

DEPARTMENT OF COMMERCE

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**CIRCULAR**  
OF THE  
**BUREAU OF STANDARDS**

S. W. STRATTON, DIRECTOR

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No. 14

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**ANALYZED IRONS AND STEELS—  
METHODS OF ANALYSIS**

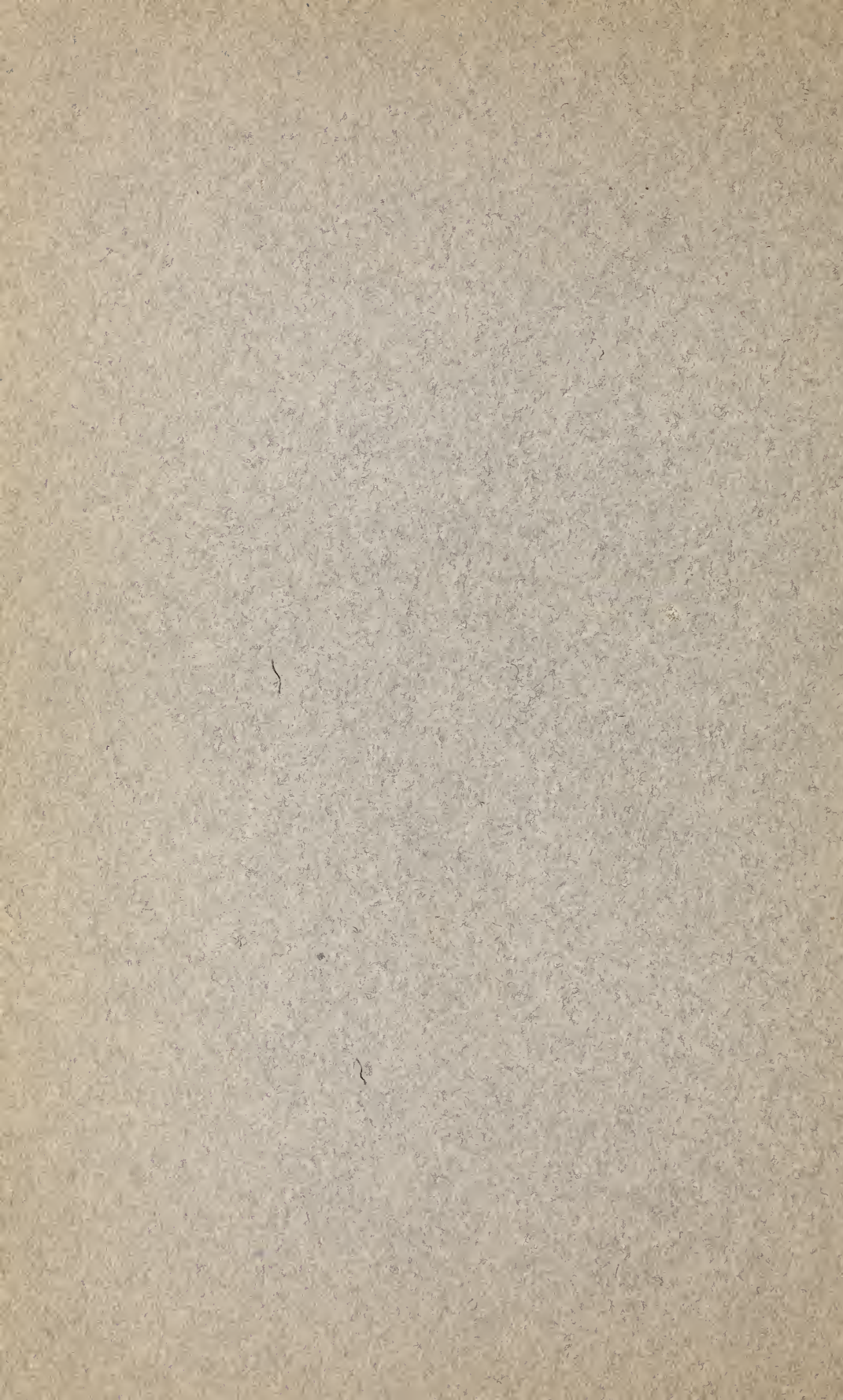
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5th Edition

