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A NEW X-RAY SPECTROGRAPHIC TECHNIQUE APPLIED TO THE ELEMENTARY ANALYSIS OP MILLIGRAM SCALE SAMPLES

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A NEW X-RAY EMISSION SPECTROGRAPHIC TECHNIQUE APPLIED TO THE ELEMENTARY ANALYSIS OF MILLIGRAM SCALE SAMPLES

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ABSTRACT

A radically new type of x-ray emission spectrograph has been used to demonstrate elementary analysis of milligram amounts of materials. Characteristic x-rays excited in an analytical specimen by nuclear radiations emitted by a radioactive source are absorbed in a lithium-drifted semiconductor diode of silicon or germanium. The pulse heights of the electrical signals produced in the circuit of which the semiconductor diode is a part are analyzed by a multichannel pulse height analyzer and displayed as a pulse height or energy spectrum. The capability of the new instrument is tested for the analysis of representative elements in samples of milligram to 10 milligrams magnitude. It is shown that the new instrument holds much promise for rapid analysis of samples of this size and possibly of several orders smaller size. A combination of Am^{2+1} exciting radiation and silicon semiconductor analyzing detector is considered useful for elements in the atomic number range 20-68. For heavier elements the combination of Co^{57} exciting radiation and a germanium detector is chosen. Advantages and limitations of the new analytical technique are discussed.

UCRL-16578

INTRODUCTION

_1.

An attractive new technique for x-ray emission spectroscopy has been described recently by Bowman, Hyde, Thompson, and Jared (1). In this technique a nuclear γ -ray source of moderate strength is used to excite characteristic x rays of the constituent elements of an analytical specimen, and a lithiumdrifted silicon or germanium semiconductor diode is used to analyze the excited x rays. The principle of the method is presented in Figure 1. Recent improvements in semiconductor detector manufacture and in the associated electronic circuitry have made it possible to generate output signal pulses directly proportional to the energy of the input x rays with sufficient energy resolution that x rays of neighboring elements can be distinguished. The efficiency of the detectors lies in the range 50-100 percent and the main limitations in sensitivity arise from geometry factors and from various scattering and background effects. The system has a number of advantages over more usual analytical techniques which make use of x-ray tubes and analyzing crystals operating on the Bragg scattering principle. These factors are discussed in the cited paper (1).

It is the purpose of the present paper to demonstrate the performance of the semiconductor x-ray emission spectrograph for rapid analysis of various materials at the milligram level. This is by no means the lower limit of sensitivity of the method, but it is a level at which analysis is exceedingly simple and rapid with apparatus presently available in our laboratory.

Equipment of equivalent or superior ability to ours should be available from commercial suppliers in the near future.

EXPERIMENTAL APPARATUS

Two detector systems were used. A sketch of the first is shown in Figure 2. A lithium-drifted silicon diode 1.4 cm. in diameter with a 2.5 mm. deep sensitive layer was mounted in an aluminum housing behind a thin beryllium window. A preamplifier incorporating a field effect transistor (FET), designated 2N3823, was located immediately behind the silicon detector. The FET served as the first stage of a charge sensitive preamplifier (2). The detector was maintained at about -130° C. and the FET at liquid nitrogen temperature. The housing containing the semiconductor diode and the FET was evacuated. Details of the FET preamplifier assembly are given by Elad (2). Standard commercial amplifiers were used.

A source of radioactive Am^{241} was prepared by depositing approximately 10⁹ alpha disintegrations per minute in the bottom of hole in a lead cylinder of 4 mm. diameter. The activity was covered and sealed with epon cement. This source was cemented to the front surface of the beryllium window in front of the detector. A sample holder was mounted a few millimeters from the opening of the Am^{241} source. This holder consisted of a 2-1/2" \times 3-1/2" aluminum sheet with a 1-1/4 in. hole punched in the center. A piece of Scotchbrand tape was placed across this opening. The samples to be analyzed were various chemical compounds of milligram size mounted on 3/4 in. filter papers. These filter papers were positioned in the middle of the opening in the sample holder.

