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DETERMINATION OF LESS THAN 10 PARTS PER MILLION CARBON IN TUNGSTEN

by William A. Gordon, Judson W. Graab,
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SUMMARY

W. Cb, Ta, Ti, Cr

In the combustion-conductometric method the smallest detectible quantity of carbon is limited by errors introduced by a spurious carbon blank. Also, the accuracy of the method for refractory metals is unknown since quantitative calibration is based on recoveries of carbon from steel samples or on organic compounds. Studies of the control of these possible sources of error resulted in establishing conditions whereby carbon concentrations of 4 ppm may be determined with a precision of ±12 percent and an estimated accuracy of ±15 percent relative. These conditions appear to be directly applicable to other metals, including niobium, tantalum, titanium, zirconium, chromium, and copper.

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INTRODUCTION

Tungsten metal that has been processed by floating-zone refining commonly contains less than 10 parts per million (ppm) of carbon by weight. The quantitative determination of carbon in this material is of interest because it affords a method for measuring the effectiveness of the purification process and also because carbon apparently has an appreciable effect on the mechanical properties of the metal (ref. 1). Quantitative determination of the carbon in this low range has proved, however, to be a difficult task with existing methods.

The combustion-conductometric technique has found wide application as a method of analysis for carbon in a variety of metals (refs. 2 to 8). This method has also been applied to refractory metals in the original or modified form. The primary steps in the method are (1) the combustion or oxidation of the sample by inductive heating in oxygen, which converts carbon in the sample to carbon dioxide; and (2) the detection of the evolved carbon dioxide by measuring the change in electrical conductivity when carbon dioxide is reacted with a dilute solution of barium hydroxide. The change in conductivity is

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*This report is an expanded version of the article entitled "Combustion-Conductometric Determination of Less Than 10 Parts per Million Carbon in Tungsten" by the same authors published in Analytical Chemistry, vol. 36, no. 7, June 1964, pp. 1936 to 1938.

related to the carbon content in the samples by calibration with standard samples of the material being analyzed. In the case of refractory metals, standard samples are not available. Calibrations must, therefore, be based on the recovery of carbon from National Bureau of Standards samples for carbon in steel or from synthesized standards such as organic compounds in which the carbon content is precisely known. The validity of the carbon determination in tungsten is therefore uncertain, because it is based on the assumption that carbon is quantitatively evolved from the sample under experimental conditions. A second limitation of the method is the inability of the detector to distinguish between the signal due to the carbon dioxide from the sample and that arising from sources other than the sample (i.e., the "blank"). The lower limit of detection for carbon in this procedure is determined by the variation of this blank, which makes the precise determination of carbon below 10 ppm particularly difficult.

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[The purposes of this investigation were to achieve improved precision at the lower carbon concentrations and, in addition, to prove the accuracy of the method when applied to refractory metals, particularly tungsten.] The oxidation step was investigated, and a new combustion technique is recommended. The precision and accuracy are reported, and the resultant analytical procedure is outlined in detail.

EXPERIMENTAL PROCEDURE

Effect of Additive Materials on Blank

A major part of the blank originates from materials such as iron, tin, lead, and copper (refs. 2, 5, 6, and 7), which are introduced into the crucible with the sample to promote coupling with the high-frequency field and to aid in the formation of a less viscous mixture. In many cases the use of additive materials is highly desirable, if not essential, in achieving quantitative liberation of carbon from the sample metal (ref. 9). At concentrations as low as 10 ppm, however, the errors introduced by variable amounts of carbon contributed by the additive limit the precision of the method. Experiments were conducted to obtain definitive data on the limitation imposed by a variety of typical additives alone. The limitations of the instrument itself in this respect would be indicative of the ultimate detection limit for carbon by this procedure.

