Dec. 13, 1960 S. J. BENNETT ET AL 2,964,389

APPARATUS FOR DETERMINATION OF OXYGEN IN METALS

Filed Aug. 15, 1958 2 Sheets-Sheet 1

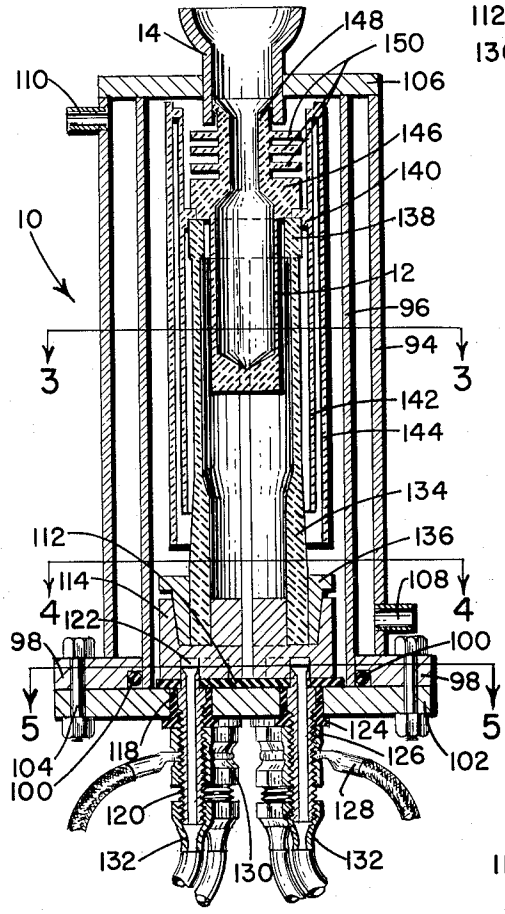


Fig. 2

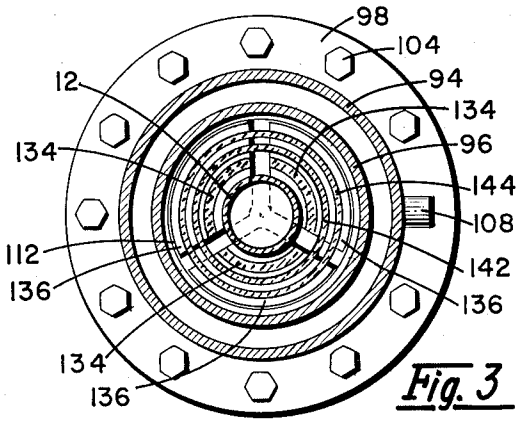


Fig. 3

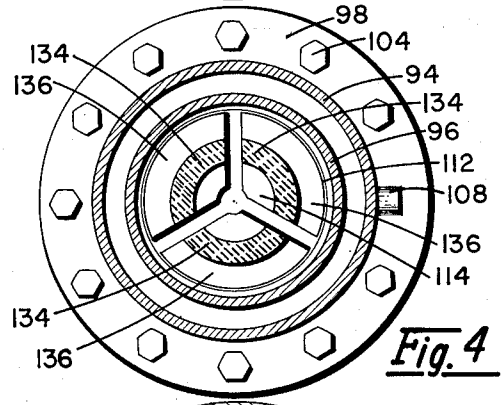


Fig. 4

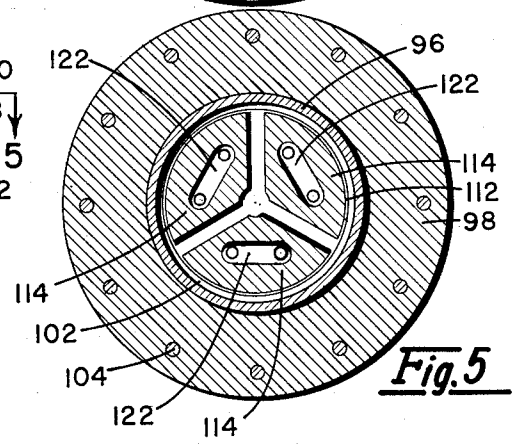


Fig. 5

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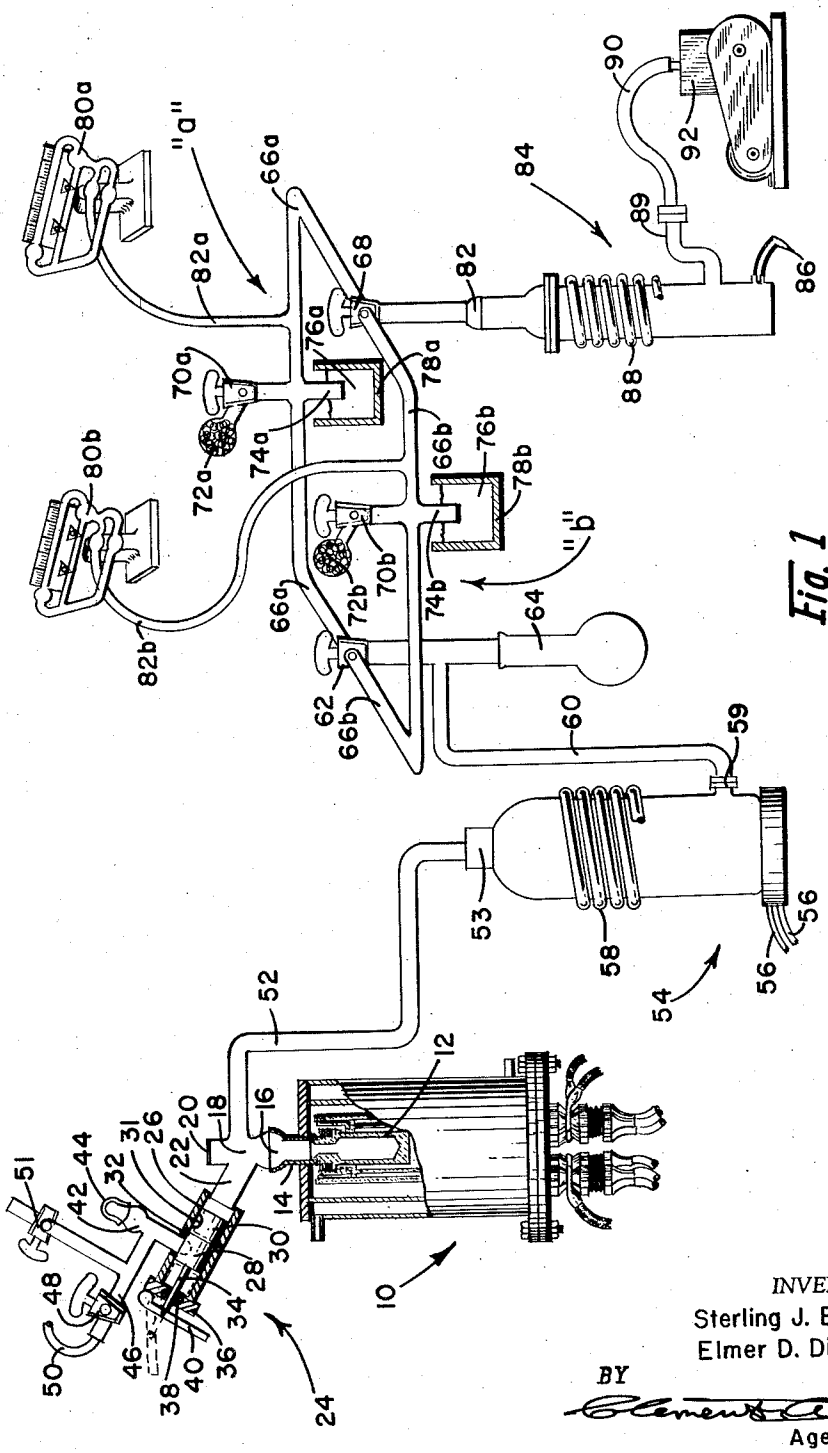


Fig. 1

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APPARATUS FOR DETERMINATION OF OXYGEN IN METALS

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3 Claims. (Cl. 23—253)

This invention relates to apparatus for the determination of oxygen in metals, particularly for the determination of oxygen in those metals adapted for such analysis for oxygen by the so-called "vacuum fusion" technique.

