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CRUCIBLE FOR ANALYTIC PURPOSES HAVING A ROTARY  
COVER VALVING ARRANGEMENT

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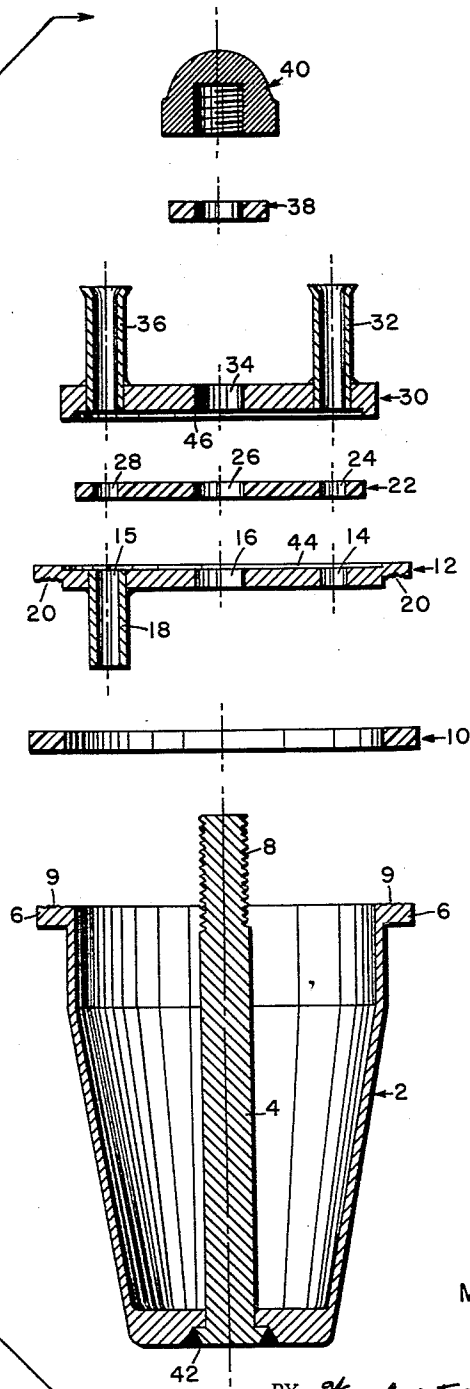


FIG. 1

ATTORNEYS

Nov. 22, 1960

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2,961,228

Filed Dec. 13, 1957

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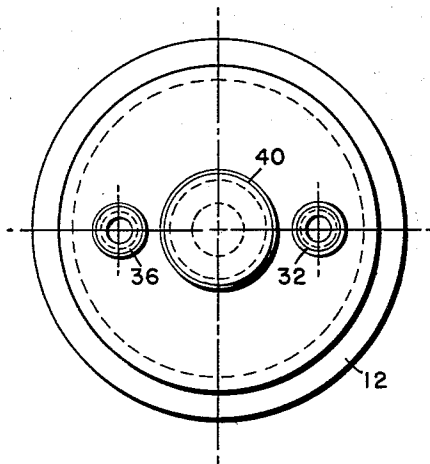


FIG. II

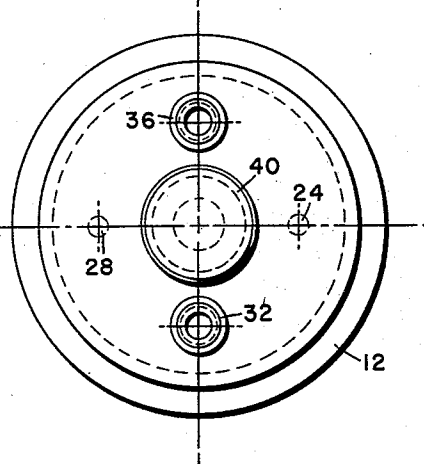


FIG. IV

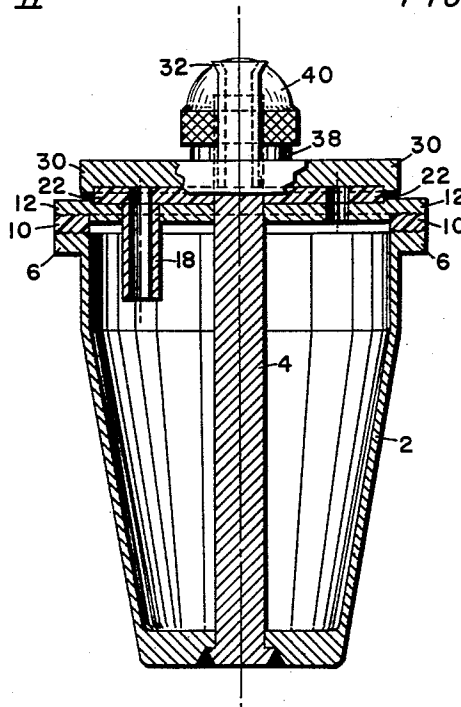


FIG. III

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## CRUCIBLE FOR ANALYTIC PURPOSES HAVING A ROTARY COVER VALVING ARRANGEMENT

