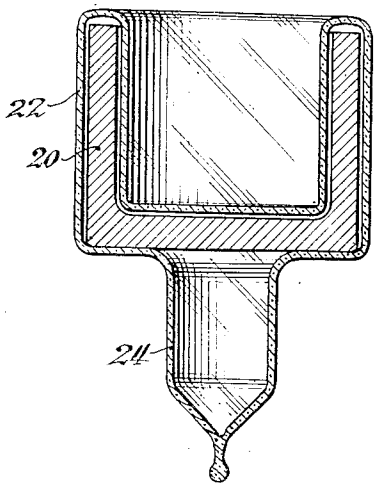
Jan. 29, 1957 E. L. BENNET 2,779,581

COMBUSTION ANALYSIS CRUCIBLE

Filed July 23, 1953

Fig. 1.



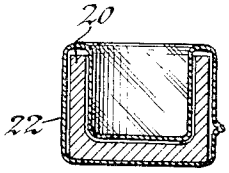


Fig. 2.

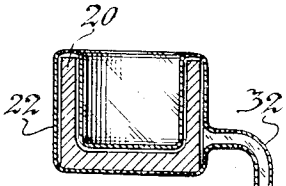


Fig. 9.

Fig. 3.

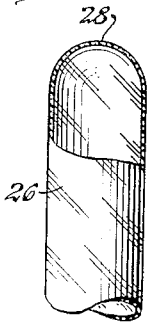


Fig. 5.

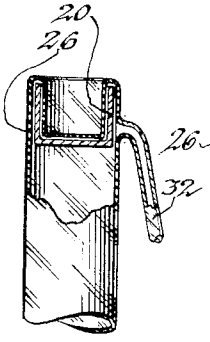


Fig. 6.

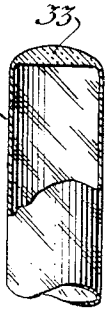


Fig. 8.

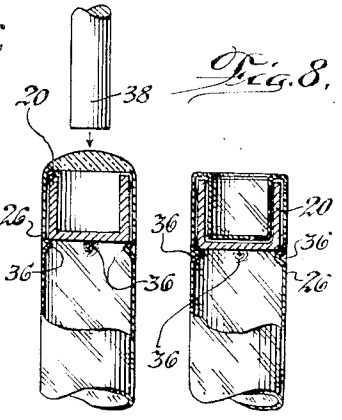


Fig. 4.

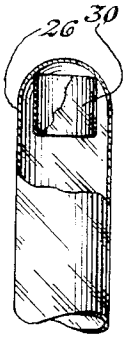


Fig. 7.

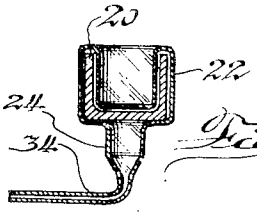


Fig. 10.

Inventor:
Eugene L. Bennet
By Ahlberg, Hespenheide & Graham
Attorneys.

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COMBUSTION ANALYSIS CRUCIBLE

Eugene L. Bennet, St. Joseph, Mich., assignor to Laboratory Equipment Corporation, St. Joseph, Mich., a corporation of Michigan

Application July 23, 1953, Serial No. 369,809

5 Claims. (Cl. 263—48)

The present invention relates to small crucibles of the type used for combustion analysis.

In the copending application of George J. Krasl, Serial No. 226,017, filed May 12, 1951, for Combustion Analyzer and Method of Analysis, there is shown and described an apparatus of the type the crucible of the present invention is intended to be used with. Briefly, in that application there is shown an arrangement in which a small cup shaped crucible containing a sample to be analyzed for carbon, for instance, is placed upon the upper end of a hollow vertical pedestal and elevated into a radio frequency induction field. The sample in the crucible if of proper constitution is quickly heated to ignition temperature by the induction field acting thereon. When the sample is hot, a jet of oxygen is blown against it so as to convert the sample into products of combustion which will include all of the carbon in the sample in the form of carbon dioxide gas. The quantity of carbon dioxide is determined in an essentially conventional manner.