Table I is a compilation of results from experiments for various additive materials. Each number in the line labeled Total blank (μg of carbon) represents an average of 10 determinations made over a period of 2 days for materials of different lots. Determination on iron chips were made after screening to reduce variation in replicate runs. In these experiments only that material retained on 10 mesh was used. Results are also shown for commercially available tin granules and tin capsules and for low-carbon, electrolytic iron. Finally, the apparatus blank was determined by heating a quartz-enclosed graphite susceptor (Laboratory Equipment Corp. No. 550-182) to approximately 1200° C to simulate heating of the sample. Data in the lines labeled Total blank, μg of carbon, and Standard deviation, μg of carbon, were obtained for

TABLE I. - COMPARISON OF BLANK LEVELS WITH AND WITHOUT ADDITIVE

	Additive material							Series of apparatus blanks (direct-combustion)
	Iron chips screened[b], 1 g				Tin granules[b], 1 g	Tin capsules[b]	Electro-lytic iron, 1 g	
	Lot designation							
	1	2	3	4	5	6	7	
Total blank, μg of carbon	26.0	39.4	39.5	40.1	12.3, 10.8	12.0, 15.7	19.2, 11.3	4.9, 3.7, 4.3, 2.8
Standard deviation[a], μg of carbon	4.0	1.4	2.5	4.0	3.7, 2.4	3.0, 6.0	1.1, 2.7	1.3, 2.0, 1.8, 1.5
Limits of detection per gram sample, ppm (range of 3s[a])	4.2 to 12.0				7.2 to 8.1	9.0 to 18.0	3.3 to 8.1	3.8 to 6 (1.7 to 3.0[c])

[a] $s = \sqrt{\frac{\sum d^2}{n - 1}}$, where n is number of tests and d is the deviation of a test from the average.

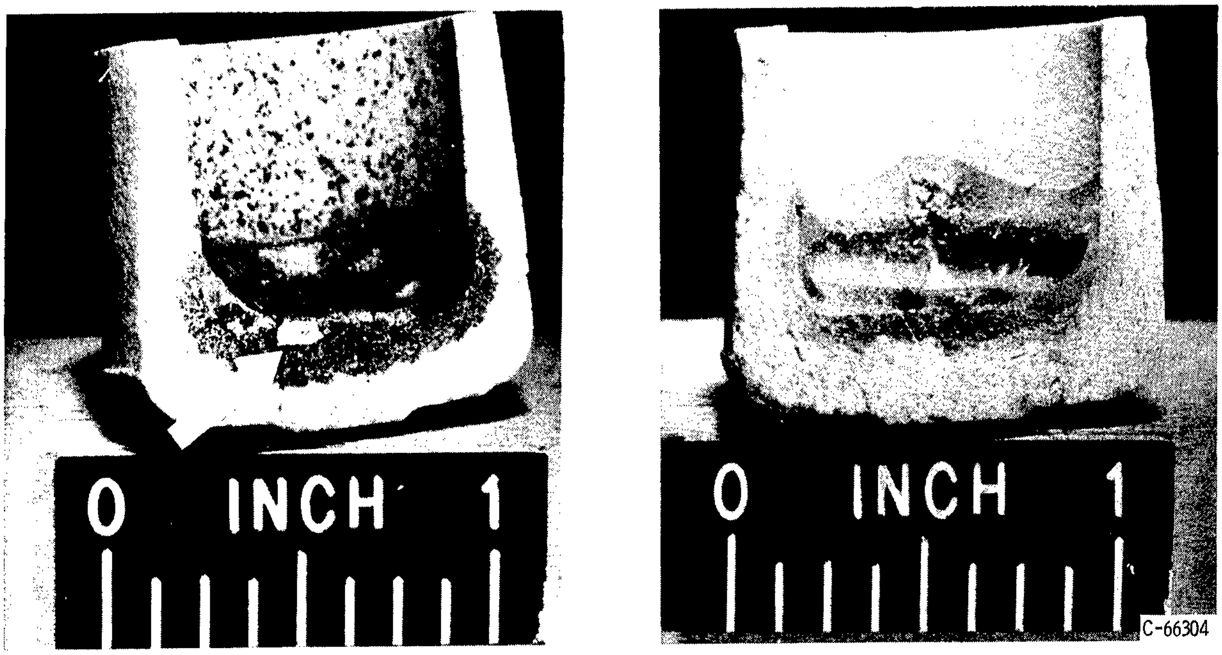
[b] Laboratory Equipment Corporation.