Issued March 20, 1916



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1916



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# ANALYZED IRONS AND STEELS—METHODS OF ANALYSIS

Used at the Bureau of Standards

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**I. INTRODUCTION**

General information regarding standard samples, including descriptions of the methods used in their preparation, is published in Circular No. 25. The information and methods given in the present circular have special reference to the irons and steels listed in Circular No. 25. Former editions of Circular No. 14 gave outlines of the methods of analysis used at the Bureau of Standards for the earlier samples. Some of those methods have undergone modification from time to time and as herein given apply mainly to the irons and steels recently issued.

The earlier samples were analyzed usually only by the rapid technical methods used more or less generally by the steel trade. The analyses made by the other cooperating chemists were mostly of the same type. The object on the one hand was to indicate the order of agreement which might be expected from careful analysis, using these methods and working on a homogeneous material, and on the other hand to supply samples of which the composition was known within the limits of error indicated by the results published on the certificates. It is therefore emphasized here that the Bureau's analyzed irons and steels are "standardized" only in this sense. In other words, the figures on the certificates are perhaps to be regarded as a criterion of the existing state of the analytical chemistry of this class of materials and of methods, and they no doubt represent fairly well the result which may be expected by attempting to fix the composition of such materials in this way. It should be remembered, however, that the composition so determined is not necessarily the absolute or correct one, and that judgment should be used in the application of the data afforded by the certificates. An illustration will make this clear.

Scrutiny of all the earlier iron and steel certificates issued shows that the color method for manganese has furnished results over 5 per cent higher on the average than were obtained by the other methods combined. Without raising the question whether the average of the color results or that of the others is nearer the truth, it is clear that both can not be correct and that their "general average" must probably be in error. Hence, an analyst, using the

samples, is justified in rejecting the "general average" if experience has convinced him that the average for any particular one of the methods tabulated is worthy of greater credence. Further, for instance, it would be manifestly wrong to use any one of the general averages for manganese (possibly in error by one or more per cent of the manganese) for the standardization of a volumetric solution that is to be used for a ferromanganese alloy or a manganese ore.

No doubt the above cautions will be found applicable to some of the other common elements as well as to manganese, but, owing to the much smaller percentages of most of those others, their errors are not as a rule so immediately apparent nor so amenable to the corrective study which the Bureau hopes to take up in time.

Closer approximation to the truth might be obtained through determinations by a greater variety of technical methods, or with better promise of success by the longer and perhaps more exact methods not generally used for technical work, or by correcting for some more or less obvious sources of error often overlooked by the technical analyst. The amount of work to be thus expended on a given sample must be regulated by the importance of higher accuracy to those who use the sample. The Bureau endeavors to have the results by its own chemists well within the limits of error permissible in practical work. These in turn are governed by such factors as the degree of segregation in ingots, castings, etc., the difficulties of sampling large lots of material, etc. At present it is believed that in the steel and iron industry most of the technical analytical methods are capable of yielding results quite up to if not in advance of the requirements of practice. However, as already intimated, the Bureau will, when opportunity offers, investigate sources of error in present analytical methods and endeavor to devise new ones, when it is deemed necessary. The amount of such work which can be undertaken at present is limited.

It follows from what has been said that sometimes a method used by the Bureau to secure a somewhat higher degree of accuracy is too cumbersome or slow for technical needs, and this fact should be borne in mind by those employed at works.

A further caution is called for with respect to the methods detailed in the pages that follow. It should not be inferred, in the absence of a direct implication or statement to that effect, that the Bureau regards them as superior to others that might have been selected.

