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Chemistry

DETERMINATION OF ALUMINUM, SILICON,
IRON, URANIUM, AND THORIUM IN
BERYLLIUM BY X-RAY FLUORESCENCE (U)

G. E. Walden
Alma D. Condrey

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ABSTRACT

X-ray emission spectroscopy has been successfully applied to the determination of aluminum, silicon, iron, uranium, and thorium in beryllium. Beryllium metal was converted by pyrohydrolysis to the oxide and the oxide pressed into a hygienically safe pellet using a thermal-setting resin as a binder. Concentration levels for all elements except aluminum were those normally encountered in beryllium metal. Aluminum concentration was determined at levels up to 1.0%.

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INTRODUCTION

The analysis of trace levels of impurities in a beryllium matrix has been of considerable interest to many laboratories. Much research effort has been expended to improve the precision and economy of the methods of analysis for these impurities.

X-ray fluorescence has been utilized for the analysis of certain impurities in beryllium metal powder⁽¹⁾ or chip samples.⁽²⁾ The experience with these methods which required finely divided metal powders or extreme pressures to produce uniform pellets made further extension of X-ray techniques unattractive if not impractical. The health hazards involved in handling beryllium oxide preclude its use on a routine basis. However, with the availability of plasticizers, the possibility of preparing hygienically safe pellets appeared feasible. With such pellets, the routine analysis of a number of impurities in beryllium would be possible.

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SUMMARY

X-ray emission spectroscopy has been successfully applied to the determination of aluminum, silicon, iron, uranium, and thorium in beryllium. Strontium Ka energy served as an internal standard for uranium and thorium while its La₁ energy was utilized for aluminum and silicon. Cobalt was added as the internal standard for iron.

Samples of beryllium metal were pyrohydrolyzed at 900° C to the oxide. The oxide was blended with a thermal-setting phenolic resin, pressed into a pellet, and heat set to produce a hygienically safe pellet. Vacuum X-ray fluorescent techniques were used for the determination of aluminum and silicon; conventional approaches were employed for all other analyses.

An evaluation of the calibration data gave the following absolute limits of error (0.95) for a single measurement:

	<u>Concentration</u> %	<u>LE</u> %
Aluminum	0.015 - 0.25	± 0.015
	0.25 - 1.00	± 0.033
Silicon	0.016 - 0.25	± 0.016
Iron	0.0032 - 0.50	± 0.0032
Uranium and Thorium	0.0010 - 0.050 (10 - 500 µg/gm)	± 0.0010

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ANALYTICAL PROCEDURE

PRECAUTIONS

Analysts working with beryllium metal and its oxide should become familiar with the hazards involved.⁽³⁾ Beryllium metal turnings present no particular hazard; however, the oxide obtained by converting the metal must be handled with extreme caution since it is a very fine powder.

EQUIPMENT

X-Ray Spectrometers

The X-ray spectrometer used for aluminum and silicon was equipped with a chromium target X-ray tube, a gypsum analyzing crystal, a vacuum path, a flow proportional counter, and a suitable counting and recording device with a single-channel pulse height analyzer.

The X-ray spectrometer used for iron, uranium, and thorium was equipped with a lithium fluoride analyzing crystal, a molybdenum target X-ray tube, a scintillation detector, 0.010-inch exit and receiving collimators, a 1 3/8-inch Teflon sample cup, and an electronic panel with a single-channel pulse height analyzer.

Glove Box

A standard-design glove box, equipped with an analytical balance, was used and maintained at a slightly reduced pressure by exhausting through an absolute filter.

Muffle Furnace

A platinum-lined furnace, capable of reaching 900° C, and equipped with a standard steam generator, was used. A slight flow of air was used to force steam through the furnace.

Platinum Dish

A platinum dish having a 100-milliliter capacity was used.

Blender

A Mixer/Mill was used to blend the powder.

Die

A standard 1 1/4-inch-diameter die was used.

Press

A 20-ton capacity laboratory press was used.

Vials

Plastic vials, 1 1/4 by 3 inches, were used.

Ball Pestle

A 3/8-inch-diameter plastic ball pestle was used.

REAGENTS

A phenolic resin-internal standard mixture, consisting of 70.0 milligrams of strontium carbonate, 60 milligrams of cobalt oxide, and 20.000 grams of a commercial phenolic resin powder, (a) was blended on a Mixer/Mill for 20 minutes. Other reagents used included: uranium oxide (U_3O_8), -325 mesh powder; thorium oxide (ThO_2), -325 mesh powder; ferric oxide (Fe_2O_3), -325 mesh powder; aluminum oxide (Al_2O_3), -325 mesh powder; silicon dioxide (SiO_2), -325 mesh powder; and spectrographically pure beryllium oxide (BeO), -200 mesh powder.

