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A SIMPLE SPECTROPHOTOMETRIC
METHOD FOR DETERMINATION
OF ZIRCONIUM OR HAFNIUM IN
SELECTED MOLYBDENUM-BASE ALLOYS

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16. Abstract <p>This report describes a simple analytical procedure for accurately and precisely determining the zirconium or hafnium content in molybdenum-base alloys. The procedure is based on the reaction of the reagent Arsenazo III with zirconium or hafnium in strong hydrochloric acid solution. The colored complexes of zirconium or hafnium are formed in the presence of molybdenum. Titanium or rhenium in the alloy have no adverse effect on the zirconium or hafnium complex at the following levels in the selected aliquot: Mo, 10 mg; Re, 10 mg; Ti, 1 mg. The spectrophotometric measurement of the zirconium or hafnium complex is accomplished without prior separation with a relative standard deviation of 1.3 to 2.7 percent.</p>			
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A SIMPLE SPECTROPHOTOMETRIC METHOD FOR DETERMINATION OF ZIRCONIUM OR HAFNIUM IN SELECTED MOLYBDENUM-BASE ALLOYS

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SUMMARY

The molybdenum-base alloys are dissolved in aqua regia. Nitrates are removed by repeated evaporation, a minimum of three times, with hydrochloric acid. The sample solution is then made to volume with 9-normal hydrochloric acid.

An appropriate aliquot, containing 0.1 to 20 micrograms zirconium or 2 to 50 micrograms hafnium, is placed in a 50-milliliter volumetric flask. The aliquot is diluted with 9N HCl, and 4 milliliters of 0.10-percent weight-per-volume solution of Arsenazo III is added. The solution is made to volume with 9N HCl and thoroughly mixed.

The color intensity of the zirconium- or hafnium-Arsenazo III complex is measured spectrophotometrically at 670 nanometers. For a zirconium or hafnium content of 0.1 to 10 micrograms, a 2-centimeter cell is used; for 10 to 50 micrograms, a 1-centimeter cell is used. The zirconium or hafnium content is determined from an appropriate calibration curve.

The proposed analytical procedure is simple, requires no prior separation, and is accurate and reproducible.

INTRODUCTION

The reagent Arsenazo III (1, 8-dihydroxynaphthalene-3, 6-disulfonic acid-2, 7-bis (azo-2) phenylarsonic acid), in addition to zirconium (Zr) and hafnium (Hf), gives marked color reactions with a number of elements, including thorium (Th), scandium (Sc), uranium (U), actinium (Ac), and lanthanum (La). The reagent forms stable metal chelates. The high stability of the metal complexes permits the elements to be photometrically determined in strongly acidic media. Photometric measurements in strongly acidic media are useful, since partial hydrolysis of certain elements is eliminated. A

high degree of selectivity is attained when determining the quadrivalent elements zirconium and hafnium in the strongly acidic medium, 9-normal hydrochloric acid (9N HCl).

It has been reported in reference 1 that no color reactions are observed for elements the cations of which have a radius of less than 0.7×10^{-10} to 0.8×10^{-10} meter (0.7 to 0.8 Å). In table I the ionic radii, in angstroms, are given for those elements that might be components of molybdenum-base alloys.

Zirconium has been photometrically determined in uranium by using Arsenazo III (ref. 2). The zirconium was removed by solvent extraction prior to the formation of the Zr - Arsenazo III complex. The photometric measurement was performed in 6N HCl.

Since molybdenum (Mo) does not form a colored complex with Arsenazo III, it was assumed that a direct photometric measurement of the Zr or Hf complex, without a prior separation, might be feasible.

The molybdenum-base alloys listed in table II were investigated to determine Hf and Zr content.

The molybdenum-base alloy designated TZM (MAB) was included because it had been analyzed by numerous laboratories (ref. 3) at the request of the Materials Advisory Board.

Samples of the alloys selected for investigation were dissolved in aqua regia. The oxidants, essentially nitrates, were displaced by repeated evaporation, a minimum of three times, with HCl. Stable solutions of the samples were obtained in 9N HCl.

EQUIPMENT AND MATERIALS

The following equipment and materials were used for the analytical procedure described herein:

- (1) Spectrophotometer, with 1-, 2-, and 5-centimeter cells
- (2) Reagents
 - (a) Hydrochloric acid (HCl), concentrated, reagent grade
 - (b) Hydrochloric acid, 9N (9 vol. HCl + 3 vol. H₂O)
 - (c) Nitric acid, concentrated, reagent grade
 - (d) Arsenazo III, 0.10-percent weight-per-volume solution (100 mg reagent in 100 ml H₂O containing 0.5 ml of a 5-percent solution of Na₂CO₃)
 - (e) Sodium carbonate (Na₂CO₃), reagent grade, 5-percent solution
 - (f) Zirconium, reactor grade, 99.9-percent pure
 - (g) Hafnium, 99.8-percent pure, with zirconium content of 245 ppm
 - (h) Molybdenum, 99.9-percent pure
 - (i) Rhenium (Re), 99.9-percent pure
 - (j) Titanium, high purity, produced by the iodide process

PROCEDURE

Preparation of Standards

A standard Hf solution was prepared from Hf metal (containing 245 ppm Zr). A 0.4977-gram quantity of the metal was oxidized to hafnium oxide (HfO_2) at 800°C . The oxide was fused with 2 grams of sodium carbonate sodium tetraborate ($\text{Na}_2\text{CO}_3 \cdot \text{Na}_2\text{B}_4\text{O}_7$). The clear melt was dissolved in HCl, transferred to a 500-milliliter volumetric flask and diluted to volume with 9N HCl.

