Dec. 7, 1965 H. L. ASHMEAD 3,222,135

APPARATUS FOR THE PREPARATION OF FLUID SAMPLES

Filed Nov. 27, 1962 2 Sheets-Sheet 1

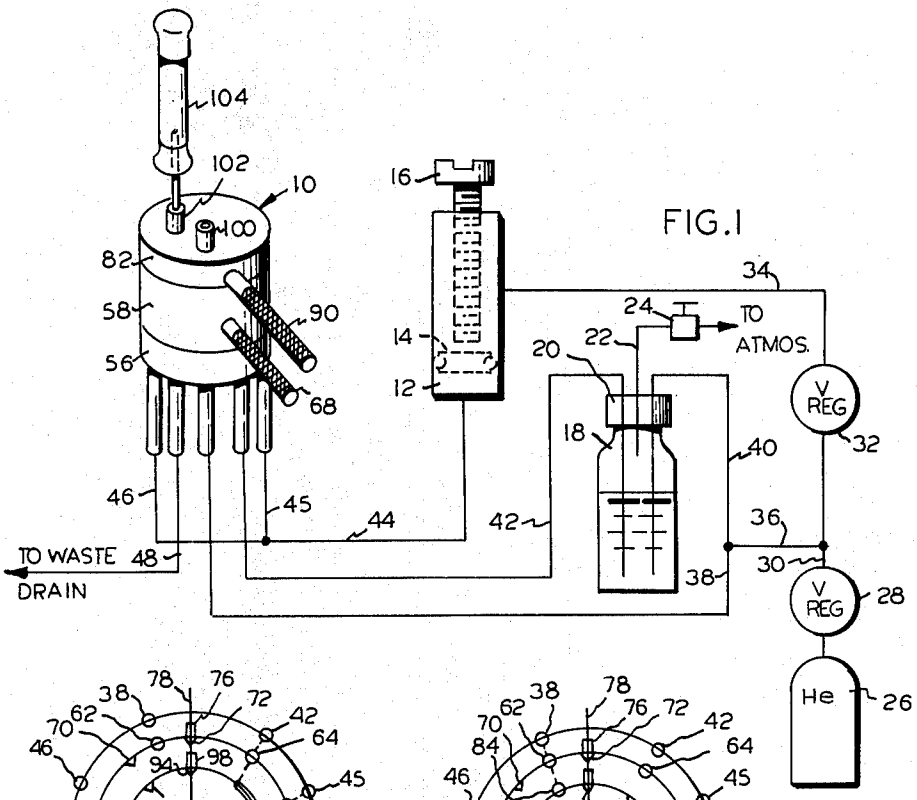


FIG.1

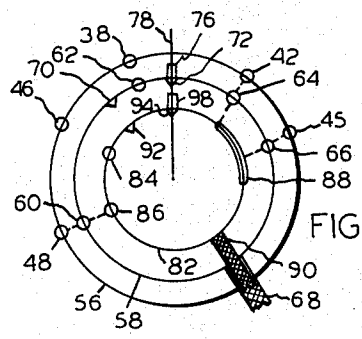


FIG.2

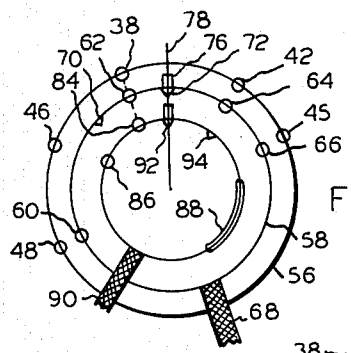


FIG.3

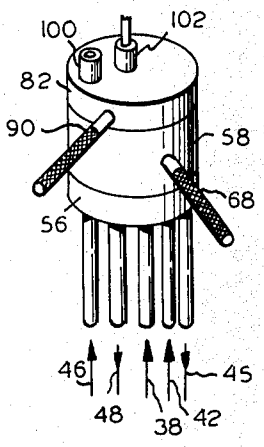


FIG.4

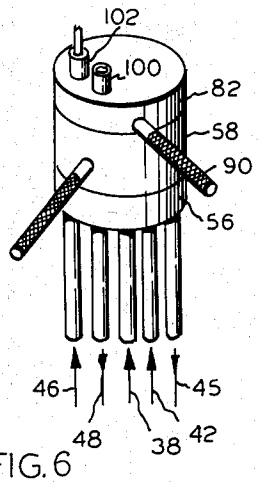


FIG.6

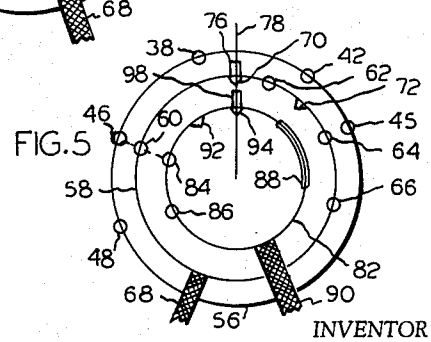


FIG.5

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APPARATUS FOR THE PREPARATION OF FLUID SAMPLES

Filed Nov. 27, 1962 2 Sheets-Sheet 2

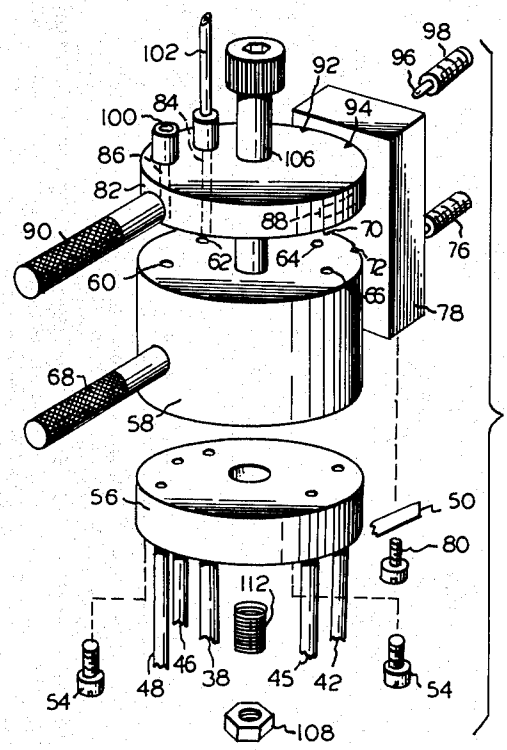


FIG. 7

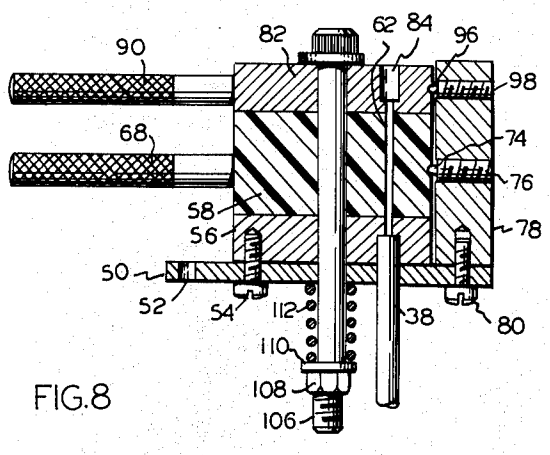


FIG. 8

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APPARATUS FOR THE PREPARATION OF FLUID SAMPLES

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Filed Nov. 27, 1962, Ser. No. 240,314
16 Claims. (Cl. 23—253)

This invention relates to fluid sample measuring apparatus particularly adapted for use in combination with devices for analyzing dissolved and/or chemically bound gases in fluid materials and more particularly to novel apparatus capable of consistently measuring a fluid sample of predetermined volume and injecting same along with releasing agent into a reaction capsule.

While the present invention may be utilized in the analyses of dissolved and/or chemically bound gases in various types of liquids, it will be described in connection with apparatus for analyzing dissolved and/or chemically bound gases in whole blood or serum. It is conventional procedure in the practice of a blood gas analysis to release the gases chemically and then determine the total volume of the released gases by a measuring instrument, such as a manometer. Another conventional procedure involves chemically releasing the gases from the blood while simultaneously stripping the gases from solution and injecting them into a gas chromatograph. However, such conventional procedures and the apparatus therefor are unduly complex; are incapable of injecting a measured volume of the sample; are subject to inefficiency because of the prolonged time necessary for a complete analysis, and often give inaccurate analyses because the rate of chemical reaction is not reproducible.

Accordingly, it is an object of this invention to provide means for accurately obtaining a measured sample of a fluid from which it is possible to obtain a complete gas analysis of said fluid sample in a relatively short time.

Another object of this invention is to provide gas analyzing apparatus with a sampling device operable to measure a volume of the sample to be analyzed.

It is another object of this invention to extract various blood gases by relatively simple and efficient apparatus.

