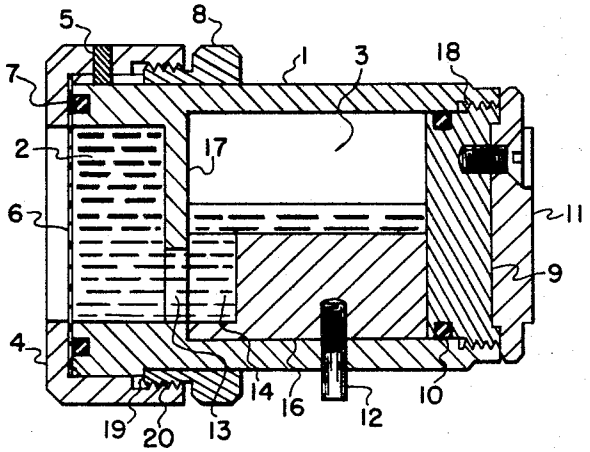
Nov. 16, 1965 J. R. BENS 3,218,459

FLUID SAMPLE HOLDERS FOR X-RAY SPECTROMETERS UNDER VACUUM

Filed Oct. 9, 1963



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United States Patent Office 3,218,459 Patented Nov. 16, 1965

3,218,459

FLUID SAMPLE HOLDERS FOR X-RAY SPECTROMETERS UNDER VACUUM

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Filed Oct. 9, 1963, Ser. No. 315,031
Claims priority, application France, Oct. 25, 1962, 913,395
5 Claims. (Cl. 250—51.5)

The present invention concerns improvements in sample holders that are used for the analysis of fluids in a vacuum X-ray spectrometer.

When low atomic number elements are analyzed in an X-ray spectrometer it is necessary for the X-ray path to be in a vacuum in order to avoid absorption of the low energy X-rays by air or other gas. Therefore, in a vacuum spectrometer, the X-ray tube, the sample, the analyzer crystal and the detector are all in a vacuum ambient.

Difficulties are encountered when fluid samples are undergoing analysis in a vacuum spectrometer. If the fluid is exposed, it is not unusual for it to degas or boil when the vacuum is created. Therefore, the fluid must be contained in a receptacle which isolates it from the vacuum. Such receptacle has a thin radiation permeable window that is traversed by radiation such as X-rays which excite the sample and which permits exit of the excited radiation that is characteristic of the elements in the sample. To minimize absorption, the window must be a thin film and it follows that it will have low rigidity. A commonly used film material for such a window is therphane which in one form is known by the trademark Mylar.

Two approaches to overcoming the above noted difficulties are as follows:

(1) The first contemplates the use of a leakproof receptacle that is completely filled with the fluid sample to be analyzed. The receptacle, or sample holder, may then be introduced into the enclosure which is to be evacuated. There is a serious drawback to this approach, however, because the interior of the sample holder is under its original pressure, usually atmospheric, while the outside is exposed to a vacuum ambient. The pressure differential causes the window to distend and this creates a void in the fluid which, in turn, allows gas bubbles to be liberated from the fluid. Liberation of gas bubbles is caused by the known phenomena that gases are soluble in a fluid in proportion to the temperature and pressure of the fluid. The gas bubbles migrate to the window with a random distribution in which case they distort the intensity measurements of the characteristic radiation which indicate constitution of the sample. It is easy to see that reproducible results would be difficult to obtain with consecutive samples of the same composition.

(2) The second approach is to use a sample holder whose interior remains in communication with the atmosphere in which case the fluid is not degassed because of the absence of the pressure differential, but the window must withstand the entire pressure of the atmosphere. Under this stress the window undergoes a plastic deformation, a progressive flow, which displaces the exposed and active volume of the fluid sample with respect to the geometry of the spectrometer. This results in an error in the quantitative measurements.

