Nov. 1, 1966 A. FERRARI ETAL 3,282,651

SAMPLE AND REAGENT AND/OR WASH LIQUID SUPPLY APPARATUS

Filed July 12, 1963

FIG. 1

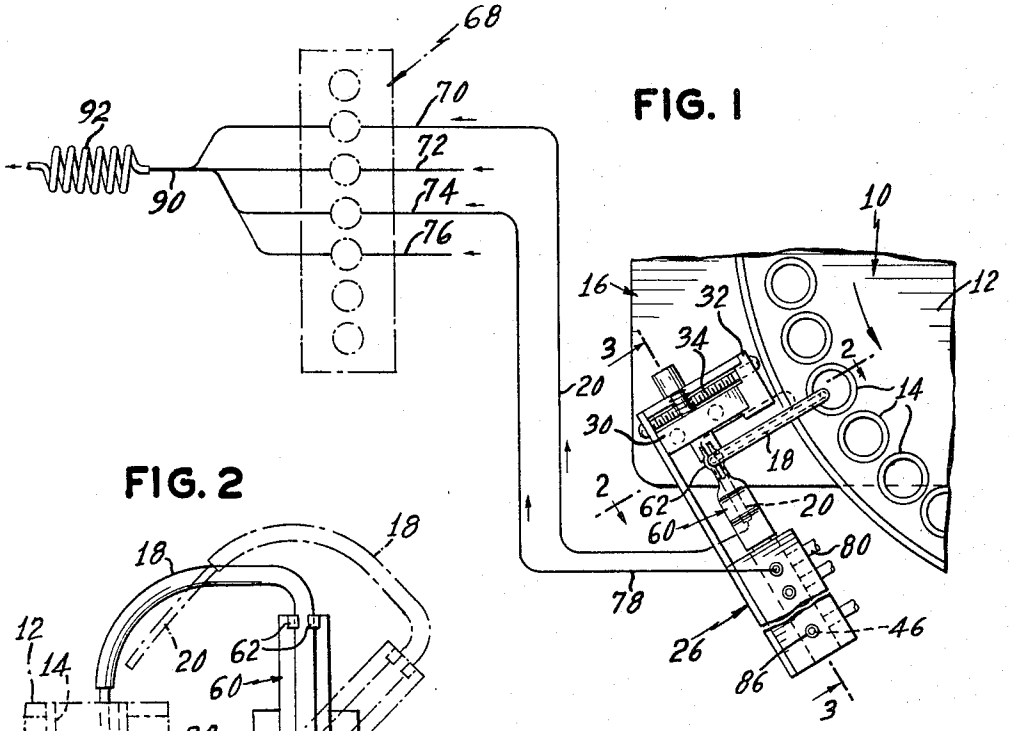


FIG. 2

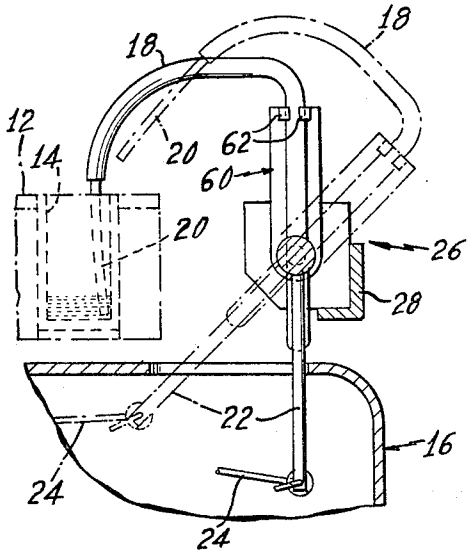


FIG. 3

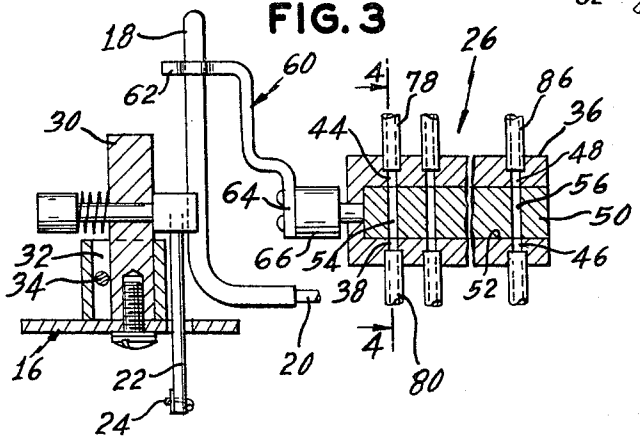
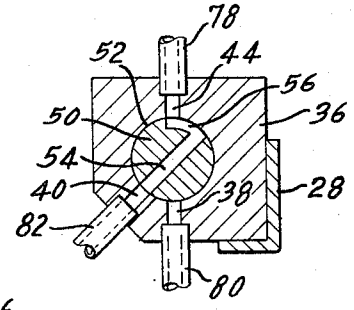


FIG. 4



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SAMPLE AND REAGENT AND/OR WASH LIQUID SUPPLY APPARATUS

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This invention relates to an automatic analysis apparatus or system for quantitatively analyzing a series of sample liquids in respect to a known substance therein, the samples being transmitted one after another in stream-like fashion from automatic sample supply apparatus and treated with a reagent for analysis while flowing in the system.

The primary object of the invention is to provide means for automatically interrupting the supply of reagent to the system when the flow of sample liquid from the sample supply apparatus is interrupted.

Another object is the provision of means to automatically supply a wash or other liquid to flow through the system instead of the reagent, during the intervals between the flow of sample liquids and reagent, whereby to cleanse the tubing and other parts of the analysis apparatus or at least in some cases to inhibit an undesirable reaction or contamination.

Pursuant to another object of the invention, provision is made to automatically introduce wash liquid into the sample supply tube itself when sample flow is interrupted.

The above and other objects, features and advantages of the invention will be fully understood from the following description of the presently preferred mode of practicing the invention, reference being had to the accompanying drawings, in which:

FIG. 1 is a plan view of apparatus embodying the present invention showing a part of the sample supply apparatus and illustrating schematically a pump and tubes which constitute parts of the analysis apparatus;

FIG. 2 is a view partly in elevation and partly in section along the line 2—2 of FIG. 1;

FIG. 3 is a sectional view along the line 3—3 of FIG. 1; and

FIG. 4 is a sectional view along the line 4—4 of FIG. 3.