Previous apparatus for determination of oxygen in metals by vacuum fusion has not provided the desired accuracy or speed. Furnace units proposed and utilized have been cumbersome and involved relatively large volumes; outgassing of powders and packing employed for insulation has proved particularly troublesome. There is an established need for apparatus of this type which would permit a determination of oxygen within a period of a few minutes.

It is therefore the principal object of this invention to provide an improved apparatus for the determination of oxygen in metals. Another object of this invention is to provide apparatus which will make possible rapid determination of oxygen in metals. These and other objects of this invention will be apparent from the following description thereof and from the annexed drawings in which:

Fig. 1 is a general view of apparatus embodying features of this invention.

Fig. 2 is a vertical sectional view of the furnace unit employed in the apparatus of Fig. 1.

Fig. 3 is a horizontal sectional view of the furnace of Fig. 2 taken along the line 3—3.

Fig. 4 is a horizontal sectional view of the furnace of Fig. 2 taken along the line 4—4.

Fig. 5 is a horizontal sectional view of the furnace of Fig. 2 taken along the line 5—5.

Referring now to Fig. 1, the apparatus comprises a resistor type electric furnace indicated generally at **10** in which is provided a graphite crucible **12**. The furnace design and construction are of importance since the temperature and conditions obtained by its employment result in an advantageous combination with other elements; therefore the furnace itself is described hereinafter in more detail.

Removably engaging the top of crucible **12** and projecting upwardly from the furnace is a short connecting tube **14** to which is attached by globe seal **16**, vertical charging and gas takeoff tube **18**. The top of tube **18** is preferably sealed by optical flat **20** which permits sighting therethrough to determine the temperature of the crucible by, for example, use of an optical pyrometer. Communicating with side arm **22** of vertical tube **18** is a sample charger indicated generally at **24**. This unit comprises a tubular barrel **26** suitably connected and sealed to side arm **22** and in which rides solid piston **28** having an open end tubular extension **30** provided with port **31**. Gasket ring **32** is arranged to provide a sliding seal between piston **28** and the inner surface of barrel **26**. Piston rod **34** attached to the upper end of piston **28** projects through end closure member **36**, its passage therethrough being protected by seal **38**. Pivotably attached to the exterior end of piston rod **34** is lever handle **40**

by means of which the piston **28** and its tubular extension **30** may be actuated up and down in barrel **26**.

When the piston **28** is in lower position, as illustrated, it seals the lower portion of barrel **26** and thereby acts as an end closure seal for side arm **22**. When piston **28** is actuated by exterior handle **40** to its upper position in barrel **26** as shown in dotted lines, port **31** in tubular extension **30** will align and communicate with sample charging tube **42** which projects from the side of barrel **26** as shown and which may be opened and closed by removal or replacement of sealing cap **44**. A sample of metal to be analyzed disposed in charging tube **42** with piston **28** in lower position will, on raising piston **28** to upper position, fall through port **31**, into piston extension **30**, side arm **22**, tubes **18** and **14** and finally into crucible **12**. Charging tube **42** is also provided with communicating side arm **46** to which is attached through stockcock **48** a lead pipe **50** which is connected to a suitable source of vacuum (not shown).

Bleed valve **51** is suitably let into side arm **46** so that vacuum in charging tube **42** and side arm **46** may be relieved, when desired, by admission of air and cap **44** readily removed for sample insertion.

Also communicating with and projecting from tube **18** is gas delivery tube **52**. This tube **52** leads directly to the intake **53** of a diffusion pump indicated generally at **54** which may be of commercial and conventional design, and which is illustrated for clarity more or less diagrammatically, being provided with suitable heating means such as electric power input leads **56** and cooling means such as cooling coil **58**. Preferably this pump employs mercury as its working fluid. The outlet **59** of diffusion pump **54** is connected through a suitable tube or pipe **60** to the bore of three-way stopcock **62**. Since during operation of the apparatus, pipe or tubing **60** will be employed to collect and contain gas pumped from the furnace **10**, the volume of this part of the system may be increased by provision of connected flask **64**. Three-way stopcock **62** is arranged as shown to permit flow of gas from pipe **60** and flask **64** to, selectively, either connecting tubes **66***a* or **66***b*. These tubes **66***a* and **66***b* each connect similar absorption trains which communicate at their other ends with three-way stopcock **68**, and which for clarity will be referred to as the "*a*" train and the "*b*" train. Connected to tube **66***a* is stopcock **70***a* which controls connection and disconnection of tube **66***a* with an absorption bulb **72***a* connected therewith. Absorption bulb **72***a* is filled with suitable material for oxidizing carbon monoxide to carbon dioxide and also for absorbing carbon dioxide. Such materials are commercially available and generally comprise mixtures of various oxides including copper oxide, nickel oxide, manganese dioxide and rare earth oxides for oxidation, and include alkali substances such as calcium oxide and sodium oxide for absorption of carbon dioxide. Also communicating with tube **66***a* is cold trap well **74***a*. This well **74***a* is partially immersed in a cold producing mixture such as a combination of acetone and Dry Ice illustrated at **76***a* maintained in a suitable container **78***a*. Also communicating with tube **66***a* is a vacuum indicating instrument such as McLeod gage **80***a* which is suitably connected thereto by flexible hose **82***a*.

