Nov. 2, 1948. W. S. YOUNG 2,452,623

APPARATUS FOR TRANSFER OF LIGHT LIQUID

Filed Jan. 19, 1946

FIG. 1.

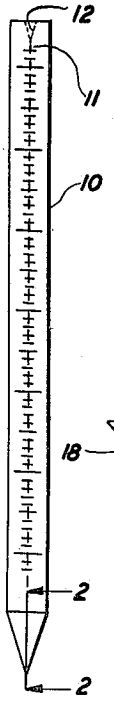


FIG. 3.

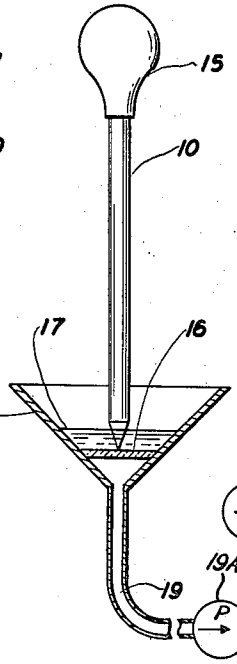


FIG. 4.

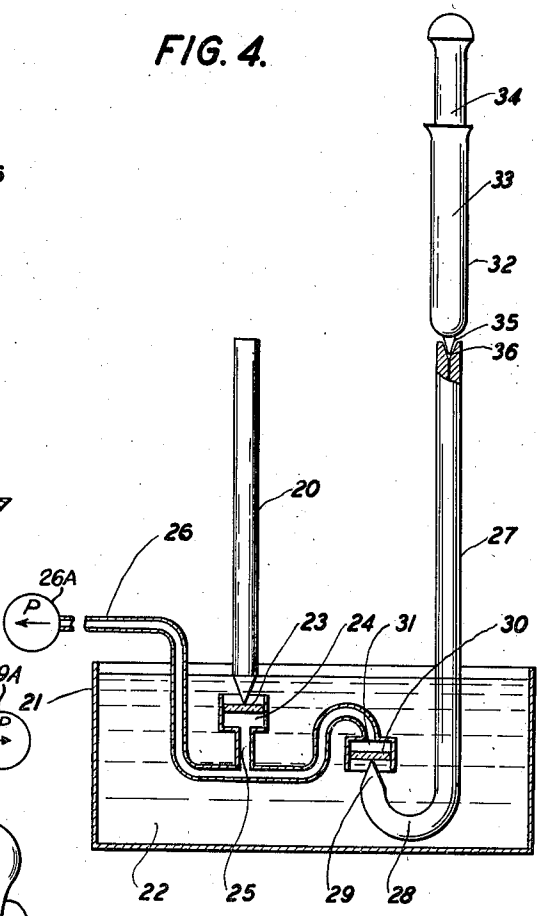


FIG. 2.

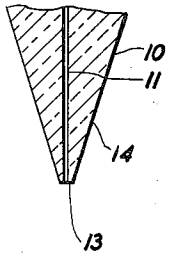
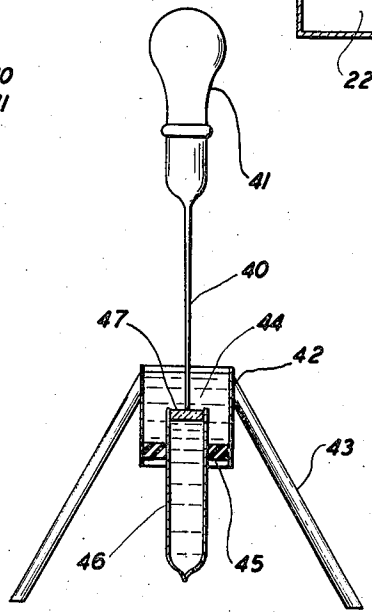


FIG. 5.



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APPARATUS FOR TRANSFER OF LIGHT LIQUID

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12 Claims. (Cl. 137—78)

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This invention is concerned with the transfer of light liquids from one container to another and provides novel means for such transfer while maintaining a seal between the containers.

In the introduction of samples into analytical apparatus such as mass spectrometers and infra-red spectrometers, a liquid to be analyzed should be introduced, preferably in predetermined quantity, without introducing a contaminant, for example air. This has been accomplished heretofore through valves, stop cocks, and the like, but such devices are not entirely satisfactory since the moving parts of valves, cocks, etc. present an opportunity for leakage and lubricants employed to seal the moving parts may contaminate the sample or absorb part of it, thus making it unrepresentative.

