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WATERTOWN ARSENAL LABORATORIES

APPLICATIONS OF A SPECTROGRAPHIC-SOLUTION
TECHNIQUE TO ANALYSIS OF IRON, TITANIUM,
VANADIUM, AND ZIRCONIUM ALLOYS

TECHNICAL REPORT WAL TR 823.5/1

BY

FRED G. HOWELL

AND

WILLIAM K. MURRAY

DATE OF ISSUE - MARCH 1962

OMS CODE 5010.11.842

MATERIALS FOR ARMY WEAPONS AND COMBAT MOBILITY

D/A PROJECT 593-32-007

WATERTOWN ARSENAL
WATERTOWN 72, MASS.



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TITLE

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OF IRON, TITANIUM, VANADIUM, AND ZIRCONIUM ALLOYS

ABSTRACT

A spectrographic-solution technique has been applied to the analysis of certain iron, titanium, vanadium and zirconium alloys. The samples, after dissolution in an appropriate acid, are excited with a high-voltage spark using a commercial rotating-disk unit. Suitable line pairs are selected and calibration curves drawn using synthetic alloy solutions prepared from pure metals. The coefficient of variation for analysis of representative samples of each of the above alloy systems was determined.

Fred G. Howell

FRED G. HOWELL
Chemist (Anal)

William K. Murray

WILLIAM K. MURRAY
Supervisory Chemist (Anal)

APPROVED:

J. F. Sullivan

J. F. SULLIVAN
Director
Watertown Arsenal Laboratories

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INTRODUCTION

Many varied materials can be analyzed by preparing a solution of the sample and then analyzing the solution spectrographically. This technique is particularly effective when dealing with unusual metals or with a small lot of samples for which it is not practical to set up procedures which are unfamiliar to the "wet" chemist. This report describes six determinations which were performed by the spectrographic-solution technique. The approach to the development of the method is similar in each case. The samples were dissolved with appropriate acids, and synthetic standard solutions corresponding to the unknown samples exactly in composition and amount of acid present were prepared. These solutions were then excited spectrographically. The spectra were examined for suitable line pairs, modifications were made in the techniques, if required, to achieve the necessary accuracy, and, finally, calibration curves were prepared from the data.

EXPERIMENTAL

A. Selection of Line Pairs

In selecting line pairs for spectrochemical procedures, a line pair is considered to be well matched if the excitation potentials are nearly equal for both the analytical line of the element being determined and the internal standard line of the base element. A well-matched pair is less affected by variations in excitation parameters.

Even after selection of possible line pairs on the basis of excitation potentials and other factors, it is still necessary to check to determine if the intensity ratios of the lines remain essentially constant under experimental conditions. In order to minimize the amount of data needed for selection of the best line pairs, some parameter which affects the intensity ratios can be deliberately varied beyond its normal experimental range. Frisque in a study of matrix effects¹ varied the matrix and decided that the major cause of the changes in intensity ratios was due to temperature variations in the discharge.

Experimentally it is easier to vary the temperature of the discharge by varying the length of the analytical gap. This was done by Mahoney² who found that the analytical curves underwent parallel horizontal shifts as the length of the spark gap (and temperature) varied if the lines were not well matched. Consequently, it is simple to employ the constancy in the intensity ratio of the line pairs as the discharge gap is varied as a measure for matching the lines.

For the alloy systems under study, it was necessary to select suitable line pairs experimentally, since none were available in the literature. Trial exposures of the various alloy systems were made to establish optimum exposure times for covering the concentration range of the minor

constituent. For the minor constituents, only the more sensitive arc or spark lines were visible and their excitation potentials were available in the literature. Among the matrix element lines, only the weaker ones had proper transmittances, within the desirable range of 20 to 60 percent, for use as internal standard lines. Excitation potentials were not available for most of these matrix lines.

To determine what line pairs would be well matched, exposures were made in duplicate, first with an analytical gap of 3 mm, then with a gap of 5 mm. The log intensity ratios were calculated for each tentative line pair. Those line pairs that showed the least change in ratio for the two gap widths were chosen for further study.

Standard solutions covering the desired range of concentration were sparked and those line pairs giving the best analytical curves were chosen to complete this investigation.

B. Preparation of Analytical Curves

The transmittances of the selected alloy and internal standard line pairs from each set of standard solutions were converted to log intensity values by reference to the emulsion calibration curve.³ $\log I_a/I_b$ was calculated for each line pair. (I_a = intensity of alloy line; I_b = intensity of internal standard line.)