Americium-241 is an alpha emitter with a half-life of 470 years. The alpha particles are totally absorbed in the epon cement over the source. In addition, Am^{241} emits the x rays and γ rays shown in Figure 3. These radiations:

-2-

can be absorbed by the K or L electrons of any atom in the analytical sample whose electrons are lower in energy. This absorption results in the ejection of a K or L electron and subsequently in the emission of a K or L x ray characteristic of the absorbing atom.

A certain fraction of the secondary x radiation reaches the semiconductor diode, where it is absorbed by the photoelectric process, giving rise to an electrical pulse proportional in energy to the absorbed x ray. Individual pulses are analyzed in a pulse-height analyzer and stored in the memory of the analyzer. A commercial 400 channel analyzer was used for this puspose (3). At the end of the data collection period, which was typically only two minutes, the collected data were printed out on paper tape and plotted out with a Moseley XY plotter. The figures shown in this paper were traced from the Moseley plots. In each experiment the gain controls of the amplifier and of the pulse-height analyzer were set so that the x rays of particular interest would be suitably displayed on the output XY plots.

The above apparatus is suitable for the analysis of elements from about calcium (element 20) to erbium (element 68). For the analysis of elements of higher atomic number, the apparatus sketched in Figure 4 was used.

The detector in the second set of apparatus was a lithium-drifted germanium diode prepared at the Livermore branch of this Laboratory. It was a 1×3 cm. crystal with a sensitive layer of 0.3 cm. The detector bias was -2000 volts. The detector and field effect transistor (FET 2N3823) were mounted in a vacuum chamber and operated at liquid nitrogen temperature. The detector was electrically insulated from ground potential by means of an insulating rod which also served as a heat conductor. A 10 mil beryllium window

-3-

UCRL-16578

covered the front wall of the chamber in front of the counter. The amplifier and pulse-height analyzer units were the same as described above for the silicon crystal unit.

A radioactive source of 270 day Co^{57} (~2 × 10⁹ disintegrations per minute) was mounted in a lead block positioned in front of the beryllium window. Cobalt-57 dećays by orbital electron capture and emits prominent γ rays of 122 and 136 k.e.v. energy. These gamma rays lie higher in energy than the binding energy of the K electron of any chemical element so that K x radiation of any element can be excited by them. For higher energy K x rays, germanium is a more suitable detector than silicon because photon absorption is a strong function of the atomic number of the absorber. Hence Co^{57} primary radiation and a germanium detector constitute an excellent combination for excitation and analysis of characteristic x radiation of heavy elements. Of course the Co^{57} source could be used with the silicon detector with a reduced efficiency.

EXPERIMENTAL PROCEDURE

A series of samples was prepared in the following way. Solutions of compounds of single elements or mixtures of elements were prepared by dissolving weighed samples in water and diluting to a definite volume in a calibrated flask. Measured aliquots of these solutions were pipetted into a test tube and mixed with ammonium hydroxide or other reagents to form an insoluble compound. This compound was filtered onto a 3/4 in. filter paper (Wattmann No. 40) and air dried. The filtered deposit was covered with a 0.00025 in. Scotch-brand tape and mounted in the center of the 1-1/4 in. opening in the aluminum sample holder.

-4-

The sample holder was placed in front of one of the semiconductor detectors and a spectrum of the characteristic x rays was taken for a period of a few minutes. Then the sample was removed and a blank piece of filter paper was mounted in the same sample holder. The multichannel analyzer was set on subtract mode of operation and turned on for the same time period as used for the sample itself. This operation automatically subtracted, channel by channel, any background contribution from scattered γ rays or other sources. Then the data were printed out of the memory of the multichannel pulse height analyzer onto paper tape and onto graph paper by means of the Moseley XY plotter. The results are shown in the form of tables and figures.