## II. METHODS FOR IRONS AND ORDINARY STEELS

### 1. TOTAL CARBON

One to four grams of the metal is placed in a platinum boat lined with alundum and is burned in a current of oxygen, as described below.

#### DETAILS OF DIRECT-COMBUSTION METHOD

*Furnaces and Temperature of Burning.*—Porcelain tubes wound with "nichrome" wire, provided with suitable heat insulation and electrically heated, are used, and readily give temperatures of 1000° C to 1100° C. Type FB 301 Hoskins tube furnace is also used, and is satisfactory. The temperature control is by means of an ammeter and rheostat in series with the furnace, with occasional check by a thermocouple.

*Boats and Lining.*—Platinum boats provided with a long platinum wire for manipulation in the tube are mostly used; alundum ones occasionally. The bed or lining on which the steel rests is the 90-mesh "RR alundum, alkali-free, specially prepared for carbon determination." A layer of this alundum is also placed in the bottom of the combustion tube to prevent the boat sticking to the glaze. A platinum cover for the boat is sometimes used, and is essential when the combustion is forced.

The nature and quality of the bed material are matters of great importance. Alumina as prepared from the sulphate or from alum may not be free from sulphate or alkali, both of which have given serious trouble at the Bureau. The alkali, if present, may not manifest itself by an alkaline reaction until after one or two combustions have been made, using the same bed material. Even the ordinary white "alundum" on the market carries a few hundredths of 1 per cent of alkali. Iron oxide has been tried, and when pure should, apparently, give good service. As yet, however, it has been difficult to obtain or prepare acceptable material for



use with steels. Quartz sand gives rise to a fusible slag which, melting before combustion is complete, incloses bubbles of carbon dioxide gas. (Personal communication from Mr. George M. Berry, of the Halcomb Steel Co.) This defect would probably inhere in any other material of an acid character. Mr. Berry has also occasionally noted the presence in the silica bed after combustion of crystals which appear to be carborundum.

*Purity of Oxygen. Blanks.*—The Bureau makes its oxygen electrolytically, and its content of this element is usually 99 to 99.5 per cent, and sometimes higher. Even with this gas a slight blank is usually obtained. When running a blank, in addition to the usual precautions, the rate at which the oxygen is introduced should be the same as when burning a sample, and the time should be three to five times as long.

*Method of Admitting Oxygen and Rate of Combustion.*—The furnace being at the proper temperature, the boat containing the sample is introduced. Oxygen is admitted either at once or after the boat has reached the temperature of the furnace, as the operator prefers or as the nature of the steel may demand. The rate of flow of the oxygen varies with the absorption apparatus used and with the preference of the operator, and may be considerably more rapid when absorbing carbon dioxide in soda-lime than in an alkaline solution. A rapid flow of oxygen also facilitates the burning of resistant samples. A continuous forward movement of the gas current is maintained at all times. The time for a determination varies, of necessity, with the nature of the sample and the rate of flow of the oxygen, ranging from 10 to 30 minutes. The endeavor is to obtain a well-fused oxide. With all samples close packing in a small space is conducive to rapid combustion and to fusion of the resulting oxide.

Authorities differ as to the advisability of allowing the oxide of iron to fuse thoroughly. Even when fusion does take place additional carbon dioxide is obtained very frequently by grinding the oxide and reburning. Often more than one grinding and reburning is necessary in order to reduce the amount of carbon dioxide obtained to that of the constant blank. Until further light can be thrown on the subject the Bureau will proceed in future on the assumption that the oxides should be fused and reburned.

(Consult Bureau of Standards Technical Paper No. 69, "Determination of Carbon in Steels and Irons by Direct Combustion in Oxygen at High Temperatures," by Cain and Cleaves.)

Oxides of sulphur have been found very difficult to eliminate from the gases leaving the tube. Lead peroxide ("nach Dennstedt") heated to 300° C, and zinc at room temperature, appear to retain them best.

Attention is called to the inadmissibility of using drying agents of different absorptive power in the same train, in positions where a difference could possibly affect results.

