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[54] METHOD AND APPARATUS FOR DETERMINING THE CONTENTS OF CONTAINED GAS SAMPLES

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[58] Field of Search: 250/41.9 S, 41.9 G; 73/23.1, 73/421.5

[56] References Cited

UNITED STATES PATENTS

3,458,699 7/1969 Padra250/41.9 S
2,486,199 10/1949 Nier250/41.9 G

2,736,810 2/1956 Clark250/41.9 S
3,534,945 10/1970 Sweeny250/41.9 S
3,520,176 7/1970 Becker250/41.9 S

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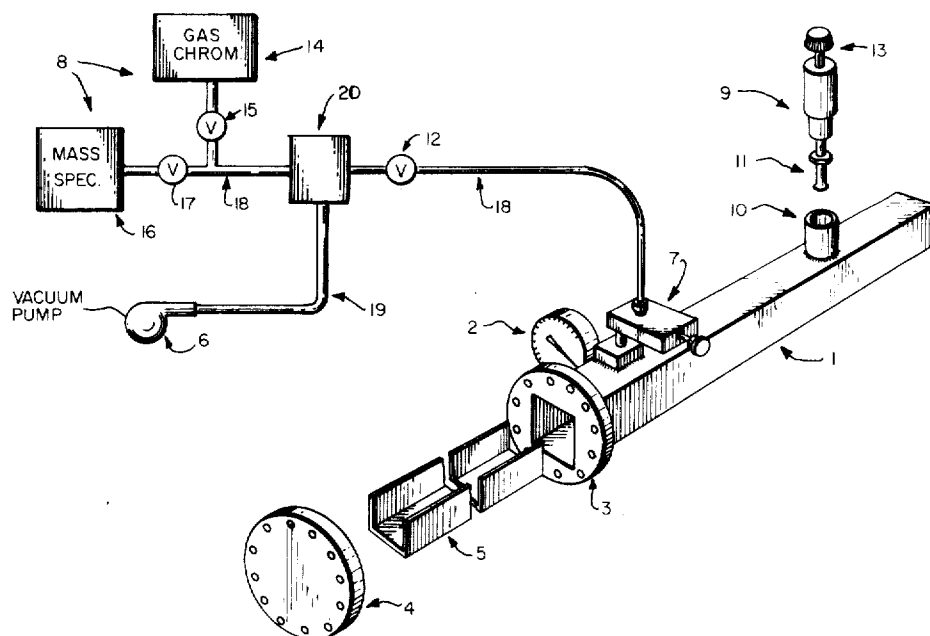
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[57] ABSTRACT

An apparatus for analyzing gas samples in containers comprising a vacuum chamber, a sealable opening in said vacuum chamber for inserting a sample holder, means to open said sample and release the gas in the container, pressure sensing means, and means for analyzing the gas released by opening the sample. The apparatus is operated by placing a sample containing the gas to be analyzed on the sample holder, sealing and evacuating the chamber, taking a background gas analysis, opening the sample container to allow the gas contained therein to escape into the evacuated chamber and withdrawing the released gas to the analysis apparatus.