The purpose of the experiments was to demonstrate how easy or how difficult it is to distinguish a particular element above the background at various sample levels. In particular we wished to show that most elements can be distinguished and estimated at the milligram level with equipment available at the present time. For our purpose we have not deemed it necessary to make a detailed analysis of the errors which one would have to consider for routine quantitative work. This is a new technique and it is our belief that our curves and tables should be valuable in indicating its potential usefulness. We wish also to emphasize the simplicity of the system and the rapidity of analysis. Ours is a multichannel system which collects and displays results on all elements simultaneously.

The sensitivity levels which are indicated by our curves are far from the possible lower limits. The strength of this primary γ source can be increased, and its geometrical form and its location with respect to the semiconductor and the specimen can be altered to give an order of magnitude or

-5-

perhaps several orders of magnitude increased sensitivity. There is also the possibility of better design in the dimensions and configurations of the semiconductor detectors. Any further reductions in electronic noise in the preamplifier or amplifier will cause further improvement in the energy resolving capability, which also will improve the sensitivity of the technique.[†]

DISCUSSION OF RESULTS

In Figure 5 and in all subsequent figures in this report, the abscissa is given in units of pulse height. This is directly proportional to energy, but the energy scale varied from figure to figure owing to the use of different gain settings on the amplifier and pulse-height analyzer in different runs. The energies of all pertinent x rays are collected in Table III.

The results for iron and yttrium are displayed in Figures 5 and 6, respectively. It is clear that the K x-ray peak of these elements can easily be resolved from the background at the 1 milligram level and that significant results can be achieved at the 0.1 milligram level. The yttrium and iron x rays are strongly excited by the neptunium L x radiation in the primary radiation. The preparation of the yttrium or iron in the form of a thin deposit is essential because of the strong absorption of their low-energy gamma rays in any material. For the same reason the use of a thin beryllium window in front of the detector is important.

^TThe resolution achieved by our apparatus in most of these experiments was l.l k.e.v. (full width at half maximum). Later work in our laboratory resulted in a reduction of this figure to<0.7 k.e.v (M. Nakamura and E. Elad, unpublished results).

-6-

The energy resolution in these curves, expressed as full width at half maximum, is l.l k.e.v. This resolution figure is approximately constant over the range of energies reported in this study. In the case of iron a constant our resolution is not sufficient to separate the components of the K x rays; e.g., the K α and K β groups of iron.

-7-

The AgBr analysis shown in Figure 7 is interesting because the K electron energy of bormine lies lower than the energy of the Np L x rays coming from the Am^{241} source, whereas the K-electron energy of silver lies higher. Hence, the bromine x rays are more strongly excited than the silver x rays which are produced only by the absorption of the 59.57 k.e.v. γ photons. Nonetheless 17,000 x rays were recorded in the K x-ray peaks of silver, showing that 10 mg. of silver can easily be analyzed. In the case of silver there is a clear separation of the K α and K β peaks.

In Figure 8 there appears the result of the analysis of a four-component mixture of 5 mg. each of chromium, cobalt, zinc and arsenic precipitated as mixed hydroxides. These elements are separated by steps of three in atomic number.

Results for lanthanum are shown in Figure 9. The L x rays are excited by the Np x rays coming from the Am^{241} source, and the K x rays are excited by the 59.57 k.e.v. radiation. If we compare the K x-ray peak of the 10 mg. yttrium sample to that of the 10 mg. lanthanum sample (Figure 6) we notice a decrease in sensitivity for the heavier element. This is caused by the decreased absorption of the exciting radiation. The half thickness of 59.57 k.e.v. radiation in lanthanum is about 150 mg./sq. cm., whereas the half thickness of a 17 k.e.v. Np x ray in yttrium is about 20 mg./sq. cm. Thus the major fraction of the 59.57 k.e.v. radiation passes through the sample, and it would be possible to increase the counting rate by use of a thicker sample. If the purpose of an analysis is to measure a small percentage of an element as heavy as lanthanum, an increased sensitivity could be achieved by using a thicker sample.