Semi-logarithmic paper was used for preparing analytical curves by plotting the concentration of the alloying element on the log axis versus the ratio of log intensities ($\log I_a/I_b$) on the linear scale.

GENERAL PROCEDURE

A. Apparatus

The apparatus employed for this investigation is as follows:

Spectrograph	Baird Associates 3-meter concave grating instrument in Eagle mounting, with a reciprocal linear dispersion of 5.5 Å per mm.
Excitation Source	Jarrell-Ash Co. Custom Varisource, Model No. 4075.
Microphotometer	Jarrell-Ash Co. Non-Recording Microphotometer and Comparator, Model No. 2100.
Rotating-Disk Unit	Spex Industries, Inc. Speed of rotation is 10 rpm.
Plate Processor	Jarrell-Ash Co. Photoprocessor, Model No. 3410.

Spectrographic Plates	Eastman Kodak Spectrum Analysis Plates No. 1, 4 inches by 10 inches.
Teflon Cups	Machined from Teflon disks 1 inch in diameter and 3/8 inch thick.

B. Reagents

Standard solutions were prepared as described under the various determinations performed.

Metals of the highest purity obtainable should be used in making up standard solutions. If the best grade of metal available is contaminated with the element being determined, the impurity should be determined chemically or by the method of Ahrens.⁴

C. Excitation Parameters

The following settings on the Varisource were used for exciting the samples:

Input voltage (line)	240
Powerstat output voltage	180
Primary resistance, setting	No. 5
Auxiliary spark gap, mm	6 (approx.)
Secondary resistance, ohm	Residual
Inductance, μ h	310
Capacitance, μ f	0.005
Breaks per half cycle	4
Radio frequency current, amps	$3-1/2 \pm 1/4$

D. Electrode System

A high-purity graphite disk 1/2 inch in diameter and 1/8 inch thick rotates in a vertical plane perpendicular to the optical axis. The disk rotates at 10 rpm with its lower edge in contact with the solution contained in a Teflon cup. The upper or counter electrode is a graphite rod 1/4 inch in diameter and 2 inches long, pointed to a cone of 120 degrees included angle and spaced 4.0 mm above the upper edge of the disk.

E. Exposure Procedure

The following steps were involved in the exposure procedure:

1. Make duplicate exposures of both standard and unknown samples.
2. Fill the Teflon cup with the solution to be analyzed, using a plastic dropping pipet for solutions containing hydrofluoric acid.

3. Place the cup on the platform of the rotating-disk assembly and adjust the disk so that it rotates freely partially immersed in the solution without touching the cup.

4. Center the counter electrode directly above the rotating disk and adjust the gap to 4.0 mm.

5. Make the exposures using the exposure conditions listed under each determination.

F. Photographic Processing

The photographic processing included the following steps:

1. Process the Eastman S. A. No. 1 plates at a temperature of 70 F.

2. Develop in Kodak D-19 developer for 4-1/2 minutes, then place in an acetic acid (5%) short-stop bath for 20 seconds.

3. Fix the plates for 5 minutes in Kodak Rapid Fixer and wash for 5 minutes in running water.

4. Rinse with distilled water and dry with a warm air blower.

G. Photometry

Measure the percent transmittance of the analytical line pairs on the developed plates with the microphotometer using a slit 5 microns wide and 1.2 mm long.

RECOMMENDED PROCEDURES

Determination of Manganese in Iron

This method is recommended for the analysis of manganese in iron in the range from 0.001 to 0.02 percent.

A. Preparation of Standard Solutions

1. Iron Stock Solution (50 mg of Fe per ml)

Dissolve 10 grams of iron, which is spectroscopically free from manganese, in 150 ml of HCl (1:1). Oxidize with a minimum of HNO_3 , cool, transfer to a 200-ml volumetric flask, and dilute to volume with HCl (1:1).

2. Manganese Stock Solution (0.10 mg of Mn per ml)

Dissolve 10.0 mg of pure manganese metal in HCl (1:1). Transfer to a 100-ml volumetric flask and dilute to volume with HCl (1:1).

3. Manganese Stock Solution (0.01 mg of Mn per ml)

Transfer 10.0 ml of the manganese stock solution (0.10 mg of Mn per ml) to a 100-ml volumetric flask and dilute to volume with HCl (1:1).

