March 22, 1966 A. FERRARI 3,241,923

METHOD AND APPARATUS FOR THE TREATMENT OF LIQUIDS

Filed Sept. 27, 1960 2 Sheets-Sheet 1

Fig. 1.

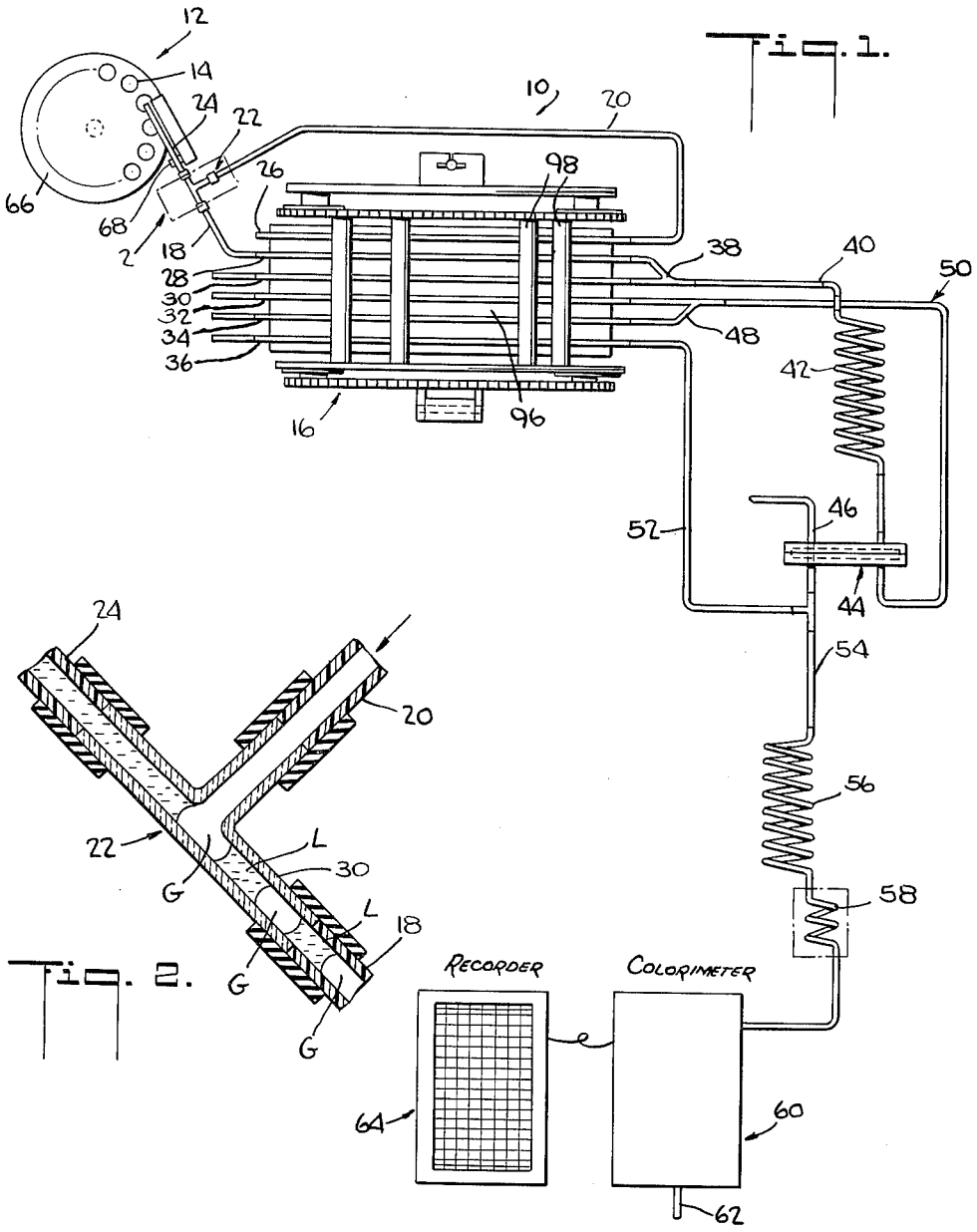


Fig. 2.