SAMPLE PREPARATION

Observe the following procedure in preparing the beryllium samples:

1. Wash the beryllium metal chip samples with acetone to remove grease, rinse with ether, and allow to air dry. (Omit this step for beryllium metal powder samples.)
2. Weigh duplicate 2.000-gram portions of the sample into numbered 100-milliliter platinum dishes.
3. Place the dishes into a platinum-lined, 900°C muffle furnace.
4. Connect the steam line from the steam generator to the furnace.

(a) Union Carbide BPR 5905.

5. Sweep steam through the furnace with a gentle flow of air for two hours to convert the beryllium metal to beryllium oxide.
6. Remove the dishes from the furnace and cool them in the hood. (Caution: Beryllium oxide presents a dust hazard.)
7. Transfer (in the hood) the beryllium oxide quantitatively to a plastic vial containing 1.000 gram of the phenolic resin-internal standard mixture and two 3/8-inch plastic balls.
8. Place each vial in a plastic bag and tape securely. Mix in a Mixer/Mill for 15 minutes.
9. Transfer (in the hood) the sample to the pellet die, assemble the die, encase the die in a plastic bag, and seal the bag with tape.
10. Press at 20 tons for 30 seconds with a standard laboratory press.
11. Remove the pellet from the die, place it on a flat polished surface, and cure it in an oven at 120° C for 20 minutes.
12. Store the pellets in clean plastic vials until ready for analysis.

ANALYSIS

Steps in the analysis for the various impurities are given in the sections that follow.

Aluminum and Silicon

1. Set the X-ray generator for 50 kv and 36 ma. Allow a few minutes for the X-ray beam to stabilize.
2. Turn on the vacuum pump.
3. Determine the pulse height analyzer (PHA) baseline settings for strontium, silicon, and aluminum. (A window width of 12 volts should permit a common baseline setting for both strontium and silicon.)
4. Transfer the sample pellet to a plastic holder and position the pellet in the X-ray beam. (Care must be exercised to prevent the plastic holder from becoming contaminated with silicone oil from the vacuum chamber.)
5. Allow the system to evacuate to 0.1 torr or less.
6. Set the electronic panel to a "preset time" of four minutes.

7. Set the PHA for the setting determined for strontium and silicon, and count those positions for four minutes each.
8. Set the PHA for the setting determined for aluminum and count the background and aluminum peak.
9. Obtain the net counts per four minutes (c/4 min) by subtracting the background c/4 min from the gross peak c/4 min.
10. Calculate the intensity ratios for silicon and aluminum by dividing each net c/4 min by the net c/4 min obtained for strontium.
11. Calculate the percent silicon and aluminum by substituting the intensity ratio in the appropriate calibration equation.

Uranium, Thorium, and Iron

1. Set the X-ray generator for 50 kv and 25 ma. Allow a few minutes for the X-ray beam to stabilize.
2. Adjust the helium flow rate to four cubic feet per hour.
3. Determine the pulse height analyzer (PHA) baseline setting for the element or elements of interest.
4. Set the goniometer to the counting positions given in Table 1 and count in the "fixed-count" position for a length of time in excess of 90 seconds at each position.
5. Calculate the counts per second (c/s) for each element and background by dividing the total counts accumulated by the time in seconds.
6. Calculate the net c/s for each element by subtracting from the c/s for the element the c/s obtained for the background.
7. Calculate the intensity ratios for the element of interest by dividing its net c/s by the net c/s obtained for the appropriate internal standard.
8. Calculate the concentration for each element from the intensity ratio and the appropriate calibration equation.

CALIBRATION OF THE INSTRUMENT

To calibrate the X-ray spectrometer, the following steps are suggested:

1. Prepare a master mix to contain 5000 $\mu\text{g}/\text{gm}$ each of iron, aluminum, and silicon, and 1000 $\mu\text{g}/\text{gm}$ each of uranium and thorium by weighing out: 4.7 milligrams