A 50-milliliter aliquot of this solution was taken and mixed with mandelic acid. The resulting precipitate was filtered out of the solution, washed, ignited to hafnium oxide (HfO_2), cooled, and weighed. Thus, the Hf content of the solution was found to be 0.4961 gram.

Two standard solutions were prepared from this master solution. One solution contained 9.92 micrograms Hf per milliliter in 9N HCl; the other solution contained 1.98 micrograms Hf per milliliter in 9N HCl.

A standard Zr solution was prepared from reactor-grade metal. A 1.0210-gram quantity of Zr was dissolved in 1:1 HCl, and a minimum of hydrofluoric acid was added drop-wise. The solution was evaporated to dryness three times with HCl. The sample was taken up with 9N HCl, transferred to a 500-milliliter volumetric flask, then made to volume with 9N HCl.

A 50-milliliter aliquot of this solution was taken and mixed with mandelic acid. The resulting precipitate was filtered out of the solution, washed, ignited to zirconium dioxide (ZrO_2), cooled, and weighed. Thus, the Zr content of the solution was found to be 1.0200 grams.

Two standard solutions were prepared from this master solution in 9N HCl containing 2.00 and 10.20 micrograms Zr per milliliter.

A working solution of Mo was prepared as follows: A 2-gram sample of Mo was dissolved in nitric acid. The nitrates were displaced by repeated evaporation, a minimum of three times, with HCl. The sample was taken up with 9N HCl, transferred to a 200-milliliter volumetric flask and made to volume with 9N HCl (1 ml = 10 mg Mo).

A working solution of rhenium was prepared as follows: A 2-gram sample of Re metal powder was fused with 5 grams of potassium carbonate. The clear melt was dissolved in HCl, transferred to a 200-milliliter volumetric flask and made to volume with 9N HCl (1 ml = 10 mg Re).

Calibration of the Absorption Spectra

An optical scan of the absorption spectra of the reagent Arsenazo III and of the Zr and Hf complexes of this reagent showed a well-defined peak at 670 nanometers. There was a minimal absorption of the reagent at this wavelength, as shown in figures 1 and 2.

Solutions of Zr and Hf, at a concentration of 50 micrograms in a 50-milliliter final volume, were reacted with Arsenazo III at HCl acid concentrations of 2.76N, 5.52N, and 8.28N. A plot (fig. 3) of the optical densities measured showed that a far greater sensitivity was obtained at an HCl concentration of 8.28N than at lower concentrations. However, it might be possible to photometrically measure higher concentrations of Zr or Hf at a lower level of acid concentration (fig. 3).

Since the data shown in figure 3 clearly indicated that a greater sensitivity for Zr and Hf was obtained at an 8.28N HCl concentration than at lower concentrations, all projected analytical work was performed at this acid level.

A calibration curve for Zr was established by first placing 0, 2, 6, 10, 14, 16, and 20 micrograms of a standard Zr solution in 50-milliliter volumetric flasks, each containing 25 milliliters of 9N HCl. To each flask 4 milliliters of Arsenazo III were added. The volumes were adjusted to 50 milliliters with 9N HCl and thoroughly mixed. The photometric measurement of the Zr complex, when blank corrected, indicated that Beer's law was obeyed in this range of Zr (fig. 4).

To ascertain whether there is an effect of Mo on the Zr complex, a repeat of the above calibration curve was made but 10 milligrams of Mo were added to all solutions. There was no adverse effect from Mo (see fig. 4).

To ascertain whether there is an effect of Re on the Zr complex, the Zr calibration curve was repeated but 10 milligrams of Re were added to all solutions. There was no adverse effect from Re on the calibration curve (see fig. 4).

Another calibration curve was established for Zr by placing 0, 2, 4, 6, 8, and 10 micrograms of a standard Zr solution in 50-milliliter volumetric flasks, each containing 25 milliliters of 9N HCl. To each flask 4 milliliters of Arsenazo III solution were added. All solutions were made to volume with 9N HCl and thoroughly mixed. The Zr complex was measured at 670 nanometers by using a 2-centimeter cell (see fig. 5).

To ascertain the effect of Mo on the Zr complex, the above calibration curve was repeated but 10 milligrams of Mo were added to each flask. There was no adverse effect of Mo on the calibration curve (see fig. 5).

A lower level calibration curve was established for Zr by placing 0, 1, 3, 5, 7, and 10 micrograms of a standard Zr solution in 50-milliliter volumetric flasks, each containing 25 milliliters of 9N HCl. To each flask 4 milliliters of Arsenazo III solution were added. All solutions were made to volume with 9N HCl and thoroughly mixed.

The Zr complex was measured at 670 nanometers by using a 5-centimeter cell (see fig. 6).

