

[54] **METHOD AND APPARATUS FOR FLUID INJECTION**

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[51] Int. Cl.F16k 19/00, F17d 1/08

[58] Field of Search.....137/1, 13, 154, 602, 604; 23/230 A, 253 A; 73/53, 423 A; 259/4; 356/179, 181

[56] **References Cited**

UNITED STATES PATENTS

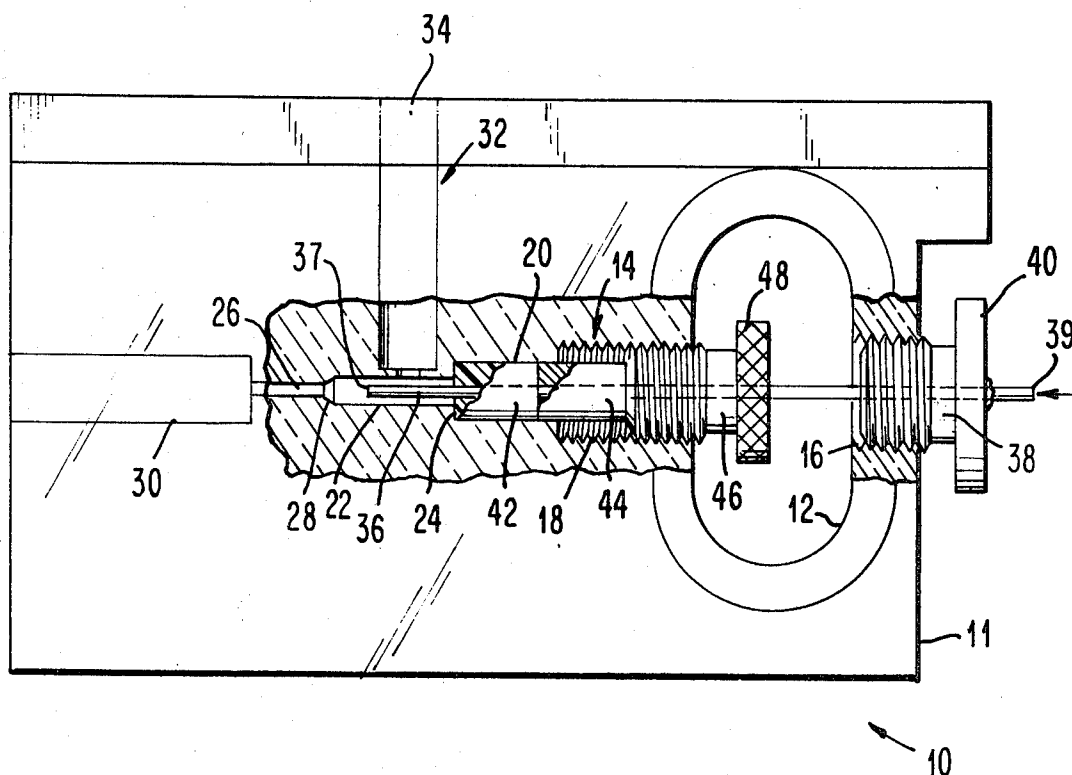
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[57] **ABSTRACT**

A fluid injection device for incorporation in automated apparatus for the quantitative analysis of a substance transported in a flowing liquid stream segmented by gas bubbles or other immiscible fluid segments. The device includes a body defining an elongated fluid passageway portion. One end of the passageway portion provides an inlet for fluid or gas under pressure through a tube extending into the passageway portion from the last-mentioned end toward the other end thereof, which tube has a discharge end portion received axially within the passageway portion. An inlet for unsegmented liquid under pressure communicates with the passageway portion, upstream of the aforementioned discharge end portion of the tube, providing for axial injection of gas into the stream of liquid flowing from the liquid inlet toward the other end of the passageway portion. By varying the resistance to liquid flow in the area around the discharge end of the tube the size of the bubbles injected into the liquid stream may be changed.