1 Claim, 2 Drawing Figures



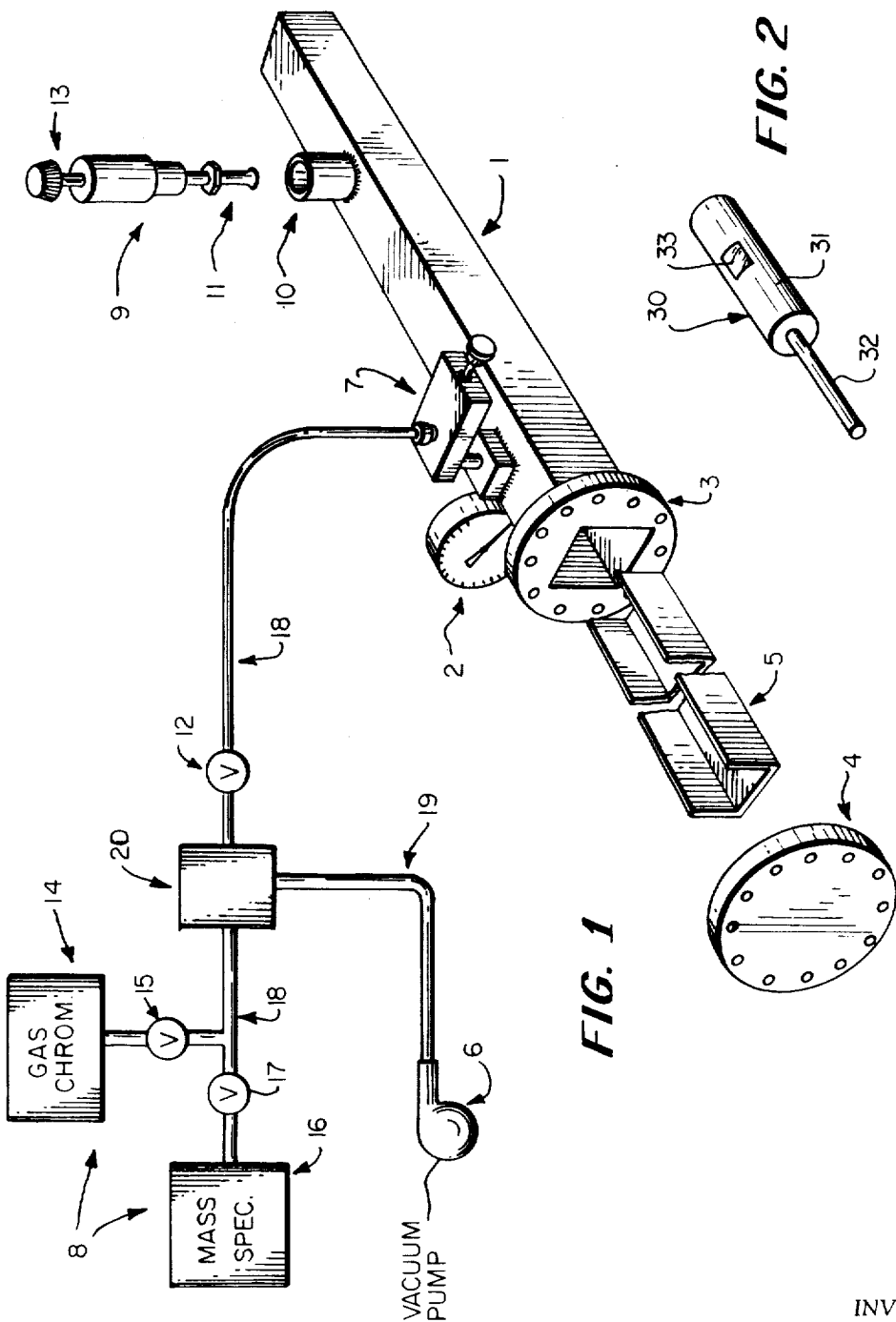


FIG. 1

FIG. 2

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METHOD AND APPARATUS FOR DETERMINING THE CONTENTS OF CONTAINED GAS SAMPLES

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention is directed to a method and apparatus for analyzing contained gas samples. More particularly, the present invention is directed to a method and apparatus for supplying the contents of a gas containing piece to gas analysis apparatus of both quantitative and qualitative types. Specifically, this invention is directed to gas analysis apparatus wherein the gas containing piece is not destroyed during analysis but rather may be reconstructed to function as originally intended.

2. Description of the Prior Art

It has long been known to use instruments such as the gas chromatograph or the mass spectrometer in analytical work in order to determine the contents of gaseous samples. Such samples are often contained in sealed units (pieces) and are placed in chambers capable of vacuum evacuation prior to opening for testing. These chambers vary in size and often contain internal mechanism, such as cutting and backing tools or actual analysis apparatus, to perform the various functions required under vacuum conditions. Also, many chambers contain exit valves or other apparatus by which samples of the gas contained in the vacuum chamber may be removed for external analysis. In view of these factors, the existing vacuum chambers are so shaped that a small sample of gas released into the chamber will be diluted and the ratio of contaminants will be high, rendering extremely accurate analysis quite difficult.