The sample supply apparatus, a part of which is indicated at **10** in FIG. 1, is fully described in the United States patent of Jack Isreeli, No. 3,038,340, issued to and owned by the assignee of this application. As this sample supply apparatus is well known in the art, as well as being described in said patent, and does not in itself form part of the invention, it will be described only to the extent necessary to the understanding of its relation to the invention. Said supply apparatus, hereinafter sometimes referred to as a sampler, comprises a circular plate **12** provided with a series of openings in which cups **14** to contain the liquids to be analyzed are supported. Plate **12** is turned intermittently by motor actuated mechanism (not shown) disposed within the housing **16** to bring each cup **14** to a liquid takeoff station at which a tubular member or crook **18** forming part of a liquid intake device is positioned. Takeoff tubing **20** (FIGS. 2 and 3), usually polyethylene tubing, is threaded through the crook and has an inlet end portion which is moved by the crook into and out of the cup **14** at the takeoff station while plate

12 is stationary. The crook is carried by a rod **22** which is mounted for pivotal movement to and from the operative and retracted positions shown in full and broken lines, respectively, in FIG. 2 while plate **12** is turned to move the cups **14** to and from the takeoff station. A link **24** connected to rod **22** is actuated by mechanism within the sampler housing **16**, as described in said patent, to move the crook tube **20** into the cup **14** at the takeoff station as soon as plate **12** comes to rest and to move crook tube **20** out of said cup and clear of the latter at the end of the takeoff operation just before plate **12** is turned to advance the next cup to the takeoff station.

In accordance with the present invention, provision is made for interrupting the flow of reagent in the system when the flow of the sample liquid is interrupted, i.e., when the crook tube **20** is withdrawn from a cup **14** at the takeoff station. For this purpose valve means **26** is utilized. Said valve is carried by a bracket **28** which is secured in position on housing **16**, adjacent the sample takeoff station, by clamping said bracket against a fixed block **30** on said housing by the clamping member **32** operated by the clamping screw **34**.

