

**Procedures for the Analysis of Metals, Alloys,
and Other Materials of Research**

**Part 2 - Determination of Silver and
Indium in Magnesium-Base Alloys,
Lead in Indium, and Zirconium in Hafnium**

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PREVIOUS REPORTS IN THIS SERIES

Part 1 - "Procedures for the Chemical Analysis of Copper-Base Alloys," O. R. Gates and O. Mylting, NRL Report 6357, Mar. 1966

ABSTRACT

A series of methods is presented in sufficient detail for direct analytical application to the determination of silver and indium in magnesium-base alloys, lead in indium, and zirconium in hafnium. The analytical approaches are combinations of chemical and instrumental techniques involving the flame spectrophotometer, the polarograph, and the x-ray fluorescence equipment, respectively.

PROBLEM STATUS

This is the second in a series of progress reports. Work on the problem is continuing.

AUTHORIZATION

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PROCEDURES FOR THE ANALYSIS OF METALS, ALLOYS, AND OTHER MATERIALS OF RESEARCH

PART 2 - DETERMINATION OF SILVER AND INDIUM IN MAGNESIUM-BASE ALLOYS, LEAD IN INDIUM, AND ZIRCONIUM IN HAFNIUM

INTRODUCTION

This is the second of a series of reports which will present progress in analytical methods developed by the Analytical Chemistry Branch, Metallurgy Division. This analytical development project has direct relevance to Navy materials needs because it is stimulated by feedback from materials research projects, the success of which is often vitally affected by analytical characterization. The analytical requirements may involve the determination of trace quantities of impurities, additives in high-purity materials, or the determination of major constituents in newly developed alloys. The swift-moving pace of materials development does not always permit either the comprehensive compositional evaluation of families of potential impurities in a given matrix or the evaluation of an impurity in a family of potential matrices. Most often the analytical methods development satisfies the needs of a specific path of materials research, but by implication the methods also lead to a broad panorama of analytical possibilities.

Two types of information will be presented in this series of reports: (a) methods that have been completed and which can be reported in sufficient detail to be followed in other laboratories, and (b) progress on productive facets of particular analytical investigations which will require further work for completion.

METHODS OF ANALYSIS

Flame Photometric Determination of Silver and Indium in Magnesium-Base Alloys

Introduction

The use of flame spectrophotometry is well known for the determination of trace amounts of certain elements that are excitable by the thermal energy provided by a flame. Less well known is the use of this analytical approach for the determination of elements in alloying concentrations. The tradition of the trace should not become the enemy of analytical progress, however. The following method describes the adaptation of flame spectrophotometry to magnesium-base alloys of a research nature, in which relatively large amounts of silver and indium are to be determined. More significant than the actual procedural details for these specific alloys is the implication it holds for major constituent analysis of a potentially wide scope of research-type alloys.

Scope

This method covers the analysis of magnesium alloys for silver and indium in equal alloying concentrations up to 10 percent.

Apparatus

Spectrophotometer—A Beckman, Model DU, with flame attachment, oxyhydrogen atomizer-burner, and blue-sensitive photomultiplier tube.

Special Beakers—Beakers of 5-ml capacity, from which solutions can be aspirated for flame excitation. A separate beaker should be provided for each solution to be aspirated.

Pipets and Volumetric Flasks—Volumetric ware should have certified calibrations.

Reagents

Magnesium Stock Solution (1 ml = 10.0 mg of Mg)—Magnesium turnings of high purity (2.00 grams) are placed in a 300-ml Berzelius beaker, covered with a watch glass, and dissolved carefully in nitric acid. The nitric acid is added cautiously, in 2-ml portions, until the metal is dissolved and a total of 30 ml of concentrated nitric acid has been added. The mixture is then boiled for several minutes, allowed to cool, transferred to a 200-ml volumetric flask, diluted to the mark, and mixed.

Silver Stock Solution (1 ml = 1.0 mg of Ag)—Reagent-grade silver nitrate (0.7875 gram) is dissolved in 300 ml of water. The solution is acidified with 10 ml of nitric acid, transferred to a 500-ml volumetric flask, diluted to the mark, and mixed.

