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THE K SPECTRA OF ELEMENT 61

by

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ABSTRACT

The K lines of the x-ray spectra of element 61 were obtained on a transmission type spectrograph. The wavelength of the lines and the experimental method is discussed.

INTRODUCTION

X-ray spectra have been widely used in establishing positive identification of the elements from the time of Moseley's first investigations. It is known that x-ray spectra are simple and that wavelengths can be readily calculated from the Moseley law.

Previous workers in the field of rare earth chemistry have made many unsuccessful attempts to isolate the missing rare earth element of atomic number 61. All early work was done on naturally occurring material and, at best, only traces contaminated by neighboring elements were collected.

The K lines of the x-ray spectrum of a sample of element 61 supplied by G. W. Parker and P. M. Lantz of Oak Ridge National Laboratory have been recorded and the $K\alpha_2$, $K\alpha_1$, $K\beta_1$, and $K\beta_2$ lines appear in their appropriate places in the simple x-ray spectra. The photographs shown herein leave no doubt as to the identity of the material.

SPECTROGRAPH

A transmission type x-ray spectrograph was used. The instrument is indicated schematically in Figure 1.

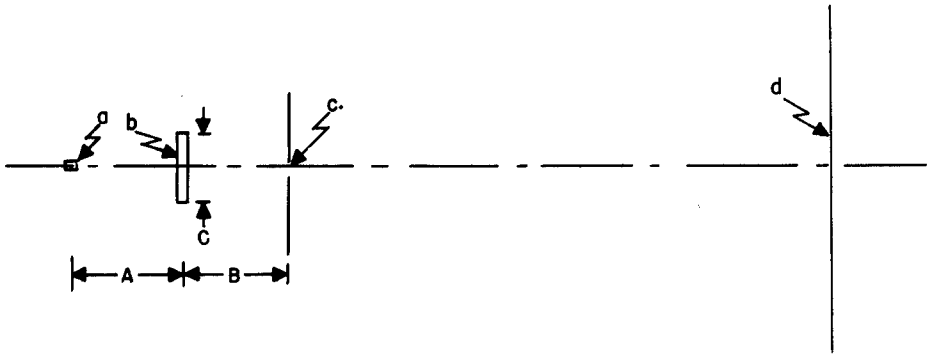


Figure 1. (a) Source of x-rays; (b) calcite crystal; (c) slit; (d) film plane.

X-rays of all wavelengths are incident on the crystal face from 0° to (arc tan $\frac{C}{2A}$)°. The rays which satisfy Bragg's law $n\lambda = 2d \sin \theta$ undergo constructive interference. If the distance A equals distance B, the diffracted radiation passes through the slit c and is recorded on the film at d. A complete K spectra of the material on the target is recorded on the film on both sides of the center line passing through the target and crystal. A calcite crystal 15 x 30 x 2.5 mm was used as a diffracting medium. The crystal was located with the 15 x 30 mm face parallel to the film and equidistant from the target and the slit. The 15 mm edge was fixed in a vertical position. The crystal was cut with the (100) planes (Rhombohedral System) normal to the 15 x 30 mm edge and parallel to the 15 mm edge. In this type spectrograph the crystal is not oscillated.

A demountable target x-ray tube with a body made of 4-inch Pyrex pipe fittings was used. The anode was made of copper, with the face cut at a 20° angle to the axis through the center of the crystal and the film of the spectrograph; a recess 1/8-inch wide, 1/16-inch deep and 1/2-inch long was cut in the target face to hold the sample. The filament assembly consisted of a tungsten helix mounted in a copper shield profiled to give a line focus of electrons on the anode. During operation, the tube was pumped continuously with a 6-inch oil diffusion pump and a 1-1/2 hp Kinney pump. Normal tube operating pressure was from 2×10^{-5} to 5×10^{-6} mm of mercury. A power supply capable of delivering 130 Kv, center grounded was used. High voltage was measured with two calibrated 2 ma meters and series resistors in the high voltage direct current circuit. The meters were connected so as to measure filament-to-ground and anode-to-ground voltages. Tube emission current was measured with a 7.5 ma meter connected in the negative high voltage circuit.

EXPERIMENTAL METHOD

The sample presumed to be element 61, was loaded on the target by evaporating a solution of the chloride salt into the 1/8- x 1/2-inch recess milled in the target face. Heat was supplied with an infrared lamp. Drying of the sample was completed by baking the target in a vacuum desiccator. When completly dry, the target was heated with a torch to fuse the sample to the target face.