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Filed Dec. 13, 1957, Ser. No. 702,563

1 Claim. (Cl. 263—48)

This invention relates to crucibles used in analytical chemistry, particularly to crucibles of the type used in determining the amount of one or more components released from a substance in the molten state, and more particularly to crucibles of the above type wherein may be placed a sample of material which is taken directly from its place of production to some other location and an analysis made thereon, without exposing the sample to the atmosphere, or otherwise altering its composition.

In almost all fields of manufacture, whether mechanical or chemical, the taking of samples of various materials in the steps of a manufacturing process is essential to maintain control over the quality of the product which is produced. Particularly in the manufacture of chemicals, the taking of samples is especially important to insure that the product will have the required uniformity of composition. By analyzing the samples taken at different stages in the manufacturing process, it is possible to determine whether or not any changes must be made so as to insure that the product will meet the required standards.

However, in order for a sample to be a true representation of the mass of material from which it was taken, it is necessary that it be maintained under such conditions as will prevent any change from occurring between the time the sample is taken, and the time when the analysis thereof is made. With many materials, this is extremely difficult, because impurities are readily picked up both from the container in which the sample is placed, as well as from the surrounding atmosphere, even after only a very short exposure thereto. Because of this situation, it is very difficult to maintain quality control on the manufacture of these materials, and hence the analysis of the final product often deviates greatly from the standard which has been set up.

Particularly susceptible to this difficulty are the hygroscopic anhydrous hydroxides of the alkali metals and some compounds of alkaline earth metals. These materials readily pick up moisture and carbon dioxide from the earth's atmosphere even after only a very short exposure thereto. As a result, analyses of samples taken of these materials, during the manufacturing process, often deviate greatly in moisture and carbonate content from the main body of the material and it is therefore extremely difficult to maintain the desired amount of moisture in the final product.

In the past, procedure for taking samples of material of this type has generally been to remove a small quantity of the material from the main body of the material at the point of production, then take the sample to the laboratory where it is weighed. After weighing, the sample is transferred to a fusion crucible and then heated to the fusion point to evolve any moisture, which is measured, as for example, in a gas absorption train. The crucibles generally used have been rather large and cumbersome, i.e., of such a size and weight as not to be readily weighable on any ordinary analytical balance.

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Moreover, these crucibles do not have any suitable valving mechanisms to prevent air from contacting the sample before it has been analyzed. Thus, because of the tendency of materials of this type to take up moisture and carbon dioxide upon exposure to the air, samples taken and analyzed by this method do not give true determinations of the amount of moisture in the main body of the material, since at each exposure to the air, however slight, the moisture content of the sample is changed.

It is therefore an object of this invention to provide an analytic crucible of the fusion type, which crucible may be used to take samples directly from the main body of the material.

A further object of this invention is to provide a fusion crucible of the above type, having a simple and highly effective valving arrangement, whereby samples taken directly from the main body of the material may be analyzed without further exposure to the atmosphere.

These and other objects will become apparent to those skilled in the art from the description and drawings of the invention which follows.

In the accompanying drawings which are attached hereto and made a part hereof:

Fig. I is an exploded view, in cross section, of the crucible of the present invention, wherein the parts thereof are shown in the order in which they are assembled;

Fig. II is a plan view of the assembled crucible of Fig. I showing the valves in open position;

Fig. III is an elevation in cross section of an assembled crucible of the present invention showing the valves in closed position and having portions thereof broken away for clarity;

Fig. IV is a plan view of Fig. III.

Pursuant to the above objects, the crucible of the present invention, which may be used to take samples of a material from the main body of the material, includes a cup-shaped base member and cover means therefor, said base member having means for attaching the cover means thereto, said cover means including upper and lower members having a sealing means therebetween, said members having perforate and imperforate portions forming a valve means, said members being rotatable so as to bring said perforate portions into and out of register with each other, thereby to permit or prevent ingress and egress of gases into said base member, and means for securing the said cover means to the said base member in gas tight relationship.

Referring now to the drawings and in particular Fig. I, cup-shaped member 2 is the main body of the crucible of the present invention, and has a shaft 4 therein, having threads 8 at one end thereof. Shaft 4 extends above the rim of cup-shaped member 2 and is secured to member 2 in any suitable manner, as for example, by being welded to the bottom thereof, as shown at 42. The outer rim of member 2 has a lip 6 formed thereon, with grooves as shown at 9, which lip overhangs the main portion of member 2.