Figure 10 shows an analysis performed on a mixture of rare-earth hydroxides consisting of 5 milligrams each of lanthanum (element 57), neodymium (element 60), samarium (element 62) and holmium (element 67). The K α peak fo each element is observed. The K β peak of lanthanum and neodymium is buried under the K α peak of the next higher element. However, from the known shape of the K α K β spectrum of each pure element it would not be difficult to make a proper subtraction of the K β peak of one element from the K α peak of another. A composite unresolved peak of L x rays is also observed. The heaviest element, holmium, makes the major contribution to this peak because the neptunium L x rays of the Am²⁴¹ source are most strongly absorbed in holmium.

Figure 11 shows the results for a mixture of 1 mg. tantalum and 10 mg. niobium in the form of the pentoxides. The silicon detector was used, and the counting period was two minutes. The spectrum shows the L x rays of tantalum separated from the K x rays of niobium. The K x rays of tantalum are not excited by the 59.57 kme.v. γ ray of Am²⁴¹. We chose to show this case because it is an example of a mixture which poses a difficulty in the traditional x-ray emission spectrometer. This difficulty arises from the fact that the second order Bragg reflection of the niobium K x rays occurs at the same Bragg angle as the first order reflection of the tantalum L x rays. This difficulty can be taken care of by pulse-height analysis of the output of the proportional counter used as the final detector after Bragg reflection. However, in the new system proposed here this interference difficulty never arises.

-8-

The tantalum content of such a mixture can be better determined by exciting the K radiation of tantalum as is discussed below.

-9-

Figure 13 shows the performance of the Am^{241} and silicon detector combination on a lead sample, taken as representative of heavy elements. Only the L x rays are excited, but the intensity of the L x-ray peak is large because of the strong absorption of the neptunium x rays in the L-electron shell of lead. Hence, this would be a sensitive way to detect lead in a sample which contained no light elements which could emit K x rays of the same energy. Otherwise it would be better to excite lead K x rays and perform the analysis with the germanium detector as is shown below in Figure 15.

Now we turn to a discussion of the results with the Co^{57} and germanium detector combination. Figures 13-16 show the results of analyses of 10 milligram samples of lanthanum, gold, lead and thorium. In the last three the resolving power of the system is sufficient to separate the K x rays into the K α_2 , $\operatorname{K}\alpha_1$, $\operatorname{K}\beta_1$, and $\operatorname{K}\beta_2$ groups. The subgroups of the $\operatorname{K}\beta_1$ and $\operatorname{K}\beta_2$ peaks are not resolved. The separation of comparable K x rays of neighboring elements is several kilovolts in these heavy elements so that it is possible for our equipment to separate rather clearly the K x rays of neighboring elements.

The Co^{57} and germanium detector combination is excellent for selective analysis of a heavy element in the presence of light element impurities. As an example of this we reanalyzed the tantalum-niobium mixture. The analytical result, shown in Figure 17, clearly shows the K α and K β groups of tantalum standing out well above the background. There is no niobium contribution to these x-ray peaks. In fact niobium K x rays are off scale to the left and not shown in the figure. As another example of the ease of identification of a heavy element in the presence of numerous light elements, we examined a stainless steel sample containing 2.4 percent tungsten. The sample was a polished block weighing many grams. We used the National Bureau of Standards sample 1187 with major components of chromium, iron, cobalt, and nickel. The alloy also contained minor components of sodium, manganese, niobium, molybdenum, tungsten, and tantalum. We alhowed the Co^{57} radiation to shine on the surface of this metal block and analyzed the excited K x rays with our germanium detector system. As Figure 18 shows, we had no difficulty in identifying the K x rays of the 2.4 percent tungsten component. We did not observe the 0.04 percent component of tantalum.