5 Claims, 3 Drawing Figures



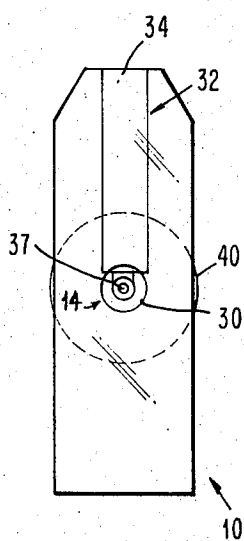


FIG. 1

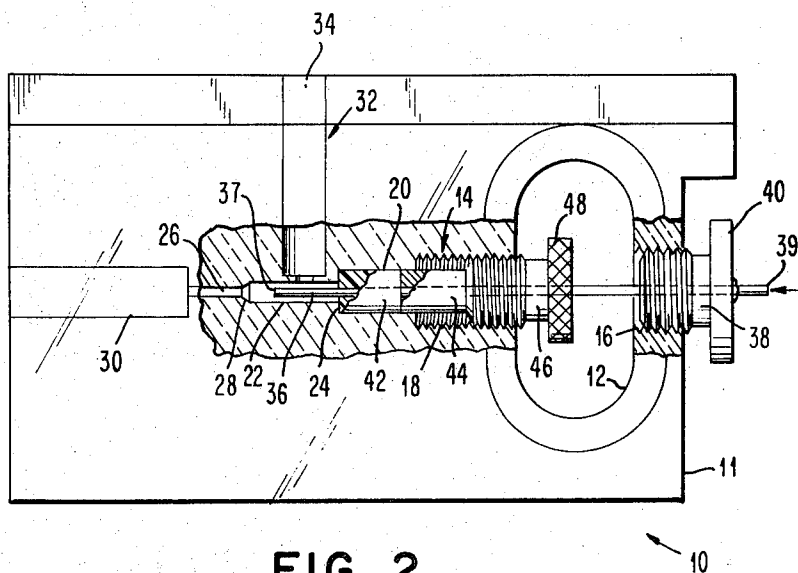


FIG. 2

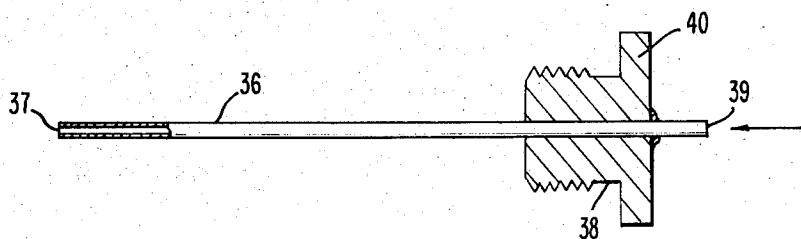


FIG. 3

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METHOD AND APPARATUS FOR FLUID INJECTION

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to a device, useful in automated quantitative analysis of samples flowing in a liquid stream, for adjustably injecting gas bubbles or other immiscible fluid segments into the stream in a manner to control the size and frequency of the injected bubbles or segments, the size and frequency being interdependent.

2. Prior Art

In analysis systems of this kind, a flowing stream of sample liquid, which stream may be a continuous monitoring stream, or a stream of sequential liquid samples, may be continuously mixed in a predetermined proportion with one or more reagents, and otherwise processed to provide a color reaction, for example, the optical density of which at a particular wave length is responsive to the concentration of a constituent of interest in the original sample. Such a system is disclosed in Skeggs U.S. Pat. No. 2,797,149 issued June 25, 1957. Customarily, the flowing stream of sample plus reagent is divided into sequential segments, each succeeding segment of sample plus reagent being spaced from the preceding segment by a gas segment. Each segment of sample plus reagent is then mixed as it flows along in the stream, as shown for example in Ferrari, Jr. U.S. Pat. No. 2,933,293 issued Apr. 19, 1960. The streams of sample and reagent are supplied in a predetermined proportion to a junction by a peristaltic proportioning pump such as is shown in Bilichniansky et al. U.S. Pat. No. 3,425,357 issued Feb. 4, 1969 wherein it is pointed out that a transient change in proportions between the sample and the reagent or reagents may affect the accuracy of the analysis system, which accuracy is premised on constant proportions, referring to the solution to this problem taught by earlier Smythe U.S. Pat. No. 3,306,229 issued Feb. 28, 1967.

