Title
THE L X-RAY SPECTRA FROM RADIOACTIVE DECAY OF TRANSURANIUM ELEMENTS

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Publication Date
1950-07-28

UCRL-341

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THE L X-RAY SPECTRA FROM RADIOACTIVE DECAY OF TRANSURANIUM ELEMENTS

G. W. Barton, Jr., H. P. Robinson, and I. Perlman

July 28, 1950

Berkeley, California

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G. W. Barton, Jr., H. P. Robinson, and I. Perlman
Radiation Laboratory and Department of Chemistry
University of California, Berkeley, California
July 28, 1950

ABSTRACT

A bent crystal x-ray spectrometer is described and some results are given on the analysis of L-series x-rays produced in radioactive decay processes of transuranium elements. There is generally good agreement between measured energy values of L-series lines and those predicted by the Moseley relationship. The relative intensities of the various lines produced in this case from γ-ray internal conversion, are compared with those emitted from uranium excited by electron bombardment and values reported of internal conversion excited x-rays in the region of lead.

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INTRODUCTION

The x-rays accompanying radioactive decay processes are capable of yielding a considerable amount of information on the nature of these processes. For some purposes simple detection may be adequate, but much potential information can only come to light if the x-ray spectra are resolved. As an example, spectrometry of x-rays enables one to distinguish between electron capture and isomeric transition processes and in particular to aid in the resolution of decay schemes for nuclei which undergo more than one mode of decay. In another study[1] the identification of x-rays has revealed an unsuspected electron-capture branching in the decay of Am^{242m}.

In many transitions the K-electron levels are "excited" and for these considerable information can be obtained from weak sources by absorption methods because of the simplicity of the K x-ray spectra. However, the complexity of L x-ray spectra renders absorption techniques largely ineffective and a method capable of greater discrimination is needed. Among the heaviest elements, for which K-electron binding energies are around 100 Kev, it would seem from the scanty data so far available that many of the decay processes do not excite the K level and that only L x-rays are observable.

It is with the measurement of the L x-ray spectra from the decay of heavy nuclei that the present communication is concerned. The method used is capable of a moderately high degree of precision and consists of diffraction separation using an oriented thin bent crystal. Since the x-rays examined were those of

[1] O'Kelley, Barton, Crane, and Perlman, University of California Radiation Laboratory Report UCRL-807 (July, 1950); to be submitted to Physical Review.

transuranium elements, the x-ray energies obtained are of interest in themselves in seeing whether or not they agree with extrapolations from lower elements.

The x-rays with which the present report is concerned have their origin in the internal conversion of gamma-rays. In addition to the goals of x-ray spectrometry already mentioned, the measurement of relative intensities of various transitions as they are related to the relative conversion coefficients in the various levels are of obvious importance in understanding the process of γ-ray internal conversion. Kinsey[2] has reviewed the problem in the heavy element region, and in a second paper[3] gives data on ThC and RaD taken by absorption and coincidence counting methods. The data to be reported here are in some details at variance with the generalizations made from the observations on ThC and RaD.

The possibility of making a focusing x-ray spectrometer was examined originally by de Broglie[4] and by Darbord.[5] Later a more complete study of the practical aspects of the problem was made by DuMond and Kirkpatrick.[6] Shortly after this the first generally satisfactory instrument was devised by Cauchois,[7] and her approach is that which has been adopted by a number of others. The essential feature of a Cauchois instrument is the use of an elastically bent crystal to permit focusing of radiation with high resolution and without structural complications.

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[2] B. B. Kinsey, Can. J. Research 26A, 404 (1948).

[3] Ibid., 421.

[4] L. de Broglie, Compt. rend. 158, 944 (1914).

[5] R. Darbord, J. phys. radium 3, 212 (1922).

[6] J. W. M. DuMond and H. A. Kirkpatrick, Rev. Sci. Instruments 1, 88 (1930).

[7] Y. Cauchois, J. phys. 3, 320 (1932); 4, 61 (1933).

Abelson[8] was the first to use a bent crystal spectrograph to observe x-rays from a radioactive decay process, and among other measurements he identified molybdenum x-rays from the electron-capture decay of element 43 (technetium). Pool and co-workers[9] have made extensive use of this technique in their identification of radioactive species as has the group at Zurich.[10] A large radius spectrometer has been built and used by DuMond[11] for precision measurement of photon energies from the conventional x-ray region into the range of an Mev.

The present report describes a bent crystal spectrometer and its use to determine the L x-ray spectra of two of the transuranium elements, neptunium and plutonium. These x-rays arise from internal conversion of gamma-rays accompanying the alpha-decay of isotopes of americium and curium, respectively.