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METHOD AND APPARATUS FOR THE TREATMENT OF LIQUIDS

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Fig. 3.

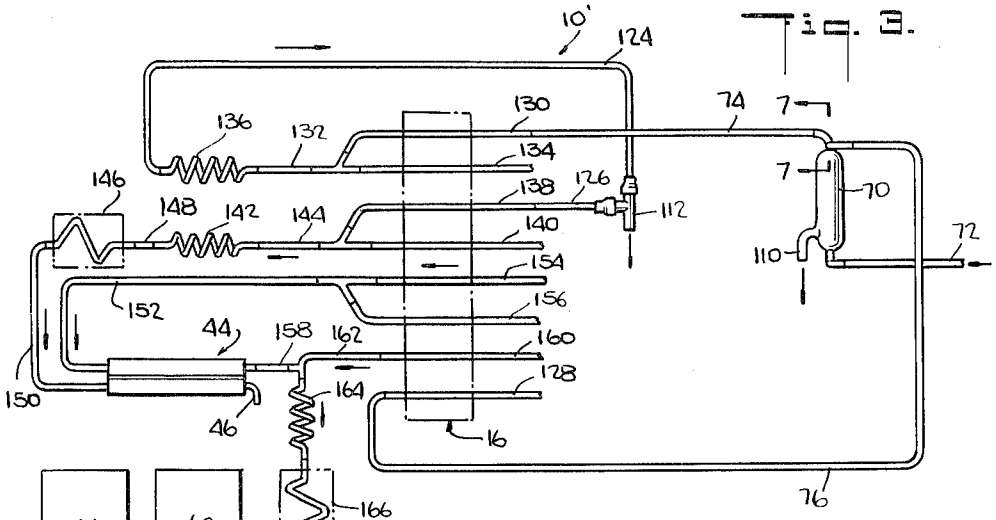


Fig. 6.

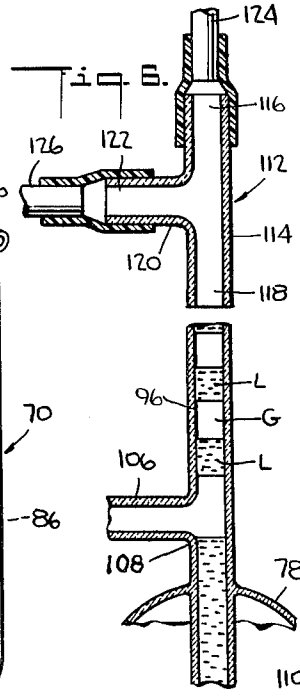


Fig. 5.

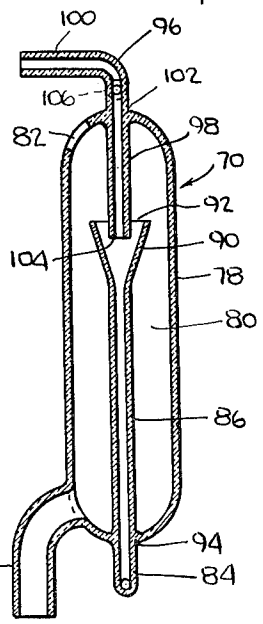


Fig. 4.

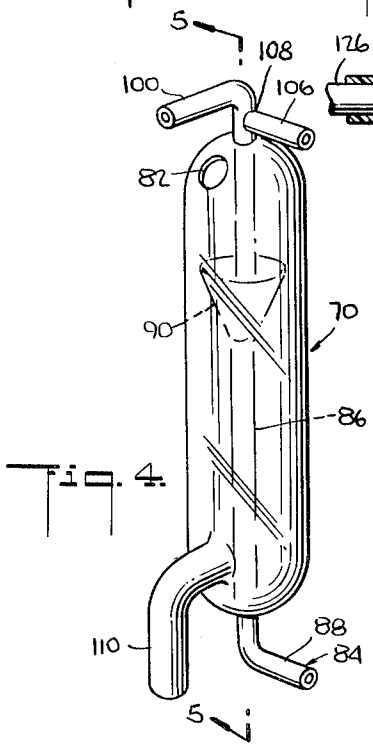


Fig. 7.