To ascertain whether there is an effect of Ti on the Zr complex, 0, 2, 4, 6, 8, 14, and 20 micrograms of a standard Zr solution were placed in 50-milliliter volumetric flasks, each containing 25 milliliters of 9N HCl. To each solution 4 milliliters of Arsenazo III were added. The volumes were adjusted with 9N HCl and thoroughly mixed. The Zr complex was measured at 670 nanometers by using a 1-centimeter cell. Then, 1 milligram of Ti was added to a 2- and a 14-microgram Zr standard solution. There was no adverse effect on the Zr complex with this concentration (1 mg) of Ti (see fig. 7).

Based on the data presented in figures 4, 5, and 7, the following conclusions can be drawn:

(1) The Zr - Arsenazo III complex can be photometrically measured in the presence of 10 milligrams of Mo.

(2) The Zr - Arsenazo III complex can be photometrically measured in the presence of 10 milligrams of Re.

(3) The Zr - Arsenazo III complex can be photometrically measured in the presence of 1 milligram of Ti.

It is evident that the Zr content of the molybdenum-base alloys under investigation can be photometrically measured without prior separation.

A calibration curve for Hf was established by placing 0, 10, 30, and 50 micrograms of a standard Hf solution in 50-milliliter volumetric flasks, each containing 25 milliliters of 9N HCl. To each solution 4 milliliters of Arsenazo III solution were added. The volumes were adjusted to 50 milliliters with 9N HCl and thoroughly mixed. The Hf complex was photometrically measured at 670 nanometers by using a 1-centimeter cell. A plot of the optical densities measured indicated that, at this Hf concentration, Beer's law was obeyed (see fig. 8).

Another calibration curve for Hf was established by placing 0, 2, 4, 6, 8, and 10 micrograms of a standard Hf solution in 50-milliliter volumetric flasks, each containing 25 milliliters of 9N HCl. To each solution 4 milliliters of Arsenazo III solution were added. The volumes were adjusted to 50 milliliters with 9N HCl and thoroughly mixed. The Hf complex was photometrically measured at 670 nanometers by using a 2-centimeter cell. A plot of the optical densities indicated that Beer's law was obeyed (see fig. 9).

VERIFICATION OF PROPOSED TECHNIQUE

Appropriate sample weights of the four molybdenum-base alloys pertinent to this report were dissolved in aqua regia. When the metal sample had completely dissolved, the solutions were evaporated to dryness under infrared drying lamps.

The nitrates were displaced by repeated evaporation to dryness, a minimum of three times, with HCl. Prior to each addition of HCl, the sides of the beakers were washed down with H₂O.

The metal salts, now free of nitrates, were dissolved in 9N HCl. The solutions were transferred to volumetric flasks, adjusted to volume with 9N HCl, and thoroughly mixed. The solutions were stable for 2 months, and no insoluble material was visible.

Aliquots containing not more than 20 micrograms of Zr nor more than 50 micrograms of Hf were selected from the sample solutions. The aliquots were placed in 50-milliliter volumetric flasks containing 25 milliliters of 9N HCl. A similar reagent blank was prepared. To each flask were added 4 milliliters of Arsenazo III solution. The solutions were adjusted to volume with 9N HCl and thoroughly mixed.

The spectrophotometer was set at a wavelength of 670 nanometers. The instrument was adjusted to zero optical density (absorbance) against distilled water. Portions of the blank and each test solution were placed in 1-centimeter or 2-centimeter cells, depending on the concentration. The Zr or Hf complex was photometrically measured. After correcting the optical densities for the reagent blank, the concentration of Zr or Hf was obtained by reference to a standard calibration curve.

When multiple aliquots of a sample solution were carried through the Arsenazo III complex forming step, the measured optical densities of the complexes showed good agreement. This indicated that the outlined photometric procedure for determining Zr or Hf was reproducible at the concentration levels previously mentioned.

Summaries of the analytical values obtained for four molybdenum-base alloys are presented in tables III to VI.

Based on the data presented in this report, a spectrophotometric procedure for determining Zr or Hf in molybdenum-base alloys is proposed. The reagent Arsenazo III is used to form the colored complex for the photometric determination of the concentration of Zr or Hf.

RECOMMENDED PROCEDURE

Based on the experimental data presented, the following analytical procedure is recommended:

Weigh an appropriate-sized sample, based on the expected homogeneity of the alloy, and place in a 250-milliliter beaker. Dissolve the sample in aqua regia. When the sample has completely dissolved, remove the cover glass and evaporate the solution to dryness under infrared drying lamps.

Cool the residue. Rinse down the sides of the beaker with water, add 20 milliliters of HCl, and again evaporate the solution to dryness under the lamps. Repeat this oper-

ation at least twice more for the complete removal of all nitrates. Cool the residue. Add 20 to 30 milliliters of 9N HCl to dissolve the metal salts.

Transfer the sample solution to a suitable volumetric flask. Make to volume with 9N HCl and thoroughly mix the solution.

Transfer a suitable aliquot, containing not more than 20 micrograms of Zr nor more than 50 micrograms of Hf, of the sample solution to a 50-milliliter volumetric flask containing 25 milliliters of 9N HCl. Prepare a similar reagent blank. Add 4 milliliters of Arsenazo III solution to each flask. Make to volume with 9N HCl and mix the solution thoroughly.

