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SPECTROPHOTOMETRIC DETERMINATION OF MOLYBDENUM
IN URANIUM ALLOYS

TECHNICAL REPORT NO. WAL TR 425/1

BY

E. F. JACOBSON

APRIL 1959

O.O. PROJECT: TN2-8054, SPECIAL
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Uranium alloys,
chemical analysis

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TITLE

SPECTROPHOTOMETRIC DETERMINATION OF MOLYBDENUM IN URANIUM ALLOYS

ABSTRACT

The spectrophotometric thiocyanate determination of molybdenum without extraction was applied to uranium alloys. It was shown to be rapid, accurate, and free from interferences by alloying elements. A detailed procedure is given for the determination of molybdenum in uranium alloys.



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INTRODUCTION

Molybdenum in steels, iron, and other materials may be determined gravimetrically by precipitation with hydrogen sulfide¹ or by precipitation with alpha-benzoinoxime.² It is also determined volumetrically,³ spectrographically,⁴ and by X-ray fluorescence. Ordinarily it is determined spectrophotometrically, usually as the thiocyanate complex.⁵

Since molybdenum-uranium alloys have recently been developed, a rapid yet accurate method for the determination of molybdenum in these alloys is required.

There is little information in the available literature concerning the determination of molybdenum in uranium alloys. Several methods are mentioned by Rodden,⁶ but the details are sketchy. Milner and Bacon⁷ describe a spectrophotometric method for binary alloys of molybdenum and uranium using "high precision spectrophotometry;" this, however, was not of general application to rapid analyses of other types of uranium alloys. Milner, Barnett, and Bacon⁸ give details of a spectrophotometric-thiocyanate method for molybdenum in uranium-columbium-molybdenum alloys. This method was of particular interest since a spectrophotometric-thiocyanate method without extraction has been employed routinely at the Watertown Arsenal Laboratories for a considerable time for the analyses of molybdenum in steel⁹ and in titanium.¹⁰ Since the method of Milner, Barnett, and Bacon differs considerably from the Watertown Arsenal Laboratories method in regard to reagents and other details, it was not considered desirable to introduce this method to the laboratory and to test it for suitability to other types of uranium alloys. Consequently, the Watertown Arsenal Laboratories spectrophotometric-thiocyanate molybdenum determination was modified to adapt it to the analysis of uranium alloys and tested for precision and accuracy in the presence of a number of alloying elements.

EXPERIMENTAL DATA

Procedure

The procedure for the determination of molybdenum is essentially as follows: a sample is dissolved in hydrochloric and nitric acids, followed by fuming with perchloric acid. Ferric iron is added to act as an oxidation-reduction buffer, and the molybdenum thiocyanate color is developed with the addition of stannous chloride and sodium thiocyanate. Measurement of the color is described below.

Apparatus and Reagents

All measurements were made with a Beckman Model B Spectrophotometer using 1-cm rectangular cells. All reagents were of Reagent Grade.