4. Standard Working Solutions of Manganese in Iron

Transfer 20-ml aliquots of the iron stock solution (50 mg of Fe per ml) to each of six 50-ml Erlenmeyer flasks numbered from 1 to 6.

To flasks 1, 2, and 3 add 1.0, 2.0, and 5.0 ml, respectively, of the manganese stock solution (0.01 mg of Mn per ml). To flasks 4 and 5 add 1.0 and 2.0 ml, respectively, of the manganese stock solution (0.10 mg of Mn per ml).

Add HCl (1:1) to each of the six flasks so that each solution will have a volume of 25 ml.

The standard working solutions now represent iron solutions containing 0.000, 0.001, 0.002, 0.005, 0.010, and 0.020 percent manganese.

B. Preparation of Samples

Transfer 1-gram samples of iron alloy to 50-ml Erlenmeyer flasks and add 20 ml of HCl (1:1). Heat gently until dissolved. Oxidize by the dropwise addition of HNO_3 . Cool, dilute to 25 ± 1 ml with HCl (1:1).

C. Exposure Conditions

The following represent the exposure conditions:

Spectral region, angstroms	2405-3105, 2nd order
Slit width, microns	25
Plate line-mask width, mm	2
Spark preburn period, sec	15
Spark exposure period, sec	40
Manganese line, angstroms	2576.10
Iron line, angstroms	2565.07*

D. Analytical Curves

Prepare as described under EXPERIMENTAL, Section B.

Determination of Zirconium in Titanium

This method is recommended for the analysis of zirconium in titanium and titanium alloys at the 2 to 8 percent level.

A. Preparation of Standard Solutions

1. Accurately weigh 20, 40, 60, and 80 milligrams of pure zirconium metal and transfer each to a 100-ml plastic dropping bottle. Add to each dropping bottle accurately weighed portions of pure titanium metal so that the total weight of the zirconium and titanium in each bottle is one gram.

* This line is not listed in the M.I.T. Wavelength Tables⁵

2. Add 10 ml of water to each of the four bottles and then 10 ml of HF slowly. Add 10 ml of HCl and digest in a hot water bath until the samples are completely dissolved. Oxidize the solutions with 1 ml of HNO_3 , cool, and dilute to 50 ml with water. Place cover containing dropping pipet on each bottle.

3. Correct the standard solutions for any zirconium present in the titanium base metal (see GENERAL PROCEDURE, Section B). The standard solutions will now represent 2, 4, 6, and 8 percent zirconium plus any zirconium present in the base metal.

B. Preparation of Samples

Transfer 1-gram samples of titanium alloy to 100-ml plastic dropping bottles. Dissolve the samples as described under Preparation of Standard Solutions of Zirconium in Titanium, Paragraph 2.

C. Exposure Conditions

Spectral region, angstroms	2750-4100, 1st order
Slit width, microns	25
Plate line-mask width, mm	2
Spark preburn period, sec	15
Spark exposure period, sec	30
Zirconium lines, angstroms	3095.07
Titanium line, angstroms	3096.42

D. Analytical Curves

Prepare as described under EXPERIMENTAL, Section B.

Determination of Titanium in Vanadium

This method is recommended for the analysis of titanium in vanadium at the 0.02 to 0.5 percent level.

A. Preparation of Standard Solutions

1. Titanium Stock Solution (0.50 mg of Ti per ml of HCl (1:1)).

Transfer 100.0 mg of pure titanium metal to a 200-ml volumetric flask.

Add 100 ml of HCl (1:1), place the flask in a beaker of boiling water, and heat until the titanium is completely dissolved.

Remove the flask, cool, and dilute to volume with HCl (1:1).

2. Standard Working Solutions of Titanium in Vanadium

Transfer six 2-gram samples of pure vanadium to six 100-ml plastic dropping bottles.

Add 10 ml of HF to each bottle, and then add 2 ml of HNO₃ dropwise. Keep the bottles immersed in cold water to moderate the reaction, and let stand until the vanadium is dissolved.

To five of the bottles add 0.4, 1.0, 2.0, 4.0, and 10.0 ml of the titanium stock solution (0.50 mg of Ti per ml of HCl, (1:1)).

Add HCl (1:1) to each of the six bottles to bring the total amount of HCl (including the HCl that was added with the titanium stock solution) to 40 ml. The total volume will be approximately 50 ml.

Correct the standard working solutions for any titanium in the vanadium base metal. The standard solutions will now represent 0.02, 0.05, 0.10, 0.20, and 0.50 percent titanium plus any titanium present in the base metal.