[c] Based on 2-g sample.

an operation cycle consisting of 2 minutes of heating and 6 minutes of flushing. The data in these lines show the magnitude of the average blank and the statistical variation associated with these determinations. These values represent an undesirable signal at the detector, which tends to mask the detection of carbon recovered from a sample such as high-purity tungsten. The values in the last line are the limits of detection in ppm of carbon for the additives listed. These values were determined by arbitrarily establishing the limit at three times the standard deviation to represent the 95-percent confidence level. The last figure in this line also shows the results for a 2-gram sample, since this weight is the minimum recommended for the direct combustion of tungsten.

A conclusion drawn from the data obtained is that a significant source of error in the determination at low carbon concentrations is the carbon content of the additive. Observations also indicated that the presence of iron in the crucible was unnecessary in achieving quantitative evolution of carbon from tungsten, since little or no intermetallic reaction occurs. P4

When iron chips were fused in an induction furnace in the presence of tungsten metal, unreacted tungsten chips were formed into an agglomerate, apparently by the stirring effect of the high-frequency field. This occurrance has been observed by other investigators, such as Huber and Chase (ref. 7), who recommend the crushing and optical examination of the fused sample for unreacted metal. In the present work, rarely would a crushing procedure alone disclose the presence of unreacted tungsten. Careful sectioning of the crucibles, however, showed that in about 80 percent of the fusions the tungsten metal could be found intact (fig. 1 shows one such example). It appeared that the elimination of all additives to the crucible by heating the sample directly would not only simplify the operation, but would be a major step toward achieving a reproducible blank and, therefore, a lower detection limit for carbon. The latter was achieved by optimizing conditions for completely oxidizing tungsten metal by direct-combustion.



(a) Fused tungsten plus iron additive.

(b) Fused tungsten, direct-combustion.

Figure 1. - Cross sections of refractory crucibles used for combustion of tungsten samples (with and without an additive). Note agglomerate of unreacted tungsten in (a).

Direct-Combustion of Tungsten by Induction Heating

The thermodynamics for oxide formation of tungsten and other refractory metals appear to make these metals especially adaptable to combustion methods. Although tungsten metal was readily converted to tungstic oxide in a resistance furnace, the time required for complete oxidation did not permit the attainment of as low a blank as was possible by rapid oxidation in an induction furnace.

Attempts to reduce the blank level by careful selection of fluxes with especially low carbon contents have been reported. The method of reference 6 reduces contamination from this source by prefusing an electrolytic iron bath in a helium atmosphere. Reference 6 suggests that the residual carbon under these conditions is liberated by combining with the oxygen in the iron to form carbon dioxide. This method was successful in reducing the residual carbon from the iron to a negligible amount. The procedure requires slight modifications of the standard instruments and introduces an operation that would be unnecessary if conditions for directly heating the specimen could be devised (e.g., as in an induction furnace).

Providing effective coupling between the high-frequency field and a tungsten sample without resorting to the use of an additive requires considering the relation between the physical form of the sample and some principles of induction heating. Determinations were most often required for metal rods of 1/8- to 1/4-inch diameter and for metal powders with particle sizes of the order of 200 mesh. The small diameter and relatively small surface area of the rods are not conducive to efficient induction heating in the megacycle frequency range, which is based on a skin effect. Fine powders, while advantageous with respect to oxidation, provide a discontinuous path for high-frequency energy and thus are also ineffective in transferring the energy. A study of the effect of particle size on heating under these conditions showed that metal granules sized between 16 and 80 mesh can be coupled effectively. Once the oxidation of tungsten commences, the metal is converted into a fused oxide mass by an exothermic reaction at temperatures above 1450° C.