Gasket 10 in the form of an annulus rests upon lip 6 on grooves 9 of cup-shaped member 2, which with gasket 22 forms the seal between member 2 and the cover means 12 and 30. Gasket 10 may be of any suitable material which form a gas-tight seal between the main body of the crucible and the cover means. Examples of such materials are rubber, nylon, and polytetrafluoroethylene, commonly known as "Teflon" (Reg. T.M.). However, because of its resistance to high temperatures and to the corrosive action of both acids and bases, as well as its inertness, the preferred material for gasket 10 is polytetrafluoroethylene.

Cover member 12 has formed therein, shoulder 20, which rests on gasket 10, and central hole 16, through

which shaft 4 extends through recess 44. Cover member 12 is formed with holes 14, 15 and 16, and tube 18, all of which extend therethrough. Although holes 14 and 15 (tube 18) may be placed anywhere on cover 12, as will be explained hereinafter, for ease in utilizing the apparatus, it is preferred that they are positioned in opposition to each other at 180°.

A second sealing gasket 22 is placed in recess 44 of cover member 12. Gasket 22, like gasket 10, is preferably made of polytetrafluoroethylene, although any other suitable material may be used. Holes 24, 26 and 28 are formed therein, which holes correspond to and are in register with holes 14, 15 and 16 and tube 18 of cover 12. Gasket 22 serves as a gas-tight seal between cover member 12 and cover member 30, which latter member is placed on top of gasket 22 so that gasket 22 fits into recess 46 formed therein. Cover member 30 has a central hole 34 formed therein, which hole registers with holes 16 and 26 of gaskets 10 and 22 and cover member 12, and through which extends shaft 4 of member 2. Cover member 30 is formed with tubes 32 and 36, which tubes extend therethrough and correspond to and are in register with holes 24 and 28, respectively of gasket 22, as well as holes 14 and 15 of member 12. A suitable securing means, such as cap-nut 40, having threads on the interior thereof, corresponding to threads 8, is placed on the threaded end of shaft 4.

When cap-nut 40 is tightened on shaft 4, cover members 30 and 12 with their respective gaskets 22 and 10 are held in gas-tight sealing relationship with the main body member 2 of the crucible. In order to insure a gas-tight seal between cap-nut 40 and cover member 30, a gasket 38, which gasket is preferably made of polytetrafluoroethylene, is placed therebetween.

Referring now to Figs. III and IV, by rotating cover member 30 so that tubes 32 and 36 are entirely out of register with holes 14, 15 and tube 18, respectively, of cover member 12, prior to tightening cap-nut 40 on shaft 4, a gas-tight container is formed, in which may be placed a sample, which will not be exposed to the atmosphere either during the time it is held in the crucible or during the time of analysis. When it is desired to conduct an analysis of a sample, a cap-nut 40 is loosened and cover member 30 is rotated so that tubes 32 and 36 are in register with holes 24, 28, 14, 15 and tube 18, of cover member 12, as shown in Fig. II, thus providing a means by which a gas or other material may be directed into the crucible and moisture or decomposition products of the sample may be removed therefrom.

With the exception of the gaskets 10, 22 and 38, all parts of the crucible of the present invention are preferably all of a corrosion resistant metal, as for example, nickel, platinum, titanium, stainless steel or the like, depending upon the material to be analyzed. Moreover, it is desirable that the crucible of the present invention be of such a size and weight as to be conveniently weighed on a standard analytical balance. Presently, it is preferred that the crucible have an overall weight when assembled of not more than about 180 grams, since on an ordinary analytical balance this assures more than adequate accuracy when samples weighing as much as 20 grams are involved, i.e., as much as one part in 20,000, where the analytical balance is capable of accurately weighing 200 grams to the nearest milligram.

In order that it may be more clearly understood how the subject crucible functions, a typical analysis in which the crucible of the present invention is used will be hereinafter described.

It has been found that a crucible designed in accordance with the present invention is particularly suited for making moisture determinations of anhydrous alkali metal and alkaline earth metal hydroxides. As set forth previously, these compounds are extremely difficult to analyze for moisture content because of their ready affinity for moisture and carbon dioxide of earth's atmos-

phere. Moisture and carbon dioxide in the atmosphere are taken up by these compounds even upon a very slight exposure thereto, and hence it is imperative that these compounds be kept from contact with the atmosphere at all times during which they are taken from the main body of material to be analyzed through the time the analysis is completed.

By alkali metal hydroxide is meant the hydroxides of lithium, sodium, potassium, cesium, rubidium and francium. By alkaline earth metals is meant the hydroxides of calcium, magnesium, strontium and barium.

