless steel. This material is not attacked appreciably by sulphur and spot welds readily to the tungsten filament. Yellow crystalline sulphur of high purity was fused in vacuum into an open-ended Pyrex ampule, which was then inserted into the x-ray tube. The sulphur was evaporated by external heating from a tungsten filament placed near the glass container, which was shielded from the heat of the x-ray filament to prevent continuous evaporation during operation of the tube.

Because of the volatile nature and low melting point of sulphur, it was necessary to keep the target cold ($-45°C$) during bombardment. This was accomplished by circulating alcohol whose temperature had been lowered by the use of solid carbon dioxide. This method of cooling was found to be quite adequate in retaining a deposit of sulphur on the target face for the duration between renewals of the sulphur coating. Indeed, for the particular thickness of target wall ($\frac{1}{32}''$), and size of focal spot (1.2 sq. cm), and a power input of 50 watts, this method of cooling was found to be more efficient than direct liquid-air cooling of the same target for the same power input. (Liquid air boils behind the focal spot, so its own vapor forms a thermally insulating layer. The use by Skinner[5] of a very thick-walled target was intended to mitigate this boiling. That it did so is, however, questionable, since calculation shows that there is no significant fringing of the lines of heat flow in a target wall whose thickness is comparable with the width of the focal spot.)

[1] J. A. Prins and A. J. Takens, Zeits. f. Physik **77**, 795 (1932).
[2] M. Siegbahn and T. Magnussen, Zeits. f. Physik **87**, 291 (1934).
[3] H. W. B. Skinner, Phil. Trans. Roy. Soc. **A239**, 95 (1940).
[4] W. M. Cady and D. H. Tomboulian, Phys. Rev. **59**, 381 (1941).
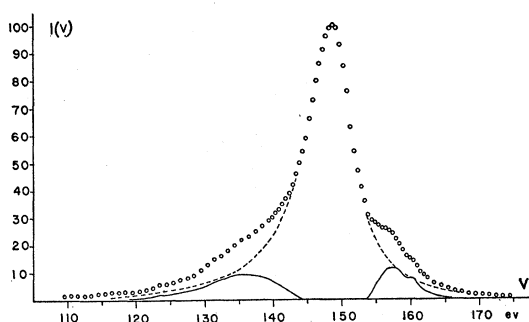[5] Reference 3, p. 101.

FIG. 1. Relative intensity I(V) in the L band of sulphur. The circles represent experimental determinations.

Five runs were made with varying exposure times (80–320 min.) and tube currents (10–20 ma) at potentials of about 2000 volts. A ballast resistance of 15,000 ohms was placed in series with the target of the tube in order to increase the stability of operation. The pressures in the x-ray tube were higher than those attained while working with aluminum and magnesium.[4]

It is difficult to know whether the deposit of sulphur on the target was orthorhombic. The appearance of the focal spot did vary from run to run both in color and texture. For tube currents of 10 ma the deposit retained a yellowish-white color. For higher currents the focal spot took on a light brownish color and a glossy appearance; upon removal of the target the deposit of sulphur was gummy in nature in these cases. However, upon standing for several hours it seemed to revert to the hard yellow form. A study of sulphur allotropes by Das[6] indicates that the gummy deposit of sulphur, often regarded as amorphous, is really crystalline and gives diffraction patterns identical with those obtained from orthorhombic sulphur. We presume that our spectra are characteristic of this modification. The loss of sulphur by evaporation from the target was found to depend strongly on the tube current. However, for currents equal to, or less than, 20 ma an exploratory run indicated that this loss was not excessive since the x-radiation did not markedly diminish during the first twenty minutes of bombardment after removing the deposit of sulphur.

[6] S. R. Das, Ind. J. Phys. **12**, 163 (1939).

RESULTS AND DISCUSSION

The points in Fig. 1 represent a plot of the data showing the intensity distribution of the radiation. The data were prepared by averaging the results of four plates representative of strong and weak exposures as well as high and low tube currents. In obtaining the average, the intensities on the high and low energy side of the main peak were weighted more heavily in the case of dense negatives; while in the case of fainter negatives the intensities near the peak were assigned greater weight. In reducing the plates the wave-length corresponding to the peak was set equal to 83.1A, the value given by Skinner,[7] and in determining the photon energies given in Fig. 1, 12,370 volt-angstroms was used as the conversion factor. Individual densitometer traces from the different runs were in good agreement in regard to the main features of the band. The plates were examined for impurity spectra due to carbon, nickel, chromium, iron and mercury. There was no indication of the presence of these impurities. On the most intense exposure, no trace of the L_I–L_{III} radiation could be found. This line is predicted[8] to be at 203A and would have been detected if its intensity were of the order of 3 percent of that of the main peak.