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United States Patent Office 3,241,923
Patented Mar. 22, 1966

3,241,923

METHOD AND APPARATUS FOR THE TREATMENT OF LIQUIDS

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Filed Sept. 27, 1960, Ser. No. 55,871
6 Claims. (Cl. 23—259)

This application is a continuation-in-part of Serial No. 849,785 filed Oct. 30, 1959 and now Patent No. 3,072,442 issued Jan. 8, 1963.

This invention relates to the treatment of liquids for analysis, monitoring, or other processing.

Apparatus especially useful for the treatment of a liquid stream for analysis is described in United States Patents Nos. 2,797,149 and 2,899,280, owned by the assignee of the present invention. As described in said patents, a series of samples of the liquids under analysis are transmitted by a pump in a flowing liquid stream through a dialyzer, and air is injected into the stream before it reaches the dialyzer to divide the stream into a series of liquid segments separated from each other by intervening segments of air. The air is introduced into the liquid stream after it leaves the pump, i.e., downstream of the pump, and the air segments are effective to cleanse the inner walls of the tubular passages through which the sample liquid stream flows from the pump and thereby prevent or greatly reduce contamination of one liquid sample by a preceding liquid sample.

I have found that in some instances contamination of one sample by another or the occurrence of spurious peaks on the graph of the recorder may take place to some extent notwithstanding the cleansing of the tubular passages leading from the pump. Although such contamination or the occurrence of spurious peaks on the graph does not ordinarily interfere with the operation of the analysis apparatus or with the reliability of the results of the analysis, it is highly desirable to eliminate or decrease the possibility of even this infrequent and slight contamination. I have discovered that optimum results can be achieved in this respect, by introducing the segmentizing air, or other inert gas, into the liquid sample in the tubular passage before the liquid is transmitted through the pump, preferably in such manner that the liquid is segmentized as soon as it is introduced into the tube leading to the inlet side of the pump, thereby preventing or greatly reducing the possibility of adhesion of particles of the liquid to the wall of the tube during the flow of the sample to the pump.

The improved cleansing action thus obtained constitutes a prime object of this invention.

The introduction of air or an inert gas into the sample liquid stream in the manner set forth above is desirable not only in the analysis of blood and other body fluids but is also useful in other processes, for example, in the treatment of a liquid stream for monitoring a manufacturing process wherein the liquid treated may be contaminated with finely divided materials, for example, diatomacious earth from filters in sugar manufacturing processes. Since the tubular passages or conduits of the treatment apparatus have very small diameters, for example, diameters in the order of about 0.030 inch to 0.110 inch, clogging of these tubular passages due to settling out of these finely divided materials is a serious problem. By introducing the air into the liquid stream being treated in the manner indicated, such settling out of the finely divided materials is eliminated.

A further object of the present invention is the provision of an improved liquid supply device which is especially suitable for introducing the air or gas into the liquid stream being treated for analysis or monitoring.

Another object is to provide an apparatus or system of the above indicated type in which a segmented liquid stream is supplied and wherein said liquid stream is diluted at an early or preliminary stage in the treatment, with means for separating a portion of the liquid from the stream whereby to reduce the quantity of liquid thereafter subject to further treatment without, however, impairing segmentation of the separated portion of the liquid stream subject to such treatment.

The above and other objects, features and advantages of the present invention will be fully understood from the following description of the invention considered in connection with the accompanying illustrative drawings.

In the drawings:

FIG. 1 is a more or less diagrammatic view of a liquid treatment apparatus or system embodying the present invention;

FIG. 2 is a fragmentary sectional view, on a larger scale, of the portion of the apparatus of FIG. 1 which is enclosed by the broken line and which is designated by the arrow 2;

FIG. 3 is a more or less diagrammatic view of a liquid treatment apparatus or system illustrating another embodiment of the present invention;

FIG. 4 is a perspective view, on a larger scale, of a liquid supply device which is used in the apparatus or system of FIG. 3;

FIG. 5 is a vertical sectional view taken on line 5—5 of FIG. 4;

FIG. 6 is a vertical sectional view, on a larger scale, of a separating device which is used in the apparatus or system of FIG. 3; and

FIG. 7 is a detail fragmentary sectional view, on a larger scale, taken on line 7—7 of FIG. 3.