Indium Stock Solution (1 ml = 1.0 mg of In)—Purified indium metal (0.5000 gram) is dissolved with 10 ml of nitric acid, diluted with 30 ml of water, and boiled gently for several minutes. The solution is cooled, transferred to a 500-ml volumetric flask, diluted to the mark, and mixed.

Primary Standards—Primary standards are prepared by diluting stock solutions as specified in Table 1.

Table 1
Primary Standard Preparation

Primary Standards	Mg Stock* [10.0 mg/ml] (ml)	Ag Stock* [1.0 mg/ml] (ml)	In Stock* [1.0 mg/ml] (ml)	Final Dilution (ml)	Percent	
					Ag	In
A	6.0	20.0	20.0	100	20.0	20.0
B	8.0	10.0	10.0	100	10.0	10.0
C	9.0	5.0	5.0	100	5.0	5.0
D	9.6	2.0	2.0	100	2.0	2.0
E	10.0	-	-	100	0	0

*Stock solutions listed under "Reagents" in above text.

Working Standards—Aliquots of the primary standards are combined, as indicated in Table 2, to provide working standards of various silver and indium percentages.

Table 2
Working-Standard Preparation

Working Standards	Primary Aliquots* Combined	Percent		Luminosity	
		Ag	In	Ag †	In †
AC	10 ml A + 10 ml C	12.5	12.5	88.4	100.0
AD	10 ml A + 10 ml D	11.0	11.0	81.7	90.0
B	10 ml B + -	10.0	10.0	77.7	81.9
BC	10 ml B + 10 ml C	7.5	7.5	65.2	62.3
BD	10 ml B + 10 ml D	6.0	6.0	56.0	50.5
C	10 ml C + -	5.0	5.0	49.0	42.0
CD	10 ml C + 10 ml D	3.5	3.5	37.1	29.6
CE	10 ml C + 10 ml E	2.5	2.5	29.0	21.1
D	10 ml D + -	2.0	2.0	24.0	17.0
E	10 ml E + -	0	0	3.0	1.0
Blank (H ₂ O)		0	0	0	0

*Primary standards prepared in Table 1.

†The silver line (338.3 m μ) and indium line (451.1 m μ) were used to obtain the luminosities.

Procedure

Preparation of Calibration Curves—A curve for the 338.3-m μ silver line is prepared by adjusting the slit width and sensitivity of the spectrophotometer so that the highest silver standard gives a luminosity reading between 90 and 100. The instrument is adjusted to zero luminosity with water. Then with the wavelength selector peaked on the 338.3-m μ silver line, the standard solutions are aspirated and measured in the increasing order of their silver content, beginning with the blank. After each measurement, water is aspirated through the siphon to reduce possible clogging and interference. The relative luminosities are plotted against the percentages of silver.

An indium calibration curve is prepared, following the same procedure outlined for silver, with the 451.1-m μ indium line. With the exception of the 10, 11, and 12.5 percentage points, the points for the indium calibration curve should form a straight line. The higher figures indicate the beginning of self-absorption. This phenomenon is much more pronounced for silver (see Fig. 1).

Preparation and Measurement of Sample Solutions—A 0.1000-gram sample of magnesium alloy is transferred to a 100-ml beaker, covered with a watch glass, and dissolved with 2 ml of nitric acid. The cover and inner wall of the beaker are rinsed with distilled water, and the solution boiled gently for a minute to remove the oxides of nitrogen. The solution is cooled, transferred to a 100-ml volumetric flask, diluted to the mark, and mixed. About 4 ml of each solution and standard is transferred to a clean, properly labeled, 5-ml beaker.

The samples and standards are atomized and their luminosities recorded, as described for the preparation of the calibration curves. The readings are referred to the appropriate calibration curves for the determination of the percentages present.