U.S. Pat. No. 3,425,357, supra, illustrates and describes apparatus tending to provide in use, over a given range of flow rates, identical liquid segments of sample plus reagent by the provision of substantially uniform periodic gas segments between the liquid segments, and which apparatus is susceptible of some adjustment to vary the size of the gas segment. The aforementioned adjustment may not be made on a single pump tube but may be made only simultaneously with reference to all of a family of pump tubes. Moreover the control of gas bubble patterns is not sufficiently fine in at least some analysis systems to meet requirements.

Modern analysis systems, making up to twelve or more tests on a single sample, employ very small quantities of sample for a single test and concomitantly small volumes of reagents. Hence, the control of proportions has become even more critical. In this connection, a need has arisen for a device to control liquid segmentation very closely and a need has also arisen to control the gas segmenting pattern in a plurality of liquid streams independently of one stream to another.

While reference has been made to the use of gas as a liquid segmenting fluid it is to be understood that the fluid need not be a gas in at least certain applications but may be a liquid immiscible with the liquid which it

segments. It is now well known that segmenting fluids are used in sample analysis apparatus of the automated type to occlude and cleanse the wall structure of the conduit in which the particular segmented stream flows and to maintain the integrity of each slug of liquid used for analysis purposes so that each will not contaminate the next succeeding one or be contaminated by the one which it follows.

SUMMARY OF THE INVENTION

One object of the invention is to provide an improved fluid injection device for use in automated apparatus for sample analysis of the continuous flow type such as characterized above. Another object is to provide a fluid injection device for segmenting any liquid with a fluid immiscible with the liquid such as a gas or another liquid, which device injects such fluid axially into the moving stream of liquid to be segmented. Still another object is to provide such a fluid injection device for each one of a plurality of segmented streams so that the operation of one is not dependent on the operation of another. A further object is to provide a fluid injection device which injects a gas, for example, into a liquid stream to segment the stream, the gas being in the form of bubbles, wherein the resistance to liquid flow acts to vary the bubble size and thereby the frequency of the gas bubbles within certain limits.

A further object is to provide a fluid injection device which includes a body defining an elongated fluid passageway portion. One end of the passageway portion provides a fluid or a gas inlet for gas under pressure through a tube extending into the passageway from the last-mentioned end toward the other end thereof, which tube has a discharge end portion received with clearance concentrically within the passageway portion. An inlet for unsegmented liquid under pressure communicates with the passageway portion upstream of the aforementioned discharge end portion of the tube, providing for a concentric injection of gas into the stream of liquid flowing from the liquid inlet toward the other end of the passageway portion, and wherein the resistance to liquid flow in the area around the discharge end of the tube may be changed to vary the size of gas bubbles injected into the liquid stream.

BRIEF DESCRIPTION OF THE DRAWING

In the drawing:

FIG. 1 is an end view of a fluid injection device shown in elevation, embodying the invention, showing the left end of the device as illustrated in FIG. 2;

FIG. 2 is a side elevational view, partially in section; and

FIG. 3 is a view illustrating a detail of the construction, partially in section.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

In the drawing, the body of the fluid injection device is indicated generally at 10. It may be formed of a clear, hard plastic material, by way of example, such as one sold under the trademark Plexiglas. As shown in the drawing, its vertical dimension shown in FIG. 2 is greater than its width shown in FIG. 1, and it is elongated.

gated horizontally as shown in FIG. 2. As shown, the end of the body remote from that illustrated in FIG. 1 is undercut at 11 as shown in FIG. 2. As shown in the last-mentioned view, adjacent the last-mentioned end of the body there is provided a vertically elongated opening 12 extending through the body from one side through the other.

The body has a longitudinal bore 14 extending therethrough approximately midway between the upper and lower extremities thereof and interrupted by the opening 12. The right end portion of this bore as viewed in FIG. 2 is threaded, as at 16, this threaded portion extending through the right end of the body into the opening 12.