Tube **66***b* which connects the elements of the "*b*" train is provided with the same unit elements as tube **66***a* and which include the absorption bulb stopcock **70***b* and its attached absorption bulb **72***b*, and also the cold trap well **74***b* immersed in cold producing material **76***b* in the container **78***b*. A vacuum indicating instrument, such as McLeod gage **80***b*, is similarly connected through hose **82***b* to tube **66***b*.

The bore of three-way stopcock **68** communicates with

the intake 82 of a diffusion pump indicated generally at 84 and which is preferably of the oil type. Such pumps are commercially available and may be of conventional design being provided with heating means such as power inlet leads 86 and cooling means such as water circulating coil 88. Suitable fore pressure is maintained on diffusion pump 84 by connection of its outlet 89 through connecting pipe 90 to a conventional mechanical vacuum pump 92.

Referring now to Figs. 2, 3, 4, and 5, the furnace indicated at 10 and referred to in the general description above comprises an outer cylindrical shell 94 and an inner cylindrical shell 96 which are joined at their bases to flange 98. An annular groove in flange 98 encloses O-ring seal 100 to form a gas tight joint between flange 98 and furnace base plate 102 to which flange 98 is attached as by removable bolts 104. The tops of shells 94 and 96 are joined as by welding to furnace top plate 106. The space between shell 94 and shell 96 forms a jacket for circulation of cooling fluid, such as water, which my be introduced through fitting 108 and exhausted through fitting 110. Inside inner shell 96 and disposed on top of base plate 102 is electrically insulating plate 112 which may be fabricated of a suitable ceramic material or organic insulating substance, such as a fluorinated hydrocarbon. Supported on insulating plate 112 are three conductor blocks 114 preferably of copper which, as shown, are constructed in the form of separated circle segments. Passing through base plate 102 and electrically insulated therefrom by bushings 118 are externally threaded tubes 120 which at their upper ends are screwed into blocks 114 and whose interior passages communicate in pairs with the ends of interior cooling ducts 122 in blocks 114. Insulating washers 124 protect the adjacent surfaces of base plate 102 and provide bearing surfaces for nuts 126 by which the threaded tubes 120 may be firmly fastened to plate 102 and also thereby securely fastening contact blocks 114 on top of insulating plate 112. Threaded tubes 120 will be in electrical contact with contact blocks 114 and suitable electrical connections may be made thereto by provision of lugs 128 which are secured around tubes 120 by locking nuts 130. The lower ends of threaded tubes 120 are provided with hose connections 132 so that a cooling fluid such as water may be circulated through each block from a suitable source not shown. It will be appreciated that the cooling water connections to each of the blocks 114 are independently electrically insulated from the furnace base plate 102 and therefore the threaded tubes 120 may be employed as conductor units for transmitting electric power to the blocks 114 inside the furnace.

Blocks 114 on their upper surfaces are provided with deep arcuate grooves into which are placed mating legs 134 of the graphite crucible heating assembly. Each of the three legs 134 is fixedly maintained in its position in the corresponding groove in block 114 by provision of wedge members 136. At their tops the three legs 134 are joined by graphite ring 138. Supported by ring 138 is collar 140 from which are suspended concentric graphite heat shield members 142 and 144.

Extending from the upper part of crucible 12 is shoulder 146 which also rests on ring 138 to support the crucible assembly. The outlet tube portion 148 of crucible 12 is provided along its sidewalls with radiating heat shields 150 and mates in a slideable joint with furnace outlet tube 14 which transfixes furnace top plate 106.