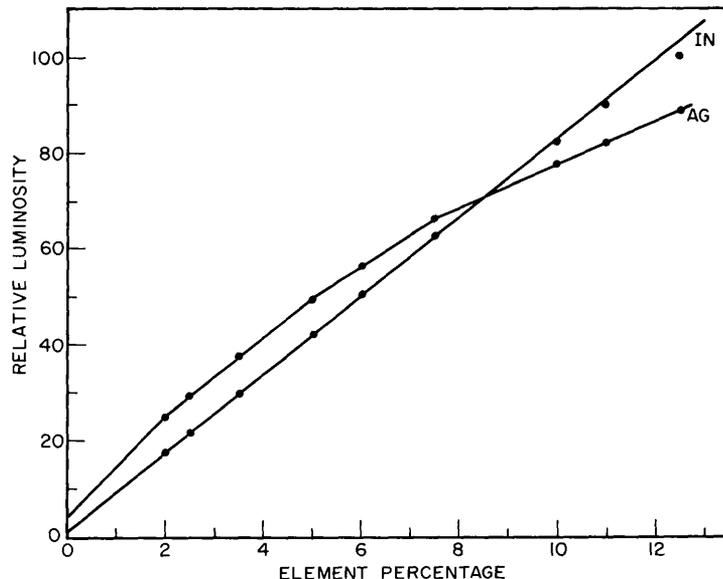


Fig. 1 - Flame photometric calibration curves for silver and indium

Appraisal of Data

National Bureau of Standards samples are available for only a few of the most common nonferrous alloys. Therefore, it is necessary to prepare synthetic standards for many of the alloy systems encountered. If analytically feasible, flame spectrophotometric data should be spot checked with data obtained from another acceptable procedure, until confidence is established in the flame procedures.

Table 3 compares the data obtained for various synthetic standards in which the silver content was checked by direct titration with standard ammonium thiocyanate, using ferric alum solution indicator.

Flame spectrophotometric values for indium compare so favorably with the synthetic standards that a refined gravimetric procedure was not considered necessary for cross-checking.

The ability of the flame spectrophotometer to discriminate between synthetic standards whose silver and indium content differ by small decrements is shown in Table 4.

Table 3
Comparative Analyses for Silver

Synthetic Sample	Silver Present (%)	Silver Found (%)	
		Titration	Flame Photometry*
A	10.0	10.15	10.3
B	10.2	10.05	10.0
C	4.0	4.1	4.1
D	0.4	0.3	0.4

*Percentages obtained from a straight line drawn through closest bracketing standard points.

Table 4
Variations in Luminosity for Small
Decrements in Analyte Content

Content (%) Silver or Indium	Luminosity (% T. Scale)	
	Silver	Indium
10.2	96.2	97.0
10.1	95.0	96.0
10.0	95.0	95.0
7.1	81.7	68.0
7.0	80.7	67.0
5.3	68.5	51.0
5.2	67.3	50.2
2.2	35.8	22.0
2.0	31.5	19.7

The Polarographic Determination of Lead in Indium

Introduction

This procedure was developed for the analysis of lead-indium binary alloys. Systems such as these, which are prepared from relatively-high-purity constituents, contain little or no impurities to produce annoying and interfering diffusion currents in polarographic analysis. With a supporting electrolyte capable of producing a well-defined diffusion current for the minor constituent, the analysis is rapid and straightforward.

Polarographic analysis of such materials becomes all the more attractive when used as an alternative to existing tedious wet-chemical procedures.

Scope

This method describes the analysis of lead in indium, from 5 to 25 percent, without the need for preliminary separation of the indium. The lower limit of the method may be extended to include trace quantities of lead by choice of sample size and adjustment of electrolyte volume.

Apparatus

Polarograph—A Leeds and Northrop Electro-Chemograph, Model E, was used in this work; however, any modern recording polarograph is suitable.

Electrolysis Cell—An H-type cell and dropping-mercury electrode assembly similar to that shown in Fig. 2 is used.

Reagents

Tartaric Acid (20% Solution)—Reagent grade tartaric acid (200 grams) is dissolved in water and diluted to 1000 ml.

