R-TR-75-012

APPLICATION OF ATOMIC ABSORPTION SPECTROPHOTOMETRIC TECHNIQUES TO THE ANALYSIS OF COPPER-BASE ALLOYS

by

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MARCH 1975

TECHNICAL REPORT

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REPORT DOCUMENTATION PAGE

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1. REPORT NUMBER: R-TR-75-012	2. GOVT ACCESSION NO.
3. RECIPIENT'S CATALOG NUMBER	
4. TITLE *(and Subtitle)*: APPLICATION OF ATOMIC ABSORPTION SPECTROPHOTOMETRIC TECHNIQUES TO THE ANALYSIS OF COPPER-BASE ALLOYS	5. TYPE OF REPORT & PERIOD COVERED: Technical Report Nov 73 to Jun 74
	6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s): Elizabeth A. Peterson	8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS: CDR, Rock Island Arsenal GEN Thomas J. Rodman Laboratory Rock Island, IL 61201	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: PRON A1-4-R0020-01-AW-M1 AMS Code 685801-49-11100
11. CONTROLLING OFFICE NAME AND ADDRESS: Army Materials and Mechanics Research Center ATTN: AMXMR-MQ Watertown, MA 02172	12. REPORT DATE: March 1975 13. NUMBER OF PAGES: 28
14. MONITORING AGENCY NAME & ADDRESS *(if different from Controlling Office)*	15. SECURITY CLASS. *(of this report)*: UNCLASSIFIED 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT *(of this Report)*

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT *(of the abstract entered in Block 20, if different from Report)*

18. SUPPLEMENTARY NOTES

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*

1. Atomic Absorption
2. Copper-base Alloys
3. Analysis

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

A simplified method of analysis for the determination of manganese, copper, zinc, iron, nickel, aluminum and tin in copper-base alloys has been developed in which a single dissolution of the sample is used. Results of repeated determinations on secondary standards prepared at this Laboratory and on National Bureau of Standards (NBS) reference materials in which aqueous standard solutions were used were in good agreement with accepted values.

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FOREWORD

This project has been accomplished as part of the US Army Materials Testing Technology Program, which has for its objective the timely establishment of testing techniques, procedures, or prototype equipment (in mechanical, chemical, or nondestructive testing) to insure efficient inspection methods for materiel/material procured or maintained by AMC.

CONTENTS

OBJECTIVE

The objective of this investigation was to develop an atomic absorption analysis technique for the determination of Cu, Zn, Mn, Fe, Ni, Al, and Sn in copper-base alloys.

INTRODUCTION

At Rock Island Arsenal, copper-base alloy secondary standards were prepared for use in emission and X-ray fluorescence analysis.[1] These secondary standards were thoroughly analyzed at the time that they were made. However, two of these standards, #'s 18 and 20, manganese bronzes, yield nearly identical zinc results on the emission spectrograph, although their previous analyses show them to be different. Therefore, the series of high zinc standards containing these two was included in this study in order to check the previous results.

In addition, when samples of wire or other material, received for analysis, were unsuited for X-ray fluorescence or emission spectrographic analysis, slow gravimetric methods had to be used. Development of atomic absorption methods would allow faster analyses and would provide a means of checking the concentration of zinc in the RIA standards.

PROCEDURE

A literature search was made to determine if previous work had been done on the subject. The search revealed that several copper-base alloy analyses have been developed, in which atomic absorption spectrophotometry was used.[2,3,4] The method of analysis described in Reference 4 ("AA Cookbook") was used as the basis for this study.

The optimum absorbance range in atomic absorption spectrophotometry is 0.1 to 0.5 absorbance units. To prepare solutions having absorbances within this optimum range, Reference 4 requires several different sample sizes and a number of dilutions to determine elements in copper-base alloys due to the wide spread of elemental concentrations. This investigation was initiated to determine if a method requiring only one sample size and a single sample dilution could be developed.

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1 Moorhead, L. H., and O. J. Littig, X-ray Spectrochemical Methods for Analysis of Copper Alloys, Technical Report U.S. Army Weapons Command, Rock Island Arsenal, Research & Engineering Division #67-849, 1967.

2 Capacho-Delgado, L., and D. C. Manning, Atomic Absorption Newsletter, 5,1, 1966.

3 Elwell, W. T. and J. A. F. Gidley, Atomic Absorption Spectroscopy, 2nd Rev. Ed., Pergamon Press, 1966.

4 Analytical Methods for Atomic Absorption Spectrophotometry, Perkin-Elmer Corporation, Norwalk, Connecticut, March, 1971.

Each element has a primary wavelength chosen for routine operation because the greatest amount of absorption is obtained at this wavelength. However, other wavelengths may be used which result in smaller amounts of absorption. Similarly different burner positions relative to the hollow cathode light beam are also used. A burner parallel to the light beam permits the greatest amount of absorption of the beam by atoms in the flame. By making the burner position less and less parallel to the beam, light absorption may be attenuated.

A half-gram sample of NBS 62D, manganese bronze, was dissolved by the method of Reference 4 and diluted to 100 ml. Working with the elemental concentrations found in this solution, various wavelengths and burner positions were investigated to see if absorbance values within useful limits could be obtained. Conditions were found for all elements present in the manganese bronze in low concentrations. A 1:100 sample dilution was then made and parameters were determined for the elements present in high concentrations.

A Perkin-Elmer Model 303 Atomic Absorption Spectrophotometer, equipped with a Digital Concentration Readout, DCR-1, was used for this investigation. The operating conditions for each element are listed in Table 1.

Stock solutions of known metal concentration were procured commercially with the exception of Zn, Sn, Al, and Fe. Solutions of these metals were prepared with pure metals (at least 99.9%) as described in Reference 4. All acids and other chemicals were reagent grade. One set of standard solutions containing the elements of interest in various concentrations was used. This set of standards is more fully described in Table 2. Each standard solution contained five percent by volume of concentrated HCl (hydrochloric acid) in addition to the elements of interest.

Half-gram (0.5000) chip samples were weighed into 300 ml Erlenmeyer flasks. Ten ml of 1:1 HCl and one-half ml of concentrated HNO_3 (nitric acid) were added to the flasks. The flasks were heated until dissolution was complete. In some instances, more concentrated HNO_3 was added dropwise to effect complete dissolution. Once the chips were dissolved, solutions were boiled at least five minutes longer to completely remove any excess unreacted HNO_3. The solutions were then cooled and transferred to 100 ml volumetric flasks each containing 10 ml of 1:1 HCl and brought to volume with distilled water. Finally, a 1:100 dilution of each of these chip solutions was made.

Parameters were adjusted for each element on the atomic absorption spectrophotometer, and the samples and standards were aspirated alternately from low concentration to high, and back again. Average values (absorbance or set concentration readout) of aqueous standards versus percentages of element were plotted to give calibration curves from which concentrations were determined.

In this manner, the NBS reference materials were treated as unknowns, and the results were compared with the certified values for evaluation of the method.

RESULTS AND DISCUSSION

The analysis procedure described above represents the best combination to date of sample size with a minimum of dilutions for the analysis of seven different elements in copper-base alloys. NBS reference materials were analyzed by this procedure. The results of these analyses are presented in Tables 3 through 9. Four locally prepared secondary standards were also analyzed by this method. Results of these analyses are given in Tables 10 through 16. Table 12 shows the zinc analyses for the locally prepared standards. Although the results for zinc in standards 18 and 20 are higher than previously determined, the method does show that the concentration of zinc in the two standards is not the same. Therefore, the anomaly of the emission spectrographic zinc results for these two standards 18 and 20 must be due to another factor, perhaps an optical interference.

The tables contain the arithmetic mean, standard deviation, coefficient of variation, and deviation of mean from certified or previously determined value for each elemental determination. The coefficient of variation in percentage is defined as $\frac{\text{standard deviation}}{\text{mean}} \times 100$.

In general, the results of the analyses were acceptable. The determinations of iron, copper, zinc, manganese and aluminum were straightforward and without problem. Aluminum always was determined last. Just before the aluminum determination, one-half gram of sodium chloride (NaCl) was added to each sample and standard solution, and was mixed until dissolution was complete. The addition of sodium to the solutions prevents ionization of the aluminum in the hot N_2O-C_2H_2 flame.[5]

Some difficulties were encountered with the determination of the two other elements. Each of these elements is discussed below:

Nickel. Neither the nickel plot of concentration of aqueous standards versus absorbance nor a similar plot of the NBS reference material samples (See Figure) is straight. Such curvature, known as chemical interference, is due to nickel compounds not being broken down readily in the flame to form neutral atoms suitable for light absorption.[6] If care is exercised so that samples and standards are closely matched, this curvature is no longer a problem.

Tin. In a half-gram sample diluted to 100 ml the concentration of tin ranged from 5 to 60 ppm. This level of tin is near the detection limit for this element. However, use of scale expansion, while causing increases in the coefficient of variation, does permit acceptable determinations.

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[5] Slavin, W. Atomic Absorption Spectroscopy, Interscience Publishers, 1968, p. 79.

[6] Ibid., p. 78.

SUMMARY

In the atomic absorption spectrophotometric method of analysis described, a single sample dilution and easily prepared aqueous standards are used to determine Cu, Zn, Mn, Al, Sn, Fe and Ni in copper-base alloys. The method is presently used at Rodman Laboratory where sample size or shape precludes the use of X-ray fluorescence or optical emission spectroscopy, and where appropriate spectroscopic standards are unavailable.

RECOMMENDATIONS

Since the method has proved useful to Rodman Laboratory, this method is recommended for use in other laboratories, if proper equipment is available.

TABLE 1

OPERATING PARAMETERS FOR PERKIN-ELMER
303 ATOMIC ABSORPTION SPECTROPHOTOMETER

Element	Hollow Cathode Tube Current	Wavelength	Slit Width Setting	Burner	Gases	Sample Strength	Readout Conditions
Cu	25ma	324.7nm	3(0.2nm)	3-Slot 1 notch from parallel to beam	Air-C_2H_2	1:100 dilution	Abs. Mode
Zn	15ma	213.8nm	5(2.0nm)	N_2O 1 notch from perpendicular to beam	Air-C_2H_2	1:100 dilution	Abs. Mode
Ni	25ma	232.0nm	3(0.2nm)	3-Slot	Air-C_2H_2	Full	Abs. Mode
Mn	20ma	279.5nm	4(0.7nm)	3-Slot	Air-C_2H_2 lean	1:100 dilution	Abs. Mode
Fe	30ma	248.3nm	3(0.2nm)	3-Slot perpendicular to beam	Air-C_2H_2	Full	Abs. Mode
Sn	30ma	224.6nm	4(0.7nm)	3-Slot	Air-C_2H_2	Full	Conc. Mode
Al	25ma	257.5nm	3(0.2nm)	N_2O	N_2O-C_2H_2	Full with NaCl added to samples & standards	Abs. Mode

TABLE 2

AQUEOUS STANDARDS USED TO ANALYZE COPPER-BASE ALLOYS

Standard	% Element Present						
	Al	Cu	Zn	Ni	Mn	Fe	Sn
A	4.0	48.0	4.0	0.08	0.08	0.4	0.2
B	6.0	60.0	8.0	0.20	0.20	0.8	0.4
C	8.0	68.0	20.0	0.40	0.28	1.2	0.8
D	10.0	80.0	28.0	0.48	0.40	2.0	1.0
E	12.0	88.0	40.0	0.60	0.60	2.8	1.2

All solutions contain 5% by volume HCl.

TABLE 3

ANALYSIS OF COPPER IN NBS REFERENCE MATERIAL

% Copper

Reference Material

	124	164	62D	62B	37A	37D
Certified Value	83.77	63.77	59.07	57.39	70.29	70.78
Trial 1	87.3	63.0	54.6	-	71.0	70.2
Trial 2	79.4	68.1	-	60.1	76.5	75.5
Trial 3	85.0	63.8	59.8	56.0	70.7	70.0
Trial 4	83.5	64.0	60.7	57.2	69.6	69.2
Trial 5	86.7	65.3	59.8	58.3	72.9	71.0
Trial 6	-	63.4	-	57.2	-	-
Trial 7	-	64.3	-	56.6	-	-
Trial 8	-	60.8	-	55.9	-	-
Mean	84.4	64.1	58.7	57.3	72.1	71.2
Standard Deviation	3.16	2.08	2.78	1.47	2.71	2.50
Coefficient of Variation	3.74	3.24	4.74	2.57	3.76	3.51
Deviation of Mean from Certified Value	+0.61	+0.32	-0.35	-0.06	+1.85	+0.40

TABLE 4

ANALYSIS OF ZINC IN NBS REFERENCE MATERIAL

% Zinc

Reference Material

	124	164	62D	62B	37A	37D
Certified Value	5.46	21.88	37.14	37.97	26.89	26.65
Trial 1	-	22.4	-	40.0	27.7	27.5
Trial 2	-	21.0	-	37.9	26.6	26.3
Trial 3	5.46	22.5	38.3	37.8	28.0	26.4
Trial 4	5.30	23.7	40.7	40.5	29.3	27.8
Trial 5	5.70	23.8	40.0	40.6	29.7	28.2
Trial 6	-	23.3	-	38.5	-	-
Trial 7	-	22.3	-	40.0	-	-
Trial 8	-	23.4	-	39.5	-	-
Mean	5.49	22.8	39.7	39.4	28.3	27.2
Standard Deviation	0.20	0.94	1.23	1.13	1.25	0.85
Coefficient of Variation	3.64	4.12	3.10	2.87	4.42	3.12
Deviation of Mean from Certified Value	+0.03	+0.92	+2.56	+1.43	+1.41	+0.55

TABLE 5

ANALYSIS OF TIN IN NBS REFERENCE MATERIAL

% Tin

Reference Material

	164	62D	62B	37A	37D
Certified Value	0.63	0.38	0.96	1.01	0.97
Trial 1	0.56	-	0.76	0.86	0.80
Trial 2	0.63	-	0.87	0.87	0.87
Trial 3	0.63	0.43	0.93	0.93	0.93
Trial 4	0.63	0.39	0.94	0.95	0.92
Trial 5	0.64	0.40	0.88	0.85	0.91
Trial 6	0.65	-	0.94	-	-
Trial 7	0.62	-	0.89	-	-
Trial 8	0.58	-	0.88	-	-
Mean	0.62	0.38	0.89	0.89	0.89
Standard Deviation	0.031	0.02	0.058	0.04	0.05
Coefficient of Variation	5.00	5.26	6.52	4.49	5.62
Deviation of Mean from Certified Value	-0.01	0.0	-0.07	-0.12	-0.08

TABLE 6

ANALYSIS OF IRON IN NBS REFERENCE MATERIAL

% Iron

Reference Material

	164	62B
Certified Value	2.52	0.82
Trial 1	2.47	0.78
Trial 2	2.40	0.73
Trial 3	2.48	0.77
Mean	2.45	0.76
Standard Deviation	0.044	0.026
Coefficient of Variation	1.80	3.42
Deviation of Mean from Certified Value	-0.07	-0.06

TABLE 7

ANALYSIS OF NICKEL IN NBS REFERENCE MATERIAL

% Nickel

Reference Material

	124	164	62D	62B	37A	37D
Certified Value	0.45	0.046	0.28	0.27	0.52	0.58
Trial 1	0.44	0.046	0.28	0.27	0.36	0.54
Trial 2	0.43	0.046	0.28	0.26	0.35	0.54
Trial 3	0.43	0.046	0.27	0.26	0.36	0.54
Trial 4	-	-	-	0.27	-	-
Trial 5	-	0.048	-	0.26	-	-
Trial 6	-	0.040	-	0.26	-	-
Mean	0.43	0.045	0.28	0.26	0.36	0.54
Standard Deviation	0.006	0.003	0.006	0.005	0.006	0.00
Coefficient of Variation	1.40	6.67	2.14	1.92	1.67	-
Deviation of Mean from Certified Value	-0.02	-0.001	-	-0.01	-0.16	-0.04

TABLE 8

ANALYSIS OF ALUMINUM IN NBS REFERENCE MATERIAL

% Aluminum

Reference Material

	164	62D	62B
Certified Value	6.22	1.23	0.97
Trial 1	6.49	1.25	1.02
Trial 2	6.26	1.40	0.80
Trial 3	6.60	1.22	0.90
Trial 4	6.40	-	0.79
Trial 5	6.60	-	0.40
Trial 6	5.75	-	0.80
Trial 7	6.00	-	0.60
Mean	6.30	1.29	0.76
Standard Deviation	0.32	0.096	0.20
Coefficient of Variation	5.08	7.44	26.3
Deviation of Mean from Certified Value	+0.08	+0.06	-0.21

TABLE 9

ANALYSIS OF MANGANESE IN NBS REFERENCE MATERIAL

	% Manganese		
	Reference Material		
	164	62D	62B
Certified Value	4.68	0.66	1.29
Trial 1	4.75	0.53	1.20
Trial 2	4.60	0.62	1.25
Trial 3	4.79	-	1.30
Trial 4	4.98	-	1.23
Trial 5	4.65	0.58	1.20
Trial 6	4.50	-	1.18
Trial 7	4.62	-	1.20
Trial 8	4.58	-	1.26
Mean	4.68	0.58	1.23
Standard Deviation	0.15	0.05	0.04
Coefficient of Variation	3.23	7.82	3.28
Deviation of Mean from Certified Value	0	-0.08	-0.06

TABLE 10

ANALYSIS OF COPPER IN SECONDARY STANDARDS

	% Copper			
	Secondary Standards			
	3A	18	20	21
Previously Determined Value	88.3	59.4	59.1	Not Previously Determined
Trial 1	91.4	60.1	58.9	66.0
Trial 2	89.0	58.9	58.8	64.3
Trial 3	89.7	59.7	59.3	63.5
Mean	90.0	59.6	59.0	64.6
Standard Deviation	1.23	0.61	0.26	1.28
Coefficient of Variation	1.37	1.02	0.44	1.98
Deviation of Mean from Previously Determined Value	+1.7	+0.02	-0.1	-

TABLE 11

ANALYSIS OF ZINC IN SECONDARY STANDARDS

% Zinc

	Secondary Standards			
	3A	18	20	21
Previously Determined Value	Not Previously Determined	34.6	33.4	Not Previously Determined
Trial 1	0.064	34.9	34.5	23.3
Trial 2	0.073	36.6	35.8	23.2
Trial 3	0.068	35.8	34.8	23.2
Mean	0.068	35.8	35.0	23.2
Standard Deviation	0.004	0.85	0.68	0.058
Coefficient of Variation	5.88	2.37	1.94	0.25
Deviation of Mean from Previously Determined Value	-	+1.2	+1.6	-

TABLE 12

ANALYSIS OF TIN IN SECONDARY STANDARDS

% Tin

	Secondary Standards			
	3A	18	20	21
Previously Determined Value	Not Previously Determined	0.57	0.04	Not Previously Determined
Trial 1	0.09	0.52	0.05	0.34
Trial 2	0.11	0.49	0.06	0.34
Trial 3	0.08	0.50	0.03	0.30
Mean	0.09	0.50	0.05	0.33
Standard Deviation	0.015	0.015	0.015	0.023
Coefficient of Variation	16.7	3.0	30.0	6.97
Deviation of Mean from Previously Determined Value	-	-0.07	+0.01	-

TABLE 13

ANALYSIS OF IRON IN SECONDARY STANDARDS

% Iron

	Secondary Standards			
	3A	18	20	21
Previously Determined Value	1.34	1.77	1.70	Not Previously Determined
Trial 1	1.23	1.48	1.38	3.10
Trial 2	1.22	1.50	1.47	3.15
Trial 3	1.23	1.50	1.47	3.12
Mean	1.23	1.49	1.44	3.12
Standard Deviation	0.006	0.012	0.052	0.025
Coefficient of Variation	0.49	0.81	3.61	0.80
Deviation of Mean from Previously Determined Value	-0.21	-0.28	-0.26	-

TABLE 14

ANALYSIS OF NICKEL IN SECONDARY STANDARDS

% Nickel

	Secondary Standards		
	18	20	21
Previously Determined Value	0.06	0.91	Not Previously Determined
Trial 1	0.04	0.88	0.38
Trial 2	0.07	0.78	0.37
Trial 3	0.06	0.76	0.37
Mean	0.06	0.81	0.37
Standard Deviation	0.02	0.06	0.006
Coefficient of Variation	33.3	7.41	1.62
Deviation of Mean from Previously Determined Value	0	-0.10	-

TABLE 15

ANALYSIS OF ALUMINUM IN SECONDARY STANDARDS

% Aluminum

	Secondary Standards			
	3A	18	20	21
Previously Determined Value	9.5	2.2	3.0	Not Previously Determined
Trial 1	10.4	2.1	2.9	5.6
Trial 2	9.0	2.2	2.8	5.2
Trial 3	9.6	2.3	2.6	5.3
Mean	9.7	2.2	2.8	5.4
Standard Deviation	0.70	0.10	0.15	0.21
Coefficient of Variation	7.22	4.54	5.36	3.89
Deviation of Mean from Previously Determined Value	+0.2	0	-0.2	-