Electric power, supplied from a suitable and conventional source, not shown, such as a transformer, connected to the lugs 128 will be transmitted to blocks 114 and legs 134 to ring 138 resulting in heating of the ring 138 and also to a degree the legs 134 by reason of their electrical resistance.

When it is desired to disassemble furnace 10, for example, to replace and renew crucible 12, this may be accomplished by removing bolts 104 and separating the base 102 and associated structures including the crucible support assembly and the crucible 12 from the top and side shell elements. The slideable joint between the top of crucible 12 and outlet tube 14 permits separation of the gas transfer system at this point.

The electrical resistor furnace 10 is preferably, as shown, of the type employing three phase current. This provides an efficient heating unit and, in addition, the magnetic field generated by the three phase current produces a stirring action in molten metal in crucible 12. At the temperature employed this results in rapid contact and reaction between oxygen in the metal and the graphite crucible to produce carbon monoxide gas. The metal sample must be introduced into the crucible without contaminating the atmosphere of the crucible and associated equipment with air or oxygen, and vacuum lock sample introduction means, as for example illustrated at 24, are preferred. The mercury diffusion pump illustrated at 54 provides means for pumping carbon monoxide from the crucible 12 into a collection space which in the embodiment illustrated in Fig. 1 is enclosed by pipe 60, flask 64 and selectively one of the pair of absorption trains referred to above as the "*a*" train and the "*b*" train. The space thus enclosed is calibrated, that is, its volume is determined and known under conditions when either of the absorption trains is connected to pipe 60 and flask 64 to form the complete total collection space.

Each of the absorption trains comprises, as described, a connected carbon monoxide absorption bulb, a cold trap and a vacuum measuring instrument. Each absorption bulb is disconnectable from the rest of the train of which it forms a part, and in the apparatus illustrated this is accomplished by appropriate manipulation of stopcock 70*a* or 70*b*. In addition each of the absorption trains "*a*" and "*b*" may be selectively isolated from the rest of the apparatus and means for accomplishing this are shown in the embodiment illustrated as three-way stopcocks 62 and 68. It will be apparent that stopcock 62 may be turned so as to isolate or connect either of trains "*a*" or "*b*" with pipe 60 and flask 64, and stopcock 68 can isolate or connect each train with vacuum pump 84 which provides means for evacuating the collection space, including either trains "*a*" or "*b*," intermittently during progress of analytical determinations as will be hereinafter described in more detail.

It is preferred to employ a flux of platinum metal in the graphite crucible 12 when analyzing samples. The addition of platinum in the form, for example, of wire, chips, foil or sheet, with each sample provides a molten bath in which the sample metal dissolves or is dispersed and insures better contact with the graphite of the crucible resulting in more efficient reaction therewith to produce carbon monoxide from any oxygen present.

A convenient and advantageous method for preparing samples comprises cutting an 0.1 gram piece from the parent material and pickling in 5% hydrofluoric acid solution to remove any surface oxides formed in the cutting operation. The pieces are then rinsed in water and acetone, allowed to dry in the air and then weighed. Each sample is then wrapped in a length of platinum wire weighing at least ten times the weight of the sample. Five and one-half inches of B. & S. 20 gauge wire is a convenient length for samples ranging in weight from 0.10 to 0.15 gram. The platinum-wrapped sample is degreased by rinsing in acetone, and is then allowed to dry in air. It is then ready for introduction into the vacuum fusion apparatus and should be handled only with tweezers to prevent contamination.

The collection space, when either of the absorption trains are connected to pipe 60 and flask 64, may readily be calibrated by first evacuating the entire system by means of pumps 84 and 92. Stopcock 68 is then closed. A measured volume of air at known pressure, advantageously atmospheric, is introduced conveniently through

the inlet of bleed valve 51. This air is then pumped by pump 54 into the collection space, that is, pipe 60, flask 64 and one of the absorption trains. A pressure reading is then taken on the gage connected in the absorption train used. Since the original volume and pressure of the introduced air is known and its pressure in the collection space is known, its new volume may readily be calculated. The operation is repeated with the other absorption train connected to pipe 60 and flask 64 and the volume determined for this collection space.