The fluid sample holder constituting the present invention permits making accurate quantitative measurements without the above mentioned inconveniences. The new sample holder was devised primarily for the purpose of analyzing fluids in a vacuum X-ray spectrometer. In general terms, the sample holder is characterized by it having two chambers, one of which has a radiation permable window in it. The first chamber communicates with the second chamber which contains some of the fluid being analyzed and a space for an expansible gas, air for example. The holder is constructed fluid-tight and this is facilitated by the use of toric joints, otherwise known as O-rings, for example. The volume of liquid used in the sample holder is such that the pressure caused by the volume of entrapped gas maintains a pressure on the liquid which exceeds the pressure at which it degases. Thus, after distension or deformation of the window following evacuation of the space surrounding the holder, the pressure on the fluid is maintained substantially constant by expansion of the gas rather than the pressure dropping to zero or a negative value in which case degassing would occur. Moreover, full atmospheric pressure is not imposed on the window.

Achievement of the foregoing and other objects of the invention will be evident as one proceeds through the following specification which gives an illustrative example of the invention in reference to the drawing which shows a sectional elevation view of a sample holder embodying the principles of the invention.

The sample holder comprises cylindrical body **1** which may be of metal or plastic. The body is divided into two chambers **2** and **3** by a partition wall **17** through which there is an opening **13**. As shown, chamber **2** is completely filled with a fluid sample and chamber **3** is only partially filled with the remainder of its volume being occupied by a gas such as air.

Chamber **3** contains an essentially semi-cylindrical element **16** that has a recess **14** milled in its left end as shown. Hole **13** and recess **14** align with each other to form a fluid trap below the level of the fluid in both chambers. The static pressure of the fluid in chamber **2** is thereby balanced against the combined pressure of the fluid in chamber **3** and the pressure exerted by the gas on the latter fluid. This is a condition that prevails when a sample holder is ready for use and in use in an evacuable spectrometer.

It may be seen that chamber **2** has an aperture which is covered by a thin window **6** which is permeable to low energy radiation that enters and exits the sample fluid. Window **6** may be any suitable, homogeneous material. Mylar of 0.00025 inch thickness has been found suitable for most cases, but it should be realized that the window thickness is governed by the size of the sample holder and the pressure which it may be required to oppose.

The window **6** must be sealed to the body of the housing and this is accomplished in this case by an O-ring **7** which resides in an annular recess in an end of the housing body **1** as shown. The window bears on the O-ring and is under the compressive force of a cap or cover that is generally designated by the reference numeral **4**. The cover has a window opening that is about the same size as the chamber **2**, it usually being desirable to expose as much sample fluid surface as possible to the analyzing radiation beam.

Cover **4** is engaged with body **1** through the agency of its internal threads **20** being engaged with threads of an externally threaded lock nut **8**. Lock nut **8** abuts a shoulder **19** that extends around the periphery of body **1** so that by reaction against the shoulder, cover **4** may be drawn tight to compress the O-ring **7**.

To assure that cover **4** will always take the same position relative to body **1** and to thereby insure that reproducible analyzing results will be obtainable, the cover **4** is provided with an inwardly radially extending guide pin **5** that registers in a slot which is milled axially of the end of body **1**.

To assure that the whole sample holder will be located in a repetitively identical position within the spectrometer, the holder is provided with a key pin **12** that registers in a cooperating keyway, not shown.

The rear part of the sample holder is removable to facilitate cleaning of its interior. It comprises a threaded stopper **9** which screws into internal threads **18** at the end of body **1**. In this instance a radially compressive seal is effected between the bore of housing **1** and the stopper by the use of an O-ring **10** that resides in an internal circular groove, as shown.

Also provided in this embodiment is a magnetic plate **11** which supplements the purpose of guide pin **12** to insure precise repositioning and secure holding of the sample holder when it is removed and replaced in the spectrometer. Plate **11** may be magnetized or magnetizable and adapted for mutual magnetic attraction with a similar element, not shown, that may be in the sample holder support of the spectrometer.

To anyone having minimum mechanical skill it will be obvious that to use the sample holder it may be filled with a predetermined quantity of fluid to be analyzed and that it may then be manipulated, after cover **4** is affixed, so that fluid completely fills chamber **2** and partially fills chamber **3** in which an air volume is trapped. The holder may then be placed in the spectrometer and mounted for use in a horizontal position most customarily although the instant design may also be used at an angle departing from horizontal by a certain amount.