Similar apparatus for a generally similar purpose, excepting that it is more directly concerned with gas analysis for sulphur, forms the subject matter of an application by the said George J. Krasl, Serial No. 288,878, filed May 20, 1952, for Gas Analysis for Sulphur. The crucible of the present invention is also adapted to be used for carrying out the method disclosed in said application Serial No. 288,878. Both of the above identified applications and the present application, are assigned to the same assignee of record.

As explained in application Serial No. 226,017, no particular problem is encountered in heating the sample in the induction field so as to bring about combustion if the sample to be analyzed is steel or some other substance which is readily heated by such a field. Frequently, however, it is necessary to analyze substances for their carbon or sulphur content when the samples are of such composition that there is no heating produced by an induction field, or the heating effect is too slight to bring about combustion. The arrangement suggested in the before mentioned applications for accomplishing an analysis under these conditions is to incorporate a quantity of steel or iron or other strongly heated substance with the sample to be combusted so that in the presence of the induction field, the field will heat the iron, for instance, to combustion temperature, and the burning iron will raise the temperature of the sample to be tested to the combustion temperature. This scheme is not completely satisfactory since the iron or other substance added to the crucible to bring about combustion will ordinarily contain some carbon or sulphur, and this quantity therefore must be known and must be deducted from the result in order to arrive at the carbon dioxide or sulphur dioxide content of the gases which is due to the carbon or sulphur in the sample.

It is the principal object of the present invention to provide a novel crucible for gas analysis which facilitates the induction heating of a sample to be tested to a temperature at which combustion in oxygen takes place even though the sample to be tested is not substantially heated directly by an induction field.

Yet another object is to provide an improved crucible having the above characteristics and which can be used for heating a noninductive sample in an induction field to combustion temperature without contamination of the sample by products of combustion not directly attributable to the sample itself.

Yet another object is to provide a novel crucible for use in induction heating of a sample which is not substantially directly heated by an induction field and in which a high order of accuracy in the determination can be arrived at by proceeding upon the assumption that all of the carbon dioxide or other gases of combustion are due to burning of the sample.

Yet another object is to provide a crucible which is self-heating in an electromagnetic induction field and which is inert chemically in the presence of oxygen at combustion temperatures.

Other objects and advantages will become apparent from the following description of a preferred embodiment of my invention, in which:

Fig. 1 is a vertical medial sectional view through a crucible embodying one form of the invention;

Fig. 2 is a view similar to Fig. 1 showing an alternative form the crucible may take;

Figs. 3, 4 and 5 are views partly in elevation and partly in vertical medial section, showing successive stages in the manufacture of the crucible of Fig. 1 or 2 according to one method;

Figs. 6, 7 and 8 are similar to Figs. 3, 4 and 5 excepting that they illustrate an alternative method for forming the crucible of the present invention;

Fig. 9 is a vertical medial sectional view showing the final stage in the manufacture of the crucible illustrated in Fig. 2; and

Fig. 10 is a view similar to Fig. 9 but showing the final stage in the manufacture of the crucible illustrated in Fig. 1.

As indicated above, combustion analysis which is based upon burning of a sample in oxygen and in which the sample is heated to combustion temperature by an induction field, usually depends upon the sample to be tested absorbing sufficient energy from the induction field to raise its temperature to the ignition point. There are substances, however, that do not absorb energy from an induction field sufficiently to accomplish this. As examples, an induction field will not directly raise the temperature of analytical samples of cement, earth, ores, oils or coke to their ignition point or to the point where carbon or sulphur compounds in the sample are volatilized and combusted. Some of these substances are heated to some extent, but samples of the size used in analytical determinations are in general difficult to heat to high temperature since they are quite small and therefore have considerable surface area for the loss of heat in proportion to their mass.

