NRL Report 6096

Photographic X-Ray Fluorescence Data for Fixed-Crystal Cameras

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May 18, 1964

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ABSTRACT

A compilation has been made of x-ray fluorescence data obtained through the use of a novel fixed-crystal evacuated camera. These data will be of use whenever fixed sodium chloride crystals are used as diffractors for photographic devices of this type. From the tables presented it is possible to identify fifty-one of the common elements.

PROBLEM STATUS

This is an interim report on a continuing problem.

AUTHORIZATION

NRL Problem CO1-10
Project SR 007-08-04-0617

Manuscript submitted March 23, 1964.

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PHOTOGRAPHIC X-RAY FLUORESCENCE DATA FOR FIXED-CRYSTAL CAMERAS

With the development of a versatile x-ray fluorescence camera* it soon became apparent that a tabulation of data taken from a series of pure specimens would be invaluable when the device is to be used for qualitative analysis. Since there were at hand all of the common elements which give fluorescence patterns with this camera under the conditions described below, it was necessary only to convert these specimens into forms stable under vacuum, if they were not already in such a condition, and to determine their characteristic x-ray spectra. These data will be of use whenever fixed sodium chloride crystals are used as diffractors for photographic devices of this type.

The conditions under which the data were obtained are listed in Table 1. With few exceptions the wavelengths and relative intensities listed approach theoretical values, the exceptions being due to the effects of high film background caused by fluorescence of camera parts. Since the camera was intended for use in the investigation of oil-ash problems aboard naval vessels whose boilers are constructed of common metals of the transition group of elements, for which the films are satisfactory, no attempt was made to vary materials of construction or the choice of diffracting crystal. Naturally occurring elements for which data were not determined are those whose spectra are not detected under the conditions of the experiment (atomic numbers of 18 or less), the permanent gases, the lanthanons that have atomic numbers from 59 through 71, and the very rare elements, numbers 43, 84, 85, 87, 88, 89, and 91.

Data for the other fifty-one elements are given in Table 2, where the wavelengths listed are those which are detected under routine conditions when the concentration of the element in question is high. For elements number 19 through 25 only the first-order K_α and K_β lines appear, while higher orders registered for elements 26 through 53. Beginning with cesium (55) only the L-series lines appear. In no case were α_1 and α_2 lines resolved, even at higher orders of diffraction, and for this reason the doublets are referred to as K_α and L_α, respectively, and the stated wavelength is the arithmetic average of the component wavelengths. Where other lines are not resolved a notation to this effect has been made.

An effort was made to identify all lines appearing on film, including those due to major impurities. Where such impurities are the rule rather than the exception, as, for instance, the presence of lanthanum in "pure" cerium, the extraneous data have been tabulated, also. In a number of instances one or more well-defined lines appeared at high diffraction angles, however, and since locations of these were clearly a function of the atomic number of the element in question, the θ-angles have been listed. The cause of these lines was not established, but they are being used as an additional diagnostic feature of the films, since they lie in a region where dispersion is very great.

There appeared on many of the films pairs of symmetrical curved lines which crossed at lower angles than intense K_α, K_β, and L_α lines. These were due to diffraction of the intense radiation by the (111) planes near the edges of the analyzing crystal, the

*A. J. Pollard, "An X-Ray Fluorescence Camera," NRL Report 6061, Mar. 1964.

source of the radiation being the margins of the specimens at the opposite side of the camera. Although these foreign lines could be minimized by narrowing the slit of the primary x-ray beam or by using a more narrow crystal, they could not be eliminated entirely and should be expected to interfere with quantitative analysis in cases where a line being measured is superimposed on a stray image from a different element.

