Dec. 3, 1963 P. T. GILBERT, JR 3,112,882

PUMP FOR LIQUIDS

Filed March 21, 1960

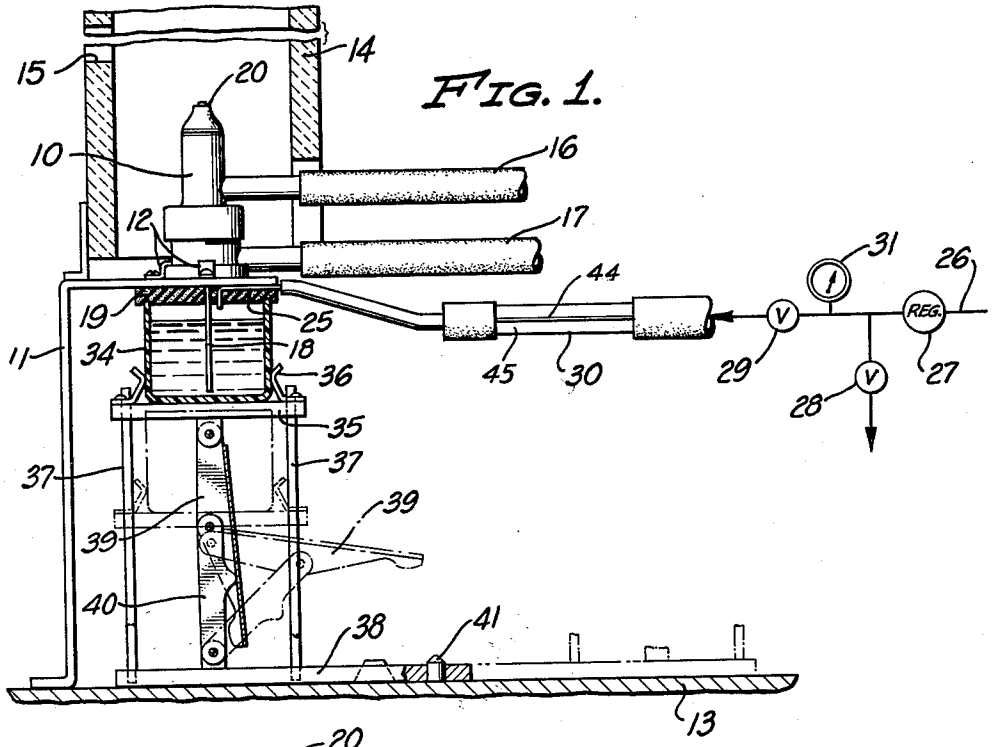


FIG. 1.

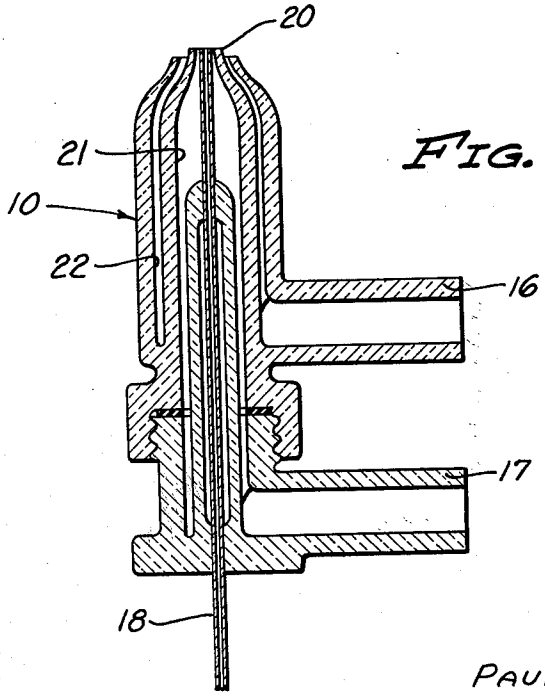


FIG. 2.