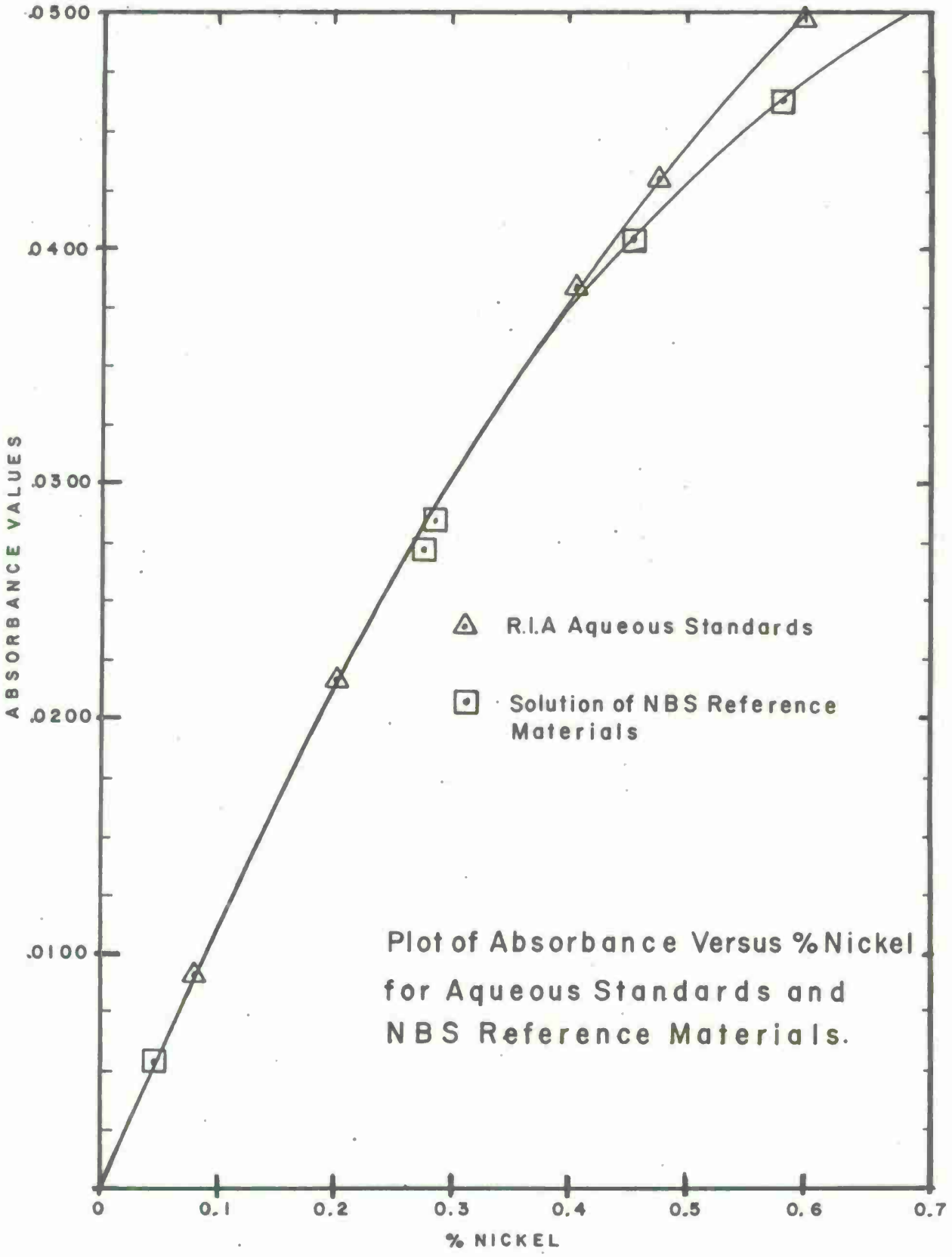
TABLE 16

ANALYSIS OF MANGANESE IN SECONDARY STANDARDS

% Manganese

Secondary Standards

Previously Determined Value	0.34	1.59	1.94	Not Previously Determined
Trial 1	0.20	1.50	1.85	4.20
Trial 2	0.20	1.60	1.80	4.25
Trial 3	0.20	1.60	1.82	4.15
Mean	0.20	1.57	1.82	4.20
Standard Deviation	0	0.058	0.025	0.05
Coefficient of Variation	0	3.69	1.37	1.19
Deviation of Mean from Previously Determined Value	-0.14	-0.02	-0.12	-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Plot of Absorbance Versus % Nickel for Aqueous Standards and NBS Reference Materials.
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