Table 1
Experimental Details

I. Camera Construction
- A. Inside diameter - 4.510 inches
- B. Materials
  - a. Case and lid - brass
  - b. Spectrometer sector - brass
  - c. Crystal mount - steel
  - d. Zero knife edge - steel
  - e. Baffles and shields - lead
- C. Crystal - NaCl (2d = 5.64 angstroms)

II. Photographic Conditions
- A. Exposure times (Using Ilford Industrial G X-Ray Film, Batch 947 X15.3)
  - a. For K_α, n = 1 2 hours max
  - b. For L_α, n = 1 2 hours min
  - c. For K_α or L_α, n > 1 4 hours min
- B. Processing Conditions (68°F)
  - a. Developer - GE Supermix MED - 5 minutes
  - b. Fixer - GE Supermix Speed Type for X-Ray Film - 10 minutes
  - c. Wash - 20 minutes in flowing water

III. X-Ray Characteristics
- A. Tungsten tube operating at 50 kv and 20 ma
- B. Camera case evacuated to less than 1 mm Hg

IV. Densitometry
- A. Recording densitometer
- B. Scanning rate 0.5 mm (1/2-degree θ) per minute
- C. Background adjacent to line equals 100% transmittance

Table 2
X-Ray Fluorescence Data for
the Common Elements

Wavelength (angstroms)	Line	Order (n)	Angle (θ)	Relative Intensity (I/I_0)
19 Potassium (Pellet pressed from KCl)				
3.543	K_β	1	37.8	30
3.744	K_α	1	42.0	100
20 Calcium (Pellet pressed from $CaCO_3$)				
3.089	K_β	1	33.2	20
3.360	K_α	1	36.5	100
21 Scandium (Sc_2O_3 pelletized with methyl cellulose)				
2.780	K_β	1	29.5	20
3.032	K_α	1	32.5	100
22 Titanium (Metal)				
2.514	K_β	1	26.5	20
2.749	K_α	1	29.2	100
23 Vanadium (Pellet pressed from V_2O_5)				
2.285	K_β	1	23.9	25
2.505	K_α	1	26.3	100
24 Chromium (Pellet pressed from Cr_2O_3)				
2.084	K_β	1	21.7	25
2.291	K_α	1	23.9	100
25 Manganese (Pellet pressed from MnO_2)				
1.910	K_β	1	19.8	30
2.103	K_α	1	21.8	100
26 Iron (Metal)				
1.753	K_β	1	18.2	30
1.937	K_α	1	20.1	100
1.753	K_β	2	38.5	5
1.937	K_α	2	43.4	30
27 Cobalt (Pellet pressed from metal powder)				
1.620	K_β	1	16.7	30
	[Ni K_α]	[1]	[17.1]	[10]
1.791	K_α	1	18.5	100
1.620	K_β	2	35.1	5
1.791	K_α	2	39.4	30
28 Nickel (Metal)				
1.500	K_β	1	15.4	35
1.659	K_α	1	17.1	100
1.500	K_β	2	32.1	10
1.659	K_α	2	36.1	40
1.659	K_α	3	61.9	10

(Table Continues)

Table 2 (Continued)
X-Ray Fluorescence Data for
the Common Elements

Wavelength (angstroms)	Line	Order (n)	Angle (θ)	Relative Intensity (I/I_0)
29 Copper (Metal)				
1.392	K_β	1	14.3	35
1.542	K_α	1	15.8	100
1.392	K_β	2	29.6	10
1.542	K_α	2	33.1	45
1.392	K_β	3	47.8	1
1.542	K_α	3	55.6	2
	?		60.0	2
	?		64.2	10
30 Zinc (Metal)				
1.295	K_β	1	13.3	30
1.437	K_α	1	14.8	100
1.293	K_β	2	27.4	10
1.437	K_α	2	30.6	40
	?		61.4	15
31 Gallium (Metal)				
1.207	K_β	1	12.3	35
1.341	K_α	1	13.7	100
1.207	K_β	2	25.3	10
1.341	K_α	2	28.4	40
1.207	K_β	3	39.9	2
1.341	K_α	3	45.5	4
32 Germanium (GeO_2 pelletized with methyl cellulose)				
1.129	K_β	1	11.5	25
1.256	K_α	1	12.9	100
	$[CuK_\alpha]$	1	15.8	10
1.129	K_β	2	23.6	10
1.256	K_α	2	26.4	40
1.256	K_α	3	41.8	10
	?		56.2	10
1.256	K_α	4	62.9	5
33 Arsenic (Pellet pressed from As_2O_3)				
1.057	K_β	1	10.8	25
1.177	K_α	1	12.0	100
1.057	K_β	2	22.0	5
1.177	K_α	2	24.7	40
1.177	K_α	3	38.7	10
1.177	K_α	4	58.2	5
34 Selenium (Pellet pressed from powder)				
0.992	K_β	1	10.1	25
1.106	K_α	1	11.3	100
0.992	K_β	2	20.6	10
1.106	K_α	2	23.1	55