Choice of Wavelength

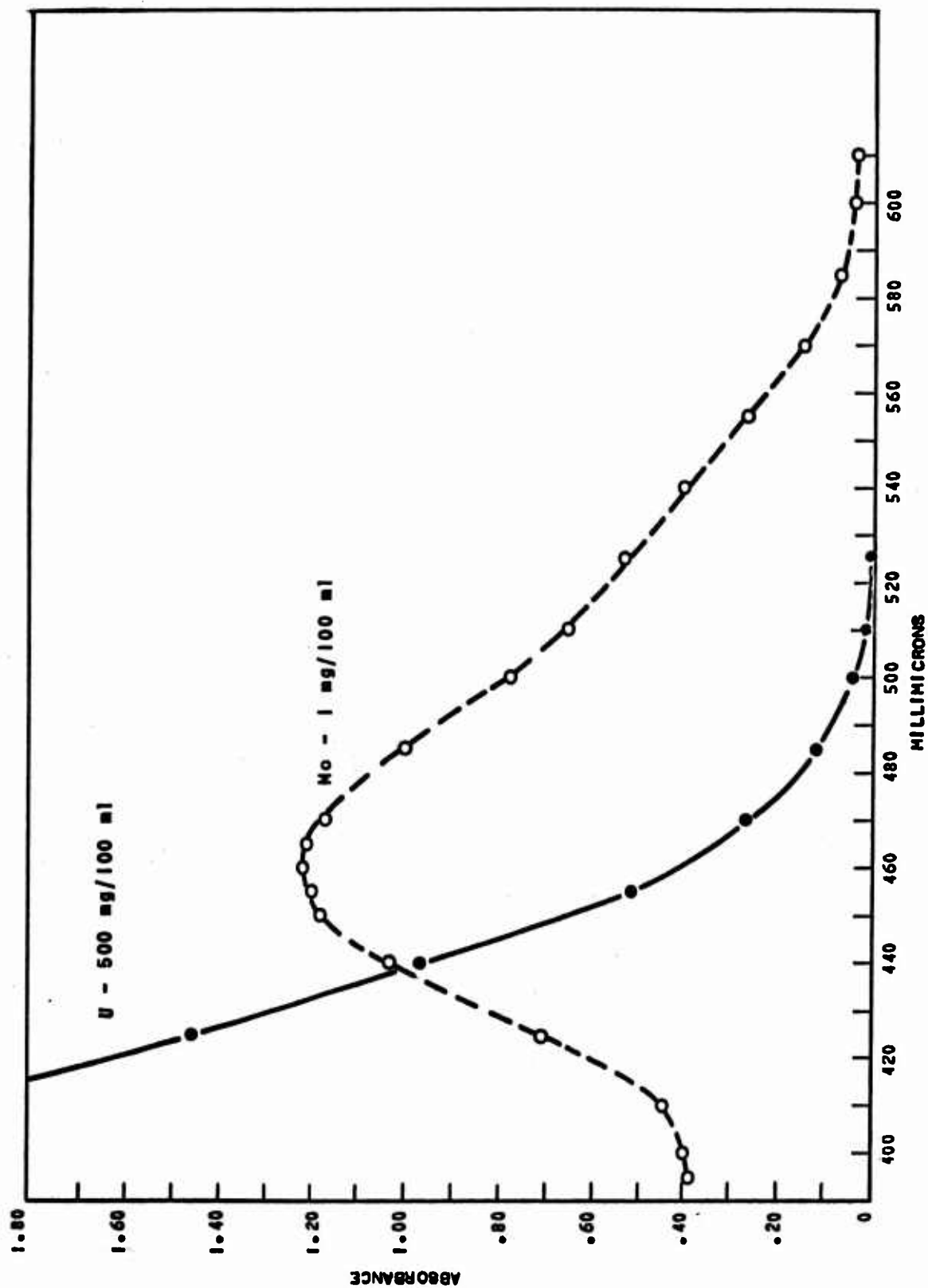
Molybdenum thiocyanate solutions show a maximum absorbance at about 460 m μ , and absorb strongly even at wavelengths well over 510 m μ . The absorbance curve of molybdenum thiocyanate and uranium thiocyanate are given in Figure 1. The absorbance of a uranium thiocyanate solution is appreciable at 460 m μ , the wavelength of maximum absorbance by molybdenum thiocyanate, but diminishes to a low value at wavelengths of 510 m μ or over. The uranium concentration of 5 mg per ml is at least ten times the concentration which is present in a determination of alloying quantities of molybdenum. The absorbance of uranium in an actual determination will be less than that shown in Figure 1 by a factor of at least 10.