Set the spectrophotometer at 670 nanometers. Adjust the instrument to zero optical density (absorbance) against distilled water. Portions of the reagent blank and each test solution are placed in 1-, 2-, or 5-centimeter cells, and their optical densities are measured. The net readings are obtained after correcting for the density of the reagent blank.

The concentration of Zr or Hf is found by reference to a standard calibration curve.

SUMMARY OF RESULTS

1. A simple analytical procedure, using the reagent Arsenazo III, was established for determining the zirconium or hafnium content in molybdenum-base alloys.

2. The spectrophotometric procedure described permits the direct determination of zirconium or hafnium content without a prior separation. Neither molybdenum, rhenium, nor titanium interfered with the color complex.

3. Repetitive analyses showed good agreement. This indicates that the outlined analytical procedure is capable of yielding reproducible analytical values.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 21, 1971,
134-03.

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TABLE I. - IONIC RADII OF VARIOUS
ELEMENTS THAT MIGHT BE
COMPONENTS OF
MOLYBDENUM-
BASE ALLOYS

Element	Ionic radius, Å
Chromium, Cr	0.52
Iron, Fe	.64
Molybdenum, Mo	.62
Niobium, Nb	.69
Rhenium, Re	.56
Tantalum, Ta	.68
Titanium, Ti	.68
Vanadium, V	.59

TABLE II. - COMPOSITION OF MOLYBDENUM-BASE
ALLOYS USED IN THIS INVESTIGATION

Alloy	Elements contained in alloy in addition to molybdenum, percent			
	Titanium, Ti	Zirconium, Zr	Hafnium, Hf	Rhenium, Re
TZM	0.50	0.08	-----	---
TZM (MAB)	.50	.10	-----	---
MoHC	----	----	0.32 to 1.22	---
MoReTiZr	1.12	.12	-----	6.6

TABLE III. - DETERMINATION OF
ZIRCONIUM IN TZM ALLOY BY
SPECTROPHOTOMETRIC
METHOD

[Nominal composition: Ti, 0.50 percent;
Zr, 0.10 percent; Mo, balance.]

Sample size, mg	Cell size, cm	Zirconium concentration, percent (a)	Average zirconium concentration, percent
50.8	1	0.090 .086	} ^a 0.090
10.16	5	0.092 .092	
25.00	1	0.092 .092	
50.03	1	0.088 .088	

^aAverage for MAB data of ref. 3 also 0.090.

TABLE IV. - DETERMINATION OF HAFNIUM
IN MOLYBDENUM-HAFNIUM-CARBON
ALLOYS BY X-RAY FLUORESCENCE
AND SPECTROPHOTOMETRIC
METHODS

Sample	X-ray fluorescence method (a)	Spectrophotometric method
	Hafnium concentration, percent	
MHC-20	0.167	0.176 .169
MHC-23	0.725	0.760 .774
MHC-24	1.08	1.06 1.09

^aData from ref. 4.

TABLE V. - DETERMINATION OF HAFNIUM IN
MOLYBDENUM-HAFNIUM-CARBON ALLOYS BY
GRAVIMETRIC AND SPECTROPHOTOMETRIC
METHODS

Sample	Hafnium concentration by gravimetric method, percent	Sample size, mg	Hafnium concentration by spectrophotometric method, percent	Relative standard deviation, percent
MHC-36	0.317	4	0.312 .312	} 1.6
		6	0.325 .325 .313 .313 .313	
		8	0.313 .313	
MHC-37	0.438	4	0.437 .437 .437 .437	} 1.4
		6	0.433 .433 .425 .425 .425 .442 .442	
MHC-391	1.22	2	1.25 1.25 1.25 1.30 1.30	} 2.7
		4	1.20 1.20 1.20 1.27 1.27 1.27 1.23 1.23 1.23	

TABLE VI. - DETERMINATION OF ZIRCONIUM IN
MOLYBDENUM-RHENIUM-TITANIUM-ZIRCONIUM

ALLOY, AC-67, BY GRAVIMETRIC AND
SPECTROPHOTOMETRIC METHODS

[Composition: Re, 6.6 percent; Ti, 1.1 per-
cent; Zr, 0.12 percent; Mo, balance.]

Zirconium concentration by gravimetric method, percent	Sample size, mg	Zirconium concentration by spectrophotometric method, percent	Relative standard deviation, percent
0.120	19.3	0.116	1.3
		.116	
		.116	
	28.8	.116	
		.116	
		.116	
	9.6	.119	
		.119	
		.119	