*Weighing of Tubes.*—There is much greater difficulty in securing constant conditions when weighing absorption tubes than is usually considered to be the case. Electrical effects, caused by wiping as a preliminary to weighing, may occasionally cause errors in weight running into the milligrams. The use of counterpoises of equal volume and similar material and shape is recommended.

If tubes are weighed full of oxygen, care is necessary to secure a uniform atmosphere in them. Even though the attempt is made to keep the apparatus always full of oxygen, some air is admitted when the boat is pushed into the combustion tube, and a much longer time is required to displace this than is usually allowed, unless the flow of oxygen during aspiration is rapid. The same is true if the tubes are weighed full of air by displacing the oxygen left in them after the steel is burned. Another source of error may arise from the air admitted when putting the boat into the tube, if this air contains much carbon dioxide, as is the case when a gas furnace is used. The boat is usually pushed at once into the hot furnace, and as combustion begins almost immediately, there is no opportunity for displacing this air before the steel begins to burn.

## 2. GRAPHITE IN IRONS

Two grams of iron is dissolved in nitric acid (sp. gr. 1.20), using 35 cc and heating very gently. The residue is collected on an asbestos felt, washed with hot water, then with a hot solution of potassium hydroxide (sp. gr. 1.10), followed by dilute hydrochloric acid and finally by hot water. After drying at 100° C the graphite is burned in the same manner as the total carbon.

### 3. COMBINED CARBON IN IRONS

The combined carbon in iron is obtained by subtracting graphitic carbon from total carbon.

### 4. SILICON

The insoluble residue obtained in preparing the iron or steel for the gravimetric sulphur determination (p. 10) is filtered off, ignited in platinum, and weighed. Evaporation with a little hydrofluoric acid and one drop of sulphuric acid and subsequent ignition gives by the loss in weight the silica corresponding to the silicon of the sample.

### 5. TITANIUM IN IRONS

Five grams of the sample is dissolved in 40 cc of hydrochloric acid (sp. gr. 1.05).<sup>1</sup> The insoluble matter is filtered off, washed with water, ignited in a platinum crucible, and treated with hydrofluoric and sulphuric acids to eliminate silica. The residue is fused with sodium carbonate, the fusion dissolved in water, the solution filtered, and the insoluble matter dissolved in sulphuric acid. A sufficient amount of ferric alum is added to the standard titanium solution to give the same tint as the sample when they are at the same dilution (for the residue from the silica always contains a little iron along with the titanium). Hydrogen peroxide is added to the solution and the standard, and these are compared in a colorimeter. (Consult Bull. 422, U. S. Geological Survey, for details regarding the determination of titanium.)

### 6. PHOSPHORUS

(a) **Preparation of Solution and Precipitation of Phosphorus.**—Two grams of sample is dissolved in nitric acid (sp. gr. 1.135) and the solution is boiled until brown fumes no longer come off. Ten cc of permanganate solution (15 grams to 1 liter) is added, and the boiling is continued. Sodium sulphite solution is added to dissolve the oxide of manganese, and the solution is again boiled and then filtered. With irons the insoluble residue should be tested for phosphorus. After cooling the filtrate, 40 cc of ammonia (sp. gr. 0.96) is added, the solution is agitated, and when the temperature is at 40° C, 40 cc of molybdate solution (Blair, Chemical

<sup>1</sup> With this strength of acid all but a negligible quantity of the titanium present remains in the residue.

Analysis of Iron, 7th ed., p. 97) is added and the solution is shaken vigorously for five minutes. After settling out, the yellow precipitate is treated according to one of the following methods, *b* or *c*:

(*b*) **Alkalimetric Method.**—The precipitate is washed 12 to 15 times with potassium nitrate solution. The precipitate is dissolved in a measured excess of standardized sodium hydroxide solution and titrated back with standardized nitric acid using phenolphthalein. The solutions are standardized by one of the well-known methods of alkalimetry or against Bureau of Standards benzoic acid, and not against a steel with a known amount of phosphorus, as formerly.

