X-Ray Spectrum of Element 43

L. E. BURKHART, W. F. PEED, AND B. G. SAUNDERS[1]
Clinton Engineer Works, Carbide and Carbon Chemicals Corporation, Oak Ridge, Tennessee
(Received October 10, 1947)

The x-ray spectrum of a sample containing Element 43 supplied by the Clinton Laboratories has been studied. Lines ascribable to $K\alpha_1$, $K\alpha_2$, $K\beta_1$, $K\beta_2$ of Element 43 were identified. Their wave-lengths followed closely the values calculated by interpolation between elements 41, 42, 44, and 45.

1. INTRODUCTION

SINCE the systematic study of the x-ray spectra of the elements by Moseley, the x-ray spectrograph has been used extensively as a research tool and especially by workers endeavoring to establish proof of new or missing elements. The striking thing about x-ray spectra is its regularity and simplicity. It will be noted in the spectra of the three elements presented in this paper that as one goes from lighter to the heavier elements, the wave-lengths of the corresponding lines decrease in a regular manner. It has long been known, therefore, that the large gap existing in the x-ray spectral region between the two elements molybdenum 42 and ruthenium 44 indicates that the K lines of missing Element 43 should fall between the K lines of molybdenum and ruthenium.

2. DISCUSSION

In the past, investigators seeking the x-ray spectra of Element 43 have been saddled with the problem of taking long exposures and finding weak lines which were assumed to arise from the presence of very small amounts of the element in unseparated compounds.

Segrè[2] states he obtained 10^{-10} g of Element 43 by cyclotron bombardment of molybdenum. He mentions that P. Abelson photographed a characteristic K line of Element 43 which was emitted through the mechanism of internal conversion.

There has been considerable work done in the search for Element 43 in minerals of the earth's crust by Noddack and Tacke,[3] Berg,[4] and others.

Despite the considerable efforts of these prior workers, it is felt that the x-ray photographs obtained by direct excitation of the sample separated by G. W. Parker of Clinton Laboratories presents the most convincing evidence of the existence and isolation of Element 43.

3. EXPERIMENTAL

Approximately 1.5 mg of sample was submitted as an ammonium salt made by dissolving the sulfide in concentrated NH_4OH and H_2O_2 and evaporating to dryness. The sample was known to contain approximately 10 percent rhenium with the chief constituent being 0.7 mg of Element 43. The problem resolved itself into determining whether or not the purified sample was 43 or some other element.

The sample was applied to the target by evaporating very small drops of the solution from a micro-pipette under an infra-red lamp. The target has been previously scored to facilitate holding the sample on the target. After the loaded target had been heated until dry, it was placed in the x-ray tube for the exposure.

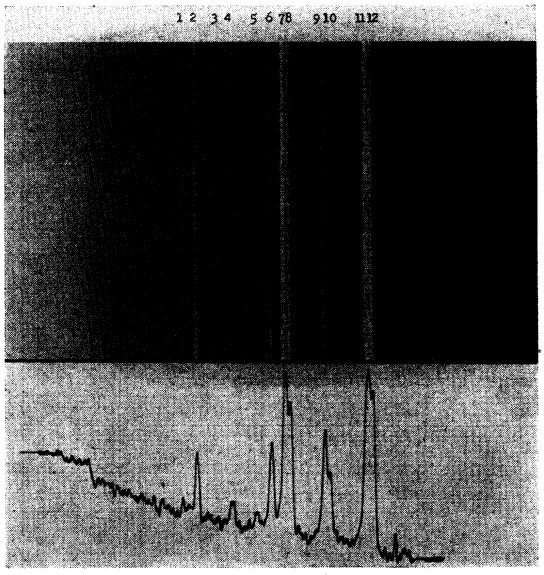
From an examination of the x-ray spectrogram, Fig. 1, it is obvious that the K lines of 43 appear between the K lines of molybdenum 42 and ruthenium 44. The separation of the densitometer peaks makes evident the resolution of the various lines. Smoother tracing was not attained because of the large grain size of the film. The calculated and observed wave-lengths of Element 43 are shown in Table I. The observed wave-length values of 43 were obtained by interpolating with a calibrated eyepiece between the wave-lengths of molybdenum, ruthenium, and the silver absorption edge listed in Siegbahn.[5]

[1] Now with the Atomic Energy Commission, Oak Ridge, Tennessee.
[2] E. Segrè, Nature **143**, 460 (1939).
[3] W. Noddack and I. Tacke, Sitz. Preuss. Akad. Wiss. **19**, 400 (1925).
[4] O. Berg, I. Tacke, Naturwiss. **13**, 571 (1925).
[5] M. Siegbahn, *The Spectroscopy of X-Rays* (Oxford, 1925).

