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MAGNETICALLY CONTROLLED SAMPLE ADDITION MEANS

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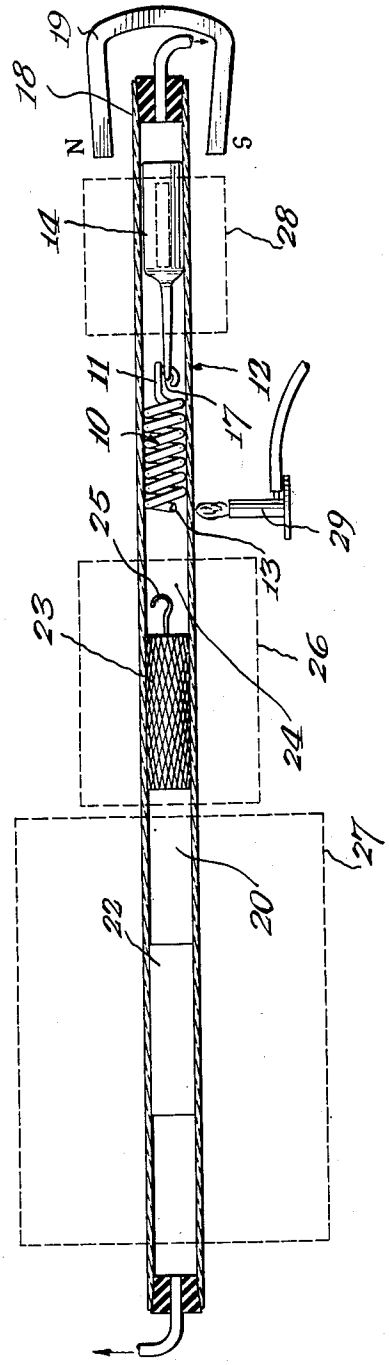


Fig. 1.

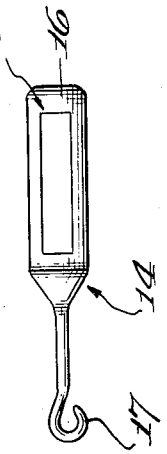


Fig. 3.

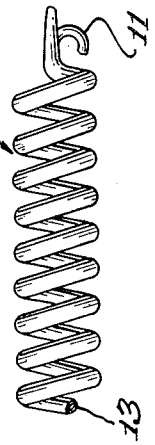


Fig. 2.

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MAGNETICALLY CONTROLLED SAMPLE ADDITION MEANS

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This invention relates to an improvement in a combustion boat and, more particularly, relates to a means for controllably introducing increments of a sample in a pyrolysis.

The Schütze method for the direct determination of oxygen in organic substances consists of pyrolysis of the sample in a stream of inert gas and conversion of the oxygen in the pyrolysis products to carbon monoxide by passage over carbon at 1120° C. or 50% platinized carbon at 900° C. The carbon monoxide may be determined in several ways and Schütze oxidized it with iodine pentoxide and weighed the resulting carbon dioxide. An article describing the general procedure for the Schütze method may be found in Analytical Chemistry, vol. 26, pages 600–602 (March, 1954).

In the determination of trace amounts of total oxygen in naphtha and other light hydrocarbons by such a combustion method, a large sample must be pyrolyzed so that a measurable amount of oxygen equivalent in the form of carbon dioxide is obtained. To prevent dangerous explosions, the pyrolysis rate must be carefully regulated. Also, the volatile sample must be maintained in a container or boat to minimize evaporation. Further, the sample container must be completely filled with the sample and have no enclosed air bubble and after the sample is placed inside the combustion tube atmospheric oxygen must be purged from the system. Also, the sample container must remain inert at 850° C. and must not react with any organic material or form a unstable metal oxide.

It is, therefore, a primary object of my invention to provide an apparatus which holds a large sample that can be controllably discharged. A further object of the invention is to provide a system which may be manipulated to deliver increments of the sample to the pyrolysis zone. Another object of the invention is to provide a combustion boat which will minimize evaporation of a volatile sample and avoid the explosion hazards heretofore encountered. These and other objects of the invention will become apparent as my description thereof proceeds.

Briefly, I provide an apparatus for direct oxygen analysis by the Schütze combustion method without an explosion hazard by releasing a relatively large sample within the pyrolysis tube in small increments for evaporation into the pyrolysis section by the heat from a portable furnace or Bunsen burner flame. The apparatus for accomplishing this comprises a spiral tubular quartz boat which can be filled by suction, a bar magnet manipulator within the combustion apparatus which is removably attached to the sample boat, and magnetic means rotatable and axially movable exterior of the pyrolysis tube for rotating the manipulator within the tube so that the boat is rotated and an increment of sample can be emptied from the boat into the tube whenever desired.

Further details of the invention will be described by reference to a preferred embodiment thereof illustrated in the drawings wherein:

Figure 1 is a schematic illustration of a combustion tube assembly embodying my invention; and

Figures 2 and 3 show details of the spiral boat and the magnetic manipulator, respectively.

Referring to the drawing, the combustion boat **10** is a spiral of quartz tubing having a small quartz hook **11** at one end. It can be made from 5 to 20 turns of 3/16 to 1/4 inch tubing, the outside diameter of the spiral being from 3/8 to 3/4 inch. The exact size of boat **10** depends upon the amount of sample required and the inside diameter of the combustion tube **12**. Thus, the boat **10** may contain from 0.5 to about 5 g. of a liquid such as naphtha, the boat **10** being filled by connecting it to a small syringe (not shown) and drawing up the sample through the open end **13**.