In accordance with my invention such devices as valves, stop cocks, etc. may be eliminated, and a sample of light liquid introduced into a conduit, say the sampling system of the mass spectrometer or the inlet to a container, through a seal containing no moving parts. This is accomplished by attaching to the conduit a seal composed of a porous member that is pervious to the light liquid to be introduced, but impervious to the heavy liquid which forms a pool covering the porous member. The heavy liquid should be such that the light liquid is insoluble in it and immiscible with it. A sample of the light liquid is placed in a tube of small bore, preferably one of capillary dimensions, having an open end. In the case of a large sample, the tube may be only part of the container. The open end of the tube is inserted into the pool and brought in contact with the surface of the porous member therein. If the pressure in the tube is made higher than that in the conduit, the light liquid will be forced through the tube into the conduit while the heavy liquid, which is displaced only from the place of contact between the tube and the porous member, will seal the member and prevent the entrance of contaminants from the outside.

Mercury is an ideal heavy liquid for use in most practices of the invention, since most light liquids, including aqueous solutions and most organic liquids are substantially insoluble in and immiscible with mercury.

The porous member may be made of any suitable material, but a disk formed by sintering together small particles of a siliceous substance, say glass, is preferred.

Preferably, the open end of the tube is of small exterior cross-section as well as interior cross-section. Thus, the tube may have a relatively heavy wall tapered to a point at the open end. In order to prevent the light liquid from creeping up the tube out of the pool of heavy liquid, it is desirable to polish the extreme end of the

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tube, i. e. across its section, and better still to polish the tapered portion of the outside wall of the tube adjacent the open end. Thus, the walls of the tube adjacent the open end may be thinned gradually toward the open end to provide a point, the open end itself and the interior tube wall adjacent thereto being polished.

If the tube is of capillary bore, a sample of liquid to be introduced through the seal will rise naturally into the tube if the open end is inserted into a body of the light liquid. However, in some instances it may be desirable to provide other means for sucking liquid into the open end of the tube, for example, an expansible bulb connected to the tube at its other end or a plunger type pump or syringe likewise connected to the tube at its other end.

As indicated above, the tube may be simply an exit from a larger vessel when a sample of considerable size is to be introduced. However, in the case of sample for analysis in a mass spectrometer or an infra-red spectrometer, a long capillary tube ordinarily is itself a sufficient container, for such samples are relatively small. In such case, the tube may have marks or graduations along its length and thus will serve as an index of the quantity of liquid passed through the seal into the conduit.

The pressure differential required to force the light liquid through the seal from the tube to the conduit may be obtained by producing a suction within the conduit or by increasing the pressure in the tube, for example with the plunger pump or with the expansible bulb.

These and other features of the invention will be more thoroughly understood in the light of the following description and the accompanying drawings, in which:

Fig. 1 illustrates a micro-burette for use in the practice of the invention as a container for transferring a light liquid;

Fig. 2 is an enlarged cross-sectional view through the tip of the micro-burette of Fig. 1 taken along the line 2—2;

Fig. 3 shows the micro-burette of Fig. 1 equipped with a suction bulb and placed in a mercury well;

Fig. 4 is an elevation partly in section illustrating two other practices of the invention; and

Fig. 5 illustrates the application of the invention to a weighing bottle for accurately determining the weights of ingredients of a sample compounded in a sealed container.

Referring to Fig. 1, it will be observed that the micro-burette 10 comprises a thick-walled glass tube having a small bore 11, preferably of capillary dimensions. The burette has graduations along its length for measurement purposes and may be made conveniently from a piece of

thermometer tubing. The upper end of the tube has an inverted conical seat 12 adapted for a tightly fitting connection with a hypodermic syringe or the like. The lower end of the tube is pointed in substantially conical shape and tapered to a very thin-walled section at the extreme end of the tube. This extreme end 13 is polished transversely to the bore, and the conical surface 14 is likewise polished. This polish is desirable, although not essential, in order to prevent creep of the light liquid upwardly in the seal.

Referring to Fig. 3, the micro-burette 10 of Fig. 1 is equipped with an expansible bulb 15 of rubber or the like fastened over its upper end. The burette stands vertically with its pointed lower end in contact with a porous disk 16. This disk is pervious to a light liquid, say a light hydrocarbon fraction, but is impervious to a pool 17 of mercury which overlies the porous disk in a well 18 of generally frusto-conical section.

The disk preferably is made by sintering together fine particles of glass and is sealed into the sides of the well which projects below and joins a tubular conduit 19.