Two choices of wavelength for measurement of the molybdenum thiocyanate complex in uranium solutions are possible. The wavelength of maximum absorbance, 460 m μ , will give maximum sensitivity. This is undesirable for two reasons: first, a sample blank must be run for each alloy to compensate for the absorbance of uranium at this wavelength, and, second, the high sensitivity at this point will necessitate the use of a very small sample in order to keep the absorbance down to a measurable level. The other alternative is to employ a wavelength at which the absorbance of the uranium thiocyanate is negligible and the absorbance of molybdenum-thiocyanate is still high enough for adequate sensitivity. The wavelength of 510 m μ was selected as meeting these requirements satisfactorily.

Conformance to Beer's Law

Having selected the wavelength of 510 m μ it was necessary to test experimentally the relationship of absorbance at this wavelength and the concentration of molybdenum thiocyanate. Ideally, if Beer's law is followed, the absorbance of a solution will be directly proportional to the concentration of the colored species being measured. There are many deviations from this law caused by many factors, both instrumental and chemical, so that its applicability can never be assumed.

Molybdenum in the form of a solution of ammonium molybdate was added to aliquots of a uranium solution, which contained 25 or 50 mg of uranium, as shown in Table I. These synthetic samples of uranium-molybdenum alloys were then treated to develop the molybdenum thiocyanate complex, and the absorbance of each was measured at 510 m μ . Aside from experimental variation, the ratio of milligrams of molybdenum to absorbance is found to be constant and, consequently, to follow Beer's law. Thus it is possible at all concentrations to relate the milligrams of molybdenum to absorbance by the constant factor of 1.52.



ABSORBANCE CURVES OF MOLYBDENUM AND URANIUM

TABLE I
CONFORMANCE TO BEER'S LAW

Uranium mg	Molybdenum mg	Absorbance	<u>mg Mo</u> Absorbance
50	.099	.065	1.52
50	.198	.130	1.52
50	.297	.195	1.52
50	.396	.260	1.52
50	.495	.325	1.52
50	.990	.650	1.52
25	1.485	.975	1.52
25	1.980	1.300	1.52
25	2.475	1.620	1.53

Interferences

A study was made of the possible interference of other alloying elements on the determination of molybdenum. The only information available in the literature was on the interference of columbium in the method of Milner, Barnett, and Bacon. In this method the columbium is complexed with oxalic acid to prevent its precipitation. Based on studies of interference in the analysis of steels and titanium alloys it was not expected that there would be interference from the common alloying elements, but the absence of interference could not be assumed without investigation.

A sample of a binary alloy of uranium and molybdenum containing 1.94% molybdenum was dissolved in acid, and aliquots containing 50 mg of the alloy were taken. To these aliquots were added 12 common alloying elements, as shown in Table II. This experiment was performed in duplicate. Although the only ternary alloy with molybdenum so far submitted for test has been a 0.5Mo-0.5Cb uranium alloy it was decided to study the interference of certain of these 12 elements at the 5 percent level and others at the 1 percent level. Aluminum, chromium, iron, manganese, nickel, tin, titanium, vanadium, and zirconium were tested at the 5 percent level, and no interference was found. At the 1 percent level, copper, columbium, and tungsten did not interfere. These percentages are believed to be the maximum normal limits for these elements to be encountered in uranium-molybdenum alloys. Copper thiocyanate does precipitate but is readily filtered out. Columbium stays in solution under normal conditions of the analysis, and tungsten is held in solution by the addition of phosphoric acid.

Accuracy and Precision

No standards are available by which the accuracy of the method can be measured. An estimate of the accuracy was gained by determining the molybdenum content of five uranium alloys by both the spectrophotometric procedure described in this report and by precipitation of the molybdenum with alpha-benzoinoxime. The alpha-benzoinoxime procedure is standard for the analysis of steels and other alloys and can be assumed to be as accurate as any of the available methods of determining molybdenum. It is not used for analysis generally, since as a gravimetric method it is somewhat time consuming compared to spectrophotometric procedures. Table III lists the results obtained, in duplicate experiments, on the five samples by both methods. The agreement between the two methods is very satisfactory and is considered sufficient for most purposes.