Valve means **26** comprises the valve housing **36** having fluid inlet openings **38** and **40** which are in circumferentially spaced relation (FIG. 4) and a common fluid outlet opening **44**. A cylindrical valve member **50** is mounted for turning movement in the cylindrical bore **52** of housing **36** and has a cross fluid passage **54** which is adapted to register with inlet opening **38** in one position of the valve and with inlet opening **40** by way of lateral passage **56** in another position of said valve for control of flow of various liquids as is obvious or will become apparent as the description proceeds.

The valve **50** is turned to its fluid-flow controlling positions by the pivotal movement of crook **18** when crook supporting rod **22** is pivoted by actuation of link **24**. The valve turning means comprises a member **60** which has a forked end **62** which receives the crook, for actuation by the latter, and an opposite end **64** which is connected to the valve stem part **66** for turning the valve about its longitudinal axis when the crook is turned.

The operation of the valve will now be described with reference to the proportioning pump **68** and tubing of the analysis apparatus. The pump **68**, which is schematically indicated in FIG. 1, is of the resiliently compressible tube type and is preferably of the construction described in United States Patent No. 2,893,324 issued to and owned by the assignee of this application. Briefly described, said pump comprises a plurality of resiliently compressible tubes, of selected internal diameters depending upon the desired liquid or other fluid proportions, and a plurality of pressure rollers which engage said tubes and move longitudinally on the tubes for compressing them progressively along their lengths for transmitting the fluids through the tubes to other modules of the analyzer. Several of the resiliently compressible tubes of the pump are shown schematically at **70**, **72**, **74** and **76**. The outlets of said tubes are connected by a tubular coupling **90** to a horizontally disposed helical mixing tube **92** in which mixing of the liquids occurs during their flow through said coil.

Sample liquid to be analyzed may be supplied directly from the crook tubing **20** to the pump tube **70**. A liquid diluent, for example distilled water, is suppled to pump

tube 72 for diluting the sample liquid. A liquid reagent is supplied under the control of valve 26 to a delivery tube 78 which is connected to pump tube 74. Air is introduced through pump tube 76 for segmentizing the liquid stream whereby to aid in cleansing the walls of the tubing, as explained in United States Skeggs Patent No. 2,797,149, owned by the assignee of this application.

As explained in detail in said Skeggs patent, the sample liquid is treated for colorimetric analysis and for this purpose a suitable reagent employed to develop the color is added to the sample liquid. In accordance with the present invention, provision is made for interrupting the flow of reagent to tube 78, which is connected to pump tube 74, when the inlet end of the crook tube 20 is withdrawn from a sample cup 14, and for supplying water or other wash liquid to tube 78 and from the latter to pump tube 74 instead of the reagent if so desired. The supply tube for the reagent is indicated at 80 and communicates with the inlet opening 38 and valve passage 54 and through the latter with valve outlet 44 to which reagent delivery tube 78 is connected. When the inlet end of the crook tube 20 is out of the cup 14, valve 50 is in the position illustrated by FIG. 4, closing reagent inlet 38 and opening inlet 40 so that wash water or other liquid can flow through valve passage 54 through outlet 44 to tube 78 so that the wash water or another liquid instead of the reagent is supplied to pump tube 74 transmitted from supply tube 82.

As shown by FIG. 3 and FIG. 4, valve 26 is provided with inlet and outlet openings, in addition to those described above, for various purposes. For example, instead of connecting the crook tube 20 directly to the pump tube 70 by which the sample liquid is delivered to the analyzer system, said tube 20 can be connected to the valve inlet 46 for supply to the pump tube 70 which can be connected to the outlet 48 by a tube, a part of which is indicated at 86 in FIG. 3. When valve 50 closes inlet 46 by the movement of crook 18 to the position in which the inlet end of tube 20 is withdrawn from the supply cup 14, the valve passage 56 is connected to another inlet similar to inlet 82, not shown, for the flow of wash water or other liquid to the outlet 48 and from the latter to a tube connected to the sample supply tube 86 so that substantially all of the sample tubing may be cleansed during the periods of sample-flow interruption at which time the supply of reagent is also discontinued. It will be understood that valve inlet 46, the other inlet, the passage 56 and outlet 48 are arranged in the relation described above with reference to FIG. 4.