This invention has another object in that a free piston valve or diaphragm device measures and injects releasing chemicals into the reaction chamber of a sample measuring apparatus.

A further object of this invention is to selectively position a sampling valve to perform purging or evacuation, sample loading, and reaction chamber loading operations in conjunction with apparatus for analyzing blood gases.

It is a further object of this invention to selectively control the various fluid flows in blood gas analyzing apparatus by means of a unitary multi-positionable valve device.

This invention has a further object in retaining the movable valve members of a blood sampling valve in selective positions by detent means.

In the preferred embodiment of the present invention, a reaction capsule disposed on a movable valve member is purged or evacuated by means of aligned bores in a second movable valve member and a valve block so as to be connected to a source of an inert purging gas such as helium and/or evacuating means such as a pump. By moving the first valve member to a selected position, a blood sample is injected into the sampling valve and releasing agents from a reservoir are conducted through the sampling valve to a free piston metering valve. Upon movement of the second valve member to another selected position, the reagents force the measured volume of the blood sample into the reaction capsule.

Other objects and advantages of the present invention

will become apparent from the following description of a preferred embodiment taken in connection with the accompanying drawings, wherein:

FIG. 1 is a schematic diagram of the entire apparatus embodying this invention;

FIG. 2 is a diagrammatic representation of the sampling valve of FIG. 1 and showing the relative positions of the communicating bores on a radical projection;

FIG. 3 is a diagrammatic representation similar to FIG. 2 except that the valve members are in another position;

FIG. 4 is a perspective view of the sampling valve shown in the position of FIG. 3;

FIG. 5 is a diagrammatic representation similar to FIG. 2 except that the valve members are shown in still another position;

FIG. 6 is a perspective view of the sampling device shown in the position of FIG. 5;

FIG. 7 is an exploded perspective view of the sampling valve of FIG. 1; and

FIG. 8 is a longitudinal section view of the sampling valve of FIG. 1.

Referring now to FIG. 1, the preferred embodiment of this invention includes a fluid sampler in the form of a selectively positionable, fluid distributing valve device, indicated generally at **10**, a reagent metering valve **12** having a differential pressure operated free piston **14** and an adjustable piston stop **16**, a reagent reservoir container **18** having an apertured plug stopper **20** with a conduit **22** leading to an atmosphere vent valve **24**, and a supply tank **26** of inert gas such as helium having a pressure regulator **28**. A conduit **30** from the pressure regulator **28** receives helium at a predetermined pressure, e.g., 25 p.s.i.g., and communicates with a pressure reducing valve **32** which supplies helium at a predetermined reduced pressure, e.g., 9 p.s.i.g., to a conduit **34**. One end of conduit **34** communicates with the low pressure chamber defined by one side of the free piston **14**.

A conduit **36** from the conduit **30** is connected to a main helium conduit having two branches **38** and **40**. The conduit branch **38** leads to the sampling valve **10** as will be described more fully hereinafter. The conduit branch **40** extends through the apertured plug stopper **20** and has its open end terminating adjacent the interior bottom of the reagent reservoir **18**. Another conduit **42** has one end similarly disposed in the reagent reservoir **18** and an opposite end connected to the sampling valve **10**. A conduit **44** has one end connected to the high pressure chamber defined by the opposite side of the free piston **14** and has its other end communicating with a pair of conduits **45** and **46**, each separately connected to the sampling valve **10**. A fifth conduit **48** is connected to the sampling valve **10** and has an opened end adapted to be connected to a waste drain of any suitable type (not shown).

As is illustrated in FIG. 7, the sampling valve **10** includes a fixed mounting plate **50** which may be secured to a suitable supporting cabinet as by bolts extending through mounting apertures **52** (only one being shown). A valve block **56** is secured to the top of mounting plate **50** as by threaded bolts **54**. The mounting plate **50** and valve block **56** are provided with five aligned bores which are radially spaced on an arc that is disposed intermediate between the center and the periphery of valve block **56**. In order to facilitate a description of the flow paths and inasmuch as the five bores in the valve block **56** and the mounting are in fixed communication with the five conduits connected to the sampling valve **10**, the five bores are designated with the reference numerals of their correspondingly connected conduits, viz., **38**, **42**, **45**, **46** and **48**.