While this procedure is satisfactory for coarse powders and solid metals that can be reduced in size, it is not amenable to fine powders (i.e., finer than 80 mesh). These fine powders will couple directly, however, if the particles are compacted to provide a more continuous path for the high-frequency field. Extremely fine powders can be sufficiently compacted with a blunt ceramic tool and hand pressure. For the majority of as-received powders, unfortunately, this procedure is not sufficiently reproducible. Several alternate methods based on simple forms of a platinum susceptor, however, have been successfully used for fine powders. A procedure suggested to the authors by G. W. Edwards of the Linde Company entails placing a piece of platinum foil in the bottom of the crucible and covering it with metal powder. Induction heating of the platinum provides sufficient heat to initiate the oxidation, which is then self-sustaining. Platinum squares as small as 1/8 by 1/8 inch and 0.003 inch thick have been used successfully. The platinum may be readily conditioned by heating in the induction furnace. A platinum disk, 1 inch in diameter and 1/32 inch thick, will similarly act as a susceptor when placed under and in good thermal contact with the crucible, which contains the powder. These disks are easily fabricated and can be used indefinitely.

When the combustion procedures described herein for metal and powders are used, it is possible to reduce the blank to a level more favorable for the determination of less than 10 ppm of carbon in tungsten.

Figure 2 demonstrates that the total instrumental and crucible blank under these conditions is essentially the same as that obtained when the susceptor described previously is used. Sample weights from 2 to 8 grams were oxidized directly in refractory crucibles, and the averages of

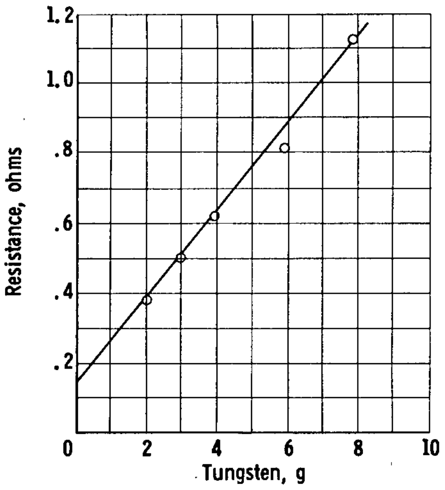


Figure 2. - Determination of blank by sample weight variation.

multiple determinations were plotted against sample weight. The extrapolation of the plot passes through about 0.15 ohm, which when converted to μ of carbon with a calibration curve is in agreement with that obtained for the susceptor alone.

Quantitative Recovery of Carbon from High-Purity Tungsten

[The accurate determination of carbon by the combustion-conductometric procedure requires consideration of (1) the calibration of the response of the conductivity cell to absolute amounts of carbon dioxide, and (2) the determination of the fraction of total carbon evolved from the sample under experimental conditions.] The calibration of the conductivity cell can be accomplished in several ways. For example, standards for carbon in steel certified by the National Bureau of Standards can be readily used for this purpose (refs. 2, 3, 6, and 8). Primary standard organic compounds, such as potassium acid phthalate, can also be used for calibration (ref. 10). As a matter of convenience, the latter approach was used in this work. Although potassium acid phthalate is available commercially in tin capsules, the blank correction for carbon in tin may prove to be undesirable at the lower concentrations of carbon (table I, p. 3). Aqueous solutions of potassium acid phthalate containing 100 micrograms of carbon per milliliter were added directly to porous ceramic disks (Laboratory Equipment No. 528-42) and oven dried at 110° C for approximately 1 hour. The dried disks were placed on top of a quartz-enclosed graphite susceptor and inductively heated to an estimated 1000° C for 2 minutes. This heating was followed by a 6-minute flushing cycle, since it had been shown that this period was required for the signal at the detector to reach a maximum plateau. The calibration and precision obtained in this procedure are shown in figure 3. The extrapolation to about 0.15 ohm again corresponds to the blanks obtained for the susceptor alone and is equivalent to approximately 2 ppm of carbon for a sample weighing 2 grams.