Also, as shown in FIG. 2, a portion of the bore, extending left from the interruption created by the opening 12, is threaded and indicated at 18. The bore 14, has a portion 20 thereof extending to the left as viewed in FIG. 2. The bore portion 20 is smooth and has a uniform diameter throughout the length thereof.

The bore portion 20 merges with a smaller smooth bore portion 22 extending to the left as viewed in FIG. 2. A shoulder 24 is formed at the junction of the bore portions 20 and 22 for a purpose which will appear hereinafter.

Again extending to the left as viewed in FIG. 2, the bore portion 22 merges with portion 26 through a bore portion 28 in the illustrated form. In the illustrated embodiment, the bore portions 26 and 28 constitute a restriction. The bore 14 at its left end as viewed in FIG. 2 terminates in a portion 30 which may be of larger cross section and receive a suitable nipple for connection to a fluid line, neither being shown.

Another bore is provided in the body 10 and indicated generally at 32. In the illustrated form, the bore 32 extends through the upper extremity of the body 10 and is provided with a portion 34 similar to the portion 30 of the bore 14 previously described. The bore 32 communicates with the bore portion 22 of the bore 14 as shown in FIG. 2. As shown in the last-mentioned view, the point of entry of the bore 32 into the bore portion 22 is in the central portion of the latter, that is approximately midway between the ends of the portion 22. The bore portion 34 may receive a suitable nipple for connection to a fluid line, neither being shown.

A tube 36 is provided in axial relation to the bore portion 22 and extending thereinto. In the form illustrated by way of example, the tube 36 is supported concentrically with clearance in the portion 22 of the bore 14, the tube having a discharge end 37 and having an inlet end 39 beyond the bore 14, as shown in FIG. 2, for suitable connection to a conventional conduit, now shown. The tube 36 extends through and is fixed to a screw member 38 in axial and concentric relation thereto. The screw member 38, best shown in FIGS. 2 and 3, provides a rigid support for the tube 36 and is threadedly received in the bore portion 16 for axial adjustment therein. The screw member 38 has a radial flange 40 to be gripped for manipulation and adjustment of the axial position of the tube 36 with the screw member 38. Both the tube 36 and the screw member 38, but particularly the tube, should be formed of a suitable corrosion-resistant metal such as platinum or stainless steel for example.

The tube 36 passes through a sealing element 42 of sleeve form and structured of a suitable elastomer material such as silicone rubber for example. The sealing element 42 is received in the bore portion 20 and bottoms on the aforementioned annular shoulder 24. The sealing element 42 is compressed to form a seal between the body 10 and the tube 36 by a thrust element 44 of sleeve-like form formed of a suitable hard plastic or metal, which embraces the tube 36 and extends into the bore portion 20 from the right of the sealing element 42 as shown in FIG. 2. The right end of the thrust element 44 receives pressure axially thereof from a clamping member 46.

The clamping member 46, also formed of a suitable corrosion-resistant metal, is of cylindrical cross section and has an axial bore extending concentrically therethrough which slidably receives the tube 36, permitting axial adjustment of the tube 36 with reference to the member 46. The clamping member 46 has an externally threaded portion threadedly received in the bore portion 18 for axial adjustment therein. The inner end of the clamping member bears against the right end of the thrust element 44, as previously indicated, and the member 46 is provided with a radial flange 48 accessible in the opening 12 in the body to be gripped and manipulated for adjustment purposes of the sealing pressure of the seal effected by the element 42 between the body 10 and the tube 36.

It will be understood from the foregoing that, when it is desired to adjust the axial position of the tube 36, the sealing pressure between the tube and the body may be relieved first, if necessary for axial movement of the tube 36, by manipulation of the clamping member 46. After this is done the tube adjusting member 38 may be manipulated as aforesaid to vary the axial position of the tube 36 carried thereby. The clamping member 46 may then be re-tightened to provide the aforementioned seal between the tube and the body in the desired axial position of the tube. The radial flange 40 of the adjusting member 38 may have on its outer face circumferentially spaced radial lines, not shown, to selectively index with a suitable reference line, not shown, on the overhanging surface 50 of the body, for the purpose of indicating the axial position of the tube 36.