Table 2 (Continued)
X-Ray Fluorescence Data for
the Common Elements

Wavelength (angstroms)	Line	Order (n)	Angle (θ)	Relative Intensity (I/I_0)
35 Bromine (Pellet pressed from CBr_4)				
0.933	K_β	1	9.5	15
1.041	K_α	1	10.6	100
0.933	K_β	2	19.3	15
1.041	K_α	2	21.7	70
	?		54.0	10
37 Rubidium (Pellet pressed from $RbNO_3$)				
0.829	K_β	1	8.4	50
0.927	K_α	1	9.4	100
0.829	K_β	2	17.1	15
0.927	K_α	2	19.2	55
0.829	K_β	3	26.1	10
0.927	K_α	3	29.5	15
	?		49.5	5
38 Strontium (Pellet pressed from $SrCO_3$)				
0.783	K_β	1	8.0	25
0.877	K_α	1	8.9	100
0.783	K_β	2	16.1	10
0.877	K_α	2	18.1	50
0.877	K_α	3	27.8	10
	?		47.5	5
39 Yttrium (Y_2O_3 pelletized with methyl cellulose)				
0.740	K_β	1	7.5	35
0.831	K_α	1	8.4	100
0.740	K_β	2	15.2	15
0.831	K_α	2	17.1	65
0.831	K_α	3	26.2	20
	?		46.1	5
40 Zirconium (Metal)				
0.701	K_β	1	7.1	20
0.788	K_α	1	8.0	100
0.701	K_β	2	14.4	10
0.788	K_α	2	16.2	50
0.788	K_α	3	24.7	10
0.788	K_α	4	33.9	5
	?		44.6	5
41 Niobium (Columbium) (Metal)				
0.644	K_β	1	6.8	40
0.748	K_α	1	7.6	100
0.644	K_β	2	13.7	20
0.748	K_α	2	15.4	70
0.644	K_β	3	20.8	10
0.748	K_α	3	23.4	30
0.748	K_α	4	32.0	5
	?		47.1	5

Table 2 (Continued)
X-Ray Fluorescence Data for
the Common Elements

Wavelength (angstroms)	Line	Order (n)	Angle (θ)	Relative Intensity (I/I_0)
42 Molybdenum (Metal)				
0.631	K_β	1	6.4	25
0.710	K_α	1	7.2	100
0.631	K_β	2	12.9	15
0.710	K_α	2	14.6	63
0.710	K_α	3	22.2	20
0.710	K_α	4	30.2	5
	?		42.0	2
44 Ruthenium (Small sample of Ru_2O_3 pelletized with methyl cellulose)				
0.643	K_α	1	6.5	100
0.643	K_α	2	13.2	60
0.643	K_α	3	20.0	25
45 Rhodium (Metal powder pelletized with methyl cellulose)				
0.544	K_β	1	5.6	20
0.614	K_α	1	6.2	100
0.544	K_β	2	11.2	10
0.614	K_α	2	12.6	60
0.614	K_α	3	19.1	15
0.614	K_α	4	25.8	5
46 Palladium (Metal powder pelletized with methyl cellulose)				
0.520	K_β	1	5.3	20
0.587	K_α	1	5.9	100
0.520	K_β	2	10.6	10
0.587	K_α	2	12.1	70
0.587	K_α	3	18.2	15
47 Silver (Pellet pressed from Ag_2O)				
0.497	K_β	2	10.1	20*
0.561	K_α	2	11.4	100
48 Cadmium (Metal)				
0.475	K_β	2	9.7	40*
0.536	K_α	2	10.9	100
0.536	K_α	3	16.5	20
	?		41.7	10
	?		44.7	20
49 Indium (Metal)				
0.455	K_β	1	4.6	20
0.514	K_α	1	5.2	100
0.455	K_β	2	9.3	20
0.514	K_α	2	10.5	60
	?		16.9	10
	?		39.3	10
	?		42.1	20

*Background sufficiently strong to obscure lines near 0°.