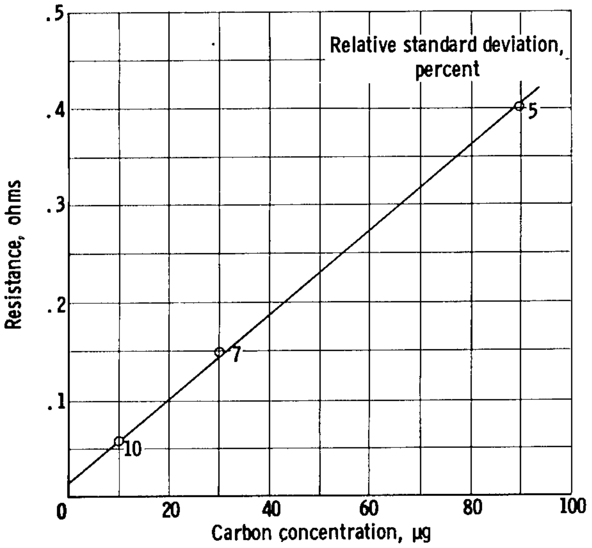


Figure 3. - Potassium acid phthlate calibration showing micrograms of carbon and relative standard deviation.

These experiments based on the oxidation of potassium acid phthalate provide the basis for the calibration of the conductivity cell but do not disclose the degree of carbon recovery from a tungsten sample. Knowing the degree is necessary to place the determination on a quantitative basis. The validity of reporting carbon in tungsten based on recoveries from iron, steel, or organic compounds appeared to warrant further testing, since these procedures are based on the assumption that the carbon contents of the metal sample and the calibrating material are totally evolved. [Recovery experiments were designed to assess the effectiveness of the evolution of trace amounts of carbon from tungsten metal, by means

→ P7

of the direct-combustion procedure, by doping samples with near-stoichiometric tungsten carbides (WC and W_2C). The carbides of tungsten were chosen because it appeared they would most nearly simulate the chemical nature of the carbon in tungsten metal. Analytical data supplied by the vendor and that obtained on the carbides at Lewis are shown in the following table. The data show that for gross amounts of tungsten carbides the deviation from the stoichiometric value was not significant for the purposes of the experiment. The stoichiometric value was therefore used in calculating quantities of carbon, since this places the quantification on a basis that does not depend on the combustion-gravimetric procedure for determining the carbon content of tungsten carbides. Weighing microgram amounts was facilitated by blending the powdered carbides with pure tungsten powder containing less than 10 ppm of carbon to form a mixture containing approximately 1 percent carbon by weight. The additional tungsten added to the crucible with the carbides was negligible and did not require a correction.

→ P8

Material[a]	Total carbon, percent		
	Stoichiometric	Vendor	NASA
W_2C	3.16	3.18	3.19
WC	6.14	6.1	5.98

[a]Purity, 99.9 percent.

TABLE II. - RECOVERY OF CARBON BASED ON POTASSIUM ACID PHTHALATE ($KHC_8H_4O_4$) CALIBRATION

[Percent deviation of 0.1 ohm in blank correction: 7 at 30 μg of carbon; 2 at 90 μg of carbon.]

Sample	Carbon added, μg	Carbon recovered, percent deviation from $KHC_8H_4O_4$
WC + 2g W	25	+28
	25	0
	28	+7
	26	+8
	33	-12
	31	+38
	28	+12
	30	+3.3
	30	-13.2
	Av.	+8.0
	46	+11
	47	+11
	57	-11
	67	+10
	74	+5
	74	+15
	80	-9
	Av.	+4.6
	96	+4.2
	90	+7.2
	95	+7.4
	96	+2.1
	93	+4.4
	90	+2.7
	Av.	+4.3
W_2C + 2g W	24	+24
	26	+10
	28	0
	31	-3
	35	0
	42	+12
	46	+2
	53	+13
	56	+11
	64	+5
	Av.	+7.4
Fe (NBS 55e)	30	+3.3
	30	+3.3
	30	+15.0
	30	+6.7
	24	0
	50	-6.0
	90	-6.1
	90	+0.78
	Av.	+2.1