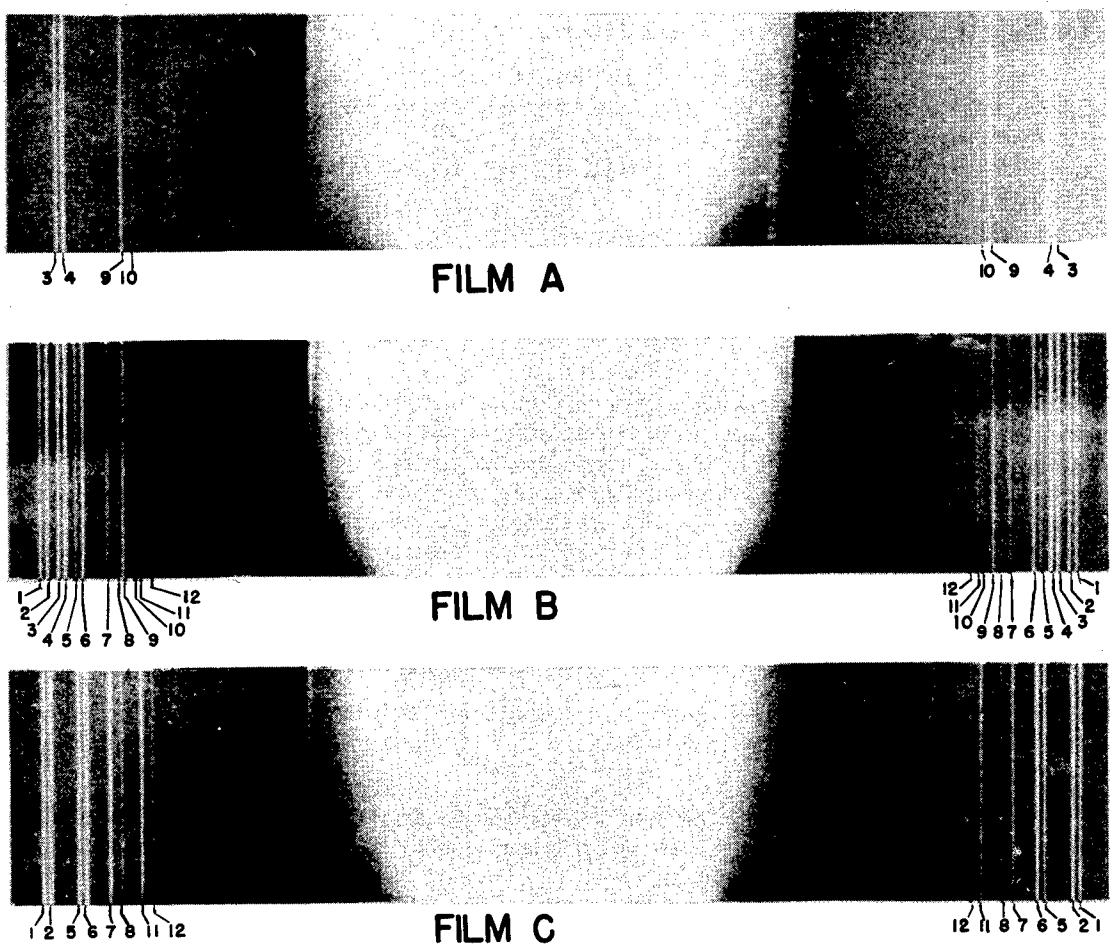
After a spectrogram of pure element 61 was recorded, 1.3 mg of Nd_2O_3 and 1.3 mg of Sm_2O_3 were added to the target, using the same general method of target-loading to provide reference lines, which is shown in Film B. The vacuum drying and fusing steps were omitted.

Table 1.

Film	Exposure time, ma hours	Voltage, kv
A	6.5	70
B	11.5	70
C	5.0	80

A densitometer trace was made of Film B on a Leeds and Northrup microphotometer using 2 mm per minute film travel and 2-inch per minute chart speed. Both film and chart on the microphotometer are driven by synchronous motors. The distance on the densitometer chart from a line on one side of center to the corresponding line on the other side of center which is proportional to the wavelength of the line was measured with an accuracy of 1 part in 9000.

In the calculations, 1/2 of the distance between corresponding peaks divided by the distance from the slit to the film plane is equal to tangent θ. From this measurement, sine θ was determined and the wavelength then calculated by means of Bragg's law. The distance from the slit to the film plane was measured with an inside micrometer and converted to the same units of distance as the microphotometer chart. Due to the paper wrapping which was used to protect the film from exposure to visible



LINE IDENTIFICATION

1 ND $K\alpha 2$	7 ND $K\beta 1$
2 ND $K\alpha 1$	8 ND $K\beta 2$
3 61 $K\alpha 2$	9 61 $K\beta 1$
4 61 $K\alpha 1$	10 61 $K\beta 2$
5 SM $K\alpha 2$	11 SM $K\beta 1$
6 SM $K\alpha 1$	12 SM $K\beta 2$

Figure 2. K series x-ray spectra. Film A, element 61; Film B, neodymium, element 61, samarium; Film C, neodymium, samarium.

light, the possible error in this measurement is 1 part in 3500.