Table 2 (Continued)
X-Ray Fluorescence Data for
the Common Elements

Wavelength (angstroms)	Line	Order (n)	Angle (θ)	Relative Intensity (I/I_0)
50 Tin (Metal)				
0.435	K_β	2	8.9	30
0.492	K_α	2	10.0	100
0.492	K_α	3	15.1	30
	?		36.9	30
	?		39.6	30
51 Antimony (Metal)				
Three bands on a high background			1.8 3.1 4.6	40 90 100
0.417	K_β	2	8.5	5
0.472	K_α	2	9.6	20
0.472	K_α	3	14.5	10
	?		35.0	10
	?		37.2	20
52 Tellurium (Pellet pressed from powder)				
0.453	K_α	1	4.6	100
0.453	K_α	2	9.2	50
0.453	K_α	3	13.9	10
53 Iodine (Pellet pressed from sodium iodide)				
0.384	K_β	1	3.9	15
0.435	K_α	1	4.4	100
0.435	K_α	2	8.8	55
0.435	K_α	3	13.3	25
55 Cesium (CsCl pelletized with methyl cellulose)				
2.511	L_{β_2}	1	26.4	20*
2.683	L_{β_1}	1	28.4	100
56 Barium (Pellet pressed from $BaSO_4$)				
2.404	L_{β_2}	1	25.2	20
2.567	L_{β_1}	1	27.1	80
2.780	L_α	1	29.5	100
57 Lanthanum ($La_2(CO_3)_3$ pellitized with methyl cellulose)				
2.141	L_{γ_1}	1	22.3	10
2.303	L_{β_2}	1	24.1	20
2.410	L_{β_3}	1	25.3	10
2.458	L_{β_1}	1	25.8	90
2.670	L_α	1	28.3	100

*Background sufficiently strong to obscure some lines.

Table 2 (Continued)
X-Ray Fluorescence Data for
the Common Elements

Wavelength (angstroms)	Line	Order (n)	Angle (θ)	Relative Intensity (I/I_0)
58 Cerium (Pellet pressed from CeF_3)				
2.048	L_{γ_1}	1	21.3	15
	[La L_{γ_1}]	[1]	[22.3]	[10]
2.208	L_{β_2}	1	23.0	25
2.311	L_{β_3}	1	24.2	25
2.356	L_{β_1}	1	24.7	85
	[La L_{β_1}]	[1]	[25.8]	[60]
2.565	L_{α}	1	27.0	100
	[La L_{α}]	[1]	[28.3]	[65]
59-71 Lanthanons (Not Investigated)				
72 Hafnium (Metal)				
1.326	L_{β_2}	1	13.8	20*
1.374	L_{β_1}	1	14.1	45
1.575	L_{α}	1	16.2	100
1.326	L_{β_2}	2	28.0	10
1.374	L_{β_1}	2	29.1	10
1.575	L_{α}	2	33.8	30
	?		65.1	15
73 Tantalum (Metal)				
1.285	L_{β_2}	1	13.1	20*
1.327	L_{β_1}	1	13.6	45
1.527	L_{α}	1	15.7	100
1.285	L_{β_2}	2	27.1	10
1.327	L_{β_1}	2	28.0	10
1.527	L_{α}	2	32.8	25
74 Wolfram (Tungsten) (Metal)				
1.098	L_{γ_1}	1	11.3	10
1.244	L_{β_2}	1	12.8	30*
1.282	L_{β_1}	1	13.2	80
1.481	L_{α}	1	15.2	100
1.244	L_{β_2}	2	26.2	10
1.282	L_{β_1}	2	27.0	20
1.481	L_{α}	2	31.7	35
	?		62.7	5
75 Rhenium (Nitrate solution neutralized with NH_4OH and dried)				
1.061	L_{γ_1}	1	10.9	15
1.206	L_{β_2}	1	12.4	25*
1.238	L_{β_1}	1	12.7	70
1.439	L_{α}	1	14.8	100
1.206	L_{β_2}	2	25.4	15
1.238	L_{β_1}	2	26.0	20
1.439	L_{α}	2	30.7	25

*Lines L_{β_2} and L_{β_3} not resolved.