In the operation of the apparatus of Fig. 3, a sample of light hydrocarbon or the like to be analyzed is sucked into the micro-burette from a supply of the liquid in a test tube or the like. If the micro-burette is of capillary bore, the liquid will rise by itself. But the operation can be aided by means of suction obtained with the bulb. When the sample is in the burette, the latter is wiped clean and introduced point down into the pool of mercury. Preferably, the point is introduced from the side, with the micro-burette held at an oblique angle with the horizontal. When the burette comes in contact with the porous disk, it is straightened up to a vertical position. As soon as the vertical position is obtained, assuming the correct porosity to be present at the zone of contact between the burette and the porous disk, the sample will be sucked out of the burette through the disk into the conduit 19 by means of suction created in the conduit by vacuum pump 19A or the like. During the transfer, the mercury in the well seals the juncture.

Fig. 4 illustrates two uses of a micro-burette constructed in accordance with the principles of the invention. Thus, the micro-burette 20 is of capillary bore and is provided with the pointed end described hereinbefore. Such a burette is filled with a sample of light liquid to be transferred and after the point has been wiped clean, it is immersed as described in the case of the micro-burette of Fig. 3 in a well 21 containing a pool of mercury 22. The point of the micro-burette is brought in contact with a porous disk 23 constructed in accordance with the porous disk of the apparatus of Fig. 3. This disk is held in a horizontal position in the enlarged end 24 of a branch conduit 25 which connects with a main conduit 26 to which suction is applied by means of pump 26A.

By means of graduations (not shown on the micro-burette 20 but indicated on the micro-burette of Fig. 1), the amount of sample introduced is controlled. In all cases, care should be taken to see that the full liquid sample in the micro-burette is not pulled through the seal since air or other gas will inevitably follow and contaminate the sample. In other words, contact between the point of the micro-burette and the porous disk should be broken after only a portion of the contents of the micro-burette has been sucked in. If the bore of the micro-burette is relatively uniform in size, the amount of sample introduced can be measured directly by means of the graduations.

Another micro-burette 27 is shown at the right of Fig. 4. It has 180° curved portion 28 (or hook) at its lower end which has its point 29 in contact with another porous disk 30 which forms the lower wall of a chamber 31 that is connected to the suction conduit 26. If the bore of this micro-burette is of capillary dimensions, a sample can be introduced merely by placing the point of the micro-burette in the container of the sample. If the bore is larger than capillary dimensions or if additional force to suck in the sample is desired, a hypodermic syringe 32 (comprising a simple cylinder 33 and a plunger 34) may be used. This hypodermic syringe has a nipple 35 which fits tightly in a socket 36 in the top of the micro-burette. This socket may be similar to the socket 12 in the micro-burette of Fig. 1.

The introduction of the sample from the micro-burette 27 is accomplished in the same way as in the previous cases, except that the sample is sucked upwardly into the conduit system 26 through the disk.

The apparatus of Fig. 5 is useful for compounding samples from known amounts of several light liquids. It employs a capillary pipette 40 having an expansible bulb 41, say a medicine dropper bulb, at its enlarged upper end. As shown in the figure, this projects downwardly into a well 42 supported on a tripod 43. The well contains a pool of mercury 44 and its bottom comprises a rubber ring 45, in the central opening of which is disposed a small sample tube 46. In Fig. 5, this sample tube is shown closed at its lower end and is sealed at its upper end by a porous disk 47 that is pervious to a light liquid to be transferred but impervious to the mercury.

In the operation of the apparatus of Fig. 5, the lower end of the container 46 is originally open and it is evacuated by means of a vacuum connection (not shown) and sealed off, as shown, about 20 mm. from the sintered disk, this operation being conducted with the tube in place and with the sintered disk underneath the mercury pool.

The weight of the complete assembly, minus the capillary pipette, should be 10 to 25 grams, depending upon the amount of mercury in the well.

A small amount of liquid to be introduced into the weighing bottle 46 is drawn up into the capillary pipette and the tip of its capillary tube is then put under the mercury to touch the sintered disk until the desired amount of the sample has been sucked through, due to the vacuum in the weighing bottle.

The weighing bottle should be tared on a micro-balance and weighed after each component is added. After all the components are blended, assuming that a blend is to be made in the weighing bottle, the weighing bottle is partly immersed in liquid nitrogen to condense the hydrocarbon vapors and distill out any impurities still in the sintered disk.

The bottle should be warmed and chilled several times in order to assure thorough mixing of the components. Thereafter, the mercury cup is removed to allow air to suck in and the sintered disk is cut off of the weighing bottle.

The blend may then be removed from the weighing bottle with a fine capillary needle and inserted into the sampling system of a mass

spectrometer or the like through a sintered disk as already described.

It will be apparent that the apparatus of the invention provides a simple and reliable procedure for introducing a sample into an analytical apparatus without contamination from the outside atmosphere.