TABLE II
EFFECT OF OTHER ELEMENTS ON THE DETERMINATION
OF MOLYBDENUM IN URANIUM

Element Added	Form	mg Added as Metal	Equivalent Percent Added	Percent Mo Found
None	-	-	-	1.94 1.94
Aluminum	Metal	25 25	5.0 5.0	1.94 1.94
Chromium	K ₂ Cr ₂ O ₇	26 26	5.2 5.2	1.95 1.95
Columbium	Metal	5 5	1.0 1.0	1.94 1.94
Copper	Metal	5 5	1.0 1.0	1.95* 1.94*
Iron	Metal	25 25	5.0 5.0	1.95 1.94
Manganese	Metal	25 25	5.0 5.0	1.94 1.95
Nickel	NiSO ₄	27 27	5.4 5.4	1.94 1.94
Tin	Metal	25 25	5.0 5.0	1.94 1.94
Titanium	Metal	25 25	5.0 5.0	1.94 1.94
Vanadium	NH ₄ VO ₃	25 25	5.0 5.0	1.94 1.94
Tungsten	Na ₂ WO ₄	5 5	1.0 1.0	1.94 1.95
Zirconium	Metal	23 25	4.6 5.0	1.95 1.95

**Colored test solution was filtered to remove NaSCN precipitate.*

TABLE III
COMPARISON OF RESULTS FOR PERCENT MOLYBDENUM

Sample No.	% Mo Alpha-benzoinoxime	% Mo Thiocyanate Complex
L-5B	0.50, 0.51	0.50, 0.50
C5TLCOS	2.00, 1.93	1.91, 1.92
C-6B	2.44, 2.44	2.41, 2.43
C-9	8.60, 8.59	8.54, 8.54
L-10	9.48, 9.47	9.45, 9.42

Discussion and Conclusions

The spectrophotometric method presented here is rapid, accurate, and simple to use. Six samples of uranium can be analyzed for molybdenum in 1-1/2 hours compared to 8 hours by the alpha-benzoinoxime method. The spectrophotometric method is convenient to use since the reagents and equipment required are the same as those used for the determination of molybdenum in steels and titanium. Since the procedure is straightforward it can be handled by technicians with limited chemical background.

Details of the spectrophotometric method, as developed in this study, are given in the Appendix.

APPENDIX

DETAILED PROCEDURE FOR THE SPECTROPHOTOMETRIC DETERMINATION OF MOLYBDENUM IN URANIUM

Scope

This method is applicable to uranium alloys containing molybdenum. It has been used for determining molybdenum in the range of 0.50% to 11.6%, but with proper aliquots the percentage may be lowered or extended. None of the alloying elements usually found in uranium interferes.

Apparatus

1. Beckman Model B Spectrophotometer.
2. 1-cm rectangular cells.

Reagents

1. HClO_4 (60%).
2. Iron Perchlorate Solution (.0025 g Fe per ml). - Dissolve 2.5 g of pure iron wire in 50 ml of HClO_4 (60%) and evaporate to fumes. While still hot add 100 ml of HClO_4 , cool, and dilute to 1 liter with HClO_4 .
3. Sodium Thiocyanate Solution (10%). - Dissolve 100 g of NaSCN in water and dilute to 1 liter.
4. Stannous Chloride Solution (25%). - Dissolve 500 g of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ in 400 ml of HCl . Digest on a hot plate until a clear solution is obtained. Cool and dilute with water to 2 liters. Add 5 g of metallic tin.
5. Reagent Mixture - Mix 1 part of the sodium thiocyanate solution with 2 parts of the stannous chloride solution, as needed. Filter through a Whatman No. 12 fluted paper or equivalent.
6. Standard Molybdenum Solution - Dissolve 0.186 g of ammonium molybdate, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$, in 800 ml of water. Transfer to a 1-liter volumetric flask, dilute to the mark with water, and mix. This solution should contain approximately 0.1 mg of Mo per ml. Standardize the solution gravimetrically as described in the 1956 *Book of ASTM Methods for Chemical Analysis of Metals*, page 32.