Referring now to the drawings in detail and particularly to FIG. 1 thereof, the liquid treatment apparatus or system 10 comprises a sample liquid feed device 12 which includes a plurality of individual receptacles 14 which are arranged in a circular row and which contain the liquid samples to be treated. A peristaltic proportioning pump 16 or other suitable pump is provided for aspirating a portion of the liquid sample in each receptacle from feed device 12, through conduit 18, to the treatment apparatus and the pump has provision for transmitting a stream of air or other inert gas to the aspirated sample liquid stream, via conduit 20, to divide the stream into a series of liquid and gas segments. More particularly, a T-fitting or three legged junction member 22 is provided in the tubular passage leading to the pump 16, preferably adjacent to the intake tube 24 of the feed device 12. One end of the fitting is connected to the intake tube 24 and the opposite end of said fitting is connected to conduit 18 while the intermediate leg of the fitting is connected to conduit 20.

Pump 16 comprises a plurality of resiliently flexible tubes including tube 26 which is connected to tube 20 for supplying air or other segmentizing or cleansing liquid to the fitting 22. The tube 18 which is connected to the outlet end of the fitting is connected to the pump tube 28. The other resiliently flexible pump tubes 30, 32, 34 and 36 are provided for supplying various fluids in the operation of the apparatus as described, for example, in the above mentioned Patent No. 2,899,280. A fitting 38 is connected to the outlet ends of pump tubes 28 and 30 and to a conduit 40 for transmission of the segmentized sample stream together with a processing liquid through a mixing coil 42 and from the latter to the inlet of the dialyzer 44 at one side of the membrane thereof. The outlet for the fluid stream at said side of the dialyzer is indicated at 46. Pump tube 32 for a processing liquid and pump tube 34 for air or an inert gas, to form a

segmented recipient stream which passes through the other side of the dialyzer, are connected to a fitting **48** which is connected to a conduit **50** for transmitting the recipient stream to the dialyzer, and pump tube **36** for another processing liquid, which may be required, is connected to the tube **52** which in turn is connected to the conduit **54** by which the recipient stream containing the dialyzed ingredient of the sample liquid is transmitted from the dialyzer to the coils **56** and **58** and from the latter through the flow cell (not shown) of the colorimeter **60**, the outlet of said flow cell being indicated at **62**. The recorder **64** is operated under the control of the colorimeter **60**. It will be understood that the sample liquids and the processing liquids transmitted by the pump **16** through the dialyzer and other parts of the apparatus depend upon the liquids to be analyzed or treated and since the present invention does not involve any one particular analysis or liquid treatment but is of general application, a detailed description of a particular analysis or specific treatment is considered unnecessary. In this connection, it will be understood, however, that the present apparatus as illustrated by FIG. 1 may be employed, for example, in the manner and for the purposes described in the above mentioned Patent No. 2,899,280. It will be noted that in accordance with the present invention and pursuant to the above stated prime object thereof the segmentizing or cleansing fluid, instead of being introduced into the liquid passage after the transmission of the sample liquid through the pump tube, is introduced into the tubular passageway which conducts the sample liquid to the inlet side of the pump.

Referring now to FIG. 2, it is seen that when pump **16** is operated, a stream of sample liquid is aspirated from one of the receptacles **14** and flows as a continuous liquid stream through intake tube **24** to fitting **22** and concurrently with the flow of the liquid, a stream of air or inert gas is transmitted by the pump, via conduit **20**, to the fitting to divide the stream into a segmentized stream consisting of a series of liquid segments L separated from each other by intervening segments G of air or other inert gas which may be supplied to tube **20**, and this segmented stream is transmitted by the action of the pump from the fitting to conduit **18** and the liquid treatment apparatus. As indicated previously, the air or other gas segments G are effective to keep the inner walls of the tubular passages of the apparatus clean of contaminants from preceding samples of liquid or portions of the preceding samples themselves which may deposit on the walls of the tubular passages and thereby possibly interfere with the accuracy of the results of the analysis.

