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The *K* Spectra of Element 61

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The $K_{\alpha 1}$, $K_{\alpha 2}$, $K_{\beta 1}$, and $K_{\beta 2}$ lines of the x-ray spectra of element 61 were obtained from 1.5 mg of sample of the chloride salt. The sample was fused on a copper target and bombarded with electrons from a 70-kv source. Wave-length measurements were made from a microphotometer tracing of the film. After one exposure of the sample, neodymium and samarium were added to the target and the spectrum of the three elements was recorded to show the position of element 61 in the periodic table.

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 wave-lengths 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 Mr. G. W. Parker and Mr. P. M. Lantz of Oak Ridge National Laboratory have been recorded and the $K_{\alpha 1}$, $K_{\alpha 2}$, $K_{\beta 1}$, 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. 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 15×30×2.5 mm was used as a diffracting medium. The crystal was located with the 15×30-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 15×30-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 $\frac{1}{8}$-inch wide, $\frac{1}{16}$-inch deep and $\frac{1}{2}$-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 $1\frac{1}{2}$-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 $\frac{1}{8}\times\frac{1}{2}$-inch recess milled in the target face. Heat was supplied with an infra-red lamp. Drying of the sample was completed by baking the target in a vacuum desiccator. When completely 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. Exposure conditions for Fig. 1 are shown in Table I.

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 wave-length of the line was measured with an accuracy of 1 part in 9000.

In the calculations, $\frac{1}{2}$ 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, $\sin\theta$ was determined and the wave-length was 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. Because of the paper wrapping which was used to protect the