If desired, of course, the capillary tube can be topped by a large container for the liquid sample, although ordinarily the capacity of the micro-burette itself is sufficient.

All of the apparatus may be made of glass, although it may be desirable to make the large well 2 in Fig. 4 of somewhat less fragile material.

Suitable sintered disks for use in constructing the apparatus may be purchased as commercial 10 mm. "immersion filters" of "fine" porosity.

The micro-burette is conveniently made from thermometer tubing. Such tubing may be obtained with an internal bore so small that it delivers only 1.5×10^{-4} ml. per cm. length.

I claim:

1. In apparatus for transferring a light liquid, the combination which comprises a conduit, a container, a pool of heavy liquid that is substantially immiscible with the light liquid disposed in the container, a porous member communicating with the conduit and covered by the pool, the member being pervious to the light liquid but impervious to the heavy liquid, a second container for the light liquid to be transferred consisting of a tube of small bore having an open end for contacting the portion of the porous member covered by the pool, and means for producing a pressure differential between the second container and the conduit to induce flow of the light liquid from the second container to the conduit through the tube and the porous member when the latter two are in contact with each other.

2. Apparatus according to claim 1 in which the heavy liquid is mercury.

3. In apparatus for transferring a light liquid, the combination which comprises a conduit, a container, a pool of heavy liquid that is substantially immiscible with the light liquid disposed in the container, a porous member communicating with the conduit and covered by the pool, the member being pervious to the light liquid but impervious to the heavy liquid, a vessel for the light liquid to be transferred consisting of a tube of capillary bore having an open end for contacting the portion of the porous member covered by the pool, and means for producing a pressure differential between the vessel and the conduit to induce flow of the light liquid from the vessel to the conduit through the tube and the porous member when the latter two are in contact with each other.

4. In apparatus for transferring a light liquid, the combination which comprises a conduit, a container, a pool of heavy liquid that is substantially immiscible with the light liquid disposed in the container, a porous member communicating with the conduit and covered by the pool, the member being pervious to the light liquid but impervious to the heavy liquid, a tube of small bore from which the light liquid is transferred and having an open pointed end for contacting the portion of the porous member covered by the pool, and means for producing a pressure differential between the tube and the conduit to induce flow of the light liquid from the tube to the conduit through the porous member when the tube and the porous member are in contact with each other.

5. Apparatus according to claim 4 in which the extreme open end of the tube is polished.

6. Apparatus according to claim 4 in which the tapered exterior wall of the tube adjacent the tube is polished.

7. In apparatus for transferring a light liquid, the combination which comprises a conduit, a container, a pool of heavy liquid that is substantially immiscible with the light liquid disposed in the container, a porous member communicating with the conduit and covered by the pool, the member being pervious to the light liquid but impervious to the heavy liquid, a tube of small bore from which the light liquid is transferred and having an open end for contacting the portion of the porous member covered by the pool, the walls of the tube adjacent the open end being thinned gradually toward the open end to provide a point, the open end and the exterior tube wall adjacent thereto being polished, and means for producing a pressure differential between the tube and the conduit to induce flow of the light liquid from the tube to the conduit through the porous member when the tube and the porous member are in contact with each other.

8. In apparatus for transferring a light liquid, the combination which comprises a conduit, a container, a pool of heavy liquid that is substantially immiscible with the light liquid disposed in the container, a porous member composed of siliceous particles sintered together communicating with the conduit and covered by the pool, the member being pervious to the light liquid but impervious to the heavy liquid, a tube of small bore from which the light liquid is transferred and having an open end for contacting the portion of the porous member covered by the pool, and means for producing a pressure differential between the tube and the conduit to induce flow of the light liquid from the tube to the conduit through the porous member when the tube and the porous member are in contact with each other.

9. Apparatus according to claim 8 in which the porous member is a sintered glass disk.

10. In apparatus for transferring a light liquid, the combination which comprises a conduit, a container, a pool of heavy liquid that is substantially immiscible with the light liquid disposed in the container, a porous member communicating with the conduit and covered by the pool, the member being pervious to the light liquid but impervious to the heavy liquid, a tube of small bore from which the light liquid is transferred having an open end for contacting the portion of the porous member covered by the pool, means for sucking the light liquid into the open end of the tube, and means for producing a pressure differential between the tube and the conduit to induce flow of the light liquid from the tube to the conduit through the porous member.

11. Apparatus according to claim 10 provided with an expansible bulb connected to the tube at its other end.

12. Apparatus according to claim 10 provided with a plunger-type pump connected to the tube at its other end.

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