Procedure

1. Transfer 0.500 g of sample to a 150-ml beaker. Cover with a watch glass. Add 10 ml of HCl (1:1) and heat gently.
2. Oxidize by the dropwise addition of HNO_3 (Note 1) and boil for a few minutes until a clear solution is obtained.

3. Cool, transfer to a volumetric flask (Note 2). Transfer a 10-ml aliquot to a 100-ml Pyrex volumetric flask.

4. Add 20 ml of the iron perchlorate solution and 5 ml of HClO_4 (Note 3).

5. Heat the sample until the perchloric acid is fuming strongly above the neck of the flask.

6. Cool, add approximately 25 ml of water, mix, and cool.

7. Add 30 ml of the reagent mixture and immediately mix by swirling. Dilute to the mark with water, mix, and allow to stand for 10 minutes (Note 4).

8. Prepare the reference solution as follows: place 25 ml of water and 25 ml of HClO_4 in a 100-ml volumetric flask, mix, and cool; add 30 ml of the reagent mixture, dilute to the mark with water, and mix well.

Photometry

Set the instrument at 510 $\text{m}\mu$ and obtain correction for cells by filling the 1-cm cells with water. Cell No. 1 is set at .000 absorbance, and the remaining cells are read against this cell. Any deviation from the .000 absorbance is recorded for each cell and later applied as a correction to the absorbance reading of the unknown. Transfer a portion of the reference solution to Cell No. 1, adjust the instrument to read .000 absorbance, and read the unknown solutions against the reference solution.

Determination of Factor

To determine the factor (mg of Mo/absorbance), dissolve 0.500 g of unalloyed uranium according to Steps 1 and 2 of "Procedure." Cool, transfer to a 100-ml volumetric flask, dilute to the mark, and mix. Transfer 10-ml aliquots to seven 100-ml Pyrex volumetric flasks. Add 0, 1, 2, 3, 4, 5, and 10 ml of the standard molybdenum solution to the flasks containing the equivalent of 50-mg portions of the uranium and continue with "Procedure," starting with Step 4, and with "Photometry." Calculate the factor by averaging the values of the ratio, mg of Mo/absorbance. The factor, which should be determined exactly for each spectrophotometer used, is approximately 1.5.

Calculation

$$\text{Molybdenum, percent} = \frac{(A - B) \times C}{D} \times 100$$

where

A = absorbance reading
B = correction for cell

C = factor
D = milligrams of sample used

Notes

1. The sample does not have to be completely dissolved before the addition of the HNO_3 . The HNO_3 speeds up dissolution of the sample.
2. If the sample contains 3% molybdenum or under, transfer the sample to a 100-ml volumetric flask; if 3 to 6% molybdenum, to a 200-ml volumetric flask; and if over 6% molybdenum, transfer to a 500-ml volumetric flask.
3. If an appreciable amount of tungsten is present, add 5 ml of H_3PO_4 before fuming the sample with HClO_4 .
4. If copper is present, the solution is filtered through a dry Whatman No. 42 filter paper to remove any precipitate of copper thiocyanate.

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SPECTROPHOTOMETRIC DETERMINATION OF MOLYBDENUM IN
URANIUM ALLOYS - E. F. Jacobson

Report No. WAL TR 425/1, Apr 1959, 9 pp - tables -
illus - appendix, DA Proj 512-15-018, 00 Proj TN2-8054,
Unclassified Report

The spectrophotometric thiocyanate determination of
molybdenum without extraction was applied to uranium
alloys. It was shown to be rapid, accurate, and free
from interferences by alloying elements. A detailed
procedure is given for the determination of molyb-
denum in uranium alloys.

I. Jacobson
E. F.

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