-10-

Our general conclusion is that an x-ray emission spectrometer consisting of a radioactive source, a specimen, and a semiconductor detector is capable of quantitative analysis of single elements or simple mixtures of elements at the milligram level. The analytical technique is extremely simple and the analysis is completed within a few minutes. With improvements, it is likely that significant results can be obtained at sample sizes 1 to 3 orders of magnitude less. Analysis of complex mixtures of elements in the region of iron will not be easy with the present apparatus, particularly for small amounts of one element in the presence of a much larger amount of a neighboring element. For these elements the higher resolution of the conventional spectrometer consisting of an x-ray tube, an analyzing crystal and a goniometer will give superior results. However, if the atomic numbers of the constituent elements differ by 3 or more, the semiconductor system has sufficient energy discrimination to make a significant analysis of a complex mixture even for light elements.

UCRL-16578

For the heavy elements the new system has some special attractive features. The conventional x-ray emission spectrographs operating in many analytical laboratories are not equipped with x-ray tubes of sufficient voltage to permit the excitation of K x radiations of the heavy elements. In the new system there is no difficulty in exciting the K radiation with a properly chosen nuclear γ source. The germanium semiconductor detectors with sensitive layers of 3 to 5 mm. are capable of absorbing the K radiations of the heavier elements with 40-80 percent efficiency (exclusive of geometry factors, which are far larger than in the conventional x-ray spectrograph). The K α_2 , $K\alpha_1$, $K\beta_1$, and $K\beta_2$ structure of the K radiation of the heaviest elements can be clearly resolved. Furthermore, the average separation of the K x-ray groups of neighboring elements is three or more kilovolts, or three-fold greater than the resolution (full width at half maximum) of the detector. Hence, there is no difficulty in separating one heavy element from its nearest neighbor.

-11-

The measurement of heavy elements and light elements simultaneously in the same sample is especially simple. In some cases there is a real advantage in the use of the new system. This is true particularly when the Bragg angle of scattering of the K radiation of one element is identical with the second order scattering angle for the x radiation of a much lighter element.

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-13-

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- (2) Elad, E., Nucl. Instr. Methods 37, 327 (1965).
- (3) We used a Radiation Industries Development Laboratory Model 34-12 Analyzer. Comparable instruments manufactured by Technical Measurements Corporation, or other manufacturers would have served as well.

UCRL-16578

FIGURE CAPTIONS

-14-

Fig. 1. Schematic arrangement of apparatus for excitation and measurement of characteristic x radiations of an analytical sample. An encapsulated nuclear γ source (Am²⁴¹, Co⁵⁷ or other nuclide) is positioned near the specimen to excite x rays in the specimen. A fraction of these characteristic x rays reaches the lithium-drifted silicon or germanium semiconductor diode and produces an electrical charge. A field effect transistor (FET) serves as the first stage of a charge sensitive preamplifier. The preamplifier output is linearly amplified and the pulse height is measured and recorded in a multichannel pulse-height analyzer. That part of the apparatus outlined in dashed lines is in a vacuum chamber and is maintained at low temperature.

Fig. 2. Experimental arrangement of Am²⁴¹ source, lithium-drifted silicon detector, and specimen to be analyzed.

Fig. 3. Gamma and x radiation emitted by Am^{241} . This spectrum was taken by a silicon semiconductor detector with source shining directly into detector. A 2 mm. thick plastic cover absorbed the alpha radiations. Intensities per α disintegration of these radiations are as follows: 59.57 k.e.v. γ -0.36, 26.36 k.e.v. γ -0.025, 13.96 k.e.v. x ray-0.12, 17.76 k.e.v. x ray-0.13, and 20.80 k.e.v. x ray-0.03.

- Fig. 4. Experimental arrangement of Co⁵⁷ source, lithium-drifted germanium detector and specimen to be analyzed.
- Fig. 5. K x rays of iron excited by radiations of Am²⁴¹ in samples containing 10 mg. and 1 mg. of iron and analyzed with lithium-drifted silicon semiconductor detector. Two-minute runs. X-ray energies are given in Table III.