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PUMP FOR LIQUIDS

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This invention relates to a pump for generating a continuous and uniform flow of liquid. Whereas it is particularly effective for generating very low flow rates, such as 0.01 ml./min., and has been used for rates up to 3 ml./min., its utility is not necessarily confined to this range. Further, whereas the pump is particularly adapted for feeding sample to a flame for purposes of flame-spectrophotometric analysis, it is not limited to this application.

In flame photometry, the sample is most commonly supplied to the flame by means of a pneumatic atomizer. The suction created by the Venturi effect of a stream of gas flowing at high speed past or around the end of a capillary tube causes liquid, applied to the other end of the tube, to flow through it, being atomized on emerging into the stream of gas. The device thus serves the dual purpose of pumping the liquid and atomizing it. The gas used for atomization conveys the spray to the flame, of which it forms one of the constituents. A typical atomizer-burner is shown in my U.S. Patent No. 2,714,833.

In pneumatic atomization, the rate of liquid flow depends upon and is usually proportional to, the suction, which in turn is governed by the rate of gas flow. The liquid flow rate thus cannot be controlled independently of the flow of gas that supplies the flame. This is inconvenient, for it is desirable in any such technique to have independent control of the significant variables, to permit their separate optimization. Accordingly, it is an object of the present invention to provide a pump for liquid having an independent control of flow rate.

Further, in pneumatic atomization the liquid flow rate will be altered by any accidental alteration of the gas flow rate, such as results from partial clogging of the gas port by dirt; or by any accidental change of configuration of the parts of the atomizer, such as results from thermal expansion of parts on heating by the flame, shifting of an insecurely fastened capillary, or accumulation of deposits (e.g., salts left by evaporation of the sample solution) upon the tip of the capillary or gas port. The liquid flow rate is also affected by the effect of nature of the gas and of the liquid itself upon the suction. Thus, the presence of hydrogen, which diffuses very rapidly, in the proximity of the capillary tip will drastically reduce the suction. If hydrogen is used for atomization, it will generate much less suction than an equal flow of heavier gas. Since the liquid itself forms part of the environment of the capillary tip, it is likely that different liquids, with their different vapor pressures, densities, etc., will somewhat affect the suction in the same way. It is another object of the invention to provide a liquid pump that does not utilize suction and is independent of the vagaries of flow rate inherent in suction type pumping.

Further, in causing the liquid to flow, the suction must act against any hydrostatic head that is effective. The liquid is commonly fed in a vertical direction, usually upward but often downward. Any change in the hydrostatic head of the liquid sample will correspondingly change the net or total effective suction available for pumping the liquid. Such change occurs as the sample in a cup or in a funnel declines in level as it is consumed. This causes a gradual decline of the flow rate of the liquid. Another object is to provide a liquid pump in which the rate of flow is independent of the quantity of liquid present.

Further, uniformity of liquid flow rate depends upon constancy of the impedance imposed upon the liquid by the capillary. This impedance depends upon the dimensions of the capillary bore and upon the viscosity of the liquid. Anything that alters either of these will change the flow rate. Thus, particles caught in the narrow capillary will obstruct the flow, as will also a deposit gradually forming upon the inner walls of the capillary. Blood plasma readily forms such a deposit. This difficulty becomes the greater, the smaller the capillary tube. Small tubes—i.e., tubes of high impedance—are desirable in flame photometry because they permit and require the high suctions generated by high gas pressures, which cause finer atomization and more efficient excitation in the flame and also suppress the effects of hydrostatic head, while keeping the sample flow down to the low rates necessary for optimal excitation of the elements in the sample. But realization of the best conditions in these respect may require a capillary tube only a few thousandths of an inch in diameter, which becomes clogged with extreme ease.