Fig. 1 of the drawings shows a crucible embodying the present invention illustrated in medial section. One element of this crucible is a cup-like element 20 formed of a material which is strongly heated in an induction field. For this purpose carbon, iron or of course other substances may be used. The choice will depend to a large extent upon the strength of the induction field available and upon the size of the crucible. For use with the equipment described in the beforementioned patent application, carbon has proved to be entirely satisfactory.

The cup-like element 20, whether formed of carbon, iron or other material, is completely enclosed in an envelope 22 which preferably should fit the cup 20 comparatively loosely so that when the crucible is shaken, the

cup 20 will rattle slightly within the envelope. This loose fit is more important if the cup 20 is formed of a material such as iron which expands considerably when raised to high temperature. It is less important when substances such as carbon are used. In fact the envelope may fit a carbon cup quite closely.

The envelope is formed of a substance which is inert at high temperature and impervious to the passage of gas. Also this material should be one which will not fracture when its temperature fluctuates widely or when it is subjected to considerable localized temperature differences. Fused quartz is admirably suited for this purpose as are certain low expansion, high silica content glasses which are customarily considered to be artificial quartz. A glass product having these characteristics is supplied under the designation "Vycor," by the Corning Glass Works. This product is understood to be about 96% silica and has an extremely low coefficient of thermal expansion. Some vitrified ceramic products are also suitable for this purpose.

After the cup-shaped element 20 has been loosely enclosed in the quartz or artificial quartz or similar envelope, most of the remaining air within the envelope is exhausted and the envelope sealed. This exhausting step may be conducted with any suitable commercial evacuating equipment and need not be carried to an extreme extent. The purpose is to remove the gas sufficiently so that sudden heating of the crucible, particularly the cup-shaped element 20, will not result in subjecting the envelope to bursting pressures due to expansion of the gas within the envelope. The evacuation also is helpful in that it prevents any substantial oxidation of the hot cup 20.

In Fig. 1 the crucible is shown with a stem 24 extending downwardly from the center thereof. This stem in this instance is formed of the envelope material and its purpose is to center the crucible at the top end of a hollow pedestal upon which it is supported in an induction field. This particular crucible, therefore, is advantageous for use with the apparatus described in the beforementioned copending applications. The crucible of Fig. 2 is similar to that of Fig. 1 excepting that it has a flat base and therefore has a more universal application, since it can rest upon any reasonably flat surface.

One method for constructing a crucible of the type discussed above from fused or artificial quartz is illustrated in Figs. 3 to 5, where it will be seen that as shown in Fig. 3 a tube of the quartz or artificial quartz or similar material indicated at 26 is provided with a closed end as at 28. A separately formed cup 30 made of the same material is inserted into the tube so that the open end of the cup is against the closed end 28 of the tube 26. The circular line of contact where the top edge of the cup touches the end of the tube is then heat-sealed, following which, the end of the tube is drawn out and broken away so as to leave a structure which is essentially a tube with a reentrant end. Down from the end a short distance a tube 32 is attached to the side wall of the tube 26 and serves as a handle to facilitate further manufacture of the crucible. It also has an additional function as will appear presently. The cup-shaped element 20 is then inserted from the open end of the tube as shown in Fig. 5. The portion of the tube below the cup 20 is then sealed off as shown in Fig. 9. In the alternative, the main portion of the tube 26 below the cup 20 can be constricted and drawn out as shown in Fig. 10, so as to form the stem 24 ending in a small diameter tube 34. In either case a connection is made to vacuum equipment by means of the tube 32 or 34 and the space within the envelope is pumped down to the desired pressure. During the pumping the cruicible is heated if this appears advisable to aid in the degassing. Thereafter the tube 32 or 34 is sealed off so as to leave the structure shown in Fig. 2 or Fig. 1 as the case may be.