- Fig. 6. K x rays of yttrium excited by radiations of Am²⁴¹ in samples containing 10 mg., 1 mg., and 158 μg., respectively, of yttrium and analyzed with lithium-drifted silicon semiconductor detector. Two-minute runs except for 158 μg. sample for which analysis time was 10 minutes. X-ray energies are given in Table III.
- Fig. 7. K x rays of silver and bromine excited by radiations of Am²⁴¹ in 17 mg. AgBr and analyzed with lithium-drifted silicon semiconductor detector. Two-minute run. X-ray energies are given in Table III.
- Fig. 8. Analysis of mixed hydroxide sample containing 5 mg. each of chromium, cobalt, zinc and arsenic (elements 24, 27, 30, and 33). K x rays excited by radiations of Am²⁴¹ and analyzed with lithium-drifted silicon semicon-ductor detector. Two minute run. X-ray energies are given in Table III.
 Fig. 9. K and L x rays of lanthanum excited by radiations of Am²⁴¹ in a 10 mg.
- sample and analyzed with lithium-drifted silicon semiconductor detector. Two minute run. X-ray energies are given in Table III.
- Fig. 10. Analysis of mixture of 5 mg. each of lanthanum, neodymium, gadolinium, and holmium. K and L x rays excited by radiations of Am²⁴¹ and analyzed by lithium-drifted silicon semiconductor detector. Two-minute run. X-ray energies are given in Table III.
- Fig. 11. Analysis of mixture of 1 mg. tantalum and 10 mg. niobium. The L x rays of tantalum and the K x rays of niobium were excited by the radiations of Am²⁴¹ and analyzed by the lithium-drifted silicon semiconductor detector. The K x ray of Ta are not excited by the Am²⁴¹ source. Twominute run. X-ray energies are given in Table III.

- Fig. 12. L x rays of lead excited in 10 mg. sample by radiations of Am²⁴¹ and analyzed with lithium-drifted silicon semiconductor detector. Time of runtwo minutes. X-ray energies are given in Table III.
- Fig. 13. K x rays of lanthanum excited in 10 mg. sample by γ rays of Co^{γ'} and analyzed with lithium-drifted germanium semiconductor detector. Time of run—two minutes. X-ray energies are given in Table III.
- Fig. 14. K x rays of gold excited in 10 mg. sample by γ rays of Co⁵⁷ and analyzed with lithium-drifted germanium semiconductor detector. Time of run—two minutes. X-ray energies are given in Table III.
- Fig. 15. K x rays of lead excited in 10 mg. sample by γ rays of Co⁵⁷ and analyzed with lithium-drifted germanium semiconductor detector. Time of run-two minutes. X-ray energies are given in Table III.
- Fig. 16. K x rays of thorium excited in 10 mg. sample by γ rays of Co⁵⁷ and analyzed with lithium-drifted germanium semiconductor detector. Time of run-two minutes. X-ray energies are given in Table III.
- Fig. 17. Analysis of mixture of 1 mg. tantalum and 10 mg. niobium. The K x rays of tantalum were excited by the 122 k.e.v. γ ray of Co⁵⁷ and analyzed with the lithium-drifted germanium semiconductor detector. The K x rays of niobium are off scale to the left. Time of run-two minutes. X-ray energies are given in Table III.
- Fig. 18. Tungsten K x rays excited in N.B.S. standard sample 1187 by Co⁵⁷ γ rays and analyzed with lithium-drifted germanium semiconductor sample. Time of run-two minutes. Tungsten content 2.4 percent. X-ray energies are given in Table III.