A movable valve cylinder of the sampling valve **10** is defined by a central rotor **58** having four bores, **60**, **62**,

64, and 66, which are radially spaced on the same arc as the five bores in the valve block 56 so as to selectively control the flow therethrough. Movement of the rotor 58 is effected by a handle 68 protruding perpendicularly from one wall thereof whereby the rotor 58 is selectively moved to two controlling positions defined by a pair of spaced V-shaped notches 70 and 72 longitudinally grooved in the exterior of the rotor 58. The notches 70 and 72 provide alternate grooves for a detent mechanism including a spring-biased plunger 74 contained in a threaded bolt 76 which is carried by a detent block 78. As is shown in FIG. 7, the detent block 78 is fixed to the mounting plate 50 as by threaded bolts 80.

A second movable valve cylinder 82 is disposed on top of the central rotor 58 and has a pair of through bores 84 and 86 and an elongated arcuate channel 88 cut into the undersurface. The bores 84 and 86 and the channel 88 are radially spaced on the same arc as the bores in the rotor 58 so as to selectively establish communication therewith. In the same manner as rotor 58, the top valve member 82 is selectively moved to two controlling positions by a handle 90 which effects alternate engagement of its spaced V-shaped notches 92 and 94 for its detent mechanism including spring-biased plunger 96 and threaded bolt 98. The outer surface of the top valve member 82 has an adapter 100 which receives the specimen to be analyzed as from a tube, syringe, capillary tube, or pipette. The adapter 100 communicates with the bore 86 and a capsule loading needle 102 communicates with the bore 84 and is used to support a reaction capsule 104. The reaction capsule 104 comprises a piece of glass tubing having serum bottle caps on each end, one of such caps being punctured by the loading needle 102 when placed thereon. The two rotatable valve members 58 and 82, the valve block 56 and the mounting plate 50 are retained in a stacked assembly by means of a headed shaft 106 centrally extending therethrough and having a threaded nut 108 and a spring retaining washer 110 on its free end. A coil spring 112 surrounds a portion of the shaft 106 and is mounted in compression between the washer 110 and the exterior surface of mounting plate 50. The coil spring 112 thus biases the central rotor 58 into surface engagement with adjustment surfaces of the valve block 56 and the top valve member 82 to effect a seal therefor and the biasing force may be adjusted by means of the threaded nut 108.

In the following description of a sequence of operation of the apparatus, a clear understanding of the present invention will be facilitated by reference to FIGS. 2, 3 and 5, wherein the various bores of the sampling valve 10 are projected radially to illustrate the flow paths and alignments between the bores; the outer arcuate portion represents the valve block 56 and the five bores thereon are fixed as are the detent block 78 and the two detents 76 and 98; the intermediate arcuate portion represents the central rotor 58 which is movable relative to its detent 76 between alternate engagement thereof by its detent notches 70 and 72; and the inner arcuate portion represents the top valve member 82 which is similarly movable relative to its detent 98 between its detent notches 92 and 94. Assuming now that it is desired to purge the reaction capsule 104, the central handle 68 is moved to its right, as viewed in FIG. 4, until the detent 76 engages the notch 72 and retains the central rotor 58 in a selected position relative to the valve block 56; the top handle 90 is moved to its left until the detent 98 engages the notch 92 and retains the top valve member 82 in a selected position relative to the central rotor 58. There are two methods which can be employed in controlling the atmosphere of the reaction capsule, i.e., by purging or evacuation of the capsule. Thus, when it is desired to evacuate the capsule, the valve elements are now placed in an evacuation position which, as shown in FIGS. 3 and 4, establishes only single flow path through the sampling valve 10 by way of connecting a vacuum pump to the conduit 38 whereby the reaction capsule 104 is evacuated in a flow path therefrom through the capsule loading needle 102 and thence through the aligned bores 84, 62 and 32 to the vacuum pump. The other method of controlling the atmosphere of the capsule involves purging the same by the use of the helium from tank 26 whereby helium flows through the conduits 30 and 36, the aligned bores 38, 62 and 84, the capsule needle 102, the reaction capsule 104, and out through the top of capsule 104 by means of a vent needle (not shown) to the atmosphere.