Table 2 (Continued)
X-Ray Fluorescence Data for
the Common Elements

Wavelength (angstroms)	Line	Order (n)	Angle (θ)	Relative Intensity (I/I_0)
76 Osmium (Metal powder pelletized with methyl cellulose)				
1.025	L_{γ_1}	1	10.5	5
1.171	L_{β_2}	1	11.9	30*
1.197	L_{β_1}	1	12.1	70
1.397	L_{α}	1	14.4	100
1.171	L_{β_2}	2	24.5	5
1.197	L_{β_1}	2	25.2	5
1.397	L_{α}	2	29.6	10
77 Iridium (Metal powder pelletized with methyl cellulose)				
1.15	L_{β}	1	11.7	95†
1.357	L_{α}	1	14.0	100
1.15	L_{β}	2	24.0	25
1.357	L_{α}	2	28.8	30
78 Platinum (Metal)				
1.11	L_{β}	1	11.4	85†
1.319	L_{α}	1	13.4	100
1.102	L_{β_2}	2	23.0	5*
1.120	L_{β_1}	2	23.4	25
1.319	L_{α}	2	27.9	25
79 Gold (Metal)				
0.927	L_{γ_1}	1	9.5	5
1.07	L_{β}	1	11.0	70†
1.283	L_{α}	1	13.2	100
	[Cu K_{α}]	[1]	[15.8]	5
1.07	L_{β}	2	22.5	25†
1.283	L_{α}	2	27.0	35
80 Mercury (Pellet pressed from Hg_2Cl_2)				
0.897	L_{γ_1}	1	8.8	20
1.04	L_{β}	1	10.6	60†
1.247	L_{α}	1	12.8	100
1.04	L_{β}	2	21.6	10†
1.247	L_{α}	2	26.2	30
81 Thallium (Tl_2O_3 pelletized with methyl cellulose)				
0.868	L_{γ_1}	1	8.9	20
1.01	L_{β}	1	10.3	85†
1.213	L_{α}	1	12.4	100
1.01	L_{β}	2	21.0	20†
1.213	L_{α}	2	25.5	25

*Lines L_{β_2} and L_{β_3} not resolved.
†Lines L_{β_1}, L_{β_2}, and L_{β_3} not resolved.

Table 2 (Continued)
X-Ray Fluorescence Data for
the Common Elements

Wavelength (angstroms)	Line	Order (n)	Angle (θ)	Relative Intensity (I/I_0)
82 Lead (Metal)				
0.840	L_{γ_1}	1	8.6	15
0.97	L_{β}	1	10.0	80*
1.180	L_{α}	1	12.1	100
0.97	L_{β}	2	20.3	30*
1.180	L_{α}	2	24.7	30
83 Bismuth (Metal)				
0.813	L_{γ_1}	1	8.3	10
0.95	L_{β}	1	9.7	70*
1.149	L_{α}	1	11.7	100
0.95	L_{β}	2	19.6	20*
1.149	L_{α}	2	24.1	35
90 Thorium (ThO_2 pelletized with methyl cellulose)				
0.653	L_{γ_1}	2	13.4	40
0.77	L_{β}	2	15.9	100*
0.962	L_{α}	2	20.0	60
92 Uranium ($UO_2SO_4 \cdot 7H_2O$ pelletized with methyl cellulose)				
0.720	L_{β_1}	1	7.3	20
0.755	L_{β_2}	1	7.7	25
0.911	L_{α}	1	9.3	100
0.720	L_{β_1}	2	14.8	20
0.755	L_{β_2}	2	15.5	15
0.911	L_{α}	2	19.0	40
0.911	L_{α}	3	29.2	15

*Lines L_{β_1}, L_{β_2}, and L_{β_3} not resolved.