	Sample	Integrated counts in peak (background subtracted)	Figure	Notes	
10 mg	. Fe as $Fe(OH)_3$	21,280	. 5		
l mg	. Fe as Fe(OH) ₃	1,465	5		
lO mg	Y as Y(OH)3	31,161	6		
l'mg.	Y as Y(OH)	3,376	6		
158 µg.	Y as Y(OH)	2;487	6	10.0 min. count	
8 mg.	Br as AgBr	79,309	- 7		
10 mg.	Ag as AgBr	17,104	7		
10 mg.	La as La(OH) ₃	6,607	9	K peaks	
	•	3,779	9	L peaks	
5 mg.	each La as M(OH)	3 2,857	10	Ka only	
<u>.</u>	Na	2,651	10	Ka only	
	Sm	2,299	10	Кα + β	
- · · ·	(_{Ho})	2,307	10	Ka only	
l mg.	Ta as Ta ₂ 0 ₅	4,457	11.	L x rays	
10 mg.	Nb as Nb ₂ 05	15,031	11	K x rays	
10 mg.	Pb as Pb(OH)	54,105	12	L x rays	

Table I. X-ray peak intensities in samples analyzed by silicon semiconductor detector.

In all cases, Am²⁴¹ was used as primary exciting radiation. Runs were for two minutes followed by two minutes of background subtraction unless otherwise stated. 18- -18-

UCRL-16578

Sample	Integrated counts in peak (background subtracted)	Figure	Notes	
l mg. Ta as Ta ₂ 0 ₅	15,151	17		
10 mg. Nb as Nb ₂ 0 ₅			Not on scale	
10 mg. La as La(OH) ₃	8,522	13	· · · · · · · · · · · · · · · · · · ·	
10 mg. Au as Au ₂ 0 ₃ •xH ₂ 0	14,187	14		
10 mg. Pb as PbO	7,362	15	KØ peaks	
	1,868	15	Kβ peaks	
10 mg. Th as ThO ₂	9,789	16	Kα peaks	
	1,925	16	Kβ peaks	

Table II. X-ray peak intensities in samples analyzed by germanium semiconductor detector.

In all cases $\operatorname{Co}^{57} \gamma$ rays of 122 k.e.v. and 137 k.e.v. energy were used as primary exciting radiation. Runs were for two minutes followed by two minutes of background subtraction.

UCRL-16578

Z	Element	κα	Ka2	кв _l	К _{В2}	$L\alpha$	Lβ	Lγ
24	Cr	5.42	5.41	•	5.95		<u> </u>	
26	Fe	6.40	6.39	•	7.06		. ,	
27	Co	6.93	6.92		7.65			
30	Zn	8.64	8.62	·	9.57	,		·
. 33	As	10.54	10.51	·) 1	1.73			· ·
35	Br	11.92	11.88	1	3.29			,
39	Y	14.96	14.88	16.74	17.01			. v
41	, ND	16.61	16.52	18.62	18.95			<u>к</u> .
47.	Ag	22.16	21.99	24.94	25.46			
57	La	33.44	33.04	37.81	38.74	4.64	5.17	5.79
60	Nd	37.36	36.85	42.27	43.34	5.22	5.91	6.60
62	Sm	40.13	39.53	45.42	46.60	5.62	6.40	7.18
67	Ho	47.53	46.69	53.86	55.29	6.70	7.72	8.75
73	Ta	57.53	56.28	65.22	67.01	8,12	9.50	10.89
74	W	59.32	57.98	67.24	69.10	8.36	9.82	11.28
79	Au	68.80	66.99	77.98	80.18	9.67	11.51	13.38
82	Pb	74.97	72.80	84.94	87.36	10.50	12.62	14.76
90	Th	93.35	89.86	,105.61	108.69	12.89	15.91	18.98
				:				

Table III. X-ray energies in k.e.v.

-19-



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-21-

MUB-9132



-22-



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MUB-9134







-25-



Fig. 6

MU 8 · 9125



Fig. 7

59



MUB-9123





MU B · 9121





MUB-9113







•





Fig. 14

-33-



MUB-9115











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