The experimental procedure consisted of adding a few milligrams of the WC-tungsten or W_2C-tungsten mixtures directly to a refractory crucible. The additive was then covered by 2 grams of the tungsten base-material, which was prepared from electron-beam-melted tungsten that produced a signal of 0.4 ohm for a 2-gram sample. Table II is a summary of recovery experiments for WC and W_2C. Data are shown for carbon recoveries from carbides in the presence of tungsten metal and oxidized by direct-combustion in a refractory crucible. Comparative data are also shown for small amounts of a standard sample of iron, designated by the National Bureau of Standards as 55e. The amount of carbon recovered in each of these experiments was compared with the data on potassium acid phthalate, and the percentage difference is shown. The recoveries from the carbides were consistently greater than those obtained for the organic compound or the standard iron sample. The higher percentage deviation at the lower amounts of carbon suggested a constant systematic error, such as might be caused by errors in the blank corrections of the various experiments. The average deviations range from approximately +4 to + 8 percent for additions of carbides representing from 24 to 96 micrograms of carbon. The relative deviations would be expected to be somewhat greater for the smaller amounts of carbon normally obtained from samples of pure tungsten. The accuracy of the procedure, therefore, for pure tungsten was estimated at ±15 percent relative at a concentrational level of 5 ppm. The agreement of the recoveries (table II) was satisfactory and showed that in the direct oxidation of tungsten metal and in the formation of fused oxide the liberation of carbon from the sample was virtually complete.

RECOMMENDED PROCEDURE

The pertinent procedural details are summarized in table III. Samples obtained as solid metal are crushed in a hardened steel mortar until the bulk of the material passes a 16-mesh screen and is retained on an 80-mesh screen. The small amount of material passing the fine screen is combined with the

TABLE III. - CONDITIONS FOR COMBUSTION-CONDUCTOMETRIC

DETERMINATIONS OF CARBON IN TUNGSTEN

Instrumentation	Laboratory Equipment Corporation Model 515 (Furnace no. 521)
Crucible	Laboratory Equipment Corporation Aluminum oxide (no. 528-25)
Oxygen flow rate	250 to 300 ml/min
Electrolyte	1 g/liter $Ba(OH)_2 \cdot 8H_2O$
Sample weight	~4 g (2 to 8 g)
Sample form	Powders - less than 80 mesh
	Granules - 16 to 80 mesh (and fines)
Susceptor	Platinum foil or disks for powders
	None for granules
Calibration	W_2C+W mixture
	WC+W mixture
	Potassium acid phthalate
Combustion and collection time	8 min total

sample granules, because carbon segregation may occur. A considerable amount of iron may be introduced into the sample during this operation and will result in high values for carbon it not completely removed. Removal is accomplished by digestion in 1:1 hydrochloric acid followed by thorough rinsing with water and drying with ether. Approximately 4 grams (2 to 8 g depending on carbon content) of the prepared sample is added to a crucible, which must be previously conditioned by heating in flowing oxygen at 900° C for 15 minutes. After the sample is placed in the induction furnace, pure oxygen is flushed through the furnace and conductivity cell for 2 minutes to divest the system of carbon dioxide, which may be picked up during the loading operation. The furnace is then energized for an 8-minute combustion cycle, although the formation of tungstic oxide is generally complete in 2 minutes (as indicated by the plate current meter). The carbon dioxide formed during this combustion cycle is collected at the conductivity cell. Resistivity readings in equivalent ohms are taken at a selected period in the temperature cycle of the thermostatically controlled bath near the end of the collection period. (The instrument used in this work gave the most reproducible readings 60 seconds after completion of a heat-on cycle.) The instrument readings are converted to micrograms of carbon by using a potassium acid phthalate calibration curve. Since blanks for the calibration and tungsten procedures are the same, it is convenient to plot all values as uncorrected.