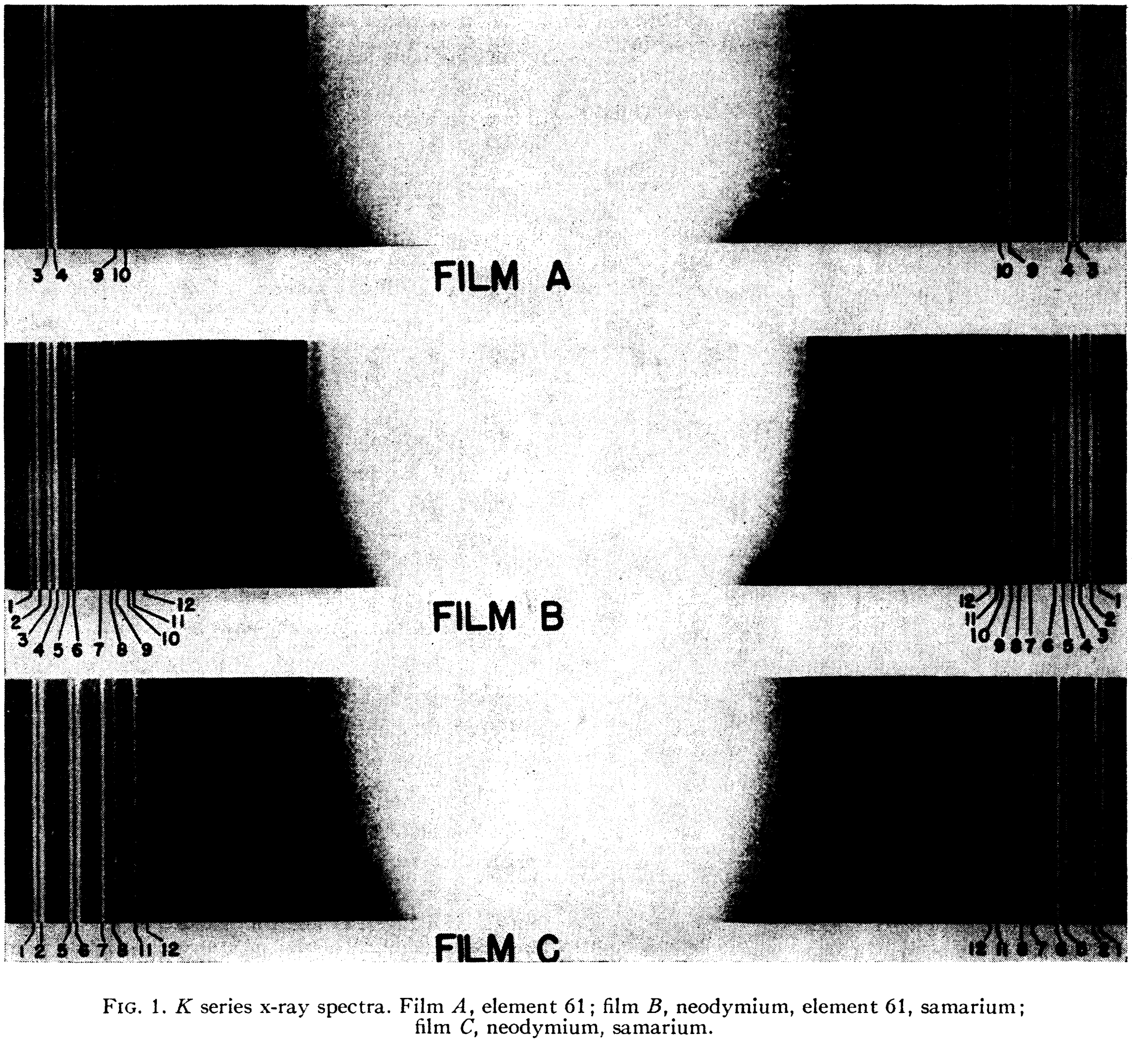

FIG. 1. *K* series x-ray spectra. Film *A*, element 61; film *B*, neodymium, element 61, samarium; film *C*, neodymium, samarium.

Line Identification

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

TABLE I.

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

film from exposure to visible 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 wave-length and to determine the wave-lengths of lines of element 61 by interpolation between these standards. It was observed, however, that there is a discrepancy between the wave-lengths of neodymium and samarium as measured in this laboratory and those reported in the *International Critical Tables*.[1] This difference is shown in Table II.

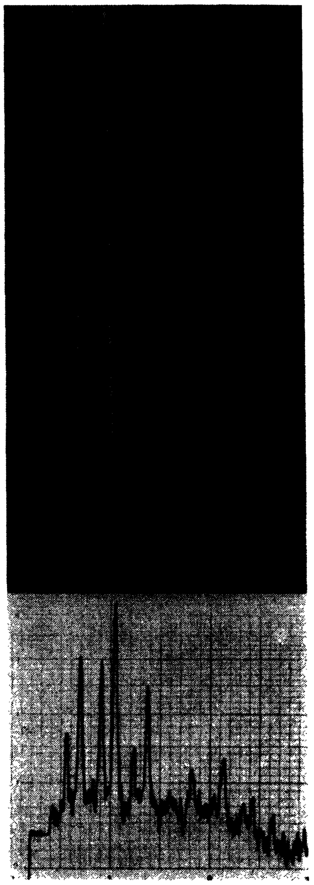

FIG. 2. In the above figures the lines reading left to right are: Nd $K\alpha 2$, Nd $K\alpha 1$, 61 $K\alpha 2$, 61 $K\alpha 1$, Sm $K\alpha 2$, Sm $K\alpha 1$, Nd $K\beta 1$, Nd $K\beta 2$, 61 $K\beta 1$, 61 $K\beta 2$, Sm $K\beta 1$, Sm $K\beta 2$.

TABLE II.

Line	Wave-lengths XU Reported in ICT	Observed
Ndα2	335.96	335.74
Ndα1	331.31	330.95
Smα2	313.20	312.66
Smα1	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 3029.04 XU 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.[3]

DISCUSSION OF RESULTS

Photographic prints of three films are shown in Fig. 1. Film *A* shows the *K* spectrum of element 61. In the original film, all four lines, K_{α_2}, K_{α_1}, K_{β_1}, and K_{β_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_{β_2} wave-length = 168.75, second order wave-length 337 XU), is the longest wave-length that will interfere with any of the elements shown in Fig. 1. Threshold voltage for osmium is 74 kv and, therefore, there is no possibility of inter-

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[1] *International Critical Tables*, Vol. VI, p. 39.

[2] J. A. Bearden, J. App. Phys. **12**, 402 (1941).

[3] C. E. K. Mees, *The Theory of the Photographic Process* (The Macmillan Company, New York, 1942), p. 907.

ference from second order lines of the elements of higher atomic number.

Figure 2 shows one end of Film *B* with a densitometer trace which more clearly shows the relative position of the lines and their resolution.

Because of the very large grain size of the high speed film which was used, the β_1 and β_3 doublet lines were not resolved. Predicted wave-lengths of element 61 were calculated by calculating screening constants from the Moseley lay for elements 58, 59, 60, 62, 63 and 64 and determining the value of the screening constant for element 61 graphically. Wave-lengths of reference lines were taken from the *International Critical Tables*.[4] The measured and calculated wave-lengths of the lines of element 61, together with the possible interfering lines, are shown in Table III.

CONCLUSIONS

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

TABLE III.

Element 61 *K* spectra lines	Wave-lengths XU		
	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_2 285.73 Tb α_2 282.94 Tb α_1 278.19
			Dy α_2 275.64
β_2	275.03	275.33	Sm β_1 272.50

The authors wish to acknowledge the assistance of C. E. Normand and A. C. Eckert for their many suggestions while the spectrograph was in its early stages of development, and to thank L. G. Lankford for the excellent machine work in construction of the spectrograph and tube components.