Table 1
OPERATING PARAMETERS FOR DETERMINING IMPURITIES IN BERYLLIUM

Element	Emission	Theoretical Angle		Excitation		Counting Equipment (voltage)		
		Gypsum	LiF	kv	ma	Baseline	Window	Detector
Strontium	La ₁	53.72	-	50	36	36	12	1860
Silicon	Ka ₁₋₂	55.95	-	50	36	36	12	1860
Background	-	60.00	-	50	36	28	12	1860
Aluminum	Ka ₁₋₂	66.59	-	50	36	28	12	1860
Strontium	Ka ₁₋₂	-	25.15	50	25	24	20	1050
Uranium	La ₁	-	26.14	50	25	24	20	1050
Thorium	La ₁	-	27.46	50	25	24	20	1050
Background	-	-	28.50	50	25	24	20	1050
Background	-	-	50.50	50	25	9	20	1060
Cobalt	Ka ₁₋₂	-	52.80	50	25	9	20	1060
Iron	Ka ₁₋₂	-	57.52	50	25	9	20	1060

of U₃O₈, 4.6 milligrams of ThO₂, 28.6 milligrams of Fe₂O₃, 37.8 milligrams of Al₂O₃, 42.8 milligrams of SiO₂, and 10.9827 grams of BeO. Blend this mixture for 20 minutes in a Mixer/Mill.

2. Prepare duplicate standards at each concentration level from the data given in Table 2.
3. Blend each mixture for 20 minutes in a Mixer/Mill.

Table 2
COMPOSITION OF STANDARDS FOR THE ANALYSIS OF IMPURITIES IN BERYLLIUM

Composition		Grams Added				
Fe, Al, Si (%)	U, Th (µg/gm)	Master Mix ⁽¹⁾	BeO	Resin Mix ⁽²⁾	Al ₂ O ₃	Fe ₂ O ₃
Blank	Blank	0.00	5.5504	1.000	0.0	0.0
0.0100	20	0.1110	5.4394	1.000	0.0	0.0
0.0250	50	0.2775	5.2729	1.000	0.0	0.0
0.0500	100	0.5550	4.9954	1.000	0.0	0.0
0.1000	200	1.1100	4.4404	1.000	0.0	0.0
0.2500	500	2.7752	2.7752	1.000	0.0	0.0
0.5000	-	0.00	5.5172	1.000	0.0189	0.0143
1.000	-	0.00	5.5126	1.000	0.0378	0.0

(1) Master mix contains: 4.7 mgs of U₃O₈, 4.6 mgs of ThO₂, 28.6 mgs of Fe₂O₃, 37.8 mgs of Al₂O₃, 42.8 mgs of SiO₂, and 10.9827 gms of BeO.

(2) Resin-internal standard mix contains: 20.000 gms of resin, 70.0 mgs of SrCO₃, and 60.0 mgs of Co₂O₃.

4. Press each mixture into a pellet as per Steps 9 - 12 of the Sample Preparation Procedure (Page 12).
5. Analyze for each element following the appropriate procedures [Steps 1 - 10 of the Aluminum and Silicon Procedure (Page 13), and Steps 1 - 7 of the Uranium Iron, and Thorium Procedure (Page 14)] .
6. Plot the concentration of each element as a function of its intensity ratio.
7. Calculate the equation of the curve. Since each relationship is linear, equations should take the form: $Y = mX + b$.

DISCUSSION

Although satisfactory pellets can be prepared from beryllium metal chips,⁽²⁾ the technique is not readily applicable to large numbers of routine samples. Early efforts to analyze aluminum in beryllium involved the grinding of beryllium chip samples in a beryllium-lined crucible in a heavy-duty shaker.⁽⁴⁾ About 45 minutes of grinding were required to produce 10 grams of -40 mesh beryllium powder from normal turnings or chips. Since this approach also presented some difficulty in preparing satisfactory standards, it was abandoned. If X-ray fluorescence is to be applied to the analysis of a number of impurities in beryllium, some more suitable form of sample preparation will be required.

Beryllium metal can be converted quantitatively to the oxide by pyrohydrolysis. The conversion takes place at 900° C very rapidly if a slight flow of air is used to force steam into the furnace. Although the dilution of the sample by converting to the oxide was undesirable for the determination of low levels of aluminum and silicon, this approach was more suited to routine applications than the other methods if the health hazards involved in handling the oxide could be overcome. Research efforts were then directed toward the preparation of hygienically safe pellets from beryllium oxide. After evaluating a number of standard binders without success, some pellets were prepared utilizing a commercial phenolic resin. These pellets were heated on a flat metal sheet at 120° C for about 20 minutes. This heating process partially cured the resin and produced a brick-like disc that could be handled without chipping, breaking, or crumbling. This technique of pyrohydrolyzing the metal and binding the beryllium oxide with a phenolic resin made feasible the application of X-ray fluorescence to the task of analyzing for a number of impurities. Generally speaking, only elements which are volatile at 900° C are outside the scope of such a procedure.