METHODS

The spectrometer used in the present studies was designed as a monochromator using a counter tube as detector. A scale drawing of the essential parts may be seen in Fig. 1 in which the sample (2), its collimator (4), the crystal (1), and the counter (9) with its collimator (8) are shown in line. The sample holder

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[8] P. Abelson, Phys. Rev. 56, 753 (1939).

[9] Edwards, Pool, and Blake, Phys. Rev. 67, 151 (1945); Edwards and Pool, Phys. Rev. 72, 384 (1947); Coleman and Pool, Phys. Rev. 72, 1070 (1947).

[10] Marmier, Blaser, Preiswerk, and Scherrer, Helv. Phys. Acta. 22, 155 (1949); 21, 198 (1948).

[11] J. W. M. DuMond, Rev. Sci. Instruments 18, 626 (1947).

Fig. 1. Scale drawing of x-ray spectrometer.

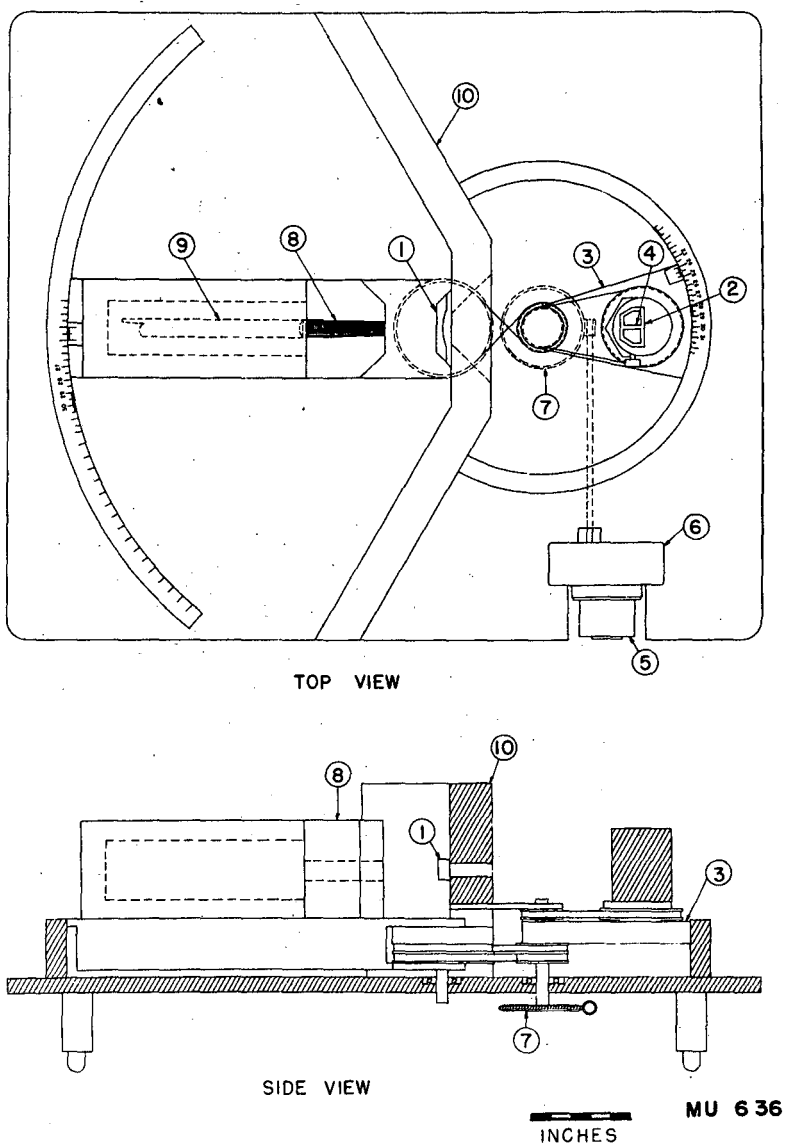

Fig. 1

is mounted on an arm which rotates it on the focal circle of 5-inch radius facing the concave side of the crystal. As pointed out by DuMond[11] if the counter is placed on the convex side and the source on the concave side, there is a considerable increase in efficiency over the opposite arrangement since all x-rays from the source are incident upon the crystal at the appropriate angle for diffraction. The crystal used for the measurements to be reported was quartz 0.008-inch thick cut perpendicular to the 310 planes. A tool steel holder with an aperture 3/4-inch long x 1/4-inch high and surfaces machined to a radius of 10 inches clamps the crystal so that its center is tangent to the focal circle and all crystal planes are directed at a point on the opposite side of the focal circle.

In operation on continuous sweep the motor (5) rotates the sample holder arm and at the same time rotates the detector arm so that the angles between crystal planes and sample and between crystal planes and detector are equal. An arrangement of metal bands turns the sample holder so that the sample and its defining slit always face the crystal aperture. The gear changing box (6), operating through the worm gear (7), permits selection of angular speeds of the sample arm of 1, 1/5, 1/20, and 1/100 degree per minute. The position of the sample arm is read on the scale by means of a vernier.