After the reaction capsule has been purged, the sampling valve 10 is moved to a blood loading position by rotating the top handle 90 to the right as viewed in FIG. 1 until the detent 98 engages the notch 94 and retains the top valve member 82 in a selected position relative to the fixed valve block 56 and the central rotor 58. The central handle 68 remains in its same position as described above in connection with FIG. 3. The valve elements are now in the positions illustrated diagrammatically in FIG. 2 wherein the flow paths are shown in dashed lines. A flow path for the releasing agents may be traced from the reservoir 18 through conduit 42, central rotor bore 64, the flow channel 88 in the top valve element, the central rotor bore 66 and conduit 45 to the common conduit 44; since the conduit 46 is blocked off by the central rotor 58, the reagents can only flow through the conduit 44 to the lower chamber of the metering valve 12 because of pressure differential between the chambers defined by the lower chamber of the metering valve 12 and the chamber defined by the upper side of the free piston 14. In the same position, a flow path for the blood sample is established by the communication between the bores 86, 60, and 48; at this time a blood sample is continuously fed through the inlet 100 so that there is a flow of blood through the bores 86, 60 and 48. As is illustrated schematically in FIGS. 4 and 6, the direction of the various fluid flows are indicated by the arrows on the external conduits 38, 42, 45, 46, and 48, however, the five fluid flows will not occur simultaneously.

As soon as the sample valve 10 and the metering valve 12 are loaded, the sampling valve 10 is moved to a capsule loading position by rotating the central handle 68 to the left as viewed in FIG. 6 until the detent 76 engages the notch 70 and retains the central valve member 58 in a selected position relative to the fixed valve block 56 and the top valve member 82. The top handle 90 remains in its same position as described above in connection with FIG. 2. The valve elements are now in the positions illustrated diagrammatically in FIG. 5 wherein the flow path is shown in dashed lines. As the central rotor 58 is rotated, the continuous flow of the blood sample is cut off because the bore 60 is moved out of communication with the bores 86 and 48; however, the bore 60 is filled with the blood sample and is positioned so as to establish a flow path between the bores 46, 60 and 84. Since the metering valve 12 has been cut off from the reagents reservoir conduit 42, the helium pressure (9 p.s.i.g.) acting on the top of free piston 14 moves the piston downward to force the reagents from the storage chamber in valve 12 through the conduit 44. Because the conduit 45 is blocked off, the reagents can only flow from the conduit 44 to the conduit 46 and thence into the bore 60 where the reagents and the specimen sample are mixed as they are forced from the bore 60 and through the bore 84 and the needle connector 102 into the reaction capsule 104. The operation is now complete with regard to the required sequence for measuring the blood sample and the gas releasing agents and injecting the measured mixture into the reaction capsule.

Provisions are also provided for degasing of the reagents in the reservoir 18 in that helium is delivered from tank 26 through conduits 36 and 40 and bubbled into the gas releasing agents. Means are provided for the escape of

gases released from the gas releasing agents by conduit **22** leading to an atmosphere vent valve **24**.

The sampling valve **10** may now be returned to the purging position of FIG. 3 in order to be set for a new sample or, if purging of the new reaction capsule is unnecessary, the sampling valve **10** may be returned to its blood loading position of FIG. 2.

With the above arrangement, the volume of the blood sample and the volume of the reagents will be the same for each sampling. The volume of blood sample may be calibrated to any desired amount by varying the cross-sectional area or the length of the chamber defined by the bore **60**. The volume of reagents may also be calibrated to a desired amount by adjusting the meter valve stem **16**.

The various bores as shown of the radial projections of FIGS. 2, 3 and 5 each define a separate chamber and each of the bores on the top valve member **82** and the valve block **56** are counterbored on their exterior surfaces as shown for bores **84** and **38**, respectively, in FIG. 8. Thus, the flows through the central rotor bores, e.g., **62** in FIG. 8, will be subject to the higher pressure in the enlarged portions of such bores.

The valve block **56** and top valve member **82** are made of stainless steel and are highly polished on their valving surfaces for sliding contact with the central rotor **58**. The rotor **58** facilitates the sliding movement between the valving surfaces and prevents the leakage of the blood sample or the reagents. The rotor **58** can be made of any desirable low friction material that will provide a seal between valve block **56** and top valve member **82**. A useful material in this respect is Teflon, the registered trademark for polytetrafluoroethylene, to E. I. du Pont de Nemours & Company, Wilmington, Delaware, although any other material possessing the necessary characteristics can be employed, if desired.

The device of the invention is useful for measuring samples of materials such as beer, liquor, water, fuels, gasoline, rocket motor fuels, lubricating oils, edible oils, essence oils, milk, cream, sewer effluents and the like. By employing the device of the invention it is possible to obtain a measured volume of material and inject gas releasing agents into the measured volume of material and subsequently measure the dissolved gases released from the aforesaid materials by the use of apparatus for analyzing blood gases such as by a chromatographic technique.