Synthetic standards were prepared for the calibration of the X-ray spectrometers for aluminum, silicon, iron, uranium, and thorium (Table 2). High-purity beryllium oxide containing less than 100 ppm total impurities was used as a base material; reagent-grade oxides were used for the other elements. All oxides except beryllium oxide were dried and ground to -325 mesh prior to using. In the case of iron, grinding to -325 mesh was necessary to overcome particle-size effects. For all other elements this particle-size effect disappeared at -200 mesh.

Internal standards were added for each element of interest in this procedure. Initial efforts to analyze iron without the benefit of internal standards produced limits of error which were considered excessively high. A dual use has been made of strontium in this procedure. Strontium Ka energy at 0.926 angstrom serves as an internal standard for uranium and thorium. Strontium La₁ energy at 6.863 angstroms is a satisfactory internal standard for aluminum and silicon. To use strontium in both cases, some compromise in the amount of strontium added was necessary. The technique of adding the internal standards with the resin was very satisfactory in that it saved con-

siderable time since batch quantities of the mix could be prepared and three constituents added in one weighing. Also, the precision with which the internal standards could be added was improved considerably.

Analysis of synthetic standards followed the conditions outlined in Table 1. Standards were prepared in duplicate and were analyzed on successive days to check for instrument setup and stability. The data obtained from these standards are presented in Tables 3 - 7 and plotted in Figures 1 - 4. Statistical evaluations and concentration ranges are summarized in Table 8. The calibration equation for aluminum has been broken into two levels, 0.0 - 0.25% and 0.25 - 1.00%. Evaluations of data

Table 3
CALIBRATION DATA FOR ALUMINUM IN BERYLLIUM

Aluminum Concentration (%)	Pellet Number	Intensity Ratio	
		Day 1	Day 2
Blank	1	0.0839	0.0981
	2	0.0924	0.0865
0.01	1	0.0979	0.1101
	2	0.0946	0.1385
0.025	1	0.1513	0.1502
	2	0.1726	0.1526
0.050	1	0.1897	0.1997
	2	0.1916	0.1824
0.10	1	0.3636	0.3786
	2	0.3644	0.3686
0.25	1	0.7496	0.7133
	2	0.7271	0.7177
0.50	1	1.480	1.572
	2	1.474	1.431
1.0	1	3.004	2.934
	2	3.052	3.096

through 0.25% gave absolute limits of error (0.95) of $\pm 0.015\%$ or 150 $\mu\text{g}/\text{gm}$ for a single measurement. Thus, the method can be applied satisfactorily to normal materials with a precision comparable to other techniques. At higher concentration levels the precision becomes poorer.

Some discussion of the slope-intercept values is appropriate since there is an intercept value in each case equivalent to or greater than the limits of error for the method. These values, in all cases except silicon, arise from counting levels of the background since no element was present in the base material at more than 10 $\mu\text{g}/\text{gm}$. For silicon, the high-intercept value arises from the silicone grease used as a lubricant in the vacuum chamber. The counting rate could be controlled at a minimum level by using only a minimum of silicone oil in the chamber. Some care must be

Table 4
CALIBRATION DATA FOR SILICON IN BERYLLIUM

Silicon Concentration (%)	Pellet Number	Intensity Ratio	
		Day 1	Day 2
Blank	1	0.8564	0.8270
	2	0.8016	0.8157
0.01	1	0.9236	0.9369
	2	0.9558	0.9810
0.025	1	1.247	1.267
	2	1.275	1.314
0.050	1	1.569	1.455
	2	1.566	1.643
0.10	1	2.000	1.966
	2	1.943	2.084
0.25	1	4.070	3.870
	2	3.812	3.823

Table 5
CALIBRATION DATA FOR IRON IN BERYLLIUM

Iron Concentration (%)	Pellet Number	Intensity Ratio	
		Day 1	Day 2
Blank	1	0.0549	0.0597
	2	0.0487	0.0518
0.01	1	0.1154	0.1224
	2	0.1258	0.1283
0.025	1	0.2227	0.2281
	2	0.2190	0.2232
0.050	1	0.3974	0.3956
	2	0.4009	0.4045
0.10	1	0.7224	0.7239
	2	0.7221	0.7262
0.25	1	1.673	1.677
	2	1.670	1.689
0.50	1	3.294	3.293
	2	-	-

exercised in handling the pellets to prevent silicon contamination from the chamber. An effort was made to find a nonsilicone lubricant for the chamber but without success.

In the apparatus for converting the beryllium metal, it was found to be necessary to insert a trap to collect the water that condensed in the steam tube. Any water carried into the furnace dropped into the samples and caused some scattering of the oxide. The beryllium metal sample should be spread loosely over the bottom of the crucible so that the pyrohydrolysis can take place rapidly.

Table 6
CALIBRATION DATA FOR URANIUM IN BERYLLIUM

Uranium Concentration (%)	Pellet Number	Intensity Ratio	
		Day 1	Day 2
Blank	1	0.0144	0.0175
	2	0.0113	0.0144
20	1	0.0182	0.0212
	2	0.0185	0.0177
50	1	0.0267	0.0280
	2	0.0252	0.0270
100	1	0.0375	0.0350
	2	0.0380	0.0387
200	1	0.0593	0.0634
	2	0.0659	0.0603
500	1	0.1351	0.1354
	2	0.1360	0.1356

Table 7
CALIBRATION DATA FOR THORIUM IN BERYLLIUM

Thorium Concentration ($\mu\text{gTh/gm}$)	Pellet Number	Intensity Ratio	
		Day 1	Day 2
Blank	1	- 0.0161	- 0.0163
	2	- 0.0167	- 0.0178
20	1	- 0.0118	- 0.0124
	2	- 0.0119	- 0.0125
50	1	- 0.0057	- 0.0063
	2	- 0.0059	- 0.0041
100	1	0.0042	0.0054
	2	0.0062	0.0057
200	1	0.0276	0.0283
	2	0.0313	0.0318
500	1	0.1015	0.0996
	2	0.1014	0.1015

Table 8
SUMMARY OF THE STATISTICAL EVALUATION FOR ALUMINUM, SILICON,
IRON, URANIUM, AND THORIUM IN BERYLLIUM

Element	Concentration Range (%)	Calibration Equation	Absolute LE (%)
Aluminum	0.0 - 0.25	% Al = 0.342 x Ratio - 0.021	± 0.015
Aluminum	0.25 - 1.0	% Al = 0.342 x Ratio - 0.021	± 0.033
Silicon	0.0 - 0.25	% Si = 0.083 x Ratio - 0.073	± 0.016
Iron	0.0 - 0.50	% Fe = 0.1547 x Ratio - 0.0100	± 0.0032
Uranium	0.0010 - 0.0500 (10 - 500 $\mu\text{g/gm}$)	$\mu\text{gU/gm} = 4123.0 \times \text{Ratio} - 57.8$	± 0.0010 (10.0 $\mu\text{g/gm}$)
Thorium	0.0010 - 0.0500 (10 - 500 $\mu\text{g/gm}$)	$\mu\text{gTh/gm} = 4237.0 \times \text{Ratio} + 73.1$	± 0.0010 (10.0 $\mu\text{g/gm}$)

Figure 1. CALIBRATION CURVE FOR ALUMINUM IN BERYLLIUM.

Figure 2. CALIBRATION CURVE FOR SILICON IN BERYLLIUM.

Figure 3. CALIBRATION CURVE FOR IRON IN BERYLLIUM.

Figure 4. CALIBRATION CURVES FOR URANIUM AND THORIUM IN BERYLLIUM.

To evaluate the new procedure as fully as possible, it was first applied to the analysis of New Brunswick Laboratory beryllium standards for aluminum and silicon. Excellent agreement was obtained except for one value for silicon (Table 9). Possibly some contamination occurred for this sample. The procedure was also applied to some routine samples as well as NBL standards for iron, and the results compared to the ortho-phenanthroline colorimetric method (Table 10). No significant differences were observed. No similar comparisons for uranium and thorium were made although a number of samples were analyzed for the two elements using this technique.

Table 9
ANALYSIS OF THE NEW BRUNSWICK LABORATORY
BERYLLIUM STANDARDS

Standard Number	X-ray Value ($\mu\text{g}/\text{gm}$)		Accepted Value ($\mu\text{g}/\text{gm}$)	
	Aluminum	Silicon	Aluminum	Silicon
85	327	572	300	580
86	1657	696	1600	650
87	1494	1410	1500	1300
88	1514	1847	1500	1450
224	650	439	630	486

Table 10
COMPARISON OF ANALYTICAL METHODS FOR IRON

Sample Number	Analytical Method	
	Colorimetric	X-ray Fluorescence
44	1319	1347
45	1368	1350
46	1146	1174
47	1228	1247
48	1353	1416
NBL 224	1335 ⁽¹⁾	1329
NBL 225	1426 ⁽¹⁾	1418

(1) Accepted value.

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REFERENCES

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