DISCUSSION OF RESULTS

The results of precision studies on the tungsten-base material for two commonly used techniques (involving commercial or electrolytic iron additives) in addition to the direct-combustion technique are shown in table IV. The carbon content of this material was approximately 3 ppm by means of the recommended procedure. The iron additives used in these experiments were selected from the lots that gave the lowest standard deviation from the data in table I (p. 3), and the comparisons, therefore, are more typical of intralaboratory precision. Thus, the method using electrolytic iron gave slightly better precision than did the direct-combustion method. A conclusion drawn from the data was that the method employing selected electrolytic iron was comparable to the direct-combustion method with both falling in the range of values reported in table I (p. 3) for the experiments on the additive alone. It is probable that the results for electrolytic iron would appear less favorable on an interlaboratory basis, which may be inferred from the spread reported in table I for the

TABLE IV. - COMPARISON OF EFFECTS OF ADDITIVES ON DETERMINATION OF CARBON IN TUNGSTEN

Additive	Standard deviation, μg of carbon	Relative standard deviation, percent	Carbon concentration, ppm
Commercially prepared iron chip, 1 g Fe + 1 g W	2.3	55.0	4.2
Electrolytic iron, 1 g Fe + 1 g W	1.3	34.0	3.8
Direct-combustion (no additive), 4 g W	1.7	12.0	3.5

additive alone. Comparison of the data in table IV shows that the method is blank limited and the precision may be further improved by increasing sample weights, for example, up to 8 grams (fig. 2, p. 5) for the direct-combustion procedure.

The advantages of performing the determination by the direct-combustion method are further demonstrated in figure 4. Carbon determinations were made on single-crystal tungsten rod by using both the direct-combustion and the iron-additive techniques. The data show that the direct-combustion procedure gives a more satisfactory representation of the carbon gradient along the rod. The direct-combustion method permits determination of carbon in tungsten at concentrations of approximately 4 ppm with a precision of ±12 percent and an estimated accuracy within ±15 percent relative to the true carbon concentration based on recoveries of tungsten carbides from tungsten metal.

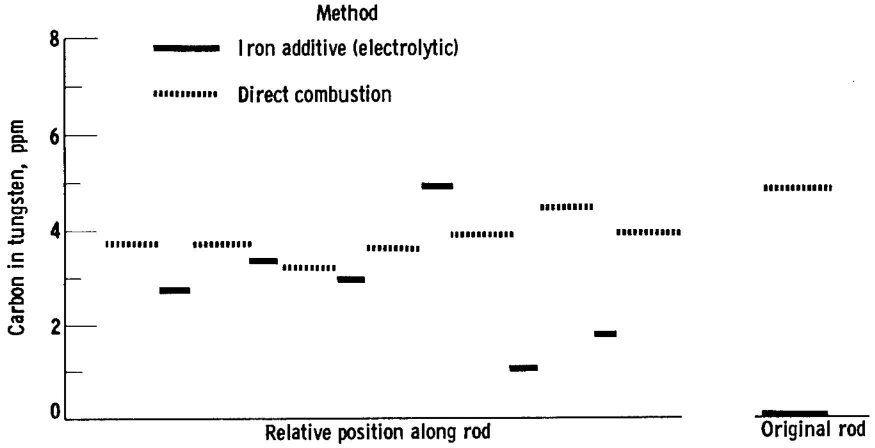


Figure 4. - Comparison of direct-combustion and iron-additive (electrolytic) methods of determining carbon gradient in single-crystal zone-refined tungsten rod. Rod diameter, 3/16 inch.

CONCLUDING REMARKS

The recommended procedure will provide quantitative results for the determination of carbon in tungsten below 10 ppm within the limits stated. Instrument reliability, however, may vary between laboratories. For this reason it is important to establish the limitations of a particular instrument. Guidelines used in this investigation consist of two basic instrumental checks. The variation in conductivity of a barium hydroxide solution subjected to a continuous flow of oxygen for a period of 1/2 hour should not exceed 0.1 ohm. Occasionally a continuous drift in the conductivity readings under these conditions has been encountered. A common cause for such a drift is the presence of liquid in the glass stem of either the reference or the sensing electrode. This condition may arise because of a faulty seal between the glass and the platinum at the tip of the glass envelope. It may be corrected by removing the liquid under vacuum and subsequently sealing the glass-platinum juncture with a bead of epoxide adhesive.