After the collection spaces have been calibrated as described above, and the apparatus degassed with the crucible assembly at a temperature suitably higher than that normally employed, the power input to the furnace is regulated to provide the desired temperature for analysis of the contemplated metal samples. The temperature of crucible 12 is conveniently measured by sighting through optical flat 20 with an optical pyrometer. With the whole system under evacuation and with pumps 54, 84, and 92 running, stopcocks 62 and 68 are manipulated to connect one of the "*a*" and "*b*" absorption trains to pipe 60 and flask 64 and to shut the absorption trains off at their other ends from pump inlet 82. For example, train "*b*" may be connected to pipe 60 and flask 64 by adjusting stopcock 62 so that its bore is connected to tubing 66*b*, and stopcock 68 adjusted so that its bore is shut off from both tubes 66*b* and 66*a*. The collection space into which carbon monoxide and other gases will be pumped from crucible 12 by diffusion pump 54 will consist of tube 60, flask 64 and train "*b*." Train "*a*" will be isolated from the rest of the apparatus.

With handle 40 down, stopcock 48 closed and the vacuum broken in tube 42 and arm 46 through bleed valve 51, cap 44 of the vacuum lock charging means 24 is removed and a sample placed in tube 42. Cap 44 is then replaced, bleed valve 51 closed and tube 42 is evacuated by manipulation of stopcock 48 to provide connection through tubing 50 to a vacuum pump or other means for evacuation, not shown. When the tube 42 has been evacuated, stopcock 48 is closed and arm 40 is manipulated from its lower position as shown in Fig. 1 to its upper position as shown in the same drawing in dotted lines, thus permitting the sample in tube 42 to fall through port 31 into open tube 30, then into side arm 32 through takeoff tube 14 and into crucible 12. Arm 40 is then returned to its original closed position thus sealing the sample charger from the rest of the system. When the sample becomes heated in crucible 12 its oxygen content is converted to carbon monoxide by reaction with the graphite crucible itself and the carbon monoxide thus formed and generated, together with incidental amounts of other gases such as hydrogen and nitrogen, are pumped through tube 52 and into the collection space formed by tube 60, flask 64 and connected absorption train "*b*." Generally it takes less than 2 minutes to convert the oxygen content of the sample and to pump the carbon monoxide into the hereinbefore described collection space. After 2 minutes it is reasonably certain that all of the carbon monoxide has been collected from crucible 12 and is now in the collection space of which absorption train "*b*" is a portion. Train "*b*" is now isolated from the rest of the apparatus by manipulating stopcock 62 so that its bore connects tube 60 now to absorption train 66*a*, and stopcock 68 is manipulated so as to connect pump 84 with the other end of tube 66*a*.

A pressure reading is now taken on McLeod gage 80*b*. Stopcock 70*b* is then operated so as to connect absorption bulb 72*b* with the remainder of the "*b*" train so that carbon monoxide now in the train collection space may be absorbed by the absorbent materials in bulb 72*b*.

At the same time the gases in pipe 60 and flask 64 are being evacuated together with the connected "*a*" train through diffusion pump 84, and mechanical pump 92. After a suitable period of evacuation stopcock 68 is then closed to both "*a*" and "*b*" trains while stopcock 62 is retained in position to connect absorption train "*a*" with pipe 60 and flask 64. At this stage the apparatus is in condition similar to that which existed before introduction of the sample as hereinbefore described with the exception that the "*a*" absorption train forms the corresponding portion of the total collection space into which carbon monoxide, generated from another sample of metal, will be pumped by diffusion pump 54.

The operation of introducing another sample is then repeated as described above with the carbon monoxide and other incidental gases being collected in pipe 60, flask 64 and the connected absorption train "*a*." During the time that this is being carried on, the carbon monoxide retained in isolated absorption train "*b*," is being absorbed by the chemicals in bulb 72*b*. When the carbon monoxide in the "*b*" absorption train has been absorbed in bulb 72*b* and this takes only a few minutes, then stopcock 70*b* is again closed, disconnecting absorption bulb 72*b* from the remainder of the "*b*" train and a second pressure reading is taken on instrument 80*b*. The difference in pressure before and after absorbing the carbon monoxide from the gases retained in absorption train "*b*" may be employed, as will be apparent to those skilled in the art, to calculate the percentage of carbon monoxide in these gases; and since the space enclosed by absorption train "*b*" is a portion of the total collection space into which all the carbon monoxide was pumped from crucible 12 then the amount of carbon monoxide evolved from the sample may readily be ascertained.