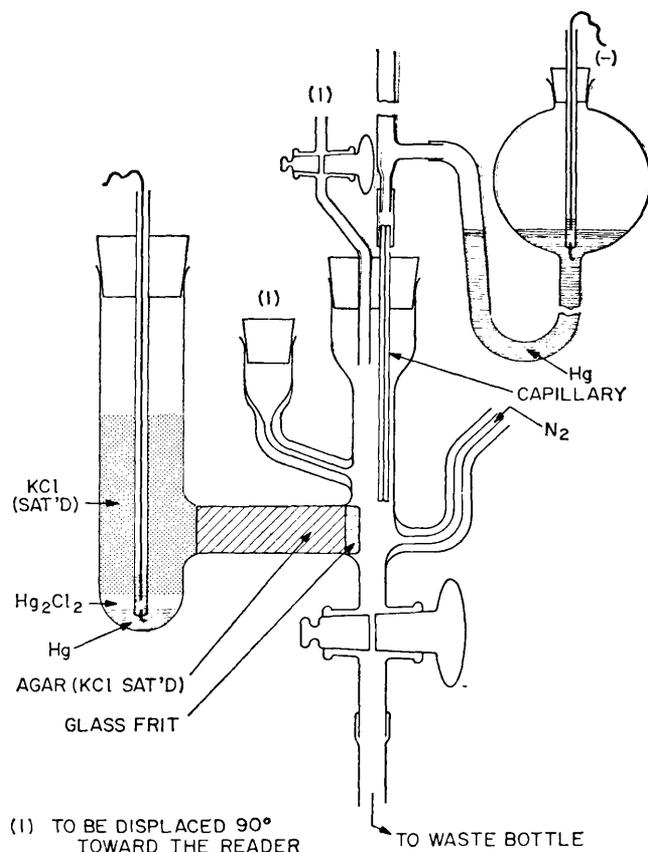


Fig. 2 - Dropping-mercury electrode assembly

Sodium Hydroxide (20%)—Reagent grade sodium hydroxide (200 grams) is dissolved in 500 ml of water, cooled, and diluted to 1000 ml in a polyethylene bottle.

Phenolphthalein (1% solution)—Phenolphthalein power (1 gram) is dissolved in 100 ml of ethyl alcohol.

Standard Lead Solution (1 ml = 0.0005 gram of Pb)—High-purity lead (0.1000 gram) is dissolved with 10 ml of nitric acid (1-1). The solution is diluted to 30 ml with water and boiled to expel the oxides of nitrogen. After cooling, the solution is transferred to a 200-ml volumetric flask, diluted to the mark, and mixed.

Standard Indium Solution (1 ml = 0.001 gram of In)—High-purity indium (0.2000 gram) is dissolved with 10 ml of nitric acid (1-1). The solution is diluted to 30 ml with water and boiled to expel the oxides of nitrogen. After cooling, the solution is transferred to a 200-ml volumetric flask, diluted to the mark, and mixed.

Reference Solutions (5-25% Pb)—To five clean 25-ml volumetric flasks, numbered 1 through 5, the following aliquots of the standard lead solution are added: 0, 1, 2, 3, and 5 ml. To these same flasks, and in the same order, the following aliquots of the standard indium solution are added: 10, 9.5, 9, 8.5, and 7.5 ml. The combined weight of lead and indium in each flask is then 0.01 gram.

Supporting electrolytes are added to the reference solutions as follows: To each of the 25-ml flasks containing the reference solutions, 5 ml of the 20-percent tartaric acid

solution is added, followed by one drop of the phenolphthalein solution. The solutions are neutralized with the 20-percent sodium hydroxide solution. After adding an exact excess of 0.5 ml to each,* the solutions are diluted to the 25-ml mark and mixed.

Preparation of Calibration Curve

Instrument and Cell Adjustment—The polarograph is conditioned and checked according to the manufacturer's recommendations.

With the electrodes immersed in water, the mercury column is adjusted to provide a standing column or "head" of about 30 cm. The cell is rinsed several times with the synthetic standard solution that contains no lead (blank), and then filled with the same electrolyte until the lower tip of the dropping electrode is well immersed. The mercury-drop interval is about 5 seconds.