Broadly considered, liquid delivered under pressure to the inlet of the passageway 32 to be segmented may be any liquid, which includes sample streams and reagent streams but is not limited thereto, with which liquid it is desired to provide an interface with a fluid immiscible therewith. The liquid to be segmented may be delivered in any conventional manner as from a pressurized container of such liquid or from a suitable pump such as that illustrated and described in U.S. Pat. No. 3,425,357, supra, or of the type of Ferrari, Jr. et al. U.S. Pat. 2,935,028 issued May 3, 1960. The immiscible fluid for segmentation of the liquid stream is also delivered under pressure to the inlet end of the tube 36, as previously indicated.

The immiscible fluid, which may be a gas, may be delivered to the last-mentioned inlet from a source of such fluid in a pressurized container or may be delivered by a suitable pump of the type of U.S. Pat. No. 2,935,028 supra, or of the type of U.S. Pat. No. 3,425,357. Whatever the source of pressure, that is, in-

dependently thereof, the gas line to the inlet of tube 36 may be valved in the manner of U.S. Pat. No. 3,425,357, supra, so as to be pulsed to the last-mentioned tube inlet. As previously indicated, the fluid injection device shown by way of example in the drawing may be used in any or as many liquid lines of analysis apparatus as require segmentation, one such device being used in each such liquid line. The segmented stream is discharged from the device through the bore portion 30.

From the foregoing, it will be evident that the bore portions 22, 28, 26 and 30, constitute an elongated passageway portion. One end of the last-mentioned portion provides an inlet for fluid or gas under pressure through the tube 36 extending into the passageway portion from the last-mentioned end toward the other end thereof. The bore or passageway 32 provides an inlet for unsegmented liquid under pressure, communicating with the passageway portion upstream of the discharge end 37 of the tube, providing for concentric injection of gas into the stream of liquid flowing from the liquid inlet toward the discharge end 30 of the passageway portion.

The operation of the fluid injection device will now be described. With the discharge end of the tube 36 in the position shown in FIG. 2, extending beyond the junction of the passageway portions 14 and 32 toward the aforementioned liquid outlet, liquid under pressure entering the passageway portion 14 from the passageway portion 32 flows around the tube 36 as it passes in a direction toward the discharge end of the passageway 14. As this occurs, immiscible fluid, say, gas, is discharged concentrically from the discharge end of the tube 36 into the stream of liquid. Hence a bubble balloons out of the tube in a longitudinal direction toward the outlet of the passageway 14, with liquid flowing around it.

The bubble is eventually sheared off the tube 36 and passes downstream in the bore portion 26 and beyond followed by a portion of the liquid stream, while another bubble builds from the tube 36. It has been found that gas bubbles formed in this manner are much more uniform in size than those formed customarily by injecting one fluid into another in a right angular relationship. The frequency of bubbles, or the bubble pattern in a segmented liquid, is also more uniform when the injection is made concentrically according to the invention.

Bubble size is important in sample analysis apparatus. Of course, the bubbles must be of a size to occlude analysis tubing to maintain the integrity of liquid segments. Bubbles must also reach a greater diameter than the inner diameter of the conduits used in analysis in order to cleanse the wall structure of such conduits properly. On the other hand, bubbles in such conduits should not be unduly elongated as such bubbles, which have no analysis function, only delay analysis, and because their friction with such conduit wall structures creates undesirable resistance to flow.

With constant flow rates of the liquid and the immiscible fluid to the injection device, the size of the bubbles, together with their frequency, may be varied by adjusting the outlet of the tube 36 toward or away from the narrowed bore portion 26 from the position of FIG. 2. Within practical limits, when the adjustment is away

from the bore portion 26, the resistance to liquid flow lessens and the bubbles become larger and the interval therebetween correspondingly longer; and when the adjustment is in the other direction, increasing the resistance to liquid flow, the bubbles become smaller and the interval therebetween smaller. Initially, for given flow rates, the size of bubbles formed is dependent on the ratio of the liquid flow rate to the immiscible fluid flow rate, these being delivery rates to the injection device. The higher the liquid flow rate is to the immiscible fluid flow rate, the smaller the bubbles become. The greater the resistance is to liquid flow in the passageway portion 22, the smaller the bubbles of gas become.