Capillary tubes also become clogged very easily by bubbles, which restrict the sample flow. These bubbles tend to deposit within the capillary owing to the reduction of pressure experienced by the liquid as it enters. This phenomenon can be avoided by deaerating the sample or by adding wetting agents, but both expedients are inconvenient and can in fact be interdicted by the nature of the sample. Bubbles also form very commonly by a hydrodynamic effect at the capillary inlet. If the inlet has sharp edges, the stream of liquid as it enters on first application of the sample will, by virtue of its inertia, resist the sudden change of direction imposed by the geometry of the inlet, and tend to skip a small area on the inner wall of the capillary just above the inlet. An air bubble is thereby trapped. Deaeration will not alleviate this difficulty. In some types of atomizer the formation of bubbles is one of the commonest sources of error. It is an object to provide a pump in which the rate of flow is not a function of the dimensions of the capillary tubes, permitting the tubes to be selected for optimizing other variables.

A pneumatic suction atomizer pumps liquid at a rate proportional to its fluidity. Viscosity depends upon the nature of the liquid, the concentration and nature of solutes dissolved in it, and the temperature. Flame photometry is a relative or comparative method of analysis; the sample must always be compared with a standard solution. It is of prime importance that the sample and standard solutions flow at the same rate. Since the sample is often of unknown composition, it may not be convenient to prepare a standard having the same viscosity. Further, viscosity is a very sensitive function of temperature. Any slight differences of temperature between liquids to be compared can lead to corresponding errors. Warming by the proximity of the flame or cooling by evaporation of a volatile solvent will introduce similar errors. Another object is to provide a pump having a controllable flow rate that is substantially independent of the viscosity of the liquid being pumped.

Atomizers for commercial instruments should be as nearly alike in their dimensions and characteristics as possible, to permit interchange and standardization of analytical methods. The manufacture of atomizers having the desired close tolerances of performance is exceedingly difficult, as the dimensions and smoothness of surfaces are very critical. Although subjected to the most rigorous inspection, commercial atomizers are never truly interchangeable.

In the manufacture of atomizer-burners an effort is generally made to adjust the dimensions and prescribe the gas pressures in such a way as to assure that for most

analytical emissions the intensity will be maximal with respect to sample flow rate. Unfortunately, different spectral lines or bands or different elements attain maximal intensity at different flow rates. Thus the prescribed pressures for a given atomizer-burner are necessarily a compromise. A pneumatic suction atomizer-burner does not permit simultaneous optimization with respect to all three variables: fuel flow, oxidant flow, and sample flow. This difficulty is particularly marked with hot flames such as premixed oxyacetylene and most especially with oxygen-cyanogen, in which the slightest departure from optimal flow adjustment can very greatly diminish the analytical sensitivity.

A pneumatic atomizer injecting sample directly into the base of a flame (an atomizer-burner) can operate only with flames of high burning velocity or with pilot flames to maintain combustion, owing to the necessary high speed of the emerging gas. In the case of oxyacetylene, which is particularly advantageous for flame photometry, it may be desired to atomize the sample with premixed oxyacetylene. Owing to danger of detonation, such a mixture should not be compressed above 15 p.s.i.g. Since a mixing manifold must be interposed, there must generally be a pressure drop between the pure acetylene and the mixture, wherefore the mixture itself is not readily utilized at pressures as high as 15 p.s.i.g. But suction and atomization become less efficient at the lower pressures. Further, if a pneumatic suction atomizer is to be used in a spray chamber with premixed oxyacetylene or oxyhydrogen, the high burning velocities of these mixtures entail an appreciable pressure drop across the burner port. This pressure drop imposes a burden upon the atomizer, acting like the hydrostatic head mentioned above. Accordingly, it is an object of the invention to provide a liquid pump for use with atomizer-burners in which the liquid flow rate is not dependent on burner geometry nor on fuel or oxidant flow rates.

The available amount of certain samples (e.g., blood from a small mammal) may be so small as to be inadequate for flame-photometric analysis with a given instrument. It is known that in an instrument optimized for emission intensity, reduction of sample flow rate does not entail a proportional loss of intensity. Indeed, a 10-fold reduction of flow may cause a loss of only 50% of the signal. The possibility of adjusting the sample flow rate independently of the flame adjustment would therefore be valuable in conserving a scarce sample. But with the usual suction atomizers this is not practical.