In this region the dispersion of the spectrograph is 10.84X units/mm. The calculated values for the wave-lengths of Element 43 were obtained by plotting the screening constants of elements 41, 42, 44, 45 and interpolating for the constants for Element 43. Wave-lengths were then calculated using Moseley's formula.

In checking for the possible interference of lines from elements other than 43, Table II shows the second-order wave-lengths of lines of the elements involved in this region. The threshold excitation voltages for these elements are also listed.

The difference in voltage applied, 35 kv, and the threshold voltages for the rare earths and rhenium, as shown in Table II, exclude the possibility of any interference from these elements, had they been present in the sample. The threshold voltage given for cesium is near the applied voltage; however, this is of no concern since its interference is with the molybdenum $K_{\alpha 1}$ line and in no way affects the lines of Element 43. The voltage used in the present experiment is sufficiently high to excite the L series of rhenium, but since the L_I absorption edge of rhenium lies at 987 XU, no interfering rhenium L lines of lower wave-lengths are possible.



Line identification

	Element	Line		Element	Line
1	Ru	$K\beta_2$	7	Ru	$K\alpha_1$
2	Ru	$K\beta_1$	8	Ru	$K\alpha_2$
3	43	$K\beta_2$	9	43	$K\alpha_1$
4	43	$K\beta_1$	10	43	$K\alpha_2$
5	Mo	$K\beta_2$	11	Mo	$K\alpha_1$
6	Mo	$K\beta_1$	12	Mo	$K\alpha_2$

FIG. 1. X-ray spectrograms. Molybdenum element 42, Element 43, ruthenium element 44.

TABLE I. Observed and calculated values for x-ray lines of Element 43.

Element	Line	Calculated XU	Observed XU
43	$K_{\alpha 2}$	677.77	677.8
43	$K_{\alpha 1}$	673.37	673.5
43	$K_{\beta 1}$	600.14	601.4
43	$K_{\beta 2}$	589.18	589.9

The experimental conditions of the exposures are listed in Table III.

4. APPARATUS

The x-ray spectrograph of a 25-cm radius was the conventional Bragg-focusing type, employing the internal standard method of wave-length calibration. The spectrograph chamber was pumped down to 100μ. A Be window 0.010 inch thick between the chamber and the slit permitted the x-ray tube to be evacuated to 0.02μ, the operating pressure. Eastman No-Screen safety film was exposed in the camera and developed seven minutes in DK 50.

The tube was operated with the anode at ground potential and the cathode at high negative potential. The separation of the cathode-target was set at $1\frac{1}{4}$ inches, and the distance of the anode spot to slit was fixed at $1\frac{1}{2}$ inches, the radius of the tube body. The filament assembly was insulated from the tube body by a 2×6-inch Pyrex flanged pipe and was cooled by a blast of air directed towards the flange. The regulated high voltage supply was rated at 60 kv and 100 ma.

The targets were flanged at one end to make them interchangeable and permit water cooling. They were fabricated from aluminum in order to minimize continuous background. The $\frac{3}{4}$ inch face of the target was cut with its normal at 70° to the slit-crystal-film axis.

The diffracting crystal[6] was of rocksalt and measured 70 mm across the face. It was rocked by a cam mechanism through an angle of 5°.

[6] Grown by Harshaw Chemical Company, Cleveland, Ohio.

TABLE II. Second-order interference lines.