(*c*) **Molybdate Reduction Method.**—The precipitate is washed 10 to 15 times with acid ammonium sulphate (prepared according to Blair) or until the washings no longer react for iron. It is dissolved in 25 cc of ammonia (5 cc ammonia of 0.90 sp. gr. to 20 cc water). The filter is washed well with water and 10 cc of strong sulphuric acid added to the filtrate, which is run through the reductor at once into a ferric alum solution decolorized with phosphoric acid, and titrated against a  $\frac{N}{30}$  permanganate solution which has been standardized against sodium oxalate (Bureau of Standards Sample No. 40), as prescribed by McBride.<sup>1</sup>

## 7. SULPHUR BY OXIDATION

Five grams of iron or steel is dissolved in a 400-cc Erlenmeyer flask, using 50 cc of strong nitric acid. A little sodium carbonate is added, the solution is evaporated to dryness, and the residue baked for an hour on the hot plate. To the flask 30 cc of strong hydrochloric acid is added, and the evaporation and baking are repeated. After solution of the iron in another 30 cc of strong hydrochloric acid and evaporation to a sirupy consistency, 2 to 4 cc of the same acid is added, followed by 30 to 40 cc of hot water. The solution is then filtered and the residue washed with hot water. (See paragraph below for the treatment of this residue for its sulphur content.) The sulphur is precipitated in the cold filtrate (about

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<sup>1</sup> Bull. Bur. Stds., 8, p. 641; J. Am. Chem. Soc., 34, p. 393; 1912.

100 cc) with 10 cc of a 10 per cent solution of barium chloride. After 48 hours the precipitate is collected on a paper filter, washed first with hot water (containing 10 cc of concentrated hydrochloric acid and 1 gram of barium chloride to the liter) until free from iron and then with hot water till free from chloride; or, first with cold water, then with 25 cc of water containing 2 cc of concentrated hydrochloric acid to the liter. The washings are kept separate from the main filtrate and are evaporated to recover dissolved barium sulphate.

With irons the paper containing the insoluble residue above mentioned is put into a platinum crucible, covered with sodium carbonate free from sulphur, and charred without allowing the carbonate to melt. The crucible should be covered during this operation. Sodium nitrate is then mixed in and the mass fused with the cover off. An alcohol flame is used throughout. The melt is dissolved in water and evaporated to dryness in porcelain with excess of hydrochloric acid. The evaporation with water and hydrochloric acid is repeated to insure removal of nitrates. The residue is extracted with a few drops of hydrochloric acid and water, the insoluble matter is filtered off, and barium chloride is added to the filtrate. The barium sulphate obtained is added to the main portion.

Careful blanks are run with all reagents.

#### 8. SULPHUR BY EVOLUTION

Five grams of sample is dissolved in 60 cc of concentrated hydrochloric acid<sup>1</sup> in an evolution apparatus whose connections are all of glass, the air being first displaced by hydrogen. The gases evolved are passed through a solution containing 5 cc of 3-per cent hydrogen peroxide (made by diluting "perhydrol") and 25 cc of ammonia (sp. gr. 0.90). The solution in the evolution flask is boiled vigorously for several minutes after the metal has dissolved. The contents of the absorption tube are transferred to a beaker, are made slightly acid with hydrochloric acid if not already so from acid which has distilled over, boiled, and precipitated with 5 cc of boiling barium chloride solution (10-per

<sup>1</sup> At first the practice was to use acid of half strength. Certain steels, however, do not yield as much sulphur in volatile form with the weaker acid as with the stronger.

cent). The beaker is then placed on the hot plate for 10 or 15 minutes. After two or three hours the precipitate is filtered off on paper, washed several times with hot water, ignited, and weighed.

#### 9. MANGANESE

The bismuthate method as described by Blair (*Chemical Analysis of Iron*, 7th ed., p. 121) is used at the Bureau of Standards.