A stopper is inserted in the filling tube, the stopcock at the top of the cell is opened, and nitrogen is bubbled through the electrolyte for about 5 minutes. The nitrogen flow is then stopped, and the nitrogen exit cock is closed.

Electrolysis—The solutions are arranged for electrolysis so that each succeeding sample contains an equal or higher percentage of lead than its predecessor. This can be arranged for standards and reference solutions, but may not always be possible with unknown samples.

After observing that the dropping electrode is functioning properly and that the electrode cable is attached securely, the electrolyses are conducted as follows: The instrument is set at or near its highest sensitivity, and the cell current is turned on. The chart paper is adjusted so that the recorder pen is directly over a heavy crossline, which is identified as the -0.4-volt† starting line. The polarizing voltage is set at -0.35 volt, and the polarizer is started. At the instant that the scanning polarizer registers -0.4 volt, the recorder is started and a polarogram is taken which scans through the diffusion current range to about -1.1 volts. The recorder is then stopped, and the cell current turned off.

The electrolyte is removed, and the electrodes are rinsed at least once with the next electrolyte to be analyzed. Oxygen is removed from the new electrolyte by nitrogen flushing, as previously described, and the electrolysis procedure repeated.

Plotting Calibration Curve—After all the standard solutions have been electrolyzed, the respective relative diffusion currents are determined from the polarograms and plotted against the lead percentages they represent. (Log-log paper is used for wide percentage ranges.) Figure 3 shows a typical curve obtained for synthetic solutions containing up to 25 percent lead.

Procedure

A 0.100-gram sample of the alloy to be analyzed is transferred to a 100-ml beaker, 5 ml of water is added, the beaker is covered, and 5 ml of nitric acid is added cautiously. The mixture may be warmed, if necessary, to effect sample dissolution. The solution is diluted to about 30 ml with water and boiled gently to remove the oxides of nitrogen. After cooling, the solution is transferred to a 100-ml volumetric flask, diluted to the mark, and mixed.

*A total of 4.0 ml of NaOH is generally required to neutralize and adjust each standard solution to pH 12.

†Since the reference cell is a saturated calomel electrode (S.C.E.), this and following voltages will be referred to it.