In general, the invention contemplates pumping by means of a constant but adjustable flow of gas provided by supplying gas under controlled pressure to a very fine leak. The gas emerging from the leak acts as a piston to force liquid from a closed cup through a small tube or capillary to the atomizer. The cup containing the liquid sample may be sealed by merely being pressed against a soft gasket. The liquid thus touches no additional object except the atomizer to which very little liquid adheres. In general, no rinsing is needed. By proper construction of the cup holder and the mechanism for moving it, samples can be changed in very rapid sequence.

It is an object of the invention to provide a pump for liquid including a liquid container, a cover for sealing engagement with the container, an outlet capillary projecting into the container, an inlet capillary projecting into the container, and means for supplying gas at a constant rate of volume flow through the inlet capillary for moving liquid out of the container through the outlet capillary. A further object is to provide such a pump including a leak connected between a source of gas under pressure and the inlet to the container with the leak having a flow impedance that is high relative to any impedance imposed upon the flow of liquid, so that the gas functions as a piston displacing liquid from the container, with the gaseous piston being generated at a constant rate. Another object is to provide a pump having continuously variable pumping speeds over a wide range of flow rates, with the flow rate being settable to any desired value within a range in a matter of seconds and with different ranges being available by merely changing the leak in the inlet line.

It is an object of the invention to provide a pump which reduces the hazard of analyzing highly volatile and combustible material, such as gasoline, by flame photometry. A further object is to provide a pump wherein the sample is fully enclosed and hermetically separated from the surrounding air thereby preventing evaporation and change of temperature and concentration.

It is an object of the invention to provide a pump suitable for use with atomizers and the like which eliminates the inconvenience of cleaning, washing, rinsing, or changing auxiliary equipment such as syringes, cylinders, or flexible tubing. Only the sample cup, a simple cylindrical container of polyethylene or glass, need be rinsed between uses. Often a water repellent cup, such as one coated with a silicone water repellent, can be simply shaken dry and requires no cleaning. Another object of the invention is to provide a pump for liquids that is inexpensive and requires no moving parts and one that can be incorporated into existing commercial flame photometers and other instruments with only minor modifications.

While the invention is described herein in conjunction with an atomizer-burner for a flame photometer, it is also applicable to other fields such as chromatography, electrophoresis, and fermentation, where accurate control of liquid flow is needed, often at low rates. The invention is also useful as a means of supplying sample to other spectrochemical emission sources such as the arc and spark. The drawing merely shows and the description merely describes a preferred embodiment which is given by way of illustration or example.

In the drawing:

FIG. 1 is an elevation view of the pump used in conjunction with an atomizer-burner; and

FIG. 2 is an enlarged sectional view of a portion of the apparatus of FIG. 1.

An atomizer-burner 10 is mounted on a bracket 11 by clips 12, with the bracket in turn mounted on a base 13. A tubular shield 14 with an access port 15 may be mounted on the bracket 11 around the burner 10. Fuel, such as hydrogen or acetylene, is connected to the burner through a line 16 and a combustion supporting and atomizing gas, such as oxygen, is connected to the burner through a line 17. Suitable pressure regulators and rate of flow control valves may be positioned in each of the lines for individually controlling the fuel and atomizer gas flow rates. A small diameter tubing or capillary 18 is fixed within the burner and projects downward through the bracket 11 and a gasket 19 fixed to the under side of the bracket. Typically, the tubing 18 will be a short length of stainless steel hypodermic tubing.

The internal arrangement of the atomizer burner 10 is shown in detail in FIG. 2 and may be identical to that shown in my previously mentioned patent, No. 2,714,833. The upper end of the tubing 18 terminates at the discharge nozzle 20. The line 17 for the atomizer gas communicates with an internal chamber 21 which terminates in an annular opening about the tubing 18. The line 16 for the fuel communicates with another internal chamber 22 which terminates in an annular opening disposed about the atomizer discharge nozzle 20.