**Bismuthate Method.**—One gram of drillings is dissolved in 50 cc of nitric acid (sp. gr. 1.135) in a 200-cc Erlenmeyer flask. Irons should be filtered. The solution is cooled, about 0.5 gram of sodium bismuthate is added, and it is then heated until the pink color has disappeared. Any manganese dioxide separating is dissolved in a slight excess of a solution of sodium bisulphite. The solution is boiled till free from nitrous fumes. After cooling to 15° C a slight excess of bismuthate is added and the flask is shaken vigorously for a few minutes. Then 50 cc of 3-per cent nitric acid is added and the solution is filtered through asbestos. A measured excess of ferrous sulphate is run in and the excess titrated against permanganate solution which has been compared with the iron solution on the same day. A great many steels now carry small amounts of chromium as impurity. In such cases titration against arsenite solution is recommended, or removal of the chromium by zinc oxide and subsequent determination of the manganese by the bismuthate method.

Permanganate solutions are standardized against sodium oxalate (Bureau of Standards Sample No. 40) as prescribed by McBride.<sup>1</sup>

### III. METHODS FOR SPECIAL ALLOY STEELS

#### 1. INTRODUCTION

In some cases no decision has been reached as to the best methods for the determination of certain constituents. This statement holds for some of the commoner as well as special elements, for the presence of the latter may necessitate modification or abandonment of one or another of the methods applicable to ordinary steels.

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<sup>1</sup> Bull. Bur. Stds., 8, p. 641; J. Am. Chem. Soc., 34, p. 393, 1912.

It was expected that each of the Bureau's special steels would contain of the more unusual elements only those characteristic of that steel, aside from such traces as might occur in any steel, according to the composition of the ores and fluxes used in its making. Some of them, however, were found to contain unexpected elements in determinable and even considerable amounts. Thus, the vanadium steel contains 0.006 per cent molybdenum, 0.009 per cent chromium, and 0.009 per cent nickel. The nickel steel contains about 0.15 per cent tungsten and the chrome-vanadium steel about the same amount of nickel. As to the sources of these constituents the Bureau has little positive information. They are certainly to be sought in some cases in the ores and in others perhaps in crucibles, hearth linings, etc., which had been used for other special alloys, or in scrap. It seems probable that the condition is one of common occurrence. In any case the need for watchfulness on the part of the analyst is sufficiently indicated.

The methods that differ from those given under Section II of this circular will be found below, so far as occasion has arisen hitherto for their use at the Bureau.

## 2. COPPER

Ten grams of the sample is dissolved in sulphuric acid of 10 per cent strength and the hot solution is saturated with hydrogen sulphide, then filtered through paper. The filter is washed free from iron with very dilute sulphuric acid containing hydrogen sulphide. The use of this reagent is merely precautionary, to prevent possible solution of copper while filtering, for copper seldom if ever dissolves while the steel is dissolving, if air is wholly excluded. The paper with its contents is incinerated in a porcelain crucible, digested therein with nitric acid, transferred to a platinum crucible, and evaporated with hydrofluoric and sulphuric acids till the former acid has removed silica and itself suffered expulsion. The residue, if not soluble in sulphuric acid, is fused at a low heat, and only just long enough to effect the purpose, with sodium or potassium pyrosulphate. The copper is then deposited electrolytically from the acid solution, or is determined

by colorimetry in an ammoniacal solution or as the oxide after reprecipitation as the sulphide.

The method is applicable to irons and common steels.

### 3. MOLYBDENUM (IN SMALL AMOUNTS) WITH AND WITHOUT COPPER

The treatment is the same as for copper up to the first filtration, when it is advisable to filter through asbestos in glass or porcelain, instead of through paper, and to digest with aqua regia without igniting. The filtrate is evaporated with sulphuric acid, so as to have no other acid present. The residue is taken up with water, sulphuric acid added if needed to effect complete solution, and molybdenum (copper, too, if present) precipitated by hydrogen sulphide from the hot solution, preferably in a closed pressure bottle after saturating the cold solution with the precipitant.