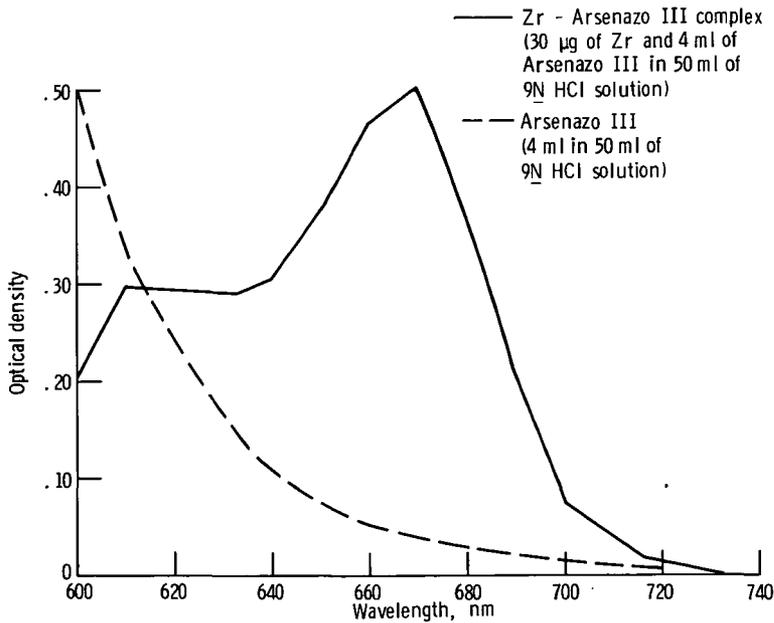


Figure 1. - Absorption spectra of Zr - Arsenazo III complex and Arsenazo III.

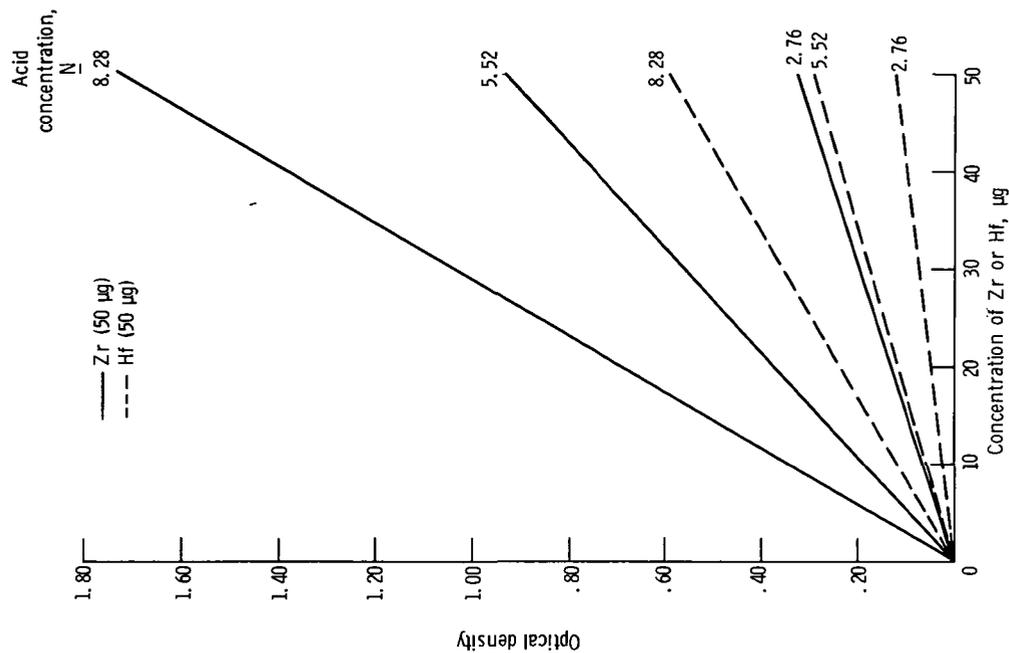


Figure 3. - Effect of acid concentration on Zr - Arsenazo III and Hf - Arsenazo III absorbance. Wavelength, 670 nanometers; 1-centimeter cell; 4 milliliters of Arsenazo III in 50-milliliter HCl solution.

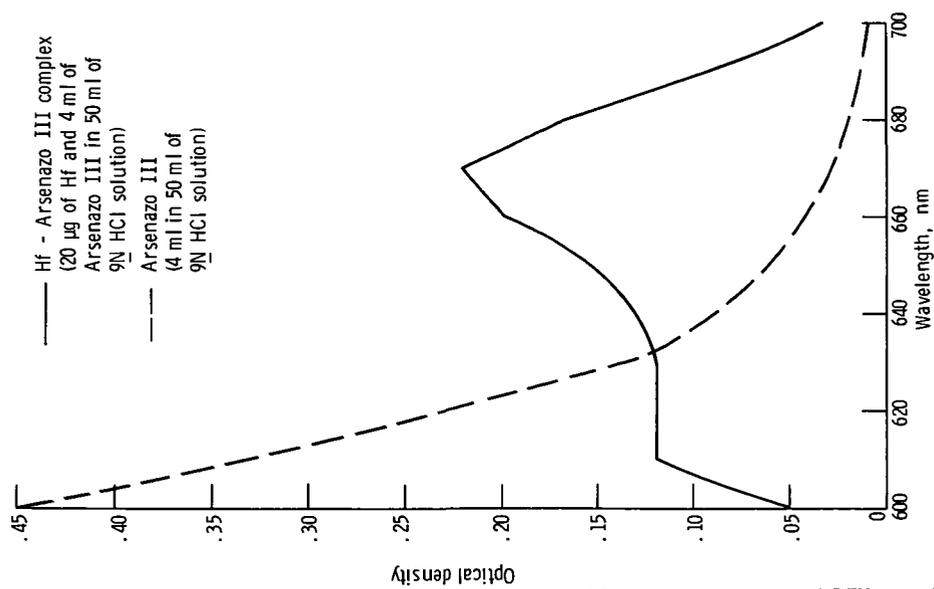


Figure 2. - Absorption spectra of Hf - Arsenazo III complex and Arsenazo III.

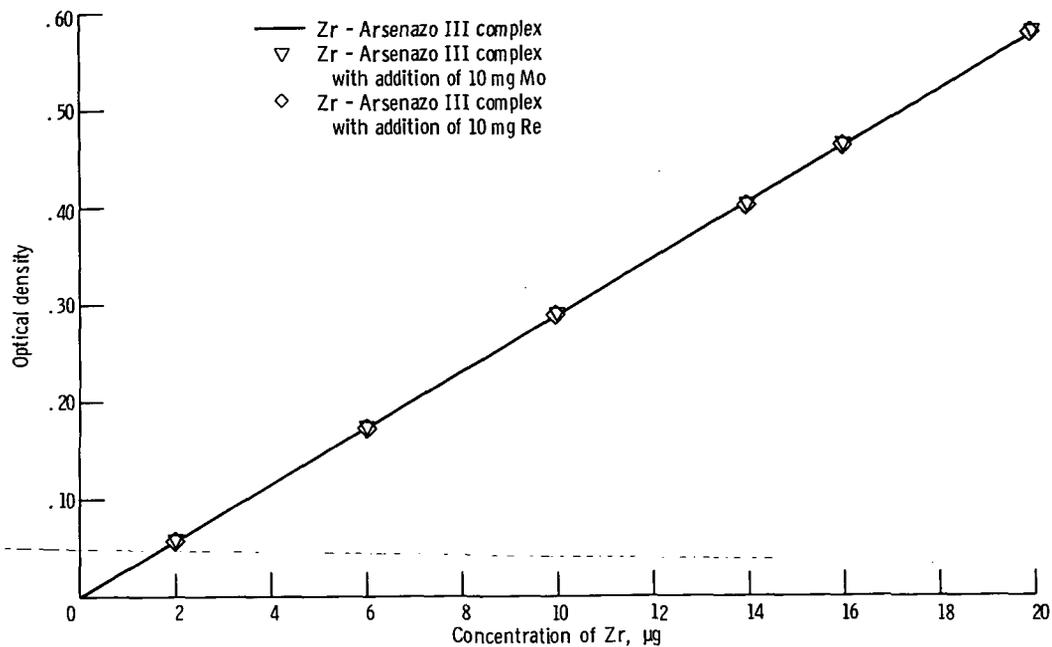


Figure 4. - Effect of Mo and Re on absorbance of Zr - Arsenazo III complex. Wavelength, 670 nanometers; 1-centimeter cell; 4 milliliters of Arsenazo III in 50 milliliters of 9N HCl solution.

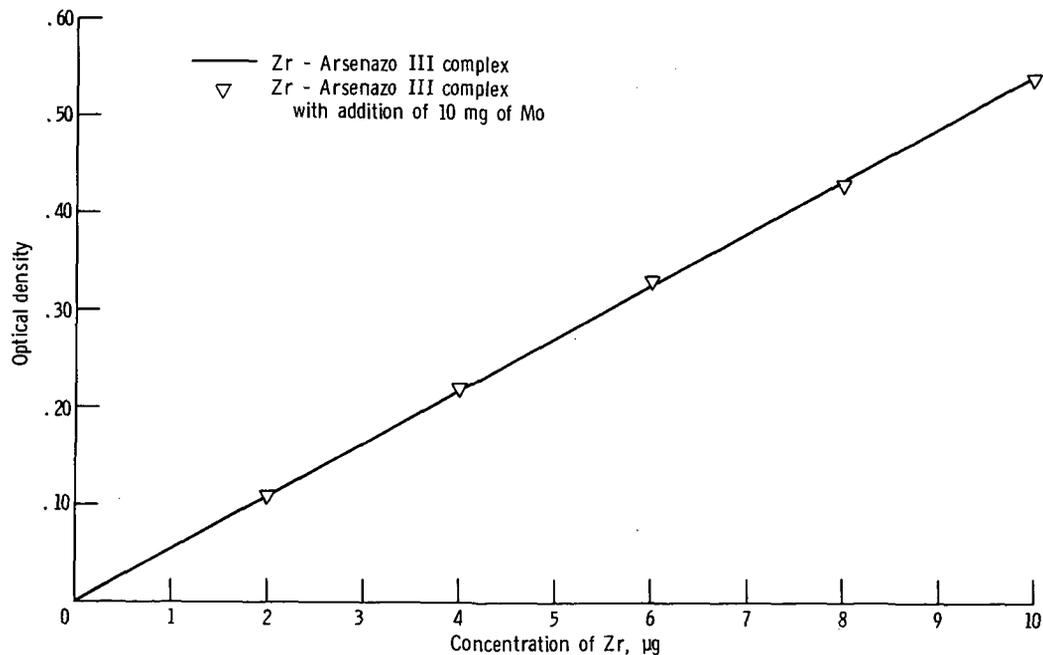


Figure 5. - Effect of Mo on absorbance of Zr - Arsenazo III complex. Wavelength, 670 nanometers; 2-centimeter cell; 4 milliliters of Arsenazo III in 50 milliliters of 9N HCl solution.