It will be understood that the present invention does not involve and is not limited to any particular analysis or the chemistry thereof. Among the advantages of the invention is the obviously important advantage that less reagent is consumed since it is supplied only when sample liquid is supplied. Another advantage of discontinuing the introduction of the reagent is that in certain analyses the continuous flow of reagent into the system might cause improper function on a long-term operation by deposition of the reagent on the inner surfaces of the tubing and other parts of the apparatus, for example deposits from microbial substrate during a microbiological assay.

Another advantage derivable by the use of the device of the present invention resides in the provision for introducing an inhibitor liquid into the system during the nonsampling periods. An example of this last mentioned advantage is the introduction of formaldehyde or other inhibitor in enzyme determinations, in the determination of amino acid with enzymes (decarboxylase), as in the analysis of l-lysine and d-glumatic acid, as well as in antibiotic assays in microbiological systems. In these provisions for introducing an inhibitor, as distinguished from a wash liquid, the inlet 46 would be connected to a supply of the inhibitor, while the outlet 48 would be connected to pump tube 70 by tube 86 so that the pump 68 would aspirate the inhibitor liquid during nonsampling periods.

While we have shown and described the preferred embodiment of the 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 in the specific manner of practicing the invention may be made without departing from the underlying idea or principles of this invention within the scope of the appended claims.

What is claimed is:

1. Automatic analysis apparatus for analyzing a plurality of liquids comprising: a sampler including a plurality of sample containers each adapted to contain a liquid sample, off-take means having an off-take tube with an inlet end, which is adapted to enter a said container, and an outlet end, support means for supporting said sample containers, for sequentially presenting said sample containers to said off-take means and for inserting said inlet end of said off-take tube in a presented sample container; sample treating means including mixing means having an inlet and an outlet, pump means having a plurality of pumping conduits each having an inlet and an outlet respectively, said outlets of said conduits being coupled to said inlet of said mixing means; said outlet end of said off-take tube being coupled to the inlet of one of said pump means conduits, whereby when said inlet end of said off-take tube is disposed within a sample container containing a liquid sample, such sample is off-taken and transmitted to said mixing means; valve means having an inlet for coupling to a source of treating liquid and an outlet coupled to the inlet of another one of said pump means conduits, whereby when said inlet is open to said outlet, treating liquid is transmitted to said mixing means; and actuating means for said valve means coupled to said support means for opening said inlet of said valve means in a predetermined phase relationship with the insertion of said inlet end of said off-take tube into a sample container and for closing said inlet of said valve means in a predetermined phase relationship with the removal of said inlet end of said off-take tube from such a sample container.

2. Apparatus according to claim 1 wherein said actuating means opens and closes said inlet of said valve substantially concurrently with the respective insertion into and removal from a sample container of said off-take tube inlet end.

3. Apparatus according to claim 1 wherein said valve means includes an additional inlet for coupling to a source of additional liquid; and said actuating means alternatively opens said first mentioned inlet to said outlet and closes said additional inlet, and closes said first mentioned inlet to said outlet and opens said additional inlet.

4. Apparatus according to claim 1 wherein said valve and actuator means comprise a lever operated multiport plug valve.

5. Automatic analysis apparatus comprising a sampler including a plurality of sample containers each adapted to contain a liquid sample, off-take means having an off-take tube with an inlet end adapted to enter a said container and an outlet end, support means for supporting said sample containers, for sequentially presenting said sample containers to said off-take means and including transport means inserting said inlet end of said off-take tube in a presented sample container; sample treating means including mixing means having an inlet and an outlet; pump means having a plurality of pumping conduits each having an inlet and an outlet respectively, said outlets of said conduits being coupled to said inlet of said mixing means; valve means having a first inlet coupled to said outlet end of said off-take tube, a second inlet for coupling to a source of cleansing liquid, and an outlet coupled to the inlet of one of said pump means conduits, whereby when said inlet end of said off-take tube is disposed within a sample container containing a liquid sample, and said first inlet is open to said outlet, such sample

is off-taken and transmitted to said mixing means, and when said second inlet is open to said outlet, such cleansing liquid is transmitted to said mixing means; and actuating means for said valve means coupled to said transport means for said off-take tube, for alternatively opening said first inlet of said valve means in a predetermined phase relationship with the insertion of said inlet means of said off-take tube in a sample container and closing said second inlet, and for opening said second inlet in a predetermined phase relationship with the removal of said inlet end of said off-take tube from such a sample container and closing said first inlet.

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