While the carbon monoxide from the first sample is being absorbed from the portion of the gases which were isolated in absorption train "*b*" the carbon monoxide evolved from the second sample will now be confined in the space of which absorption train "*a*" is a portion and after a suitable and similar time period as that previously employed, absorption train "*a*" is isolated and the carbon monoxide determined in the gases confined therein by pressures indicated on gage 80*a*. Absorption circuit "*b*" is then connected to pipe 60, flask 64 and vacuum pumps 84 and 92, so that residual gases may be evacuated and the confined space enclosed by tube 60, flask 64 and absorption train "*b*" may be readied for reception of carbon monoxide containing gases in the next sample to be introduced into crucible 12.

The cycle described above is repeated for the determination of oxygen in a number of samples. The unique apparatus in which duplicate absorption trains are employed alternately to measure the carbon monoxide content of gases evolved from samples heated in crucible 12 in the furnace 10, provides a most effective and time saving utilization of the apparatus elements. The organization as described permits a determination of oxygen to be completed within five to seven minutes, in a continuous series of analyses.

A small amount of gas is continuously evolved from the walls of the furnace and the apparatus in any vacuum system. For accurate work, it is necessary to determine the amount of this gas which is evolved during the time required to run a sample so that a correction for it may be made when the results are calculated. This is commonly referred to as the "furnace blank" and is measured by following exactly the same procedure as used to run a sample except that no sample is introduced. The difference in pressure obtained is a measure of the carbon monoxide content of these furnace gases, and is subtracted from the pressure difference obtained for a sample. This furnace blank should be measured before samples are run whenever the furnace has been opened to air. If the system is free of leaks, this blank is consistent from day to day.

Another source of error which must be determined and for which a correction must be made, is the oxygen content of the platinum introduced with the sample, if this is employed. This blank may be determined by introducing a similar piece of platinum into the crucible

and following the procedure previously described for samples. By using exactly the same length of wire, or amount of other form, for each sample, this platinum blank is constant.

The amount of oxygen in a sample may be calculated from the following formula:

$$\%O=\frac{(\Delta P)(V)(16)(100)}{(760)(R)(T)(S.W.)}$$

where

ΔP=the initial pressure in mm. Hg in the absorption train less the sum of the pressure after absorption of CO and the furnace and platinum blanks
V=volume of collection space in liters
16=wt. of oxygen per mole of CO
100=to convert to percentage
R=gas constant=0.08205 liter atmospheres per mole degree
T=absolute temperature
S.W.=sample weight in grams
760=to convert pressure readings to atmospheres

The temperature in most laboratories does not vary more than a few degrees; thus, assuming T to be constant at 300° K. will not introduce a significant error.

To illustrate the speed of operation and accuracy obtained, sixteen samples of a bar of unalloyed titanium metal were tested for oxygen in apparatus of type hereinbefore described. The oxygen content of the bar of titanium had previously been precisely analyzed by a long series of careful tests and found to be 0.092%. With the crucible maintained at 2100° C. and employing platinum wire flux material the sixteen samples were analyzed for oxygen in one hour and forty minutes. The analyses shown in Table 1 below, show acceptable accuracy.

Table 1

Sample	Analysis, percent O
Unalloyed Ti:	
0.117% O	0.110
0.117% O	0.120
0.117% O	0.130
0.117% O	0.126
0.117% O	0.123
0.117% O	0.124
0.117% O	0.120
0.117% O	0.129
0.117% O	0.126
0.117% O	0.127
0.117% O	0.125
0.117% O	0.114
0.117% O	0.119
0.117% O	0.124
0.117% O	0.128
0.117% O	0.127
Average	0.123
Range	0.019
Standard Deviation	0.006

The apparatus of this invention is useful for determining oxygen in any of the metals which may be analyzed by the so-called "vacuum fusion" technique in which the sample is melted under vacuum in the presence of carbon to convert oxygen to carbon monoxide. Such metals include, titanium, zirconium, hafnium, iron, niobium, uranium, aluminum, magnesium, beryllium, chromium, nickel, vanadium, copper, molybdenum and thorium.