→ C11

The procedure used in this investigation for conditioning the furnace prior to operation consists of heating until readings for an 8-minute period do not exceed 0.3 ohm for multiple determinations. The blank may be determined with a quartz-enclosed graphite susceptor or the platinum devices described previously.

Since this method is successful in determining the carbon content in tungsten, the extension of the direct-combustion method to metals such as molybdenum, tantalum, niobium, zirconium, titanium, chromium, and copper is also of interest. These metals will couple directly if the particle size is similar to that described for tungsten. For these more ductile metals, it is often necessary to provide the proper size by cutting or shearing. In sheet form up to 1/16 inch in thickness, these metals couple and oxidize especially rapidly when a section is placed in the bottom of the crucible with the sheet oriented perpendicular to the axis of the induction coil. Complete oxidation is observed for molybdenum only if the sublimation of molybdic oxide is allowed to go to completion. It appears, therefore, that a special trapping device is desirable to prevent contamination of the conductometric system by the volatile oxide. Tantalum, niobium, zirconium, titanium, chromium, and copper undergo fusion of the oxides similar to tungsten. Preliminary recovery experiments using tantalum carbide and niobium carbide in the metals indicate quantitative recovery of carbon under the conditions described for tungsten.

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National Aeronautics and Space Administration
Cleveland, Ohio, May 7, 1964

REFERENCES

1. Stephens, Joseph R.: Effects of Intersitial Impurities on the Ductility of Tungsten. Paper presented at AIME meeting, New York (N. Y.), Oct. 28 - Nov. 2, 1962.

2. Anon: Methods for Chemical Analysis of Zirconium and Zirconium-Base Alloys. Designation 63T, ASTM, 1962.

3. Bennet, E. L., Harley, J. H., and Fowler, R. M.: Conductometric Method for Determination of Carbon in Steel. Analytical Chem., vol. 22, no. 3, Mar. 1950, pp. 445-448.

4. Elwell, W. T., and Wood, D. F.: The Analysis of Titanium, Zirconium and Their Alloys. John Wiley & Sons, Inc., 1961.

5. Elwell, W. T., and Wood, D. F.: The Analysis of Niobium and Niobium Alloys. Metallurgia, vol. 65, no. 392, June 1962, pp. 308-310.

6. Haymes, J. E., and Ollar, Albert: Methods for Determining Microquantities of Impurities in Tungsten. BM-RI-6005, Bur. Mines, 1962.

7. Huber, F. E., Jr., and Chase, D. L.: Determination of Carbon and Nitrogen in Some Refractory Metals. Chemist-Analyst, vol. 50, no. 3, 1961, p. 71.

8. Mueller, C. P.: Molybdenum Sheet Rolling Program. Interim Reps. 1, 2, and 3, Universal-Cyclops Steel Corp., Nov. 10, 1959.

9. Green, I. R., Still, J. E., and Chirnside, R. C.: The Determination of Carbon in the Less Common Metals and in Highly Alloyed Stainless Steels. Analyst, vol. 87, 1962, pp. 530-538.

10. Tougarinoff, B., et al.: Analysis of Refractory Metals - Niobium, Molybdenum, Tantalum and Tungsten. Working Paper M33, AGARD, p. 55.

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DETERMINATION OF LESS THAN 10 PARTS PER MILLION CARBON IN TUNGSTEN. William A. Gordon, Judson W. Graab, and Zita T. Tumney.
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In the combustion-conductometric method the smallest detectible quantity of carbon is limited by errors introduced by a spurious carbon blank. Also, the accuracy of the method for refractory metals is unknown since quantitative calibration is based on recoveries of carbon from steel samples or on organic compounds. Studies of the control of these possible sources of error resulted in establishing conditions whereby carbon concentrations of 4 ppm may be determined with a precision of ±12 percent and an estimated accuracy of ±15 percent relative. These conditions appear to be directly applicable to other metals, including Nb, Ta, Ti, Zr, Cr, and Cu.

I. Gordon, William A.
II. Graab, Judson W.
III. Tumney, Zita T.
IV. NASA TN D-2153

NASA

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