The feed device **12** illustrated in FIG. 1 is preferably of the type shown in the United States patent No. 3,038,340 issued June 12, 1962 and assigned to the assignee of the present application. Briefly described, the receptacles **14** are mounted on an apertured plate **66** which is intermittently rotated to position each receptacle under the intake tube **24** for the withdrawal of portions of the liquid samples from each of the receptacles, in succession. The intake tube **24** is mounted for pivotal movement at **68** and is operated during the rotation of the plate **66** to move into and out of each succeeding receptacle. In this regard it should be understood that as intake tube **24** is moved out of the receptacle so that a succeeding receptacle may be moved into position underneath the intake tube, said intake tube aspirates air because it is not immersed in the liquid in the receptacle. Accordingly, the aspirated portion of the sample from each receptacle is separated from a portion of the sample from a succeeding receptacle by an intervening segment of air and because of the introduction of the gas or air into the sample stream, by the use of fitting **22** as previously described, each liquid sample is divided into a series of liquid segments separated from each other by intervening segments of gas. Thus, not only is each sample separated from each other by an air segment but each individual sample itself is subdivided into a series of liquid segments separated by intervening segments of gas or air.

Referring now to FIG. 3 of the drawings, there is shown another treatment apparatus **10'**, according to the invention, which is especially suitable for monitoring a manufacturing process wherein a stream of liquid from the manufacturing process is treated for analysis concurrently with the operation of the manufacturing process. The stream of liquid is transmitted to a liquid supply device **70** from the manufacturing apparatus, via a conduit **72** and a portion of the liquid is aspirated from the liquid supply device, by the action of the proportioning pump **16**, via a conduit **74**. Concurrently with the aspiration of the liquid from the supply device, a stream of air or other gas is transmitted through conduit **76** to divide the aspirated liquid stream into a series of liquid segments separated from each other by intervening segments of air or gas, so that a segmented stream is formed before the liquid reaches the pump **16** and, more particularly, the segmented stream is formed adjacent or near the position at which the liquid stream is introduced into the treatment apparatus through the supply device **70**. The division of the liquid stream into liquid and gas segments occurs in the same manner as was previously described with respect to FIG. 2 and is illustrated in FIG. 7 with respect to liquid supply device **70**.

As best seen in FIGS. 4 and 5, the liquid supply device **70**, which is preferably made of glass, comprises a tubular member or body portion **78** which forms a chamber **80** and is provided with a vent opening **82** therein for venting the chamber. The inlet **84** of the supply device comprises a tubular member **86** which has a nipple part **88** at the lower end thereof, said nipple part being connected to conduit **72** in fluid flow communication therewith, and a flared cone-shaped part **90** at the upper end thereof. Flared part **90** is provided at its top with an opening **92** which constitutes a liquid inlet for chamber **80**. Tubular member **86** is secured to member **78** and is preferably integral therewith, as at **94**, with nipple **88** disposed externally of the chamber **80** and the vertically extending part of tubular member **86** being concentrically disposed within chamber **80**, as shown.

The liquid supply device **70** has a liquid outlet or aspirating tube **96** which comprises a tubular member **98** provided with a nipple part **100** at the upper end thereof. Nipple **100** is disposed externally of chamber **80** and is connected to conduit **74** in fluid flow communication therewith. The outlet **96** is secured to member **78** and is preferably integral therewith, as at **102**. Below connection **102**, tubular member **98** is in vertical alignment with the vertical extending part of tubular member **86** and is provided at its end with an opening **104** which is disposed within flared part **90** and below the top opening **92** thereof. Opening **104** constitutes a liquid inlet for transmitting liquid pumped from chamber **80** and, more particularly, from the flared part **90** of inlet **84**. Since the opening **104** is below opening **92** and therefore below the level of the liquid in inlet **84**, air will not be introduced into the liquid through opening **104** as the liquid is aspirated from the chamber.