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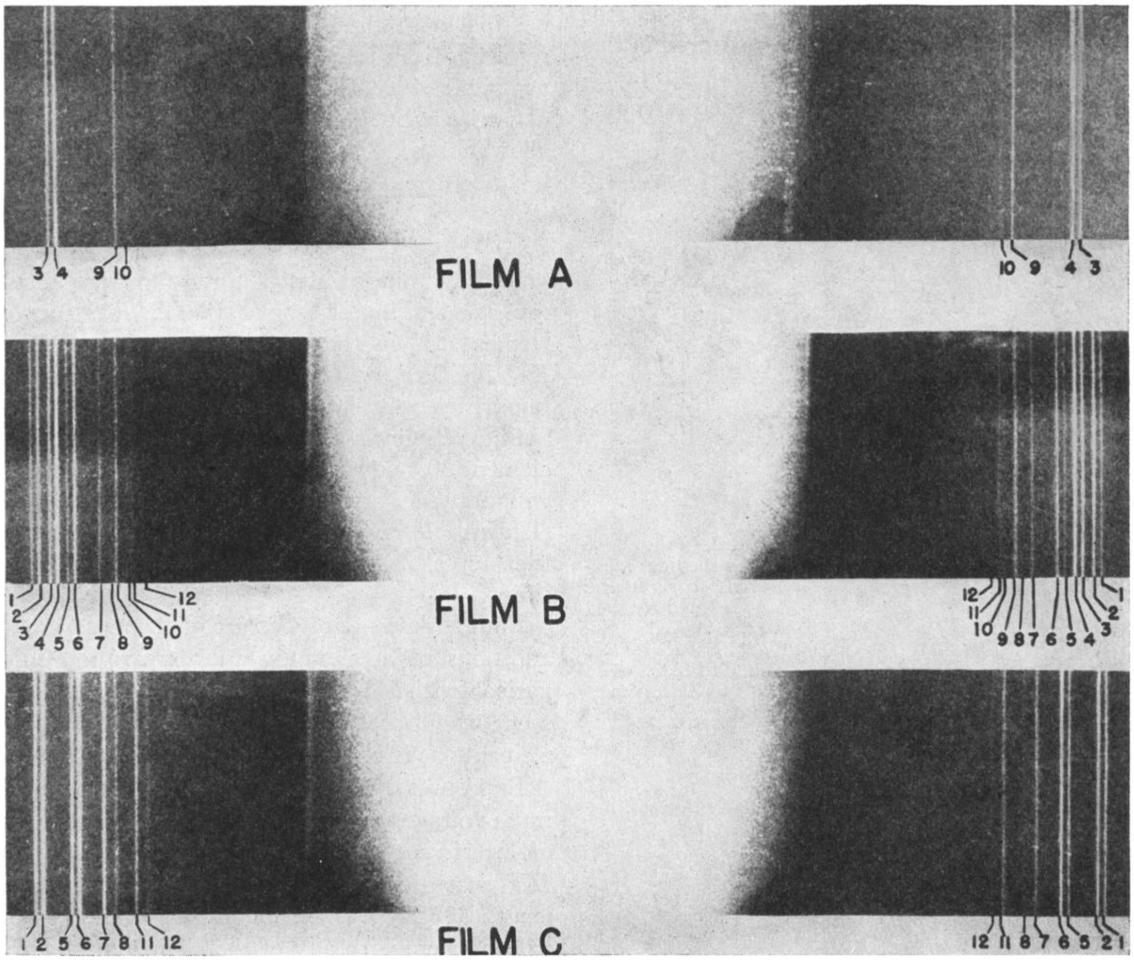
[4] *International Critical Tables*, Vol. VI, Pp. 39 and 40.



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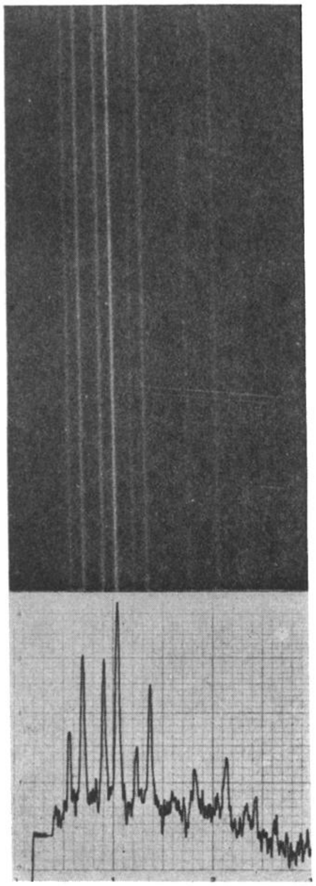

FIG. 2. In the above figures the lines reading left to right are: Nd $K\alpha 2$, Nd $K\alpha 1$, 61 $K\alpha 2$, 61 $K\alpha 1$, Sm $K\alpha 2$, Sm $K\alpha 1$, Nd $K\beta 1$, Nd $K\beta 2$, 61 $K\beta 1$, 61 $K\beta 2$, Sm $K\beta 1$, Sm $K\beta 2$.