For illustrative purposes only, the internal diameter of the tube 36 may be approximately 0.020 inch, and the bore portion 26 of the passageway 14 may be of approximately the same diameter. The clearance between the tube 36 and the bore portion 22 of the passageway 14 may be approximately 0.001 inch.

Though not limited thereto, the passageway 32 of the fluid injection device may be considered a conduit for a reagent to be segmented before the confluence of the reagent stream and a sample stream. If desired, a sample stream inlet bore, not shown, may be included in the fluid injection device communicating with the passageway 14 downstream from the inlet end of the bore portion 26.

As previously indicated, the apparatus disclosed in the drawing is for the purpose of example only. The method of injecting a fluid into a stream of liquid immiscible therewith comprises flowing a first stream constituted by a liquid under pressure on a path so that it flows along and around the discharge end portion of a conduit, and flows beyond the last-mentioned end portion toward an analysis utilization conduit, and flowing a second stream through the first-mentioned conduit in the same direction as the first stream to segment the latter, which second stream is constituted by a fluid under pressure which is immiscible with the liquid. The method further comprises predetermining the resistance to flow of the first stream around the discharge end of the first-mentioned conduit to thereby determine the size and frequency of the segments in the first stream.

The precision with which the injection device functions in sample analysis apparatus for proper proportioning of liquids, and the precision with which it forms immiscible fluid segments of the desired size are significant advantages. The feature of the device which permits the adjustment of size of, say, bubbles, within previously established flow rates is highly advantageous for reasons previously brought out.

It is believed that many advantages of this invention will now be apparent to those skilled in the art. The foregoing description is illustrative, rather than limiting, as a number of variations and modifications may be made without departing from the true spirit and scope of the invention. The invention is limited only by the scope of the following claims.

What is claimed is:

1. A fluid injection device for a continuous-flow sample analysis system utilizing at least one segmented liquid stream in a utilization conduit, comprising: a first unobstructed fluid passageway conduit for laminar

liquid flow therethrough, having an inlet and having an outlet connected to said utilization conduit, a second unobstructed fluid passageway conduit extending a distance inwardly into a portion of said first fluid passageway conduit of uniform diameter, having an outlet axially directed within said portion of said first fluid passageway conduit in a direction toward and terminating short of said first passageway conduit outlet, said second passageway conduit having an inlet without said first passageway conduit, said inlet of said first passageway conduit being adapted to receive a stream of unsegmented liquid, said inlet of said second passageway conduit being adapted to receive a stream of segmenting fluid immiscible with said liquid, and said outlet of said first passageway conduit being adapted to discharge segmented liquid, and means in said first conduit, downstream from said second conduit outlet, creating a predetermined resistance to flow of said fluid from the last-mentioned outlet, thereby determining the size and frequency of the segments of said immiscible fluid.

2. A method of injecting a fluid into a stream of liquid immiscible therewith for utilization in a sample analysis system comprising: flowing a first stream constituted by a liquid under pressure on a path toward an analysis utilization conduit, injecting axially into said first stream a second stream flowing in the same direction as said first stream to segment the latter,

which second stream is constituted by a fluid under pressure which is immiscible with said liquid, and creating by confinement a predetermined resistance to flow of said first stream in the area where said second stream is injected into said first stream, thereby determining the size and frequency of the segments in said first stream.

3. The method of claim 2, wherein: there is provided a conduit for said second stream having an inlet and having an outlet, and said first stream is flowed along and around the outlet portion of the last-mentioned conduit.

4. The method of claim 2, wherein said second stream is injected concentrically into said first stream.

5. A method of injecting a fluid into a stream of liquid immiscible therewith for utilization in a sample analysis system comprising: flowing a first stream constituted by a liquid under pressure on a path toward an analysis utilization conduit, injecting axially into said first stream a second stream flowing in the same direction as said first stream to segment the latter, which second stream is constituted by a gas under pressure which is immiscible with said liquid, and creating by confinement a predetermined resistance to flow of said first stream in the area where said second stream is injected into said first stream, thereby determining the size and frequency of the segments in said first stream.

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