From the foregoing it will be seen that as pump **16** is operated, a portion of the liquid entering chamber **80**, via inlet **84**, will be aspirated therefrom, via outlet **96**, and transmitted to the liquid treatment apparatus via conduit **74**. Concurrently with the aspiration of the liquid from chamber **80**, the liquid stream is segmentized by the introduction of air or gas via the nipple connection **106** at the upper vertically extending part of tubular member **98** which is below the turn of the nipple part **100** of outlet **96**. Conduit **76** is connected to nipple **106** which is preferably integrally connected to outlet **96**, as at **108**. The remaining portion of liquid entering chamber **80** through inlet **84** will overflow at opening **92** into the chamber and be discharged therefrom via auxiliary out-

let 110 provided in member 78. The members 98, 106 and 100 form a three legged junction member.

If desired or as required, the treatment apparatus 10' illustrated in FIG. 3 may include a device 112 for separating a portion of the liquid in the liquid-gas segmented stream without impairing the segmentation thereof and the use of this separating device is especially desirable when the sample liquid must be diluted to reduce the proportionate concentration of materials present therein. Since the quantities of liquids used in the treatment apparatus are relatively small, such separation of a portion of the liquid in the segmented stream is desirable and often necessary.

Referring now to FIGS. 3 and 6, separating device 112 comprises a T-shaped tubular member or fitting, preferably made of glass, with the vertical crossbar 114 thereof provided with an inlet 116 and an auxiliary outlet 118. The horizontal leg 120 of the fitting provides an outlet 122. Inlet 116 is connected to a conduit 124 which transmits the segmented stream to the separating device and outlet 122 is connected to a conduit 126 for transmitting a portion of the liquid, as a segmented stream, to the apparatus for further treatment. Inlet 116 is larger than outlet 122 so that the rate of flow through the inlet is greater than the rate of flow through the outlet. Accordingly, with crossbar 114 of the separating device 112 disposed in a vertical position, as shown, a portion of the liquid in the segmented stream entering inlet 116 will be separated from the stream and discharged to waste through auxiliary outlet 118 without impairing the segmentation of the incoming stream. The remaining portion of the liquid, still segmentized, will be aspirated from separating device 112, via outlet 122, by the action of pump 16 and will be transmitted to the other parts of the treatment apparatus, via conduit 126, for further treatment in accordance with the particular analysis which the liquid is undergoing.

In the use of the method and apparatus illustrated by FIG. 3, for example with respect to determining the quantities of total sugars contained in waste and sewer waters from sugar manufactories, the liquid sample is transmitted to liquid supply device 70, via conduit 72, and a portion thereof is aspirated from the device through outlet 96 by the action of pump 16. This liquid sample may contain finely divided material, as for example diatomaceous earth from filters of the sugar manufactory, and the introduction of air or other inert gas into the aspirated liquid stream, via pump tube 128 and conduit 76, is effective to prevent the finely divided material from settling out and clogging the various tubular passages of the apparatus or otherwise contaminating the fluids so that erroneous analysis results which are sometimes recorded due to this contamination are obviated.

The segmented fluid stream is transmitted, via conduit 74 and pump tube 130, to conduit 132 where it joins a stream of a diluent, such as water, supplied via pump tube 134. The joined segmented fluid stream is transmitted to a horizontal helical mixing coil 136 where the diluent is thoroughly mixed with the liquid segments of the segmented fluid stream and the concentrations of the various materials in the liquid are reduced. From the mixing coil 136, the mixed and diluted segmented fluid stream is transmitted to separating device 112 and, as previously described, a portion of the stream is separated and discharged through auxiliary outlet 118 and concomitantly with this separation a substantial part of the finely divided material in the fluid stream is also discharged through the auxiliary outlet without impairment of the segmentation of the stream. The remaining portion of the segmented fluid stream is aspirated from device 112, via outlet 122 thereof, and is transmitted to pump tube 138, via conduit 126, and joins a stream of a suitable acid, as for example, 0.25N solution of hydrochloride acid (HCl), supplied to pump tube 140. The segmented fluid stream containing the acid is then transmitted to a helical mixing coil 142, via conduit 144, wherein the acid is thoroughly mixed in each of the liquid segments of the stream. From the mixing coil the mixed stream is transmitted to a heating bath 146, via conduit 148. In the heating bath, the liquid sample is hydrolized to invert the non-reducing sugars contained in the waste and sewer water samples to reducing sugars, the treatment of the sample being for the determination of the total quantities of the sugars contained therein.