The counter is protected from stray radiation in several ways. The fine lead collimator (8) protects the counter tube from undiffracted x-rays passing through the crystal; in addition the tube is placed in a lead cylinder and the fixed shield (10) gives still more protection against direct radiation from the sample. The counter used in the measurements to be reported was an end window proportional counter filled with xenon at a pressure of 55 cm mercury and methane at 15 cm. With no sample in place, the background counting rate was in the range 3-10 per minute, depending on the amount of activity in the adjacent laboratory. Incoherent scattering contributed an additional background of about 1/2 percent

of the total x-radiation observed. This low background counting rate was made possible by operating the tube in a pulse height selection circuit to discriminate against radiation not in the energy range of the x-rays under measurement.

Fig. 2 shows a block diagram of the counting circuits. The tube is operated with the copper cathode shell at high voltage in order to simplify the coupling of the small pulses from the proportional counter to the preamplifier. The preamplifier is mounted on the counter housing and amplifies the counter pulse about one hundred fold. The pulse height discriminator which follows the second amplifier may be adjusted both in band width and pass band, and as mentioned, it is used to eliminate pulses which are not in the range of those produced by the x-rays under measurement. The recording system following the third amplifier includes a scaling circuit which actuates a recording Streeter-Amet Traficounter and a counting rate meter connected to an Esterline-Angus graphic milliammeter. The diffraction angle is read and recorded manually on the Traficounter tape at intervals. Points on the milliammeter record chart and on the Traficounter tape are related reliably to the angular position of the sample since all three are driven by synchronous motors.

The method of converting the counting data to x-ray energies is based on the grating spacing of the 310 planes of quartz which was taken to be 1.178A. From the measured diffraction angle and Bragg's law, the wave length is determined in Angstrom units and this is converted to energy in Kev units by the relation $E = 12.395/\lambda$. An internal check on the value of the diffraction angle is obtained by observing it on both sides of the spectrometer zero point. Errors caused by inaccuracies in location of the sample or orientation of the crystal planes are eliminated in this manner. Other details of the methods will be found under discussion of the particular measurements.

Fig. 2. Block diagram of electronic circuits for proportional counter detector of the x-ray spectrometer.

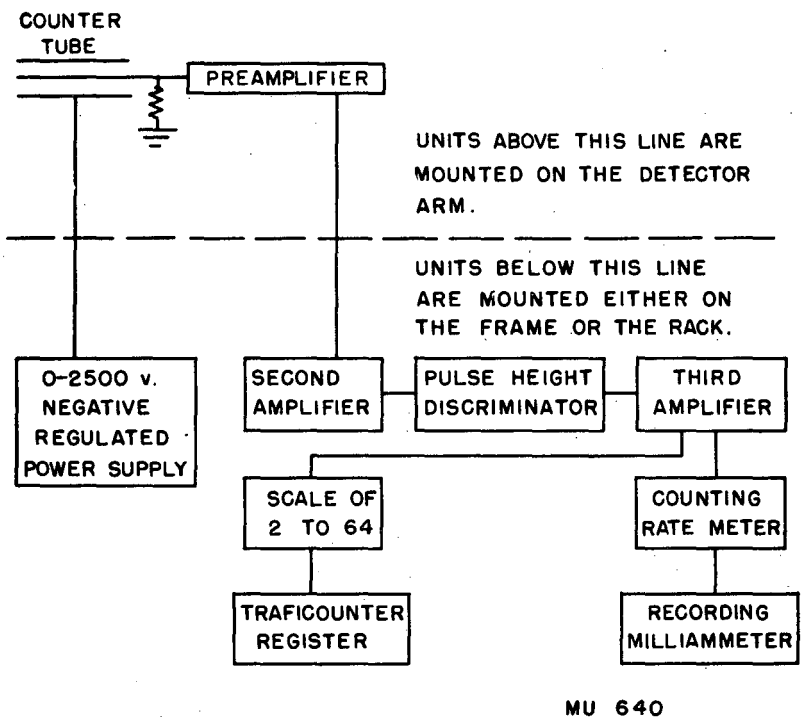
COUNTER TUBE
PREAMPLIFIER
UNITS ABOVE THIS LINE ARE MOUNTED ON THE DETECTOR ARM.
UNITS BELOW THIS LINE ARE MOUNTED EITHER ON THE FRAME OR THE RACK.
0-2500 v. NEGATIVE REGULATED POWER SUPPLY
SECOND AMPLIFIER
PULSE HEIGHT DISCRIMINATOR
THIRD AMPLIFIER
SCALE OF 2 TO 64
COUNTING RATE METER
TRAFICOUNTER REGISTER
RECORDING MILLIAMMETER
MU 640

Fig. 2

RESULTS

There will be need to refer to the different components of the L x-ray spectra and Fig. 3 shows a term diagram with the transitions observed in these studies designated according to the Siegbahn convention.[12] The particular levels shown are those for plutonium x-rays with energy values obtained by use of the Moseley relation:[13]

$$E^{1/2} = K(Z-\sigma)$$

The values for K and σ were calculated from the level energies of Th and U as given by Siegbahn.[14] The transition energies obtained in this manner have been used to identify the observed x-ray lines. The energies predicted for the major transitions from elements in the atomic number range 90-96 are listed in Table I.