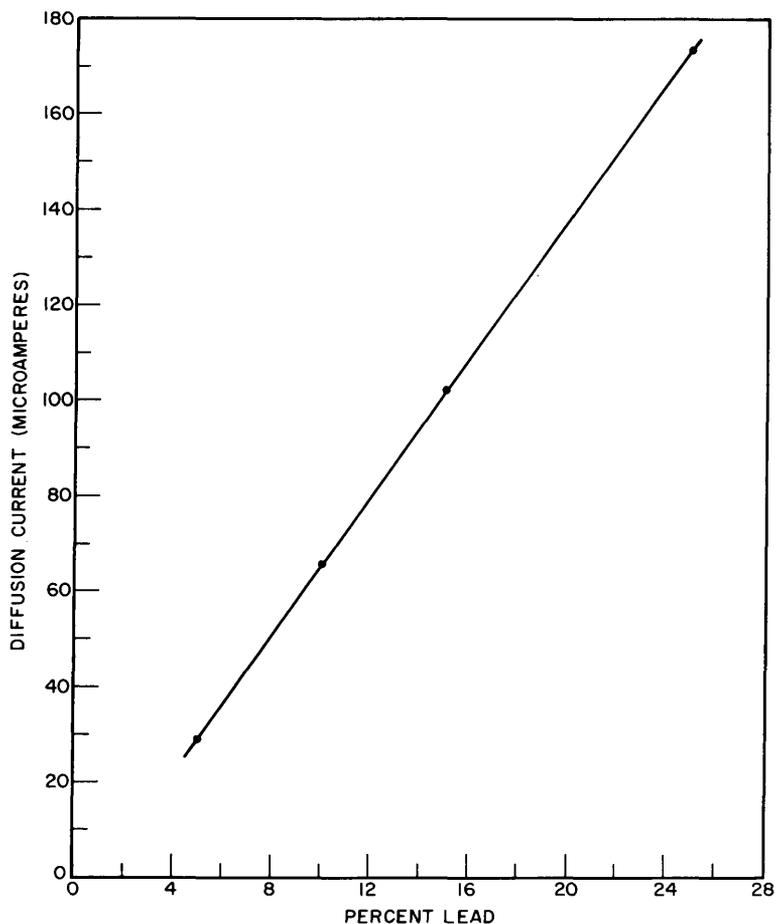


Fig. 3 - Polarographic calibration curve for lead

A 10-ml aliquot of the sample solution is transferred to a 25-ml volumetric flask. The supporting electrolyte is added as directed under the section "Reference Solutions" above.

The diffusion current for the sample is determined in the same manner as that used for the standard solutions, and the lead percentage is obtained from the standard curve.

NOTE: By using a 0.5-gram sample and adjusting the final volume of the total sample to 15 ml, it is possible to determine as little as 0.005 percent lead in indium, without resorting to preliminary isolatory techniques. This order of sensitivity can be attained only with great difficulty by usual wet-chemical procedures.

X-Ray Fluorescence Determination of Zirconium in Hafnium

Introduction

The chemical reactions of hafnium and zirconium are so similar that analyses involving both elements in the same matrix are extremely difficult by chemical means. X-ray fluorescence techniques offer the greatest analytical potentialities for hafnium-zirconium alloys, but standards covering the concentration ranges of interest must be available.

By combining the resources of chemical methods of sample and standard preparation with the analytical resolution provided by x-ray fluorescence techniques, a method for analyzing hafnium-zirconium alloys has been developed that provides both accuracy and flexibility of operation.

In the method detailed below, the metal sample is reduced to powder form, recovered completely, and cast into a sturdy homogeneous disc by briquetting with an organic binder. Many advantages are achieved by this powder method of analysis, foremost of which are the elimination of metallurgical history of the sample, the simplicity of preparing synthetic standards of the exact nature of the material to be analyzed, and the provision of a permanent form of sample and standard for future reference work.

Scope

This method is applicable to the determination of zirconium in hafnium over the 0.1-10 percent range.

Apparatus

X-ray Tube—Tungsten-target, Machlett OEG-type x-ray tube capable of operation at 50 kv and 20 ma.

Analyzing Crystal—Lithium fluoride.

Detector—Geiger tube, scintillation counter, or proportional counter.

Goniometer—Norelco Hi-Angle goniometer, or equivalent.

Power Supply—50-kv, 50-ma, full-wave rectified x-ray tube power supply.

Briquetting Press—Buehler press, capable of 12,000 psi on a 1-in.-diam piston.

Reagents

Hydrofluoric Acid (48%).

Sugar Solution—A solution of sucrose is made by dissolving 100 grams of sucrose in 100 ml of water.

Procedure

Preparation of Sample—A 0.200-gram sample of metal is weighed into a 40-ml platinum crucible, then dissolved by adding 5 ml of water, 1 ml of nitric acid, and 1 ml of hydrofluoric acid. The crucible is covered with a platinum lid and placed on an asbestos pad on a hot plate to warm. Sample dissolution is allowed to proceed, with 1-ml additions of hydrofluoric acid as required. After dissolution of the sample, the mixture is cooled, and the cover is rinsed, catching the rinsings in the crucible. The solution is evaporated to dryness, and the residue is taken up in 2 ml of hydrofluoric acid. Ten drops of sulfuric acid (1-1) are added, and the mixture is again evaporated to about a 1-ml volume. Ten drops of the sugar solution are added, and the evaporation is continued until the sugar begins to carbonize. When the solution becomes black and viscous, the crucible is carefully rotated, using a pair of Blair-type crucible tongs, until most of the wall of the crucible is coated with the syrup. The evaporation is continued to dryness, and the residue

is carefully charred by heating the crucible on an asbestos-covered gauze over a Fisher burner. Final ignition of the residue is done in a muffle furnace at 550°C. This temperature is maintained to prevent the loss of such metals as molybdenum and tungsten as their volatile oxides. Due to the sugar solution carbonization, the ignited residue may be readily scraped from the crucible wall and pulverized with a soft scraper.