Element	Line	Wave-length	Element	Line	Threshold V
Mo	$K_{\alpha 1}$ 708	704	Cs	$K_{\beta 1}$	35.9 kv
		710	Ce	$K_{\alpha 1}$	40.3
Mo	$K_{\alpha 2}$ 712	710	Ce	$K_{\alpha 1}$	40.3
No. 43	$K_{\alpha 1}$ 673	670	Nd	$K_{\alpha 2}$	43.6
No. 43	$K_{\alpha 2}$ 677	—	—	—	—
Ru	$K_{\alpha 1}$ 642	—	—	—	—
Ru	$K_{\alpha 2}$ 646	—	—	—	—
Mo	$K_{\beta 1}$ 631	628	Ce	$K_{\beta 1}$	40.3
Mo	$K_{\beta 2}$ 619	628	Ce	$K_{\beta 1}$	40.3
No. 43	$K_{\beta 1}$ 601	602	Pr	$K_{\beta 1}$	41.9
No. 43	$K_{\beta 2}$ 590	584	Nd	$K_{\beta 1}$	43.6
Ru	$K_{\beta 1}$ 574	584	Nd	$K_{\beta 1}$	43.6
Ru	$K_{\beta 2}$ 564	—	—	—	—
			Re	K series	71.5

The crystal was set at a median grazing angle of 6° 30′ by means of a bevel protractor.

The slit was composed of two $\frac{1}{16}$ inch stainless steel jaws. The slit was set under a measuring microscope to a width of 0.001 ± 0.0001 inch.

5. SUMMARY

The sample shown by the x-ray spectra is chiefly Element 43. The lines of Element 43 measured were $K_{\alpha 1}$, $K_{\alpha 2}$, $K_{\beta 1}$, and $K_{\beta 2}$. The agreement of the observed wave-length assignment and calculated values are apparent in Table I.

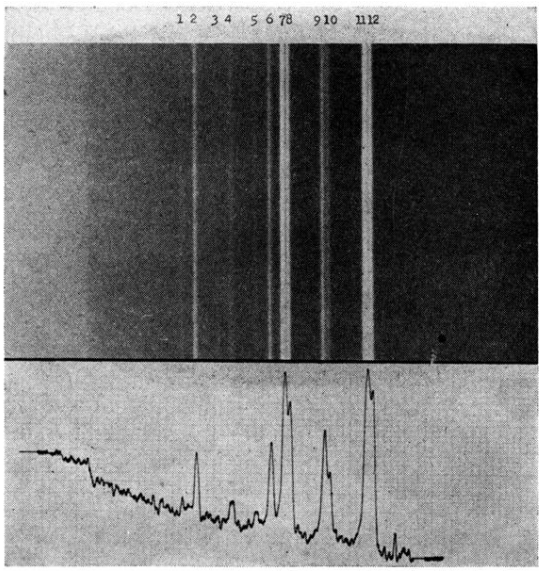
The chemical and physical history of the material together with the controlled conditions in obtaining the x-ray spectra is conclusive evidence that this material is Element 43.

This document is based on work performed under Contract No. W-7401-eng-23 for the Atomic Energy Commission at the Tennessee Eastman Corporation, Oak Ridge, Tennessee and under Contract No. W-7405-eng-26 for the Atomic Energy Commission at the Carbide and Carbon Chemicals Corporation, Oak Ridge, Tennessee.

TABLE III. Experimental conditions for the exposures.

Print No.	Element	Potential kv[a]	Emission current ma[a]	Cam	Exposure per degree	Total exposure minutes	Tube pressure[a]	Camera pressure
1	No. 43	35	7.0	5°	20	100	0.02μ	100μ
2	43 and Mo	35	8.0	5°	24	120	0.02μ	100μ
3	Mo, 43, Ru	35	8.0	5°	24	120	0.02μ	100μ
4	Mo and Ru	40	8.0	5°	8	40	0.02μ	100μ

[a] The values in columns 3, 4, and 8 are average values.



Line identification

	Element	Line		Element	Line
1	Ru	$K\beta_2$	7	Ru	$K\alpha_1$
2	Ru	$K\beta_1$	8	Ru	$K\alpha_2$
3	43	$K\beta_2$	9	43	$K\alpha_1$
4	43	$K\beta_1$	10	43	$K\alpha_2$
5	Mo	$K\beta_2$	11	Mo	$K\alpha_1$
6	Mo	$K\beta_1$	12	Mo	$K\alpha_2$

FIG. 1. X-ray spectrograms. Molybdenum element 42, Element 43, ruthenium element 44.