A convenient method for determining the moisture content of materials of this type is in a gas absorption train. A train for this type of analysis includes a source of a dry inert gas, such as dry nitrogen and a dehydrating or drying tube such as U-tube containing a moisture absorbing material such as magnesium chlorate. The dry inert gas is passed over the sample, thus removing the moisture therefrom, and is then passed through the dehydrating agent in the drying tube, which dehydrating agent removes the moisture from the inert gas. The weight of the drying tube and dehydrating agent with the moisture which is removed from the inert gas, less the original weight of the drying tube and dehydrating agent, gives the amount of moisture which was contained in the sample.

In using a crucible designed in accordance with the present invention for the determination of moisture in such a method of analysis, the crucible is taken to the place where, for example, commercially anhydrous hydroxide is being produced. A sample of the commercially anhydrous material is taken directly from the main body of the material to be analyzed, placed in the crucible, and the cover means of the crucible is assembled on the base member 2 in the manner described above, so that holes 24 and 28 are out of register with tubes 32 and 36, and tube 18 of cover member 12.

Cap-nut 40 is then tightened so that the sample is maintained in the crucible without any further exposure to the atmosphere. The crucible and sample are then weighed, thus determining the combined weight of the crucible and the sample. When the initial weight of the crucible and sample is obtained, the crucible may, for example, be heated until the sample becomes molten, cap-nut 40 having been loosened sufficiently to permit cover 30 to be rotated so that tubes 32 and 36 are in register with holes 14 and tube 18 (hole 15) of cover member 12. Tube 36 of cover member 30 is connected to a source of a dry inert gas, such as dry air or dry nitrogen, and tube 32 of cover 30 is connected to a drying tube containing a moisture absorbing material, such as magnesium chlorate, which drying tube and moisture absorbing material has previously been weighed. Cap-nut 40 is then tightened so as to maintain a gas-tight seal between the cover means and the main body 2 of the crucible. While the sample is maintained in the molten state, dry inert gas is passed through tubes 36 and 18 into the crucible whereupon the gas purges the moisture from the sample and passes out of the crucible through holes 14, 24 and tube 32, into the absorption tube (not shown).

The moisture absorbing material in the absorption tube removes the moisture from the inert gas, which gas may then be returned to the gas source where it may be passed again through the crucible. Following the complete purging of all the moisture from the sample, which complete purging may require about one hour, the absorption tube and dehydrating agent are weighed and the increase in weight over the initial weight is the amount of moisture within the sample.

If desired, hole 14 in cover member 12 may be baffled in any suitable manner, so as to prevent any carry over of the molten sample into the analysis train. The construction of such a baffle means is believed to be obvious to one skilled in the art and hence no further description

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thereof is deemed necessary. Generally, when the sample in the crucible is not heated too vigorously, entrainment of the sample with the inert gas is not experienced and hence such a baffle is not usually necessary.

It has been found, however, that by placing the inlet and outlet holes in cover members 12 and 30 on opposite sides of the center hole, a more efficient gas removal is effected, in that the gas is swept across the entire surface of the molten sample rather than just a portion thereof, as would be the case if the holes were not placed on opposite sides of the cover member, thereby effecting a more rapid removal of the moisture.

By use of the crucible of the present invention, it is thus possible to remove a sample of material from the main body of material to be analyzed and make an analysis of the sample without the necessity for further exposing the sample to the atmosphere whereby it may pick up moisture or other impurities. By this means, it is thus possible to obtain a sample for analysis which sample will have the same composition as the main body of material from which it has been removed, thereby giving a true picture of the conditions existing within the main body of the material.

Although the invention has been described with specific reference to its use in the analysis of various alkali metal and alkaline earth metal hydroxides for the moisture content thereof, it will be apparent to those skilled in the art that the subject crucible will find application in other and varied analyses. Moreover, it is to be understood that although the invention has been described with specific reference to particular embodiments thereof, it is not to be so limited since changes and alterations therein may be made which are within the full intended scope of this invention as defined by the appended claim.

What is claimed is:

An apparatus comprising a cup-shaped base member, said base member having a central shaft secured therein,

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said shaft extending above the outer rim of said base member, cover means for said base member, said cover means including upper and lower members, said lower member having a central aperture formed therein through which aperture extends the central shaft of said base member, said upper member also having a central aperture formed therein through which aperture extends the shaft of said base member, sealing means between said upper and lower cover members and between said base member and said lower cover member, said upper and lower cover members having perforate and imperforate portions forming valve means therein, said cover members being rotatable with respect to each other so as to bring said perforate portions into and out of register with each other, thereby to permit or prevent ingress and egress of gases to said base member, and means for securing said cover means to said base member in gas-tight relationship.

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