Briquetting—A total of 1.5 ml of methocellulose powder, in 0.5 ml separate portions, is added to the oxides in the crucible. A platinum spatula is used in a stirring-grinding motion to insure a homogeneous mixture. The mixture is transferred to a 1-inch cylindrical mold, the surface of the powder leveled, and compacted into a wafer at 12,000 psi in a Buehler press.

Preparation of Standards—A total of 0.200 gram of high-purity hafnium and zirconium, in the percentage ratio required for each standard, is transferred into a 40-ml platinum crucible. The mixture is dissolved, ignited, and briquetted as described under "Sample Preparation" and "Briquetting" above. (If sufficiently pure hafnium is not available, standards may be prepared by adding known weights of zirconium to weighted portions of the specimen stock, and determining the composition by transposing the calibration curve to intercept zero.)*

X-Ray Measurements—The briquetted wafer is inserted in the sample holder of the x-ray spectrometer and irradiated at 35 kv and 10 ma with a tungsten-target, OEG-50 type, x-ray tube. The Zr K_{α} x-ray line is measured at $2\theta = 22.5^{\circ}$. Background intensity is determined for the $2\theta = 22.5^{\circ}$ line by scanning the spectrum from $2\theta = 20^{\circ}$ to $2\theta = 24^{\circ}$. A calibration curve is established between the Zr K_{α} line intensity (minus the background intensity) and the weight percent of the standards.

*In general, if the desired metal is not available in sufficient purity for use as a standard, a reliable "twin" calibration curve may be obtained by choosing an element with nearly the same x-ray parameters as the desired metal. In the analysis of hafnium for zirconium, tantalum was used as the "twin" for hafnium, since the tantalum and hafnium have very similar excitation and absorption factors for a zirconium matrix. Figure 4 shows typical calibration curves for both.

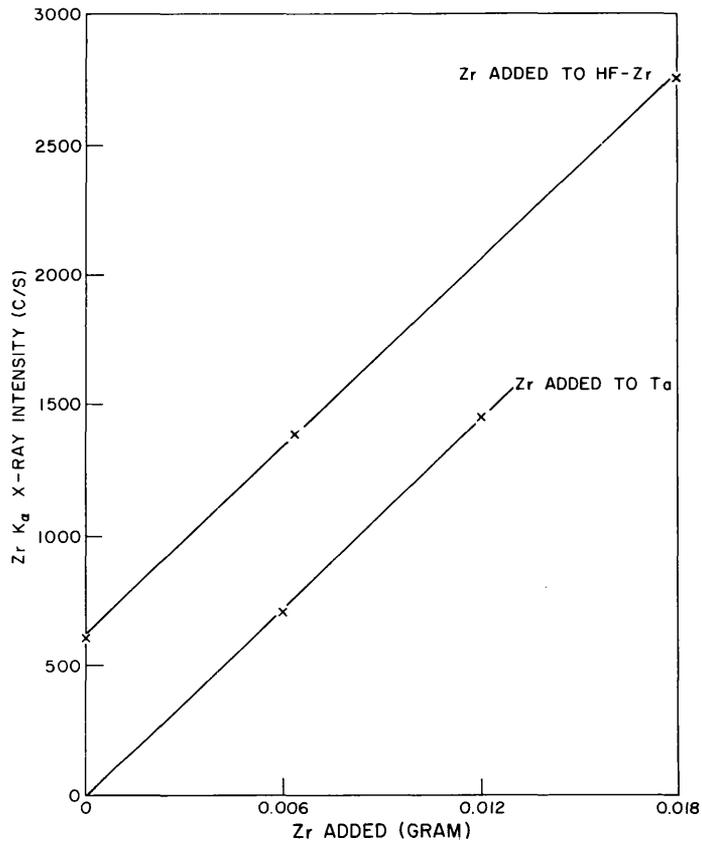


Fig. 4 - Typical x-ray fluorescence calibration curves for zirconium

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