Plutonium X-rays From Decay of Cm^{242}

The isotope Cm^{242} is an alpha-particle emitter with 162-day half-life prepared for the present study by the neutron irradiation of the 475-yr. Am^{241} according to the following reactions:

$$Am^{241}(n,\gamma)Am^{242m}$$

$$Am^{242m} \xrightarrow[16\ hr.]{\beta^-} Cm^{242}$$

The alpha-decay of Cm^{242} includes fine structure in which roughly 20 percent of the disintegrations go to an excited state of Pu^{238} about 50 Kev above the ground

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[12] See Compton and Allison, X-rays in Theory and Experiment (D. Van Nostrand Company, Inc., New York, 1935), p. 596.

[13] H. G. J. Moseley, Phil. Mag. 27, 703 (1914).

[14] M. Siegbahn, Spectroscopic der Rontgenstrahlen (Springer, Berlin, 1931).

Fig. 3. Term diagram for x-ray energy levels in the plutonium transitions observed in this study are indicated.

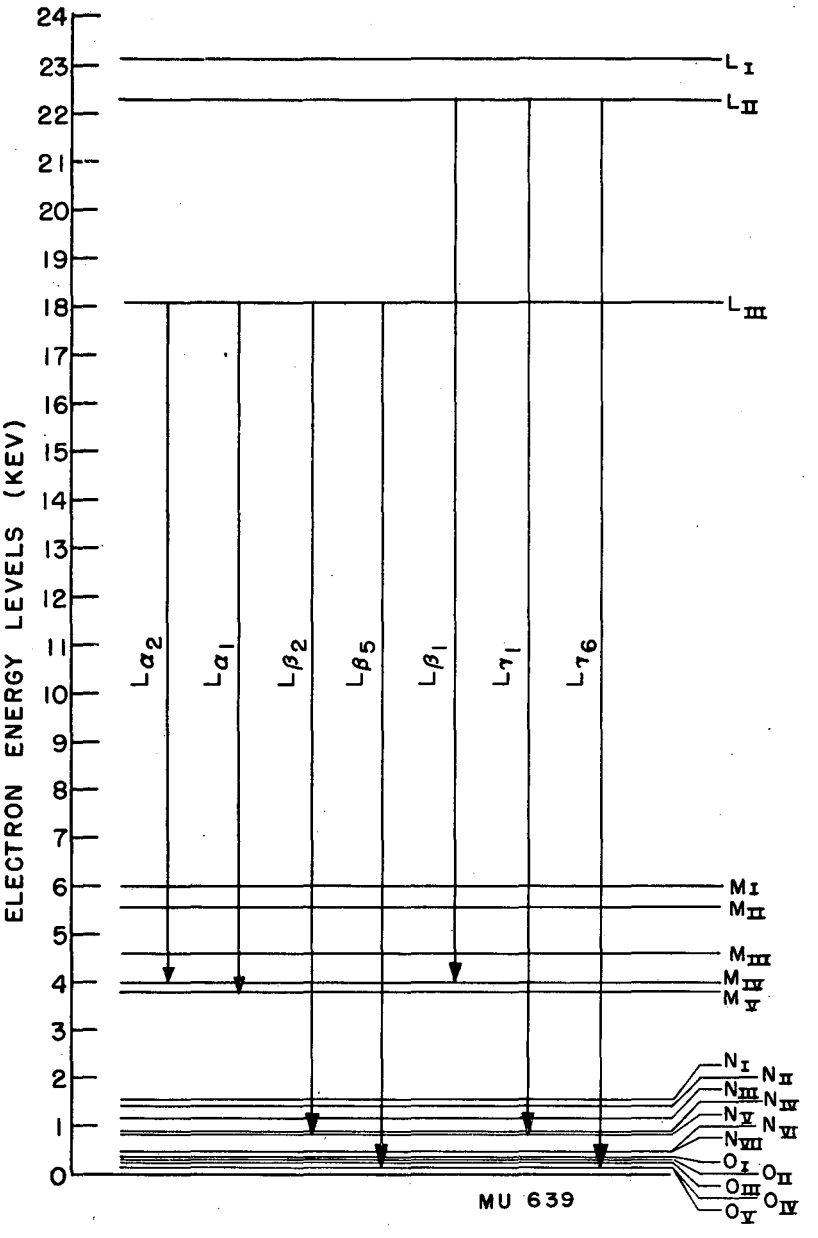
ELECTRON ENERGY LEVELS (KEV)
24
23
22
21
20
19
18
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
L_I
L_{II}
L_{III}
$L\alpha_2$
$L\alpha_1$
$L\beta_2$
$L\beta_5$
$L\beta_1$
$L\gamma_1$
$L\gamma_6$
M_I
M_{II}
M_{III}
M_{IV}
M_V
N_I N_{II}
N_{III} N_{IV}
N_V N_{VI}
N_{VII}
O_I O_{II}
O_{III} O_{IV}
O_V
MU 639