Comparison with Skinner's curve[7] on the same element indicates good agreement with respect to the wave-length. However, the present results differ widely in intensity from those of Skinner. If, as we believe, our band shape is correct, this discrepancy may perhaps be ascribed to a failure in the adjustment of the photometer, the deflections of which were assumed by Skinner[9] to be approximately proportional to the intensity of the x-radiation causing the blackening on the plate.

The interpretation of the observed band shape is hardly a task for the experimentalist, but we hazard a guess. For this purpose it is convenient to extend the contour of the main peak smoothly to the base line as indicated by the dotted lines in Fig. 1. It is then possible to sketch in the shapes of two component features with maxima at about 136 and 158 ev. If the valence electrons

[7] Reference 3, p. 108.
[8] D. H. Tomboulian and W. M. Cady, Phys. Rev. **59**, 422 (1941).
[9] Reference 3, p. 106.

of sulphur can be classified crudely as belonging to $3s$ and $3p$ bands, then the L-emission spectrum should divide itself into two parts corresponding to the electron transitions

$$3s \rightarrow L_{II, III}$$
$$3p \rightarrow L_{II, III}.$$

The main contribution to the band (peak at 149 ev) is to be ascribed to the first of these; while the structure at 158 ev may perhaps be associated with the second transition which should be relatively weak since it is nearly forbidden. The $3s$-$3p$ interval (9 ev) as deduced by this procedure is not in contradiction with the value estimated from x-ray and optical data.

The broad peak at 136 ev may perhaps arise from a "semi-Auger" process[4] in which a $3s$ electron falls into the ionized L shell and a second $3s$ electron is excited into an unoccupied level. In this process a photon is emitted of which the energy is less than the energy loss of the first electron. Such a process would demand that the interval between the main peak and the broad peak be larger than that $3s$-$3p$ interval, and indeed such is the case (13 vs. 9 ev).

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The Half-Life of Iodine (128)

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The half-life of iodine (128) has been measured with high accuracy by means of a G-M counter and amplifying circuit of tested reliability. An absolute calibration of the counter was carried out by a simple method. The half-life was determined from the data by an arithmetical, rather than a graphical method. The results of four closely concordant experiments give for the best value of the half-life, $T = 24.99 \pm 0.02$ minutes. It is suggested that this radio-element can now be used in a quick and convenient calibration of counters or other instruments for measurement of radioactivity.

IN some current experimental work by the writers it has become necessary to know the half-life of iodine (128) with greater accuracy than is characteristic of the values now in the literature. Although this radio-element was among the first discovered by Fermi in his fundamental research on the production of artificial radioactivity by neutron bombardment, and it has been used and studied by researchers too numerous to list here, yet its half-life has not been determined with an accuracy much better than 5 percent. Probably the best value is that given by Livingood and Seaborg,[1] namely, 25 ± 1 min. Of course, this situation is not unique with radio-iodine. The half-lives of very few radio-elements are known with an accuracy better than a few percent. Notable exceptions are the values for radon, determined independently by investigators in two laboratories with excellent agreement;[2,3] for phosphorus[4] (32), and for carbon[5] (11).

Since there is available in this laboratory a G-M counter and amplifier with a known reliability of about 0.2 percent in relative measurements, an attempt has been made to determine the half-life of radio-iodine with an accuracy of this order. The radio-iodine was prepared by slow neutron bombardment of phenyl iodide, concen-

* Now with the duPont Company, Wilmington, Delaware.

[1] J. J. Livingood and G. T. Seaborg, Phys. Rev. 54, 775 (1938).

[2] W. Bothe, Zeits. f. Physik 16, 266 (1923).
[3] I. Curie and C. Chamié, J. de phys. et rad. [6] 5, 238 (1924).
[4] N. B. Cacciapuoti, Nuovo Cimento 15, 213 (1938).
[5] A. K. Solomon, Phys. Rev. 60, 279 (1941).