The temperature of the furnace should be adjusted to provide rapid and efficient conversion of the oxygen content of metal samples to carbon monoxide and must be higher than the melting point of metal sample or bath in the crucible. For titanium and titanium base alloys employing a platinum bath for example, a temperature of about 2100° C. will be advantageous, and this temperature is useful for analyzing other metals also using the platinum bath. For other techniques including use of no bath, a suitable temperature dependent on the melting point, vapor pressure, rate of carbide formation, rate of carbon monoxide evolution, and other factors may be employed.

Employment of the three phase resistor furnace provides a clean simple design which promotes rapid conversion of oxygen to carbon monoxide. The stirring action of the three phase current accelerates the chemical conversion and the simple construction of the furnace, in which the graphite crucible is employed as the carbon supply, promotes rapid transfer of gases into the collection space. Provision of duplicate absorption trains, each forming alternately a portion of the collection space for succeeding analyses, results in an overlapping of time consuming operations so that in a series of determinations the average time for each may be no more than five to seven minutes. This is made possible by the unique organization described in which generation and collection of carbon monoxide is accomplishd in about the same period of time as that required for absorption of carbon monoxide from a portion of the gases generated from the previous sample. The combination of the furnace design and the collection arrangement therefore provides an improved apparatus capable of rapid operation and producing results of acceptable accuracy.

We claim:

1. Apparatus for determining oxygen in metals comprising; an electrical resistor furnace comprising a shell, a plurality of upstanding graphite legs within said shell each connectable to a lead of an electrical power source, a graphite crucible in said furnace supported by said graphite legs, means for introducing an oxygen-containing metal sample into said crucible when heated thereby to convert the oxygen content of said sample to carbon monoxide, pumping means for transferring said carbon monoxide from said crucible into a calibrated collection space, means for enclosing said collection space, a portion of said collection space being selectively enclosed by one of a pair of absorption trains, each of said trains comprising a carbon monoxide absorption bulb disconnectable from said train, a cold trap and a pressure measuring instrument, means for intermittently selectively isolating one of said pair of absorption trains from the remainder of said apparatus, and means for evacuating said collection space including the other of said pair of absorption trains while the one of said pair of absorption trains is isolated therefrom.

2. Apparatus for determining oxygen in metals comprising; an electrical resistor furnace comprising a shell, three upstanding graphite legs within said shell each connectable to a lead of a three-phase electric power source, a graphite crucible in said furnace disposed between the top portions of said graphite legs and supported thereby, means for introducing an oxygen-containing metal sample into said crucible when heated thereby to convert the oxygen content of said sample to carbon monoxide, pumping means for transferring said carbon monoxide from said crucbile into a calibrated collection space, means for enclosing said collection space, a portion of said collection space being selectively enclosed by one of a pair of absorption trains, each of said trains comprising a carbon monoxide absorption bulb disconnectable from said train, a cold trap and a pressure measuring instrument, means for intermittently selectively isolating one of said pair of absorption trains from the remainder of said apparatus, and means for evacuating said collection space including the other of said pair of absorption trains while the one of said pair of absorption trains is isolated therefrom.

3. Apparatus for determining oxygen in metals comprising; an electrical resistor furnace comprising a shell having a top plate, an outlet tube transfixing the top plate of said shell, a plurality of upstanding graphite legs within said shell each connectable to a lead of an electrical power source, a graphite crucible in said furnace and supported by the tops of said graphite legs, said crucible

having an upper extending outlet tube portion mating in the outlet tube transfixing the top plate of said shell, means for introducing an oxygen-containing metal sample into said crucible when heated thereby to convert the oxygen content of said sample to carbon monoxide, pumping means for transferring said carbon monoxide from said crucible into a calibrated collection space, means for enclosing said collection space, a portion of said collection space being selectively enclosed by one of a pair of absorption trains, each of said trains comprising a carbon monoxide absorption bulb disconnectable from said train, a cold trap and a pressure measuring instrument, means for intermittently selectively isolating one of said pair of absorption trains from the remainder of said apparatus, and means for evacuating said collection space including the other of said pair of absorption trains while the one of said pair of absorption trains is isolated therefrom.

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