Fig. 3

Table I

Calculated L-series x-ray energies.

Line	Transition	Energy (Kev)						
		$_{90}Th$	$_{91}Pa$	$_{92}U$	$_{93}Np$	$_{94}Pu$	$_{95}Am$	$_{96}Cm$
1	L_{III}-M_I	11.14	11.39	11.64	11.90	12.16	12.42	12.69
α_2	L_{III}-M_{IV}	12.84	13.15	13.47	13.79	14.11	14.45	14.78
α_1	L_{III}-M_V	13.00	13.32	13.65	13.98	14.31	14.66	15.00
η	L_{II}-M_I	14.54	14.99	15.43	15.89	16.35	16.81	17.29
β_6	L_{III}-N_I	15.00	15.37	15.76	16.14	16.53	16.92	17.32
β_2	L_{III}-N_{IV}	15.62	16.01	16.42	16.82	17.29	17.66	18.08
β_4	L_I-M_{II}	15.67	16.14	16.61	17.09	17.58	18.07	18.57
β_5	L_{III}-O_{II}	16.24	16.67	17.10	17.54	17.98	18.44	18.89
β_1	L_{II}-M_{IV}	16.24	16.75	17.26	17.78	18.30	18.84	19.38
β_3	L_I-M_{III}	16.46	16.97	17.49	18.02	18.56	19.10	19.65
γ_1	L_{II}-N_{IV}	19.02	19.61	20.21	20.81	21.43	22.05	22.68
γ_2	L_I-N_{II}	19.34	19.93	20.53	21.13	21.75	22.37	23.00
γ_3	L_I-N_{III}	19.54	20.15	20.76	21.38	22.01	22.65	23.29
γ_6	L_{II}-O_{IV}	19.64	20.27	20.89	21.53	22.17	22.83	23.49

state.[15] The accompanying γ-ray transition is largely internally converted in the L-shell and the x-rays measured in this study are those resulting from the refilling of these L-orbit vacancies. The uncertainties in x-ray counting efficiencies allow an estimation of their number only between wide limits, but which for the present we take to be 10 L x-ray quanta per 100 alpha-disintegrations.

[15] G. D. O'Kelley and W. W. T. Crane, unpublished work.

A sample of CmF_3* emitting about 3×10^8 x-rays per second was mounted in a quartz capillary tube making a line source about 1-cm long. This capillary tube was held vertically in a lucite holder in the sample housing. No sample collimator was necessary because of the good definition of the source.

Even so, it is likely that the resolution is limited by the sample width since with similar samples not so homogeneous in their geometrical distribution, identical "fine structure" appeared in each line which is interpreted as an image of the sample distribution.

The spectrometer was adjusted to sweep at a rate of 1/20 degree per minute and the counts in each 0.8-min. interval were recorded. Fig. 4 shows one segment of the spectrum including the La_1 and La_2 lines in which both the spectrometer scale reading and the energy calibration are indicated. One unit of the spectrometer scale corresponds to 4 degrees displacement of the sample arm, therefore, the angle traversed between the two peaks shown is only about 0.6 degree.

Fig. 5 shows curves obtained for the spectrum in the interval of approximately 14-22 Kev in which the two sections represent data taken on the two sides of the zero position. As mentioned, the mean diffraction angle obtained from scale readings on both sides of the zero position is the true value even though misalighment of the sample or the crystal planes would introduce an error in each of the single position readings. In obtaining these curves a system of curve smoothing was employed which averaged over each five adjacent points and plotted the number so obtained at the center of the smoothing interval. The relative heights of the peaks do not reproduce faithfully the abundances of the emitted x-ray lines because no corrections have yet been made for the differences in reflection coefficients, counter efficiencies, and several sources of absorption. These will be discussed further below.

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*We are grateful to Mr. W. W. T. Crane for placing this sample of curium at our disposal.

Fig. 4. La_1 and La_2 lines of plutonium from decay of Cm^{242}.

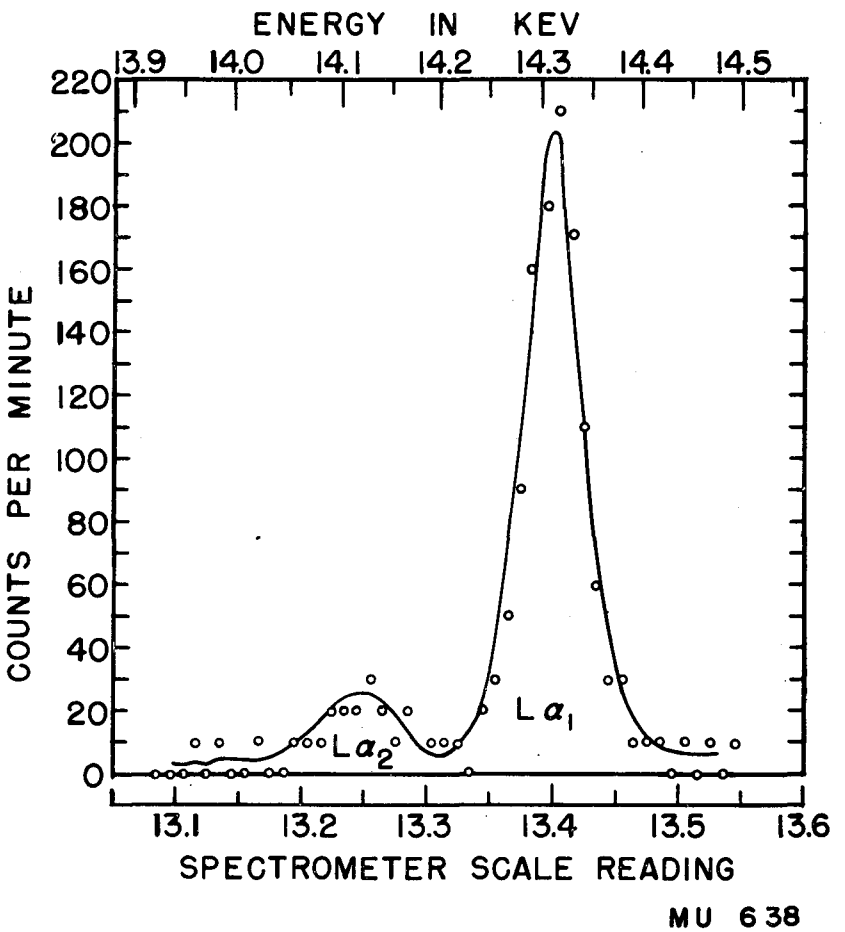
ENERGY IN KEV
13.9
14.0
14.1
14.2
14.3
14.4
14.5
220
200
180
160
140
120
100
80
60
40
20
0
COUNTS PER MINUTE
$L\alpha_2$
$L\alpha_1$
13.1
13.2
13.3
13.4
13.5
13.6
SPECTROMETER SCALE READING
MU 638

Fig. 4

Fig. 5. L-series x-ray spectrum of plutonium following decay of Cm^{242}. (Upper and lower plots show identical spectra taken on two sides of spectrometer zero position.)

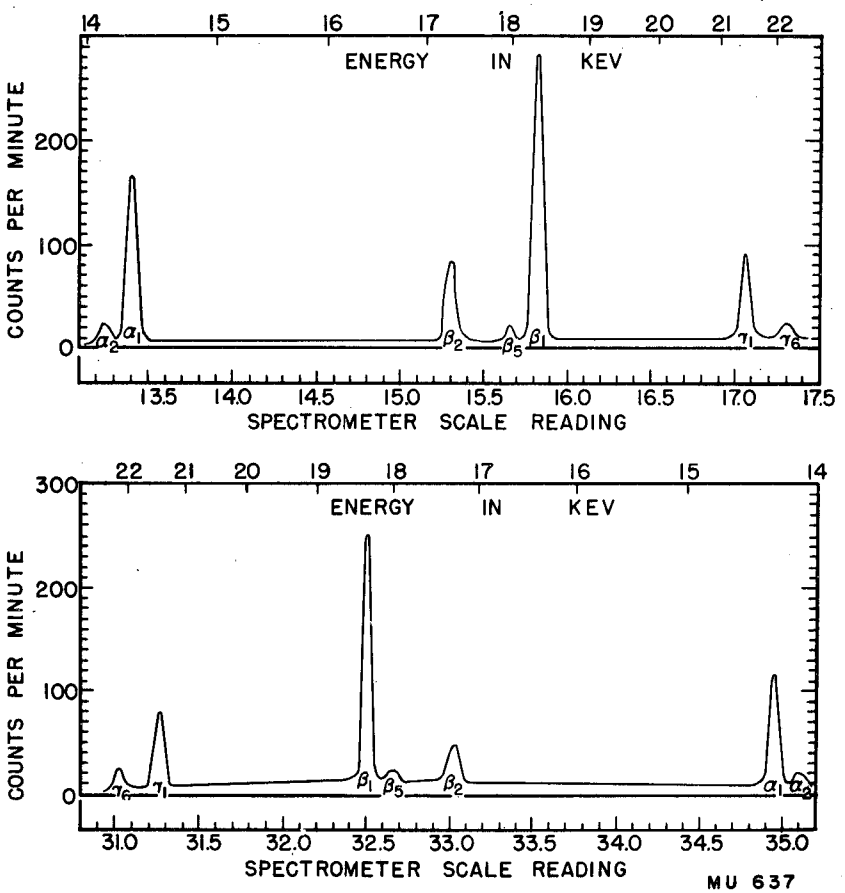
14 15 16 17 18 19 20 21 22
ENERGY IN KEV
COUNTS PER MINUTE
300
200
100
0
α_2 α_1 β_2 β_5 β_1 γ_1 γ_6
13.5 14.0 14.5 15.0 15.5 16.0 16.5 17.0 17.5
SPECTROMETER SCALE READING
22 21 20 19 18 17 16 15 14
ENERGY IN KEV
COUNTS PER MINUTE
300
200
100
0
γ_6 γ_1 β_1 β_5 β_2 α_1 α_2
31.0 31.5 32.0 32.5 33.0 33.5 34.0 34.5 35.0
SPECTROMETER SCALE READING
MU 637

Fig. 5

Table II lists in the second and third columns the spectrometer scale readings corresponding to the lines of Fig. 5 for some of which there were two measurements. Another series of measurements has been made on a different curium sample and the results agree with those reported here both in energy of the lines and intensities. The last two columns of Table II give the angles of diffraction obtained from these readings and the corresponding energies. The level transition assignments of the lines were made by comparing with the calculated values listed in Table I. Table III compares our measured energy values for the L-series x-rays of plutonium (Table II) with those obtained by extrapolation and shown in Table I and a set calculated by Monk and Allison[16] who used a formula of Sommerfeld[17] derived by the old quantum theory.

Of interest are the intensity values listed in the last three columns of Table III. The column headed "Uranium" gives relative intensities measured by Allison[18] from the electron bombardment of uranium. Comparing these values for uranium with similar measured ones for thorium indicates no gross changes with atomic number. The next column lists the observed intensities according to Fig. 5, and the last column, the corrected values normalized to the La_1 intensity taken to be 100. The corrections involved the following. The sample mounting, quartz crystal and counter window were estimated to be equivalent to 150 mg/cm^2 aluminum for x-rays in this energy region and absorption losses were calculated from the compilation of absorption coefficients of S. J. M. Allen.[19] The counting

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[16] A. T. Monk and S. K. Allison, Manhattan Project Metallurgical Laboratory Report CP-2120 (September, 1944).

[17] Compton and Allison, op. cit., p. 610.

[18] S. K. Allison, Phys. Rev. 30, 245 (1927); 32, 1 (1928).

[19] S. J. M. Allen in Compton and Allison, op. cit., p. 800.

Table II

Plutonium L x-rays from Cm^{242} decay.

X-ray designation	Spectrometer scale reading		Angle of diffraction θ	Energy
	First position	Second position		
$L\alpha_1$		34.932 34.947		
	13.404	34.940	21.536 ± 0.02	14.31 ± 0.01 Kev
$L\alpha_2$	13.244	35.090	21.846 ± 0.02	14.14 ± 0.01
$L\beta_1$		32.500 32.495		
	15.830	32.497	16.667 ± 0.02	18.35 ± 0.02
$L\beta_5$		32.650 32.660		
	15.665	32.655	16.990 ± 0.02	17.91 ± 0.02
$L\beta_2$	15.305	33.020	17.725 ± 0.02	17.28 ± 0.02
$L\gamma_1$	17.065 17.067	31.262 31.250		
	17.066	31.256	14.190 ± 0.02	21.46 ± 0.04
$L\gamma_6$	17.315	31.025	13.710 ± 0.02	22.20 ± 0.04

efficiency of the xenon tube for the different energy x-rays was estimated from calibrations by Crane and Ghiorso[20] and the reflection coefficient of the crystal is assumed to vary[21] as $1/E^2$.

[20] W. W. T. Crane and A. Ghiorso, unpublished work.

[21] Lind, West, and DuMond, Phys. Rev. 77, 475 (1950).