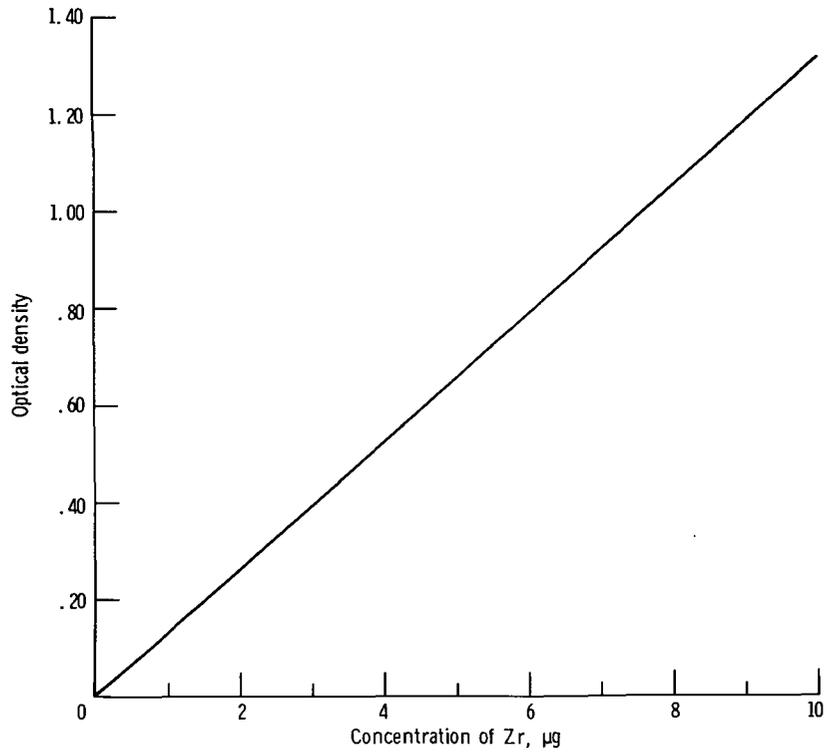


Figure 6. - Calibration curve for Zr - Arsenazo III complex. Wavelength, 670 nanometers; 5-centimeter cell; 4 milliliters of Arsenazo III in 50 milliliters of 9N HCl solution.

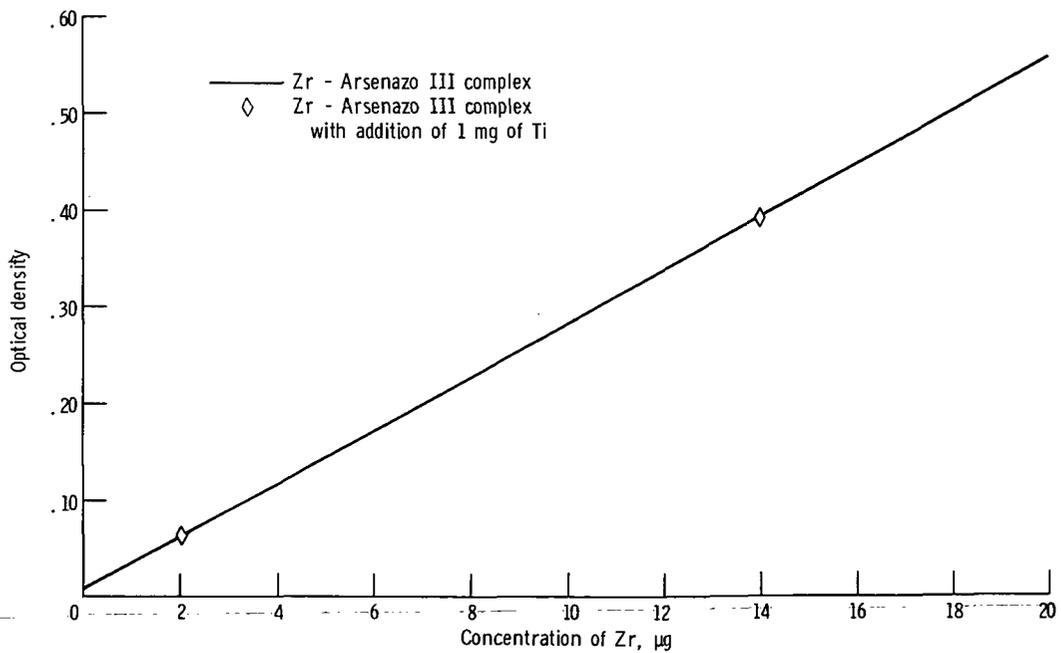


Figure 7. - Effect of Ti on absorbance of Zr - Arsenazo III complex. Wavelength, 670 nanometers; 1-centimeter cell; 4 milliliters of Arsenazo III in 50 milliliters of 9N HCl solution.