From the heating bath the hydrolized and segmented fluid stream is transmitted, via conduit 150, to the tubular passage at one side of the membrane of the dialyzer 44. The dialyzer membrane acts to hold back contaminants found in the sample waste and sewer waters and provides a convenient and advantageous means to diffuse at least a portion of the total sugars contained in the sample liquid, the quantities of the sugars diffused being in proportion to the quantities contained in the liquid sample.

Concurrently with the transmission of the hydrolized liquid sample through the dialyzer, a color processing fluid is transmitted to the dialyzer at the other side of the dialyzer membrane, via conduit 152, to provide a recipient liquid stream into which some of the sugars contained in the waste and sewer waters sample diffuse. The color processing fluid is, for example, a 0.1% solution in water of potassium ferricyanide $K_3Fe(CN)_6$ and is supplied to pump tube 154, air being supplied to pump tube 156 for segmentizing the stream of potassium ferricyanide. The quantity of potassium ferricyanide employed is not critical but enough potassium ferricyanide should be used for the reduction thereof to potassium ferrocyanide by all of the diffused sugars contained in the sewer or waste liquids being analyzed, and leave a residual amount of potassium ferricyanide which is measured and is indicative of the suger present.

The segmentized recipient stream containing potassium ferricyanide and the diffused sugars is transmitted from the dialyzer, via conduit 158, the fluid sample stream being discharged to waste from the dialyzer, via outlet 46.

A suitable alkali, such as for example, a 0.3N solution of sodium hydroxide is supplied to pump tube 160 and is transmitted by the action of the pump to conduit 162 where it joins the segmented stream containing the diffused sugars and the potassium ferricyanide solution. The alkali neutralizes any excess acid existing in the sample fluid stream due to the introduction of the acid reagent for the hydrolyzing phase of the treatment process.

The segmented sample fluid stream containing diffused sugars, potassium ferricyanide and sodium hydroxide is transmitted to a helical mixing coil 164, where the constituents of the stream are thoroughly mixed, and then to a heating bath 166, preferably operated at a temperature of 95° C., wherein the sugars in the fluid stream react with the potassium ferricyanide to reduce said potassium ferricyanide to potassium ferrocyanide, the resulting color change being proportional to the quantities of the sugars contained in the waste and sewer waters. The reacted fluid stream is then transmitted to the colorimeter 60, via conduit 168, for measurement as previously explained.

A fluid treatment apparatus for determining the quantities of total sugars in waste and sewer waters having some of the features shown therein is illustrated and described in my co-pending application, Serial No. 799,884 filed Mar. 17, 1959 and now Patent No. 3,116,754, and assigned to the assignee of the present application.

It will be understood that although the invention has been described with respect to segmentizing air or other inert gas, it is within the scope of the present invention to employ for segmentizing the sample liquid stream a fluid in liquid form, i.e., a liquid which is inert to and immiscible with the sample liquid stream.

While I have shown and described the preferred embodiment of my invention, it will be understood that the

invention may be embodied otherwise than as herein specifically illustrated or described, and that certain changes in the form and arrangement of parts and the specific manner of practicing the invention may be made without departing from the underlying ideas or principles of this invention within the scope of the appended claims.

I claim:

1. A method of continuously cleansing a peristaltic pump tube which is utilized for pumping a sample liquid stream of variable constituency, comprising: pumping a gas through an additional tube in the peristaltic pump; concurrently introducing the pumped gas from the additional tube into the sample liquid stream upstream of the first mentioned peristaltic pump tube to divide the sample liquid stream into a series of liquid segments separated from each other by intervening segments of gas.