Another tubing or capillary 25 is fixed to the under side of the bracket 11 and projects downward through the gasket 19. Typically, this tubing is also a length of stainless steel hypodermic tubing. Means are provided for supplying a gas at a constant rate to the tubing 25. Ordinarily, this gas will be the same as that used for the atomizer and oxidant gas and, hence, usually will be oxygen. In the embodiment of FIG. 1, a source of oxygen under pressure is connected to a line 26 having a pres-

sure regulator 27 therein. The output from the regulator is directed through a bypass valve 28 to the atmosphere and through a shut-off valve 29 to a leak 30. The leak is connected in turn to the capillary 25. A pressure gauge 31 provides an indication of the pressure at the output of the regulator 27.

A container 34 is removably mounted on a platform 35 for movement upward into engagement with the gasket 19. One preferred apparatus for rapidly raising and lowering and changing the container is shown in FIG. 1. The container is held onto the platform by spring clips 36. The platform moves up and down along four guides 37 which, in turn, are supported on a plate 38. A lever 39 is pivotally mounted at one end to the platform 35. Another lever 40 is pivotally mounted between the plate 38 and a central point of the lever 39. The two levers are dimensioned to coact as a toggle for locking the container in the upper position, as shown in solid lines in FIG. 1. The lever 39 may be manually rotated to the position shown in phantom lines in FIG. 1 to lower the container. When the container is lowered, the plate 38 may be rotated about the post 41 for removal and reinsertion of suitable containers.

The leak 30 has a very high flow impedance in comparison to the tubings 25 and 18 so that the leak substantially controls the rate of flow of gas into the container 34. Typically, a leak consists of a length of a noble metal wire 44, such as palladium, sealed into a hard glass capillary tube 45. The leak will be a few centimeters in length. The capacity or flow rate of the leak is a function of the diameter and length of the wire and the area of actual contact between the metal and glass. Typical leak rates for use with an atomizer-burner range from 0.03 to 0.2 ml./min. per 1 p.s.i. pressure drop across the leak. The rate for a particular leak can be varied by varying the setting of the valve in the associated pressure source. Different ranges may be obtained by substituting leaks of different lengths or different wire diameters.

Other examples of suitable leaks include a pinched metal tube, a very fine glass capillary, and a narrow tube packed with fine powder.

Typically, the pressure regulator 27 will be set to provide an outlet pressure in the range of 5 to 30 p.s.i. As the gas flow rate through the leak into the container is extremely small, it is preferred to operate the system with the bypass valve 28 partially open to provide a higher flow rate through the regulator so as to improve the regulator operation. The bypass valve 28 also permits rapid bleeding of the system on reduction of pressure to diminish the pumping rate without waiting for the gas to escape through the leak 30.

In operating the apparatus, the fuel gas and atomizer gas flow rates are adjusted to the desired values. A container with a standard liquid is raised into engagement with the gasket 19 and the rate of flow of liquid up the tubing 18 is adjusted to the desired value by choosing an appropriate leak and suitably setting the regulator 27. Then the container is lowered and a container with a sample liquid is placed on the platform and raised into the operating position. The flow of gas to the fuel line, the oxidant line and the pumping line is not interrupted as one liquid is substituted for another. After the appropriate measurements are made, additional sample and/or standard liquids are placed in containers on the platform and raised to the operating position. The apparatus may be operated with various quantities of liquid in the container and containers of varying capacities may be utilized. Of course, all the containers must have approximately the same external dimensions so as to fit onto the platform and engage the gasket.

When a container of liquid is raised to the operating position of FIG. 1, the incoming gas from the capillary 25 functions in the nature of an air piston forcing the liquid down in the container and up through the outlet capillary 18. The contact pressure between the container and the gasket must be sufficient to provide a hermetic seal adequate to retain the pumping gas. In this apparatus, the rate of flow of liquid up the capillary 18 is independent of the rate of flow of gas in the fuel line 16 and in the oxidant line 17. As the liquid is forced through the outlet capillary, any tendencies for this line to clog, as referred to previously, are substantially without effect. Also, since the rate of flow of liquid is not a function of the size of the outlet capillary, a relatively large diameter tubing can be utilized.