The precipitate is collected on a Gooch crucible, dried, and converted by careful ignition to trioxide of molybdenum if copper and other metals are absent.

If copper is present, the sulphides are dissolved in nitric acid, and the solution is boiled with potassium hydroxide to precipitate cupric oxide. After filtration and re-solution the copper can be determined in any way desired. From the filtrate, acidified with sulphuric acid, the molybdenum may be thrown out as sulphide in the manner described above.

### 4. NICKEL

The dimethylglyoxime method of Brunck<sup>1</sup> is used without other modification than the adoption of Prettnner's<sup>2</sup> recommendation to wait an hour before filtering. As here described the method applies to nickel and chrome-nickel steels, but it is equally serviceable with irons and ordinary steels, provided a larger initial weight of sample is used and the bulk of the iron is removed by an ether separation.

One-half gram of the metal is dissolved in 10 cc of fairly concentrated hydrochloric acid, and then sufficient nitric acid is added to oxidize the iron. The solution is filtered, if need be, and to it is added 2 to 3 grams of tartaric acid, and then water till the volume is 300 cc. It is then made ammoniacal, and, if no iron precipi-

<sup>1</sup> *Stahl u. Eisen*, 28 (1), p. 331; 1908.

<sup>2</sup> *Chem. Ztg.*, 33, p. 396; 1909.



tates, is again made acid and heated nearly to boiling. Dimethylglyoxime (20 cc of a 1-per cent alcoholic solution) is added and then ammonia, drop by drop, till in slight excess. After standing an hour the deep-red precipitate is collected on a weighed Munroe or Gooch crucible, washed with hot water, dried for 45 minutes at 110–120° C, and weighed. It contains 20.31 per cent nickel.

#### 5. CHROMIUM

For small amounts of chromium, as in the vanadium steel, the method now used is as follows: Five to 10 grams of the sample is dissolved in a slight excess of sulphuric acid, the solution diluted to 200–300 cc, neutralized with sodium bicarbonate and boiled. The precipitate is filtered off rapidly, ignited, fused with an oxidizing mixture, the melt extracted with water, and the filtrate compared with a standard chromate solution.<sup>1</sup>

For larger amounts Blair's<sup>2</sup> (loc. cit.) and Cain's<sup>3</sup> methods are used.

#### 6. PHOSPHORUS

The acetate separation, followed by double precipitation as magnesium phosphate, was used for phosphorus in the standard steels containing vanadium. There is great difficulty in precipitating all the phosphorus by the molybdate reagent in presence of vanadium, and the latter is apt to contaminate the phosphomolybdate that does form, but a method has been devised which obviates these difficulties.<sup>4</sup>

#### 7. MANGANESE

Cain's method<sup>5</sup> is sometimes used, either as described by the author or with substitution of sodium bicarbonate for cadmium carbonate.

For steels free from vanadium the following method has been used:

The steel is dissolved in aqua regia and the greater part of the iron is removed by an ether separation. From the aqueous solution the sesquioxides (which will include some vanadium if

<sup>1</sup> See Hillebrand, Bull. 422, U. S. Geological Survey, p. 147.

<sup>2</sup> The Chemical Analysis of Iron, 7th ed., p. 209 et seq.

<sup>3</sup> Bur. Stds. Tech. Paper No. 6; J. Ind. and Eng. Chem., 4, 17, 1912.

<sup>4</sup> Cain and Tucker, Bur. Stds. Tech. Paper No. 24; also J. Ind. Eng. Chem., 5, 647, 1913.

<sup>5</sup> J. Ind. and Eng. Chem., 3, 630; 1911.

present) are precipitated by magnesium oxide or zinc oxide, the filtrate is evaporated to a small volume, the salts are converted to nitrates, and enough concentrated nitric acid is added to make 30 per cent by volume of the solution. The manganese is then determined by the bismuthate method.

#### 8. VANADIUM

The first of the following methods was used in analyzing the Bureau's vanadium and chrome-vanadium steels, and is reproduced here on that account.

(a) **Method I.**—Five to 10 grams of drillings is dissolved in hydrochloric acid (1 : 1), a few drops of hydrofluoric acid are added, and the solution is boiled for a few minutes. The insoluble matter is filtered off, ignited, fused with a little sodium carbonate, the fusion dissolved in water and added to the main filtrate. This is then oxidized with the minimum amount of nitric acid needed and boiled till free from fumes. The iron is extracted with ether and the excess of ether removed from the aqueous layer by evaporation on the steam bath. After concentration on the bath strong nitric acid is added to the solution and it is evaporated to dryness. The residue is dissolved in strong nitric acid, the solution is diluted with water and nearly neutralized with strong sodium hydroxide solution. It is then poured slowly into 150 to 200 cc of a 10 per cent sodium hydroxide solution, stirring vigorously. The solution is filtered, and the series of operations is repeated with the precipitate until it is free from vanadium, as shown by dissolving it in nitric acid and testing with hydrogen peroxide. In the latter treatments the amount of sodium hydroxide solution used may be smaller. From the combined filtrates the vanadium is precipitated with mercurous nitrate solution, after making nearly but not quite neutral with dilute nitric acid. After settling, the precipitate is collected on paper and washed with dilute mercurous nitrate solution. The filter is burned off in a platinum crucible and the precipitate ignited till all the mercury is expelled. The impure vanadium pentoxide left is fused with a little sodium carbonate, the fusion is dissolved in water and filtered (on asbestos) from insoluble matter. A second precipitation with mercurous nitrate is then made. Sometimes a further fusion and precipita-

tion may be necessary in order to get a product sufficiently pure for the next step, which is a final fusion with sodium carbonate. The fusion is dissolved in dilute sulphuric acid and the vanadium is reduced by sulphur dioxide gas and titrated against  $\frac{N}{50}$  permanganate after complete expulsion of the excess of reducing agent.

*Option.*—Preliminary solution may be in 1.20 nitric acid, followed by evaporation to dryness, baking, re-solution in concentrated hydrochloric acid, and filtration to get rid of silica. The first method is shorter and is just as effective.

(b) **Method II.**—See Cain, Bull. Bur. Stds., 7, p. 389, Reprint No. 161; also J. Ind. and Eng. Chem., 3, p. 476, 1911.

(c) **Method III.**—See Cain and Hostetter, Bur. Stds. Tech. Paper No. 8; also J. Ind. and Eng. Chem., 4, p. 250, 1912.

#### 9. TUNGSTEN

Close attention has not been paid by chemists at the Bureau of Standards to methods for tungsten. One of them determined the tungsten content of the B. S. chrome-tungsten steel (No. 31) as follows:

The steel is dissolved in aqua regia and the solution is evaporated to dryness and baked on the hot plate. After redissolving in hydrochloric acid (sp. gr. 1.20) the insoluble matter is filtered out, washed with dilute nitric acid, and treated on the filter with ammonia (sp. gr. 0.90) until all soluble matter is removed. The ammoniacal filtrate is evaporated to dryness in a weighed platinum crucible, nitric acid being added toward the end of the evaporation. The residue is ignited and weighed, treated with hydrofluoric acid to remove silica, again ignited, and weighed as  $WO_3$ . Fusion with sodium carbonate and extraction with water will indicate whether or not there is contamination by chromium or iron. The original filtrate obtained after dissolving the steel, when evaporated a second time, yields a small amount of tungstic oxide.

#### 10. CARBON

The difficulty of obtaining all the carbon by one burning is perhaps even greater with certain alloy steels than it is with plain steels. The procedure given on pages 6 to 8 applies here without modification.