2. Apparatus for treating a liquid, comprising: a plurality of receptacles, each for containing a sample liquid; a feed device for supporting said receptacles; a first and a second peristaltic pump tube, each having an inlet and an outlet; peristaltic pump means for engaging said tubes for advancing liquids through said tubes; an off-take device coupled to said inlet of said first tube for removing sample liquid from each of said receptacles seriatim and for introducing this liquid as a stream through said first tube; a source of an inert fluid coupled to said inlet of said second tube; said outlet of said second tube being coupled to said inlet of said first tube, for introducing such inert fluid into such stream of liquid to divide the stream of liquid in said first tube into a series of flowing liquid segments separated from each other by intervening flowing inert fluid segments.

3. Apparatus for treating a liquid, comprising: a plurality of receptacles, each for containing a sample liquid; a feed device for supporting said receptacles; a first and a second peristaltic pump tube, each having an inlet and an outlet; peristaltic pump means for engaging said tubes for advancing liquids concurrently through said tubes; a first tubular three legged junction member; an off-take device coupled to one leg of said member and said inlet of said first tube coupled to a second leg of said member for removing sample liquid from each of said receptacles seriatim and for introducing this liquid as a stream through said first tube; a source of an inert fluid coupled to said inlet of said second tube; said outlet of said second tube being coupled to a third leg of said member for introducing such inert fluid into such stream of liquid to divide the stream of liquid in said first tube into a series of flowing liquid segments separated from each other by intervening flowing inert fluid segments.

4. Apparatus for treating a liquid, comprising: a first and a second peristaltic pump tube, each having an inlet and an outlet; peristaltic pump means for engaging said tubes for advancing liquids through said tubes; a source of sample liquid coupled to said inlet of said first tube for introducing this liquid as a stream through said first tube; a source of an inert fluid coupled to said inlet of said second tube; said outlet of said second tube being coupled to said inlet of said first tube, for introducing such inert fluid into such stream of liquid to divide the stream of liquid in said first tube into a series of flowing liquid segments separated from each other by intervening flowing inert fluid segments.

5. Apparatus for treating a liquid, comprising: a first and a second peristaltic pump tube, each having an inlet and an outlet; peristaltic pump means for advancing liquids through said tubes; a source of sample liquid coupled to said inlet of said first tube for introducing this liquid as a stream through said first tube; a source of an inert fluid coupled to said inlet of said second tube; said outlet of said second tube being coupled to said inlet of said first tube, for introducing such inert fluid into such stream of liquid to divide the stream of liquid in said first tube into a series of flowing liquid segments separated from each other by intervening flowing inert fluid segments; and means coupled to said outlet of said first tube, for dividing the segmented stream therefrom into two streams without impairing the segmentation thereof, including a three legged junction tubular member having a first leg thereof serving as an inlet and coupled to said first tube, a second leg thereof serving as a first outlet, and a third leg thereof serving as a second outlet, the cross sectional area of said inlet being larger than the cross sectional area of said second outlet so that the volumetric rate of flow through said inlet is greater than the volumetric rate of flow through said second outlet, whereby a portion of the stream normally flows through said second outlet and the remaining portion flows through said first outlet.

6. Apparatus for treating a liquid, comprising: a first and a second peristaltic pump tube, each having an inlet and an outlet; peristaltic pump means for advancing liquids concurrently through said tubes; a first tubular three legged junction member; a source of sample liquid coupled to a first leg of said member and said inlet of said first tube coupled to a second leg of said member for introducing this liquid as a stream through said first tube; a source of an inert fluid coupled to said inlet of said second tube; said outlet of said second tube being coupled to a third leg of said member for introducing such inert fluid into such stream of liquid to divide the stream of liquid in said first tube into a series of flowing liquid segments separated from each other by intervening flowing inert fluid segments; and means coupled to said outlet of said first tube, for dividing the segmented stream therefrom into two streams without impairing the segmentation thereof, including a second three legged junction tubular member having a first leg thereof serving as an inlet and coupled to said outlet of said first tube a second leg thereof serving as a first outlet, and a third leg thereof serving as a second outlet, the cross sectional area of said inlet being larger than the cross sectional area of said second outlet so that the volumetric rate of flow through said inlet is greater than the volumetric rate of flow through said second outlet, whereby a portion of the stream normally flows through said second outlet and the remaining portion flows through said first outlet.

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