Alternatively, as shown in Fig. 6 the end of a tube of the material of which the envelope is to be formed may be heated to form a globule 33 of thickened glass. The cup-shaped element 20 is then located inside the tube as shown in Fig. 7 and indentations 36 may be made in the side wall of the tube just below the cup to retain it in place. Thereafter the thick end 33 of the tube is heated to plastic condition and is pushed downwardly into the top recess of the cup 20 by a carbon rod 38 in the manner shown in Fig. 7. After removal of the carbon rod the main portion of the tube below the cup-shaped element 20 is closed and exhausted after the manner shown in either Fig. 9 or Fig. 10.

When a sample to be combusted is placed in the recess in the top of the crucible and the crucible is placed in a suitable high frequency induction field, the cup 20 is heated to a high temperature almost instantly and heat is radiated from the cup 20 through the envelope. The temperature of the sample quickly rises until the sample is ignited in the oxygen which will be present. This is because the cup-shaped element 20 is comparatively massive and readily absorbs energy from the high frequency field at a rate greatly in excess of its ability to dissipate heat excepting by radiation at temperatures above the ignition point of the material within the crucible. Because the envelope is inert and sealed, the burning sample is not contaminated by matter present in the heating element 20 and of course the envelope 22 also protects the cup-shaped element 20 against deterioration in the oxygen rich atmosphere.

From the above description of two similar embodiments of my invention, it will be appreciated that changes and variations in the structure may be made without departing from the spirit or scope of the invention and that therefore the invention is to be measured by the scope of the following claims. Having described my invention, what I claim as new and useful and desire to secure by Letters Patent of the United States is:

1. A crucible for combustion analysis comprising a thick walled cup formed of carbon, a thin walled envelope completely enclosing said cup, said envelope closely conforming to the internal configuration of said cup, said cup and said envelope being so sized with respect to each other that said cup is slightly loose in said envelope, the space within said envelope being at least partially evacuated to prevent said envelope from being subjected to substantial variations in internal gas pressure as the temperature of said cup fluctuates widely, said envelope being formed of artificial quartz.

2. A crucible for combustion analysis comprising a thick walled container having a cavity open at the top therein and formed of a material which is strongly heated by an induction field, a sealed thin walled envelope completely enclosing said container, said envelope having a depression therein extending into said cavity, said container and said envelope being so sized and shaped with respect to each other that said container is loosely embraced in said envelope, the space within said envelope being sufficiently evacuated to prevent said envelope from being subjected to substantial variations in internal gas pressure as the temperature of said container fluctuates widely, said envelope being formed of a material which is substantially inert chemically at high temperature and which has high resistance to thermal shock.

3. A crucible for combustion analysis comprising a container having a cavity open at the top therein and formed of a material which is strongly heated by an induction field, a sealed thin walled envelope completely enclosing said container, said envelope having a depression therein extending into said cavity, the space within said envelope being sufficiently evacuated to prevent said envelope from being subjected to substantial variations in internal gas pressure as the temperature of said container fluctuates widely, said envelope being formed of

a material which is substantially inert chemically at high temperature and which has high resistance to thermal shock, whereby said envelope protects a sample in said depression from contamination by the material of said container and protects said container from deterioration in use, and whereby said sample is heated through said envelope by radiation from said container when said container is subjected to an induction field.

4. An accessory for use in the radio frequency induction heating of an analytical sample to be combusted, which comprises a relatively massive member formed of a material strongly heated in an induction field, said member being completely but loosely sealed within an envelope, said envelope being formed of a material which is inert in oxygen at incandescent temperatures and which has high resistance to thermal shock and a high melting

point, said accessory being adapted to be placed in close juxtaposition to said sample.

5. An accessory for use in the radio frequency induction heating of an analytical sample to be combusted, comprising a relatively massive member formed of a material strongly heated in an induction field, said member being completely sealed within an envelope, said envelope being formed of a material having substantially the characteristics of fused quartz.

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