It should be noted that in operating the apparatus, a transient condition in the rate of flow of liquid up the outlet capillary occurs when the container is first raised into position. In a practical embodiment, the time constant of initial equilibration can be kept to about one second, which is equal to that often incorporated in the measuring system of a flame photometer to damp fluctuations of the flame. Apparatus constructed according to the embodiment of FIG. 1 is presently in use to provide liquid flow rates in the range of 0.01 ml./min. to 3 ml./min. and the apparatus of the invention may be constructed to provide flow rates which exceed this range in both directions.

Although an exemplary embodiment of the invention has been disclosed and discussed, it will be understood that other applications of the invention are possible and that the embodiment disclosed may be subjected to various changes, modifications and substitutions without necessarily departing from the spirit of the invention.

I claim as my invention:

1. In a pump for liquid for an atomizer or the like, the combination of: a liquid container; a cover for sealing engagement with said container; an outlet capillary projecting into said container; an inlet capillary projecting into said container; means for supplying a gas at a constant rate of volume flow to said inlet capillary for moving liquid out of said container through said outlet capillary, said means comprising a gas leak connected to said inlet capillary with said leak having a flow impedance that is high relative to the flow impedance of said outlet capillary; and a shutoff valve only upstream of said leak for controlling the pumping of liquid by said pump.

2. In a pump for liquid for an atomizer or the like, the combination of: a frame; a liquid container; a cover mounted on said frame; container support means carried on said frame for lifting said container into sealing engagement with said cover; an outlet capillary carried by said frame for projecting through said cover into said container and terminating adjacent the bottom thereof; an inlet capillary carried by said frame for projecting through said cover into said container and terminating adjacent the upper edge thereof; a source of gas under pressure; and a gas leak connected between said source and said inlet capillary for supplying gas at a constant rate of volume flow thereto, with said leak having a flow impedance that is high relative to the flow impedance of said outlet capillary.

3. In a pump for liquids, the combination of: a sealed container for the liquid to be pumped; an outlet capillary projecting into said container; an inlet capillary projecting into said container; and means for supplying gas to said inlet capillary at a constant rate of volume flow for moving liquid out of said container through said outlet capillary, said means comprising a gas leak having a fluid flow impedance that is high relative to the fluid flow impedance of said outlet capillary.

4. In a pump for liquids, the combination of: a sealed container for the liquid to be pumped; a liquid outlet line communicating with said container; a gas inlet line communicating with said container; and a source of gas under pressure connected to said inlet line, said inlet line including a gas leak connected between said source and said container for supplying gas at a constant rate of volume flow thereto, with said leak having a flow

impedance that is high relative to the flow impedance of said outlet line.

5. A method of pumping of liquids in a continuous and uniform stream for feeding to an output device such as an atomizer-burner or the like, including the steps of:

maintaining the liquid to be pumped in a closed volume with an outlet opening;

introducing a gas into the closed volume at a constant rate of volume flow to eject liquid from the volume through the outlet opening; and

directing the ejected liquid directly to the output device.

6. A method of pumping liquid in a continuous and uniform stream for feeding to an atomizer-burner with the rate of flow of liquid independent of changes in flow impedance of the atomizer-burner and independent of the rate of flow of fuel, including the steps of:

maintaining the liquid to be pumped in a closed volume with an outlet opening;

introducing a gas into the closed volume at a constant

rate of volume flow to eject liquid from the volume to the outlet opening; and

mixing the ejected liquid with the fuel at the nozzle of the atomizer-burner.

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CERTIFICATE OF CORRECTION

Patent No. 3,112,882 December 3, 1963

Paul Thomas Gilbert Jr.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 18, for "respect" read -- respects --; column 3, line 3, for "or", second occurrence, read -- of --; column 5, line 44, for "p.s.i." read -- p.s.i.g. --; column 7, line 3, strike out "of", second occurrence.

Signed and sealed this 5th day of May 1964.

(SEAL)
Attest:
ERNEST W. SWIDER

Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents