CALCIUM-LEAD ALLOY ANALYSIS BY ATOMIC
ABSORPTION AND EMISSION SPECTROSCOPY

THE DOW CHEMICAL COMPANY
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CALCIUM-LEAD ALLOY ANALYSIS BY ATOMIC ABSORPTION AND EMISSION SPECTROSCOPY

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Calcium-Lead Alloy Analysis by Atomic Absorption and Emission Spectroscopy

Harlan N. Barton, Andrew J. Johnson, and Glenn A. Shepherd

Abstract. Described are the analysis of a calcium-lead alloy by atomic absorption spectrometry and emission spectrography. Calcium in the 0.04- to 0.10-percent range is determined by an atomic absorption method. Samples are dissolved in nitric acid, lanthanum added to eliminate possible interferences, and the samples aspirated into an acetylene air flame. Recoveries on 11 synthetic samples averaged about 100 percent. The relative standard deviation on 11 aliquots of the same sample was 0.30 percent on a sample containing 640 parts per million (ppm) of calcium.

A semiquantitative emission-spectrographic method is used for the determination of 46 impurity elements. The alloy is converted to oxide, diluted with graphite, and excited by a DC arc. Spectra are recorded photographically and visually compared with standard spectra.

INTRODUCTION

The preparation of a new calcium-lead alloy at Rocky Flats required the development of analytical methods to ensure that the alloy composition met specifications. The relative accuracy of analysis requirements for the impurity concentrations were as follows: (1) for concentrations greater than 100 ppm; an accuracy of ± 50 percent, and (2) for concentrations from 0.01 to 1.0 percent, an accuracy of ± 20 percent. Calcium would have a nominal concentration of 0.07 percent and a relative accuracy requirement of ± 20 percent.

The initial preparation of the alloy was expected to be troublesome, as previous investigations¹ found there were processing problems in holding the calcium content to close limits at the low calcium content desired. Thus, a casting was analyzed as soon as possible after pouring to ensure that the calcium content was within the 0.04- to 0.1-percent range specified. This was especially important during the initial stages of the preparation when various methods of alloying were being tested.

¹ A. B. Townsend. *The Effect of Casting Variables and Heat Treatment on the Tensile Properties of Lead-0.02 to 0.10 Weight Percent Calcium Alloy*. Report Y-1307. Oak Ridge National Laboratory, Oak Ridge, Tennessee. 1960.

The desirability of a relatively fast method for a single element and the level of calcium content to be determined suggested the use of atomic absorption spectrometry. Previous experience of the plant group with a lead-tin alloy analysis indicated good accuracy and precision were possible in the range of 0.04 to 0.1 percent of an alloying metal. Therefore, a method has been developed for the calcium determination by atomic absorption spectrometry.

Emission spectroscopy appeared to offer the best choice for the determination of the remaining impurities. A large number of impurity elements, a total of 46, could be determined rapidly, along with the accuracy level required.

Because of the differences in the two methods used for the analysis of the alloy, the remainder of the report has been divided into two parts. The atomic absorption method is described in Part I and the emission spectrographic method in Part II.

SUMMARY

The analysis of a calcium-lead alloy for the concentration of the alloying constituent and for impurities has been accomplished by combined spectroscopic methods. The concentration of calcium is found using atomic absorption spectrometry, while the determination of 46 impurity elements is accomplished by emission spectrography.

Specifications on the alloy required the calcium content of the casting to be within the 0.04- to 0.1-percent range. The material to be analyzed, the amount of calcium to be determined, and the need for a fast report of the calcium concentration made atomic absorption analysis an attractive choice. The analytical procedure developed gave good analytical recoveries and a relatively low standard deviation.

The sample is dissolved in nitric acid (HNO_3), lanthanum (La) added to eliminate the effect of interfering ions, and the solution aspirated into the burner of a Perkin-Elmer 303 Atomic Absorption Spectrophotometer. The ten-centimeter (cm), laminar-flow burner was used with an acetylene air flame.

Eleven synthetic samples were prepared and analyzed. The average recovery was 101.1 percent, with individual sample recoveries in the 97- to 106-percent range. The relative standard deviation calculated for 11 aliquots of a production sample was 0.30 percent at a 640-ppm calcium concentration.

An emission spectrographic method provided for the determination of the following impurity elements in the calcium-lead alloy:

Arsenic (As)	Molybdenum (Mo)
Barium (Ba)	Nickel (Ni)
Beryllium (Be)	Niobium (Nb)
Cadmium (Cd)	Palladium (Pd)
Calcium (Ca)	Platinum (Pt)
Cesium (Cs)	Potassium (K)
Chromium (Cr)	Rhenium (Re)
Cobalt (Co)	Rhodium (Rh)
Copper (Cu)	Ruthenium (Ru)
Dysprosium (Dy)	Scandium (Sc)
Europium (Eu)	Sodium (Na)
Gadolinium (Gd)	Strontium (Sr)
Germanium (Ge)	Tantalum (Ta)
Gold (Au)	Tellurium (Te)
Hafnium (Hf)	Terbium (Tb)
Holmium (Ho)	Thulium (Tm)
Indium (In)	Tin (Sn)
Iridium (Ir)	Titanium (Ti)
Lutetium (Lu)	Tungsten (W)
Magnesium (Mg)	Vanadium (V)
Manganese (Mn)	Ytterbium (Yb)
Mercury (Hg)	Zinc (Zn)
	Zirconium (Zr)

The method consisted of conversion of the metal sample to lead oxide (PbO), dilution with graphite, and excitation by a DC arc in a crater electrode. Four determinations were required to achieve the wavelength coverage for the analysis of the previously listed elements with the spectrographic equipment. Spectra recorded in the 3375- to 4575-angstrom (Å) region were arced in an oxygen-argon atmosphere to reduce cyanogen interference.

PART I - EXPERIMENTAL

Instrumental: A Perkin-Elmer 303, Atomic Absorption Spectrophotometer was employed for all analytical measurements. The double-beam instrument was used

with a 10-cm, laminar-flow burner and acetylene air fuel. The instrumental parameters are given in Table I.

TABLE I. Atomic-Absorption Instrumental Parameters.

Wavelength	4227	angstroms
Lamp Current	15	milliamperes
Slit Width	1	millimeter
Scale Expansion	1	X
Air (Flowmeter Reading)	7.5	
Acetylene (Flowmeter Reading)	-9.0*	

* Exact setting made by aspirating calcium standard and adjusting acetylene flow to obtain maximum absorption.

Standard Preparation: The standards used to prepare working curves for the analysis contained calcium (Ca), lead (Pb), and lanthanum (La). Lanthanum was added to both the standards and the samples to eliminate the effect of interfering ions that might be present.² The following solutions were used in the preparation of the standards:

Calcium Master

Solution: A master solution of 400-ppm Ca was prepared by dissolution of reagent-grade calcium carbonate (CaCO₃) in approximately 0.1 N HNO₃.

Lead Stock

Solution: Reagent-grade Pb shot was dissolved in a small amount of HNO₃ and brought to volume with distilled water. Final solution contained 10-percent Pb by weight.

Lanthanum Stock

Solution: A 5-percent La solution was prepared by dissolving 15.5 grams of reagent-grade lanthanum nitrate [La(NO₃)₃] in 25 milliliters (ml) of concentrated HNO₃ and diluting to a 100-ml final volume.

The standard solutions were made up to cover the 1 to 25 ml/l-Ca range. Each standard, in a final volume of 25 ml, also contained 2-percent Pb and 3 ml of the 5-percent, La stock solution. Analytical curves were drawn from standards analyzed concurrently with the samples.

² *Analytical Methods for Atomic Absorption Spectrophotometry*. Perkin-Elmer Corporation, Norwalk, Connecticut. 1966.

Sample Preparation: Samples of the Pb alloy weighing 0.4 to 0.5 grams each were dissolved in 2 ml of concentrated HNO_3 and then 10 ml of water added. After dissolution was complete, 3 ml of the 5-percent La stock solution were added and the solution diluted to a final volume of 25 ml with distilled water. These sample solutions, containing approximately 2-percent Pb and 15-ppm Ca, were then aspirated directly into the burner.

RESULTS AND DISCUSSION

Accuracy: There were no standard samples available of a Pb-Ca alloy to check the accuracy of the method. Synthetic samples were therefore prepared using solutions of Ca and Pb, and submitted as unknowns. All sample solutions contained 2-percent Pb. The results obtained on these samples are given in Table II. The results indicated the absence of any bias in the analysis.

TABLE II. Results on Synthetic Samples.
(Values in parts per million, based on lead.)

Calcium Added	Calcium Found	Percent Recovery
819	812	99.2
1229	1196	97.3
1065	1037	97.3
901	875	97.1
328	338	103.1
573	584	101.9
737	768	104.2
819	829	101.2
655	681	104.0
410	402	98.1
246	261	106.1
737	768	104.2
Average		101.1

Precision: A 10-gram portion of a production sample was dissolved as in the regular sample procedure and brought to a final volume of 100 ml. Five-ml aliquots of this solution, along with 3 ml of the La stock solution, were each diluted to 25 ml and aspirated into the burner. At a Ca level of 640 ppm, the relative standard deviation was found to be 0.30 percent on the 11 aliquots analyzed.

Homogeneity of Received Samples: The material, as received from production, was in the form of several thick turnings per sample. In order to check the homogeneity of each sample submitted, specimens of alloy from each turning within a given sample were

selected. These were then individually weighed and analyzed as separate samples.

From the results shown typically in Table III, the samples submitted were homogeneous with respect to different turnings within a given sample.

TABLE III. Calcium Content of Portions from One Sample.

<u>Sample Weight</u> (grams)	<u>*Calcium Content</u> (parts per million)
0.4995	641
0.5026	633
0.5118	645
0.5078	628
0.5087	632
0.5212	616
0.5308	623
0.5238	611
0.5247	610
0.5110	626
0.4876	<u>628</u>
Average	627

*Relative Standard Deviation 1.56 percent.

Development work on the formulation of the Pb-Ca alloy and its permissible variation in Ca content was performed at the Oak Ridge, Y-12 Plant (Tennessee). The Y-12 achieved the calcium determination by flame photometry. This determination, developed before the advent of atomic absorption spectroscopy, had a relative standard deviation of approximately 10 percent.³ The use of an atomic-absorption method at the Y-12 Plant enabled the lowering of the relative standard deviation to about one percent.

PART II - EXPERIMENTAL

Reagents:

1. Lead nitrate [$\text{Pb}(\text{NO}_3)_2$].
2. G Standards, Spex Industries, Metuchen, New Jersey, [1000, 100, 10, 1 micrograms per gram ($\mu\text{g/g}$) of 49 elements in graphite].
3. Rare Earth Spex Mix, Spex Industries, Metuchen, New Jersey, [mixture of rare earth, yttrium (Y), and scandium (Sc) oxides].
4. Noble Metal G Standards, Spex Industries, Metuchen, N. J., (5000, 500, 50, 5 $\mu\text{g/g}$ of 10 noble elements in graphite).

³A. B. Townsend. *Loc. cit.*

Standard Preparation: Separate standards in a PbO and graphite mixture were prepared for elements contained in spex-mix standards, rare-earth spex mix, and noble metal standards. Lead nitrate $[Pb(NO_3)_2]$ was found to contain the lowest concentrations of impurities of interest of available Pb compounds tested. The nitrate was converted to oxide (PbO) by heating to 500°C in a muffle furnace.

Standards containing elements of interest in Spex Mix (listed below) were prepared by mixing varying proportions of powdered PbO, graphite, and G1- and G2-Spex Standards as shown in Table IV.

Antimony (Sb)	Molybdenum (Mo)
Arsenic (As)	Nickel (Ni)
Barium (Ba)	Niobium (Nb)
Beryllium (Be)	Potassium (K)
Cadmium (Cd)	Rubidium (Rb)
Calcium (Ca)	Sodium (Na)
Cesium (Cs)	Strontium (Sr)
Chromium (Cr)	Tantalum (Ta)
Cobalt (Co)	Tellurium (Te)
Copper (Cu)	Tin (Sn)
Germanium (Ge)	Titanium (Ti)
Magnesium (Mg)	Tungsten (W)
Manganese (Mn)	Vanadium (V)
Mercury (Hg)	Zinc (Zn)
	Zirconium (Zr)

TABLE IV. Preparation of Spex-Mix Standards.

Concentration (micrograms per gram)	Spex Standard Added (milligrams)	Graphite Added (milligrams)	Lead Oxide Added (milligrams)
500	100 of G1	0	215.4
200	40 of G1	60	215.4
100	20 of G1	80	215.4
50	10 of G1	90	215.4
20	40 of G2	60	215.4
10	20 of G2	80	215.4
Blank	0	100	215.4

Standards containing noble metal elements (listed below) were prepared as shown in Table V.

Gold (Au)	Palladium (Pd)
Hafnium (Hf)	Platinum (Pt)
Indium (In)	Rhenium (Re)
Iridium (Ir)	Rhodium (Rh)
	Ruthenium (Ru)

TABLE V. Preparation of Noble Metal Standards.

Concentration (micrograms per gram)	Noble Metal Standard Added (milligrams)	Graphite Added (milligrams)	Lead Oxide Added (milligrams)
500	20 of G1	80	215.4
200	8 of G1	92	215.4
100	40 of G2	60	215.4
50	20 of G2	80	215.4
20	8 of G2	92	215.4
10	40 of G3	60	215.4
Blank	0	100	215.4

Standards containing rare earth elements of interest (listed below) were prepared as shown in Table VI. Graphite standards of rare earth elements were not available; therefore these were prepared by mixing appropriate amounts of graphite and Rare Earth Spex Mix (5.28 percent of each element).

Dysprosium (Dy)	Lutetium (Lu)
Europium (Eu)	Scandium (Sc)
Gadolinium (Gd)	Terbium (Tb)
Holmium (Ho)	Thulium (Tm)
	Ytterbium (Yb)

TABLE VI. Preparation of Rare Earth Standards.

Concentration (micrograms per gram)	Rare Earth Standard Added (micrograms per gram)	Graphite Added (milligrams)	Lead Oxide Added (milligrams)
500	20 of 5000	80	215.4
200	8 of 5000	92	215.4
100	40 of 500	60	215.4
50	20 of 500	80	215.4
20	8 of 500	92	215.4
10	40 of 50	50	215.4
Blank	0	100	215.4

Spex-mix standards were analyzed using Parameter Sets 1, 2, 3, and 4 (see Exposure and Excitation Conditions Section, Table VII).

Rare-earth standards were analyzed using Parameter Set 2.

Noble metal standards were analyzed using Parameter Sets 1 and 2.

Sample Preparation: A 300- to 500-milligram (mg) Pb sample was dissolved in 8 N HNO_3 with heat and evaporated to dryness. The sample was converted to PbO by heating 15 to 30 minutes at 470 to 500°C. The

oxide was ground in a plastic vial with a plastic bar and a Wig-1-Bug® dental amalgamator. One-hundred mg of powdered graphite and 215.4 mg of the oxide were mixed with the Wig-1-Bug. Four 50-mg aliquots were loaded into electrodes and tapped to settle at the crater bottom.

Electrode System: The lower sample anode was ASTM Type S-12 (Ultra Carbon 100-L). The counter electrode was ASTM C-1 (Ultra Carbon 1992).

Spectrographic Apparatus:

1. SPECTROGRAPH, Jarrell-Ash 3.4 meter Ebert with 30,000 lines per inch grating.
2. SOURCE UNIT, National Spectrographic Laboratories Model No. 110-10.

Exposure and Excitation Conditions:

1. NONVARIABLE CONDITIONS –
 - a. Spherical condensing lens.
 - b. Entrance slit height, 1.7 millimeters.
 - c. Filter: Neutral, two-step, 10 and 100 percent transmission.
 - d. DC Arc: 16 amperes (No. 6 position, high range).
 - e. Analytical Gap: 4 millimeters.
 - f. Plate Processing: Kodak D-19, 5 minutes, 18.5°C.
2. VARIABLE CONDITIONS – Four determinations were made for each sample to permit spectral recording of 4 wavelength regions. The variable parameters used for each are shown in Table VII.

A Stallwood Jet was used for achieving the argon-oxygen atmosphere for Parameter Set 2. A flow rate of 13 cubic feet per hour (ft³/hr) of 20-percent oxygen and 80-percent argon was used.

Determination of Results: Concentrations were determined by visual comparison with standard spectra.

RESULTS AND DISCUSSION

The results of moving plate studies are shown in Figures 1 and 2. Intensities shown in Figures 1 and 2 are estimates based on visual observation of line intensities. The use of split exposures for elements determined in the 2200- to 3400-Å region (Parameter Set 1) was attempted for rapid evolving elements arsenic (As), cadmium (Cd), mercury (Hg), tellurium (Te), and zinc (Zn); and late evolving elements beryllium (Be), tantalum (Ta), tungsten (W), and zirconium (Zr). The improvement of lowered background for these elements did not justify further complexity in determination of concentrations by making exposure time a variable.

Since both standards and samples were present as the nitrate, the possibility existed of elimination of the conversion to oxide in sample preparation. However, a comparison of impurity spectra of a lead-nitrate sample before and after conversion to oxide showed greater line intensity in the oxide form.

Detection limits along with the analytical line used and parameter set (see Table VII) are shown in Table VIII.

Twenty-seven samples have been analyzed by this method. The accuracy and precision of the method were not investigated, but are estimated to be within the specified accuracy requirements by comparison with similar spectrographic methods.

TABLE VII. Variable Excitation and Recording Parameters.

Parameter Set	Grating Angle (degrees)	Focus (millimeters)	Slit Width (microns)	Exposure (seconds)	Photographic Plate	Atmosphere	Filter	Recorded Wavelength (angstroms)
1	9.52	4.5	10	120	SA1	Air	--	2200 to 3400
2	13.59	5.0	10	120	SA1	Argon-Oxygen (Ar-O ₂)	--	3375 to 4575
3	20.78	7.0	30	25	1N	Air	K2, No. 8; yellow	5400 to 6600
4	28.58	10.4	30	25	1N	Air	K2, No. 8, yellow	7500 to 8700

FIGURE 1. Time-Intensity Curves (Abcissa is 150 seconds long).

FIGURE 2. Time-Intensity Curves (Abscissa is 150 seconds long).

TABLE VIII. Detection Limits, Analytical Line, and Parameter Set by Element.

Element	Detection Limit (micrograms per gram)	Wavelength (angstroms)	Parameter Set	Element	Detection Limit (micrograms per gram)	Wavelength (angstroms)	Parameter Set
Arsenic (As)	10	2288.12	1	Nickel (Ni)	10	3412.63	1
Barium (Ba)	10	4554.04	2	Niobium (Nb)	50	4058.94	2
Beryllium (Be)	1	2348.61	1	Palladium (Pd)	10	3404.58	1
Cadmium (Cd)	10	2288.02	1	Platinum (Pt)	10	3064.71	1
Cesium (Cs)	20	8521.10	4	Potassium (K)	10	7664.91	4
Chromium (Cr)	10	4254.35	2	Rhenium (Re)	10	3460.46	2
Cobalt (Co)	10	3405.12	1	Rhodium (Rh)	10	3396.85	1
Copper (Cu)	1	3247.54	1	Rubidium (Rb)	10	7800.23	4
Dysprosium (Dy)	10	3898.54	2	Ruthenium (Ru)	20	2735.72	1
Europium (Eu)	10	3971.99	2	Scandium (Sc)	10	3613.84	2
Gadolinium (Gd)	10	3422.47	2	Sodium (Na)	10	5889.95	3
Germanium (Ge)	10	2754.59	1	Strontium (Sr)	10	4077.71	2
Gold (Au)	10	2675.95	1	Tantalum (Ta)	100	2653.27	1
Hafnium (Hf)	20	2866.37	1	Tellurium (Te)	100	2383.25	1
Holmium (Ho)	10	3891.02	2	Terbium (Tb)	20	3702.85	2
Indium (In)	10	3256.09	1	Thulium (Tm)	10	3700.26	2
Iridium (Ir)	50	2639.71	1	Tin (Sn)	10	2839.99	1
Lutetium (Lu)	10	3397.07	2	Titanium (Ti)	10	3078.65	1
Magnesium (Mg)	1	2852.13	1	Tungsten (W)	500	2724.35	1
Manganese (Mn)	10	2798.27	1	Vanadium (V)	10	3183.98	1
Mercury (Hg)	10	2536.52	1	Ytterbium (Yb)	10	3694.20	2
Molybdenum (Mo)	10	3132.59	1	Zinc (Zn)	100	3345.02	1
				Zirconium (Zr)	10	3391.98	1

APPENDIX A. Lead Determination.

During the work on the calcium determination, a request was also received for a gram per gram (g/g) determination on two lead samples. The method used was the standard lead chromate (PbCrO_4) gravimetric procedure.^{1-A} In this method, about a 0.5-g sample is dissolved in two ml of concentrated HNO_3 and then diluted to about 150-ml total volume. The solution is heated to boiling, and while stirring, a solution of 4-percent ammonium chromate $[(\text{NH}_4)_2\text{CrO}_4]$ is added dropwise, until precipitation is complete. After standing overnight, the solution is filtered and the solid dried at 140°C in a drying oven. The resultant PbCrO_4 is then weighed and the weight of Pb calculated.

Four samples of reagent-grade Pb were analyzed by this method as a test of the procedure to be used on the production Pb samples. The two production Pb samples were then analyzed. Results on the reagent-grade Pb and the two samples are given in Table I-A.

TABLE I-A. Results on Gram-per-Gram Determinations.

Sample	Lead (gram per gram)
Reagent-Grade Lead No. 1	0.9969
Reagent-Grade Lead No. 2	0.9963
Reagent-Grade Lead No. 3	0.9966
Reagent-Grade Lead No. 4	0.9962
* Sample A	0.9987
** Sample B	0.9995

* Calcium content was 4 parts per million
(Requisition No. 426591).

** Calcium content was 21 parts per million
(Requisition No. 426592).

^{1-A} Z. Karaoglanov and M. Michov: *Zeitschrift für Analytische Chemie*. 103:113. 1935.