B. Preparation of Samples

Transfer 2-gram samples of vanadium alloy to 100-ml plastic dropping bottles.

Add to each bottle 10 ml of HF and then 2 ml of HNO₃ dropwise, controlling the speed of the reaction by immersing the bottles in a cold water bath, and let stand until the samples are dissolved.

Add 40 ml of HCl (1:1) to each. The total volume will be approximately 50 ml.

C. Exposure Conditions

Spectral region, angstroms	2750-4100, 1st order
Slit width, microns	25
Plate line-mask width, mm	2
Spark preburn period, sec	15
Spark exposure period, sec	15
Titanium line, angstroms	3349.04
Vanadium line, angstroms	3376.06

D. Analytical Curves

Prepare as described under EXPERIMENTAL, Section B.

Determination of Cobalt in Zirconium

This method is recommended for the analysis of cobalt in zirconium and zirconium alloys at the 0.01 to 0.50 percent level.

A. Preparation of Standard Solutions

1. Cobalt Stock Solution (1 mg of Co per ml)

High-purity cobalt comes in small chunks very difficult to break up. A small weighed chunk of cobalt is dissolved in HCl (1:1) and transferred to a volumetric flask of suitable size and made up to volume with HCl (1:1). A volume containing 100 mg of Co is measured by means of a buret or pipet into a 100-ml volumetric flask and made up to volume with HCl (1:1).

2. Standard Working Solutions of Cobalt in Zirconium

Accurately weigh seven 2-gram samples of pure zirconium metal and transfer each to a 100-ml plastic dropping bottle.

Add 10 ml of water to each and dissolve the zirconium by the slow addition of 5 ml of HF. When dissolution is complete, dilute to 40 ml with water. Add to six of the bottles 0.2, 1.0, 2.0, 4.0, 7.0, and 10.0-ml aliquots of a standard cobalt solution (1 mg of Co per ml). Add HCl (1:1) to each of the seven bottles to bring the final volume to 50 ml.

Correct the standard solutions for any cobalt in the zirconium base metal. The standard solutions now represent 0.01, 0.05, 0.1, 0.2, 0.35, and 0.50 percent cobalt plus any cobalt present in the base metal.

B. Preparation of Samples

Transfer 2-gram samples of zirconium alloy to 100-ml plastic dropping bottles. Dissolve in HF as described under Standard Working Solutions of Cobalt in Zirconium. Add 10 ml of HCl (1:1) to each bottle and dilute to 50 ml with water.

C. Exposure Conditions

Spectral region, angstroms	2750-4100, 1st order
Slit width, microns	25
Plate line-mask width, mm	2
Spark preburn period, sec	15
Spark exposure period, sec	
Cobalt less than 0.05%	45
Cobalt greater than 0.05%	35
Cobalt line, angstroms	3453.51
Zirconium line, angstroms	3370.59

D. Analytical Curves

Prepare curves as described under EXPERIMENTAL, Section B.

Determination of Iron in Zirconium

This method is recommended for zirconium and zirconium alloys containing 0.05 to 0.25 percent iron.

A. Preparation of Standard Solutions

1. Iron Stock Solution (0.5 mg of Fe per ml)

Pipet 1 ml of iron stock solution (50 mg of Fe per ml) (as described in Section A 1 for the determination of manganese in iron) into a 100-ml volumetric flask and dilute to volume with HCl (1:1).

2. Standard Working Solutions of Iron in Zirconium

Accurately weigh five 2-gram samples of pure zirconium metal and transfer each to a 100-ml plastic dropping bottle.

Add 10 ml of water to each bottle and dissolve the zirconium by the slow addition of 5 ml of HF to each. When dissolution is complete, dilute to 40 ml with water. To four of the bottles add 2.0, 4.0, 6.0, and 10.0-ml aliquots of a standard iron solution (0.5 mg of Fe per ml). Add HCl (1:1) to each of the five bottles to bring the final volume to 50 ml.

Correct the standard solutions for any iron in the zirconium base metal. The standard solutions now represent 0.05, 0.10, 0.15, and 0.25 percent iron plus any iron present in the base metal.

B. Preparation of Samples

Transfer 2-gram samples of zirconium alloy to 100-ml plastic dropping bottles. Dissolve in HF as described under Standard Working Solutions of Iron in Zirconium. Add 10 ml of HCl (1:1) to each and dilute to 50 ml with water.

C. Exposure Conditions

Spectral region, angstroms	2405-3105, 2nd order
Slit width, microns	25
Plate line-mask width, mm	2
Spark preburn period, sec	15
Spark exposure period, sec	20
Iron line, angstroms	2973.17
Zirconium line, angstroms	2973.69*

* Not listed in the M.I.T. Wavelength Tables.⁵ Listed in the Oak Ridge Table.⁶

D. Analytical Curves

Prepare curves as described under EXPERIMENTAL, Section B.

Determination of Vanadium in Zirconium

This method is recommended for the analysis of vanadium in zirconium and zirconium alloys at the 0.05 to 0.35 percent level.

A. Preparation of Standard Solutions

1. Vanadium Stock Solution (1 mg of V per ml)

Weigh 100 mg of high-purity vanadium metal into a 100-ml beaker, add 5 ml of HNO_3 to dissolve and heat gently to complete dissolution. Add about 50 ml HCl (1:1) and transfer to a 100-ml volumetric flask. Dilute to volume with HCl (1:1).

2. Standard Working Solutions of Vanadium in Zirconium

Accurately weigh six 2-gram samples of pure zirconium metal and transfer each to a 100-ml plastic dropping bottle.

Add 10 ml of water to each bottle and dissolve the zirconium by the slow addition of 5 ml of HF . When dissolution is complete, dilute to 40 ml with water. To five of the bottles add 1.0, 2.0, 3.0, 5.0, and 7.0-ml aliquots of a standard vanadium solution (1 mg of V per ml). Add HCl (1:1) to each of the six bottles to bring the final volume to 50 ml.

Correct the standard solution for any vanadium in the zirconium base metal. The standard solutions now represent 0.05, 0.10, 0.15, 0.25, and 0.35 percent vanadium plus any vanadium present in the base metal.

B. Preparation of Samples

Transfer 2-gram samples of zirconium alloy to 100-ml plastic dropping bottles. Dissolve in HF as described under Standard Working Solutions of Vanadium in Zirconium. Add 10 ml of HCl (1:1) to each and dilute to 50 ml with water.

C. Exposure Conditions

Spectral region, angstroms	2750-4100, 1st order
Slit width, microns	25
Plate line-mask width, mm	2
Spark preburn period, sec	15
Spark exposure period, sec	30
Vanadium line, angstroms	3118.38
Zirconium line, angstroms	3115.72

D. Analytical Curves

Prepare curves as described under EXPERIMENTAL, Section B.

PRECISION OF RESULTS

The precision of the foregoing determinations was checked by running representative samples of each alloy system ten times on one spectrographic plate.

One sample solution of each alloy was prepared as described under each alloy. Five portions of each solution were then taken for analysis. This procedure eliminated the possibility of error due to segregation in the alloys. Two exposures were made with each portion of the sample solution, changing the electrodes between each exposure. This was in accord with the usual analytical technique employed.

The standard deviation (σ) was determined using the formula:

$$\sigma = \sqrt{\frac{\sum (X_1 - \bar{X})^2}{n - 1}}$$

where X_1 = percent of the element determined from each exposure

$\bar{X}$ = average of ten percentages determined

n = number of exposures

The coefficient of variation (v) is calculated from the formula:

$$v = \frac{\sigma}{\bar{X}} \times 100$$

Tables I through VI show the actual percentages obtained for each run of the samples. The coefficient of variation ranges from 0.57 percent for iron in zirconium to 6.57 percent for titanium in vanadium. The 0.57 value is probably lower than could be obtained consistently with this procedure, and likewise the 6.57 percent value should be capable of being lowered with more experience or slight changes in technique.

The coefficient of variation does not necessarily measure the accuracy of a determination. In the spectrographic analysis of solids, variations in the matrix composition or condition can cause the accuracy to be much less than the precision as measured by the coefficient of variation. It was not possible in this work to measure the accuracy directly since no samples of accurately known analysis were available. However, the advantage of solution techniques is that all variation due to matrix composition

and past history can be eliminated if the synthetic reference solutions are prepared to correspond exactly to the sample solutions. In the case of the binary alloys analyzed in this report the preparation of synthetic standards corresponding exactly to the samples was possible. The largest source of error was in the analysis of the "pure" elements used to prepare the synthetic standards. If the analysis of these elements was exact, the accuracy of the determinations should be equal to the precision which was found.