SUMMARY OF THE INVENTION

The present invention, as shown by the drawings, is a contained gas analyzing apparatus and a method for using such apparatus. The apparatus includes a vacuum chamber which has an end which is adapted for opening and placing a sample of contained gas on a tray in the chamber, means for evacuating the chamber, means to open the gas container and means for analysis. The sample opening means is usually a small saw-like apparatus such as a jeweler's saw. There is also a valved means to evacuate the chamber by, e.g., a vacuum pump, and thus allow for the removal of gas during evacuation as well as removal of the gas required for background readings and readings of the contents of the gas sample container. The valved means is arranged so as to selectively communicate with the gas analysis apparatus which may include, inter alia, a gas chromatograph and/or a mass spectrometer.

The gas sample is analyzed by cleaning the exterior of the container thereof, e.g., by swabbing with ethanol or acetone, placing it on the sample tray and putting the tray in the vacuum chamber. The chamber is then sealed and evacuated. Upon reaching the desired pressure, a background reading is taken with the analysis apparatus. The sample container is then opened and the contents allowed to escape reach an equilibrium pressure condition in the chamber. A sample of this gas is then analyzed to obtain a reading both quantitatively and qualitatively as to the composition of the gas in the container. The system is then repressurized by, for example, a release valve on the evacuating means, and the sample container removed for reuse if desired.

Thus, it is a purpose of the present invention to provide a dependable and convenient way to precisely measure and analyze the contents of a gas sample without permanently damaging the body of the container.

It is a further object of the present invention to produce an apparatus for analyzing contained gases which is both economical to utilize and capable of providing analysis, both quantitative and qualitative, with minimal space requirements.

Further objects will become obvious to those skilled in the art as the drawings and process are further described.

DESCRIPTION OF THE DRAWINGS

FIG. 1 shows a partially exploded view of the gas analysis apparatus of the present invention; and

FIG. 2 shows a proportion counter, the contents of which are to be analyzed by the apparatus of FIG. 1.

DESCRIPTION OF THE PREFERRED EMBODIMENT

In the apparatus of the present invention, the vacuum chamber, designated generally at 1, contains pressure sensing means 2, a flanged end joint 3, a cover plate 4, a sample tray 5 and a cutter inlet 10. The sample tray 5 is usually of a channel or pan-like design although it may be of any convenient shape. Cover plate 4 is adapted to seal the chamber 1 by attachment to flange 3. The chamber 1 is interconnected to vacuum pump 6 by line 18 provided with valves 7 and 12. The sample tray 5 is slidably supported in the vacuum chamber 1. A cutter, generally designated 9, is provided with a cutter edge 11 is sealed onto vacuum chamber 1 through inlet 10. The cutter may be driven either manually or by an external motor, as shown. The sample tray, while normally moved merely by lifting the apparatus at its flanged end, and allowing the tray to slide down the chamber, may be moved by means of any desired apparatus. The gas analysis apparatus indicated generally at 8 communicates with chamber 1 via valves 7 and 12, 15 and 17, line 18 and control unit 20.

In the apparatus of the present invention, the sealing means 3 and 4 has been shown as a cover plate and flanged end. However, any other means which seals the chamber may be used. Further, the pressure sensing means 2 is one conventional in the art and may be of any convenient design so long as it meets the obvious requirements of reading the reduced pressures noted below accurately.

The cutting edge 11 is shown as a jeweler's saw head and is driven from outside the chamber by tuning knob 13. The cutter inlet 10 is provided with a vacuum feed through for cutter 9; a threaded hub with vacuum seal seat integrally formed in the inlet 10.

Valve 7 is a VeeCo. valve, which is well suited for the purposes of the present invention, but, as is obvious to those skilled in the art, other valve designs could be equally well adapted to the present apparatus.

The gas analysis apparatus 8 is generally known in the art. For the preferred form of the present invention, a gas chromatograph 14 is valved into line 18 by valve 15 while the mass spectrometer 16 is connected via valve 17 to main line 18. The analysis apparatus may be varied in accordance with the analysis desired as well as

equipment availability. Further, vacuum pump 6, connected to line 18 by line 19 and control unit 20 may be of any desired type. The pump must, however, be able to evacuate chamber 1 to the pressure desired for the process of the present invention. Control unit 20 may be simple, e.g., a three-way valve, or a more complex unit, e.g. where a plurality of analysis apparatus is used, and continual vacuum pumping is desired.