The manipulator **14** contains a magnetic or paramagnetic bar **15** which is completely enclosed in quartz casing **16** with a hook **17** at one end which can engage the hook **11** on the spiral boat **10**. The bar **15** should be about 1/4 inch in diameter and about 1 inch long. During the time that the boat **10** is being filled and weighed, the manipulator **14** is disengaged.

The combustion boat **10** and the manipulator **14** can be controlled as a unit from the outside of the pyrolysis tube **18** by means of a movable bar magnet **19** which may be a horse shoe magnet. Rotating the external magnet **19** about the pyrolysis tube **18** in the region of the manipulator **14** rotates it and the spiral boat **10** in a controllable manner. By this means, the boat **10** can be rotated and a small amount of sample deposited from opening **13** with each turn. The boat **10** is then pulled back within the tube **18** about 5 inches and the deposited sample is driven into the pyrolysis section **20** with a Bunsen burner or other movable heat source **29**. The process is repeated until all of the sample has been discharged from the spiral boat **10** and at this point the entire unit is heated to about 850° C. to insure complete removal of the sample.

The vaporized sample passes through the combustion tube **18** over the platinized carbon **22** in pyrolysis section **20** and to minimize the deposition of pyrolytic carbon on the platinized carbon section **22** a roll **23** of platinum gauze may be interposed between the sample introduction section **24** and the pyrolysis section **20**.

The roll **23** may be about 1 inch long and formed from 52 mesh, 0.004 inch wire platinum gauze. A platinum wire hook **25** is provided at one end so that the roll **23** can be removed from the tube **18** to burn off the pyrolytic carbon. The roll **23** is kept at 850° C. during the course of the analysis by means of the auxiliary movable heater **26**. The platinized carbon section **22** is maintained at about 900° C. by means of the main furnace **27**.

The method and apparatus described can be used for other combustion analysis such as carbon and hydrogen, fluorine, chlorine and Ter Meulen nitrogen. When oxygen gas is used to burn the sample, the section of the tube **12** enclosing the spiral boat **10** is provided with a cooling jacket **28** which may be Dry Ice pack.

The boat **10** is brought forward by manipulator **14** and external magnet **19** to deposit a small amount of sample and is then withdrawn into the Dry Ice section **28** while the deposited portion of the sample is driven into the pyrolysis section **20** by means of heater **29**. Such a procedure as just described is used in the carbon and hydrogen, chlorine and fluorine analysis whereas in the determination of nitrogen by the Ter Meulen method the procedure is the same as that used in the Schütze oxygen analysis. It may be noted that the auxiliary platinum gauze roll **23** is not required when oxygen is used to burn the sample.

The main advantage of my spiral combustion boat **10** and manipulator **14** is that a large volatile sample can be burned a little at a time, thereby avoiding the possibility of dangerous explosions while permitting the continual

discharge of a large total sample over a period of time so that the pyrolyzed sample produces a measurable amount of oxygen equivalent in the form of carbon dioxide.

In making a direct determination of oxygen in naphtha, the sample boat containing typically about 2 gms. of sample is weighed in the spiral combustion boat and then placed in the pyrolysis tube. After the pyrolysis tube is purged of air, the sample is driven into the platinized carbon section (held at 900° C.) by employing the external magnet as described above. All the oxygen in the sample is converted to carbon monoxide which is then passed through iodine pentoxide at 120° C. and is thereby oxidized to carbon dioxide. Forty-five minutes after the sample has been driven into the platinized carbon section, the previously weighed micro absorption tube is weighed again. The amount of oxygen in the sample is calculated from the weight of carbon dioxide absorbed on the absorption tube.

Although I have described my invention with reference to a preferred embodiment of the apparatus illustrated in the drawing, it should be understood that this is by way of illustration only and that the invention is not necessarily limited thereto. Accordingly, it is contemplated that modifications can be made in the apparatus and in the mode of operation thereof without departing from the spirit and scope of the described invention.

What I claim is:

1. A combustion boat comprising a closely wound spiral of quartz tubing, said spiral having between about 5 and 20 turns, a diameter of between about 0.375 and 0.75 inch, said tubing being open at both ends, a hook means at one end of said spiral for manipulating the boat to discharge fluids therefrom, and detachable coupling means linked to the said hook means, said coupling means including paramagnetic bar means and hook means on said coupling means engaging the hook means on said spiral.

2. A magnetically controlled sample addition means including a spiral boat and a manipulator, said spiral boat comprising a length of quartz tubing, and a hook means at one end of said boat, and said manipulator comprising paramagnetic bar means within a quartz envelope, and a hook means on said manipulator adapted to engage the hook means on said spiral boat, and magnetic means for remotely turning said manipulator so as to rotate said spiral boat and thereby discharge fluids therefrom.

3. A sample boat means adapted for use in pyrolysis for the determination of oxygen in organic substances which comprises in combination a spiral boat consisting essentially of a closely wound quartz tubing having from 5 to 20 turns, a manipulator means for said spiral boat including a paramagnetic bar encased within a quartz envelope, detachable coupling means for securing said manipulator to said spiral boat, and magnet means for positioning and for rotating said manipulator whereby the discharge of fluids from said spiral boat is controlled.

4. A manipulator for controlling a spiral boat which comprises a bar magnet, a quartz envelope enclosing said bar magnet, and means for engaging coupling means on said spiral boat, said bar magnet being adapted to be rotated and translated within a pyrolysis tube by means of an external magnet.

5. The apparatus of claim 4 which includes magnet means for remotely controlling said manipulator so as to translate and rotate the spiral boat and thereby controllably discharge fluid material therefrom.

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