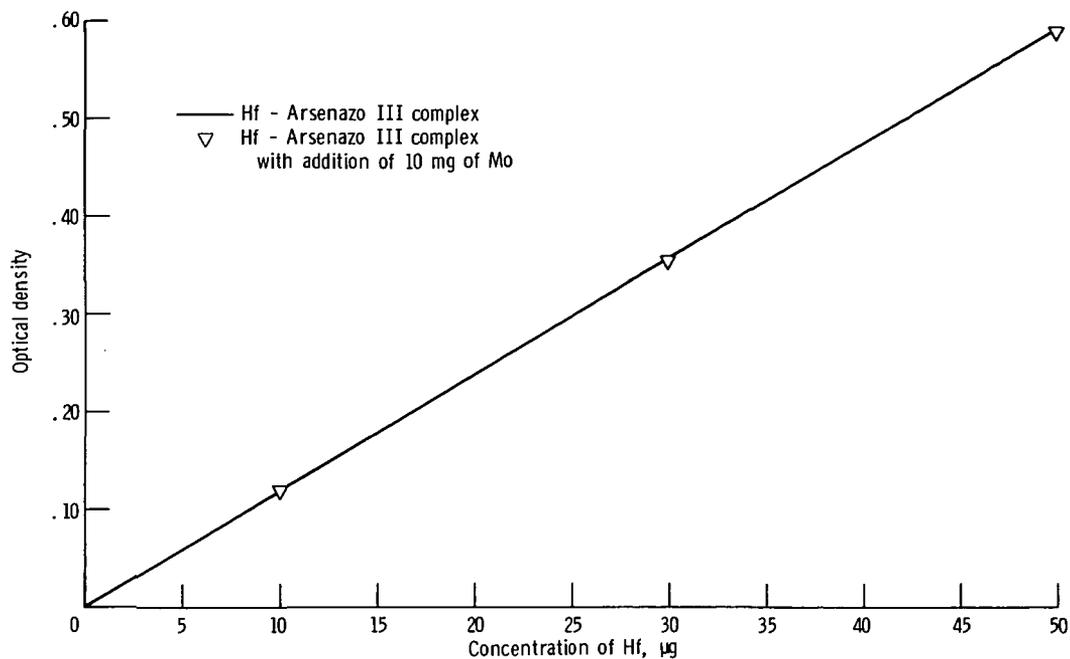


Figure 8. - Effect of Mo on absorbance of Hf - Arsenazo III complex. Wavelength, 670 nanometers; 1-centimeter cell; 4 milliliters of Arsenazo III in 50 milliliters of 9N HCl solution.

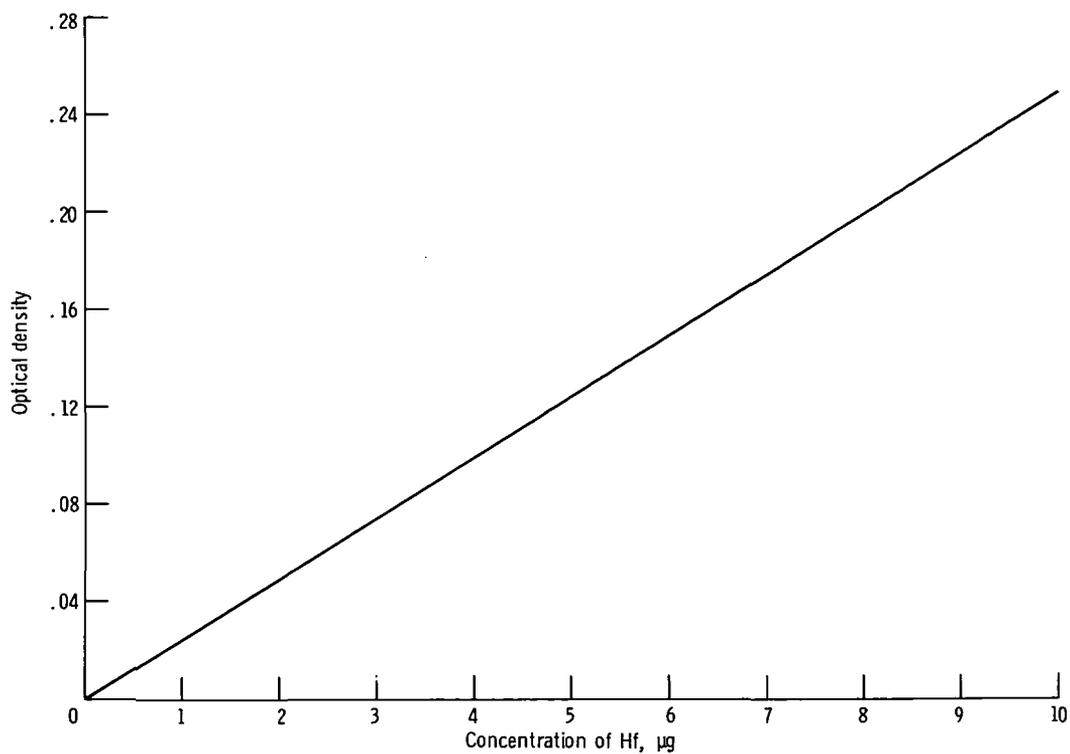


Figure 9. - Calibration curve for Hf - Arsenazo III complex. Wavelength, 670 nanometers; 2-centimeter cell; 4 milliliters of Arsenazo III in 50 milliliters of 9N HCl solution.



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