CONCLUSIONS

Spectrographic-solution analytical techniques can be successfully applied to the determination of many metallic elements in various base metals. When this method is applicable, reasonable accuracy can be obtained in the analysis of unusual alloys with a minimum of time and difficulty.

TABLE I
REPEATABILITY OF MANGANESE DETERMINATION IN IRON

Exposure No.	Manganese, % (X_1)	Difference ($X_1 - \bar{X}$)
1	0.0156	-.00038
2	0.0160	+.00002
3	0.0163	+.00032
4	0.0158	-.00018
5	0.0163	+.00032
6	0.0156	-.00038
7	0.0160	+.00002
8	0.0158	-.00018
9	0.0161	+.00012
10	0.0163	+.00032

Average ($\bar{X}$) = 0.01598

Standard deviation = 0.00027

Coefficient of variation = 1.72%

NOTE: Sample was NBS Ingot Iron Standard 55c, certified as containing 0.016% Mn

TABLE II
REPEATABILITY OF ZIRCONIUM DETERMINATION IN TITANIUM

Exposure No.	Zirconium, % (X_1)	Difference ($X_1 - \bar{X}$)
1	4.83	-.131
2	5.13	+.169
3	5.02	+.059
4	5.04	+.079
5	5.00	+.039
6	5.00	+.039
7	4.87	-.091
8	5.00	+.039
9	4.85	-.111
10	4.87	-.091

Average ($\bar{X}$) = 4.961

Standard deviation = 0.0994

Coefficient of variation = 2.00%

NOTE: Sample was MR 336, 5% Zr in Ti, nominal analysis

TABLE III
REPEATABILITY OF TITANIUM DETERMINATION IN VANADIUM

Exposure No.	Titanium, % (X_i)	Difference ($X_i - \bar{X}$)
1	0.321	+0.0257
2	0.280	-0.0153
3	0.320	+0.0247
4	0.302	+0.0077
5	0.299	+0.0047
6	0.307	+0.0117
7	0.304	+0.0087
8	0.264	-0.0313
9	0.280	-0.0153
10	0.276	-0.0193

Average ($\bar{X}$) = 0.2953

Standard deviation = 0.0194

Coefficient of variation = 6.57%

NOTE: Sample was MR 347, 0.3% Ti in V, nominal analysis

TABLE IV
REPEATABILITY OF COBALT DETERMINATION IN ZIRCONIUM

Exposure No.	Cobalt, % (X_i)	Difference ($X_i - \bar{X}$)
1	0.0495	-0.00106
2	0.0520	+0.00144
3	0.0482	-0.00236
4	0.0497	-0.00086
5	0.0512	+0.00064
6	0.0533	+0.00274
7	0.0502	-0.00036
8	0.0502	-0.00036
9	0.0500	-0.00056
10	0.0513	+0.00074

Average ($\bar{X}$) = 0.0506

Standard deviation = 0.00144

Coefficient of variation = 2.84%

NOTE: Sample was MR 672, 0.05% Co in Zr, nominal analysis

TABLE V
REPEATABILITY OF IRON DETERMINATION IN ZIRCONIUM

Exposure No.	Iron, % (X_1)	Difference ($X_1 - \bar{X}$)
1	0.150	+0.0005
2	0.149	-0.0005
3	0.149	-0.0005
4	0.149	-0.0005
5	0.149	-0.0005
6	0.150	+0.0005
7	0.150	+0.0005
8	0.151	+0.0015
9	0.148	-0.0015
10	0.150	+0.0005

Average ($\bar{X}$) = 0.1495

Standard deviation = 0.00085

Coefficient of variation = 0.57%

NOTE: Sample was MR 670, 0.15% Fe in Zr, nominal analysis

TABLE VI
REPEATABILITY OF VANADIUM DETERMINATION IN ZIRCONIUM

Exposure No.	Vanadium, % (X_1)	Difference ($X_1 - \bar{X}$)
1	0.122	+0.0089
2	0.110	-0.0031
3	0.117	+0.0039
4	0.112	-0.0011
5	0.117	+0.0039
6	0.102	-0.0111
7	0.115	+0.0019
8	0.112	-0.0011
9	0.112	-0.0011
10	0.112	-0.0011

Average ($\bar{X}$) = 0.1131

Standard deviation = 0.00528

Coefficient of variation = 4.67%

NOTE: Sample was MR 664, 0.1% V in Zr, nominal analysis

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