Table III

Comparison of predicted and observed energies and abundances in L-series x-rays of plutonium from alpha-decay of Cm^{242}

Line	Transition	Energy (Kev)			Intensity		
		Monk and Allison[16]	Calc. (this paper)[a]	Measured[b]	In uranium[18]	Observed here[c]	Corrected here[d]
α_1	L_{III}-M_V	14.30	14.31	14.31	100	160	100
β_1	L_{II}-M_{IV}	18.27	18.30	18.35	49.4	276	91
β_2	L_{III}-N_V		17.29	17.28	28	78	28
γ_1	L_{II}-N_{IV}		21.43	21.46	12	80	23
α_2	L_{III}-M_{IV}	14.11	14.11	14.14	11	20	13
β_5	L_{III}-O_V		17.98	17.91	6.4	16	6
β_3	L_I-M_{III}	18.55	18.56		4.2		
β_4	L_I-M_{II}	17.60	17.58		4.1		
1	L_{III}-M_I	12.12	12.16		3.4		
γ_6	L_{II}-O_{IV}		22.17	22.20	2.2	14	4
β_6	L_{III}-N_I		16.53		1.6		
γ_2	L_I-N_{II}		21.75		1.5		
γ_3	L_I-N_{III}		22.01		1.4		
η	L_{II}-M_I	16.28	16.35		1.0		
β_7	L_{III}-O_I		17.73		0.4		

(a) Calculated as explained for Table I and as listed there.

(b) Measured in this work. See Table II.

(c) See Fig. 5.

(d) Corrected as explained in text.

It would not be expected that there should be agreement between all of the relative intensities of x-rays resulting from electron bombardment as compared with those from γ-ray internal conversion because of differences in the relative excitation of different L-levels. However, there should be agreement in the ratios of the different transitions arising from the same L vacancy. If we normalize the α_1 intensities as in Table III, there is excellent agreement between uranium and plutonium intensities for the α_1, β_2, α_2, and β_5 lines all of which are transitions involving the L_{III} level. However, those resulting from transitions to the L_{II} level (β_1, γ_1, and γ_6) are relatively twice as abundant for the internal conversion spectrum as for the electron bombardment spectrum. Furthermore, certain transitions involving the L_I level seen in moderate abundance in the electron bombardment source are missing and therefore lower by at least a factor of 5 in the internal conversion source. From electron bombardment of uranium the ratio $L\beta_3/L\beta_1 = 0.085$ while the same ratio in our source is <0.016. Similar limits can be set for other lines representing L_I and L_{III} vacancies.

Kinsey[3] has used absorption methods to determine the L_{III} transitions as related to the sum of L_{II} and L_I transitions for internal conversion processes in RaD and ThC. The present results on the internal conversion of a γ-ray of an excited state of Pu^{238} show the ratio of L_{II} to L_{III} x-rays to be in the range reported by Kinsey, but the L_I x-rays were not detected and a limit of 20 percent of the number of L_I vacancies formed by electron bombardment could be set. This observation is not consistent with the assumption[2,3] that the L_I level is always most strongly excited in internal conversion. Almost certainly the relative incidence of vacancies is dependent upon both energy of the γ-emission process and selection rules, and differences are to be expected for different nuclei.

Neptunium X-rays From the Decay of Am^{241}

Through a mechanism similar to that in which plutonium x-rays are present following Cm^{242} alpha-decay, x-rays of neptunium result from the internal conversion of a gamma-ray from an excited state of Np^{237} following the alpha-decay of Am^{241}. The isotope Am^{241} has a half-life of somewhat less than 500 years, emitting alpha-particles measured as 5.45 Mev, a large fraction or all of which go to an excited state[22] of Np^{237}. The resulting 62-Kev transition is highly converted in the L-shell giving rise to the L x-ray spectrum. The Am^{241} was prepared by neutron capture in plutonium resulting in the β^--emitter Pu^{241} which decays to the desired product.[23]

Table IV

Neptunium L x-rays from Am^{241} decay.

Line designation	Spectrometer scale reading		Angle of diffraction	Energy (Kev)	
	First setting	Second setting		(Measured)	(Extrap.)
$L\alpha_1$	35.15	13.05	22.10 ± 0.05	13.98 ± 0.03	13.98
$L\beta_2$	33.12 33.15 33.14	15.05	18.09 ± 0.06	16.94 ± 0.05	16.82
$L\beta_1$	32.69 32.71 32.70	15.50	17.20 ± 0.04	17.79 ± 0.03	17.78

[22] Seaborg, James, and Morgan, National Nuclear Energy Series, Plutonium Project Record, Vol. 14B, "The Transuranium Elements: Research Papers," Paper No. 22.1 (McGraw-Hill Book Co., Inc., New York, 1949).

[23] Ghiorso, James, Morgan, and Seaborg, Phys. Rev. 78, 472 (1950).

The measurements of the x-rays were made in a manner similar to that already described for the curium sample. However, the americium source was some ten-fold weaker so that not so many lines could be seen and relative intensities could not be estimated. The data for the three most intense lines ($L\alpha_1$, $L\beta_1$, and $L\beta_2$) are given in Table IV and energies compared with the estimated values as listed in Table I.

This work was performed under the auspices of the U. S. AEC.