The word fluid as used herein, in reference to the sample, is intended to embrace any material that contains volatiles or that flows and includes high and low viscosity liquids, solutions, and slurries or physical mixtures.

The principles of the sample holder illustrated herein may be adapted to other designs which are intended for use with a vertical, horizontal, or inclined X-ray beam directed up or down. For instance, another embodiment has been made where a first chamber with the window is on top and a tube projects downwardly from it into a second chamber which is partially filled with liquid so that the end of the tube is sealed thereby and a gas volume is enclosed. On the basis of the above illustrated embodiment, those versed in the art will now perceive many different configurations of the invention without departing from its spirit and scope which is to be determined only by interpreting the claims which follow.

What is claimed:

1. A fluid sample holder for use in a spectrometer comprising:

(a) a hollow body having means therein that partition the body into first and second chambers which connect with each other through an opening in the partition means,

(b) the first of said chambers having an aperture and a thin radiation permeable window that is sealed over the aperture to contain a first portion of a fluid sample in the body,

(c) said window being subject to distension when the sample holder is in an evacuated ambient in a spectrometer,

(d) the second of said chambers defining a volume for a second portion of the fluid that is joined with the first portion through the opening which is below the surfaces of both portions and for the confinement of gas that may be in pressure exchange contact with the second portion of the fluid,

(e) whereby any gas that is confined may expand to compensate the volume and pressure change on the fluid in the first chamber if the window distends.

2. The invention set forth in claim **1** including:

(a) a cover that is adapted to engage the body at the end thereof at which the first chamber is located,

(b) said cover having an aperture that is aligned with the aperture of the first chamber, and

(c) an O-ring means disposed between the body and the window,

(d) whereby the window may be compressed in sealing relation with respect to the body by the cover.

3. The invention set forth in claim **1** including:

(a) a magnetizable element fastened to the exterior of the housing,

(b) said element being adapted for mutual magnetic attraction to position said holder within said spectrometer.

4. A fluid sample holder for use in a spectrometer comprising:

(a) a hollow body having means therein that partition the body into first and second chambers which connect with each other through an opening in the partition means,

(b) the first of said chambers having an aperture and a thin radiation permeable window sealed over the aperture to contain a fluid sample in the first chamber,

(c) said window being subject to distension when the sample holder is in an evacuated ambient in a spectrometer,

(d) the first chamber being adapted for being filled with fluid sample when the holder is in use,

(e) the second chamber being adapted to contain a confined volume of gas and a portion of the fluid sample which portion is in pressure exchange contact with the gas and joins the fluid in the first chamber through the opening,

(f) the said opening being located at a level that is below the fluid levels in each chamber when the holder is in a position of normal usage so that the pressure of the fluid in the first chamber balances the combined pressure of the fluid and the volume of gas in the second chamber,

(g) whereby expansion of the gas volume will maintain a substantially constant pressure on the fluid if the window is distended.

5. A fluid sample holder for use in a spectrometer comprising:

(a) a cylindrical housing body having a partition wall therein that defines first and second chambers which communicate with each other through an opening in the wall,

(b) said opening being below the level of the sample fluid which may be in a chamber when the holder is filled for use,

(c) said first chamber having an aperture communicating with the ambient surrounding the holder,

(d) a thin radiation permeable window that is adapted for being compressed in sealing relation about the margins of the aperture,

(e) a cap that is engageable with the body to effect compressive force on the window, said cap being apertured for the window,

(f) said second chamber being arranged to hold a quantity of fluid that partially fills it and permits a remaining expansible volume of entrapped gas,

(g) said first chamber being completely filled with fluid when said holder is in its position for use,

(h) whereupon said first chamber will be held under pressure by the expansible gas in the second chamber to compensate for fluid displacement due to distension of the window incident to evacuation of its ambient.

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