It was originally intended to use lines of neodymium and samarium as standards of wavelength and to determine the wavelengths of lines of element 61 by interpolation between these standards.

It was observed, however, that there is a discrepancy between the wavelengths of neodymium and samarium as measured in this laboratory and those reported in the International Critical Tables.[1]

	Wavelengths XU	
Line	Reported in ICT	Observed
$Nd\alpha_2$	335.96	335.74
$Nd\alpha_1$	331.31	330.95
$Sm\alpha_2$	313.20	312.66
$Sm\alpha_1$	308.54	307.75

Possible errors in this work which have not been investigated are: (1) variations in film dimensions and (2) variations in the interplanar spacing of the calcite crystal which was used. The value of d was taken as 3.02904XU at 18°C in order to keep these results as consistent as possible with those previously reported. Errors resulting from the change in the d of calcite are negligible, since the linear coefficient of expansion of calcite is 1.02×10^{-5} per degree centigrade.[2] No allowance has been made for film shrinkage or for the Ross effect. It does not seem possible, however, that a discrepancy as large as we have noticed can be caused by these effects alone.

DISCUSSION OF RESULTS

Photographic prints of three films are shown in Figure 2. Film A shows the K spectrum of element 61. In the original film, all four lines, α_2, α_1, β_1, and B_2, are visible and no other impurities were detected.

Film C shows the spectrum of neodymium, element 60, and samarium, element 62. The large gap between corresponding lines of these two elements clearly shows, as has been shown before, that an element of atomic number 61 should exist.

Film B shows the spectrum of element 61 after additions of neodymium and samarium. Comparison with Film C shows that the lines of element 61 fall between the lines of the reference elements as is predicted by Moseley's law.

Exposures of element 61 were made at a maximum voltage of 70 kv to eliminate the possibility of interference by second order lines. Osmium, ($K\beta_2$ wavelength = 168.75, second order wavelength 337 XU) is the longest wavelength that will interfere with any of the elements shown in Figure 2. Threshold voltage for osmium is 74 kv and, therefore, there is no possibility of interference from second order lines of the elements of higher atomic number.

Figure 3 shows one end of Film B with a densitometer trace which more clearly shows the relative position of the lines and their resolution.

Due to the very large grain size of the high speed film which was used, the β_1 and β_2 doublet lines were not resolved. Predicted wavelengths of element 61 were calculated by calculating screening constants from the Moseley law for elements, 58, 59, 60, 62, 63, and 64 and determining the value of the screening constant for element 61 graphically. Wavelengths of reference lines were taken from the International Critical Tables.[3] The measured and calculated wavelengths of the lines of element 61, together with the possible interfering lines, are shown in Table 2.

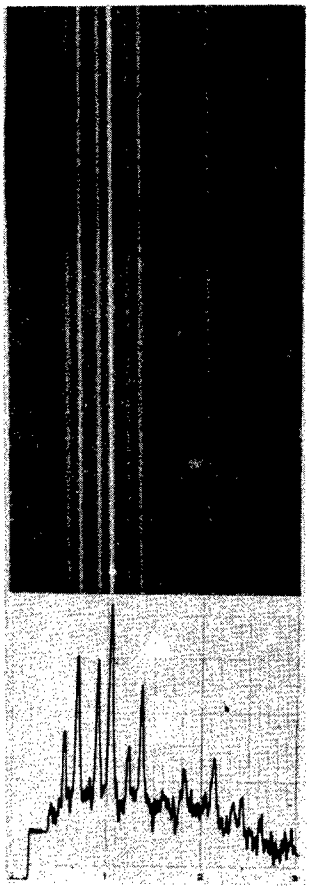


Figure 3. The lines reading left to right are Ndα_2, Ndα_1, 61α_2, 61α_1, Smα_2, Smα_1, Ndβ_1, Ndβ_2, 61β_1, 61β_2, Smβ_1, and Smβ_2.

Table 2.

Element 61	Wavelengths XU		
K spectra lines	Measured	Calculated	Possible interfering lines
α_2	323.68	324.27	La β_1 327.26
α_1	319.02	319.60	La β_2 319.66
β_1	282.00	282.99	Nd β_1 285.73 Tb α_2 282.94 Tb α_2 278.19 Dy α_2 273.64
β_2	275.03	275.33	Sm β_1 272.50

CONCLUSIONS

The K lines of the x-ray spectrum of element 61 have been recorded and their wavelengths measured and calculated wavelengths is shown in Table 1. The spectra and the controlled conditions under which they were obtained is conclusive evidence that the material is element 61. Film A in Figure 2 indicates that the sample was very pure.

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REFERENCES

1. International Critical Tables, VI, 39.
2. J. Applied Physics, 12:402 (1941).
3. International Critical Tables, VI, 39-40.

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