In the process of the present invention, a proportional counter used (for mapping X-ray fields in interstellar space) is checked to determine the content of the gases contained therein. As shown in FIG. 2, the counter 30 comprises cylindrical metal shell 31, filled with a mixture of two different gases. A cathode is formed by the shell and an anode is formed by a wire fitted along the axis of rotation of the cylinder. The X-rays enter the counter through a thin window 33 which may be beryllium of about 2 to 5 mils thickness. A fill tube 32, located at one end of the cylinder, is provided for gas charging the counter.

The counter is first measured, and mounted on the sample tray so that the cutter will strike the fill tube 32 at a point where the tube may be cut open later to refill the counter and thus reuse the counter. The counter is then cleaned, if desired, to free it of at least some of the contamination and placed in the test chamber. The chamber is sealed by attaching end piece 4 to flange 3, evacuated, and a background reading taken on the mass spectrometer which, along with the gas chromatograph, have been previously set for the gases to be encountered. The background reading is taken through line 18 in valves 7, 12, 15 and 17 while the control unit 20 is operated to allow communication with the analysis apparatus as desired. The tray 5, with the sample is then moved to contact the fill tube thereon with saw 11 by, for example, tilting the apparatus to slide the tray down the chamber. Other means of moving the tray, if necessary, may be utilized as well. Saw 11 is then operated to cut the sample tube by either manually rotating knob 13 or by modifying the apparatus shown to allow for mechanical or electrical movement of the saw. As noted above, other means of cutting the tube may be utilized, so long as the integrity of the unit is retained.

The gas analysis apparatus is then reactivated by the same procedure as that used to obtain the background reading. The results of these analyses can then be used to determine accurately the gaseous contents of the sample.

Upon completion of the second analysis, the system is repressurized by, for example, a release valve in either vacuum pump 6 or control unit 20, and the sample removed by opening the chamber at cover plate 4 and flange 3 and removing the tray. The sample container, upon separation from tray 5, may be refurbished as desired.

The pressure to which the apparatus is normally evacuated should be no greater than 10^{-4} Torr and is preferably within the range of from 10^{-4} to 10^{-6} Torr,

while the most preferable pressure is 10^{-6} Torr.

While the specific size, shape and mechanisms described above may be modified in order to operate on various size samples, i.e., different sized proportional counters, the concepts of having a means for background measuring in an evacuated chamber, means for opening the proportional counter, and evacuation means in a vacuum chamber, all in combination in order to effect the present invention, should remain. Further, the present technique could also be applied to other devices, such as scintillation tubes, small ionization chambers, Geiger tubes, etc., where fill tubes are utilized, in order to non-destructively test the units.

EXAMPLE

Two proportional counters of type described were tested. The first was of stainless steel with a circular beryllium window, and the second was of aluminum with a square beryllium window. The steel counter contained Xenon and carbon dioxide and the aluminum counter contained Xenon and nitrogen. The aluminum counter was functional, while the steel counter failed to function well electrically.

Both units were tested by placing a cleaned unit in a vacuum chamber, after alignment and fixing on the sample tray, evacuating the chamber to 10^{-6} Torr, taking a background reading, cutting the fill tube open, and taking readings on the gas chromatograph and mass spectrometer. The aluminum counter was found to contain 75 percent Xenon and 25 percent nitrogen, while the steel counter contained 91.7 percent Xenon, 8.3 percent carbon dioxide and 1.5 parts per million organic contaminants. The small amount of organic contaminants made it impossible to pinpoint their origin. Both counters were free of air and moisture contamination.

What is claimed is:

1. A method for non-destructively analyzing gas samples contained in a device having a fill tube comprising the steps of:

- a. measuring the device to determine the point where the fill tube may be cut open to refill the device;
- b. mounting the device on a sample tray to facilitate contact of the fill tube with a cutting instrument at the proper location on the tube;
- c. placing the sample tray and device in a vacuum chamber;
- d. evacuating said chamber to a pressure in the range of 10^{-4} to 10^{-6} Torr;
- e. obtaining a background gas analysis reading of the vacuum chamber;
- f. moving said device to a position to effectuate opening at the fill tube;
- g. releasing the contained sample by opening the container fill tube; and
- h. analyzing the gas contained in said sample by means of gas analysis.

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