Inasmuch as the preferred embodiment of the present invention may be subject to various modifications and changes in structural details, it is intended that all the matter contained in the foregoing description and shown on the accompanying drawings shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. In blood gas measuring apparatus, the combination comprising a reservoir of gas releasing reagents; means for degassing said gas releasing agents; a storage chamber for storing a quantity of the gas releasing reagents; a reaction capsule; a sampling valve having a fixed valve block, a rotor valve member on said block and a top valve member on said rotor valve member; said valve block having a bore communicating with a drain, a pair of bores communicating with said storage chamber, and a bore communicating with said reservoir; said top valve member being an inlet bore receiving a blood sample and a bore communicating with said reaction capsule; said rotor valve member having a plurality of controlling positions and a plurality of communication bores, one of said communication bores permitting a flow of the blood sample from said inlet bore to said drain bore and another of said communication bores permitting a flow of the gas releasing reagents from said reservoir through one of said pair of bores to said storage chamber when said rotor valve member is in one controlling position; the said one communication bore trapping a volume of the blood sample therein as said rotor valve member is rotated from said one controlling position to another controlling position, the said one communication bore permitting a flow of the gas releasing reagents from said storage chamber to said reaction capsule together with the volume of the blood sample when said rotor valve member is in said another controlling position.

2. The combination as set forth in claim **1** wherein said storage chamber, said one communication bore, and said capsule are continuously sealed from the atmosphere.

3. The combination as recited in claim **1** wherein said valve block has a purge bore, said rotor valve member has purge communication bore, and said top valve member is rotatable to a purge position in which said capsule bore communicates with said purge bore and said purge communication bore when said rotor valve member is in said one controlling position.

4. The combination as recited in claim **3** wherein said valve block, said rotor valve member and said top valve member are assembled in stacked relation and spring means biases said top and rotor valve members toward said valve block.

5. The combination as recited in claim **4** wherein detent means are operatively connected between said valve block and said top and rotor valve members to retain said top and rotor valve members in selected controlling positions.

6. In blood gas measuring apparatus, the combination comprising a reservoir of gas releasing reagents; means including an atmosphere vent valve for degassing said gas releasing reagents; a metering valve having a free piston defining a storage chamber and a pressure chamber; a tank of helium having a pressure regulator for supplying helium at a preset pressure, conduit means between said pressure regulator and said reservoir for delivering helium to the gas releasing reagents; means including a pressure reducing valve for delivering helium at a pressure lower than the preset pressure to said pressure chamber, a reaction capsule, sampling valve means controlling fluid flow to said reaction capsule and having a sampling chamber, a pair of valve members in said sampling valve means movable between a plurality of controlling positions, said valve members having first positions permitting a flow of helium from said pressure regulator to said reaction capsule for purging the same, second positions permitting a flow of blood sample through said sampling chamber to a drain and permitting a flow of gas releasing reagents from said reservoir to said storage chamber, and third positions permitting a mixture flow of blood sample and gas releasing reagents to said reaction capsule and permitting such mixture to be formed by a flow of the gas releasing reagents from said storage chamber to said sampling chamber.

7. Apparatus for processing a fluid sample with gas releasing reagents comprising:

closed means for storing a pressurized volume of the gas releasing reagents,

a closed reaction capsule,

a sampling valve having a fixed valve element and first and second movable valve members,

a plurality of bores in said fixed valve element, one of the fixed bores communicating with said storing means and another communicating with a drain,

a plurality of bores in said first movable valve member, one of the movable bores communicating with said reaction capsule and another defining an inlet port for the fluid sample,

said second movable valve member having a gas-tight chamber means and being movable between a plurality of positions,

said second movable valve member also having a first controlling position permitting a flow of the fluid sample from said inlet port through said chamber means to said drain and a second controlling position permitting a flow of the pressurized gas releasing reagents from said storing means through said chamber means to said reaction capsule,

said second movable valve member being operative during its movement from the first to the second controlling positions to trap a measured volume of the fluid sample in said chambers means, whereby the pressurized fluid sample flows to said reaction capsule with the flow of the gas releasing reagents.

8. The combination as recited in claim **7** wherein said storing means includes a valve body having a free piston defining a pressure chamber and a storage chamber,

and adjustable means to limit piston movement in the pressure chamber and to measure the volume of the stored gas releasing reagents in which the fluid sample flows from said inlet port to the drain and the gas releasing reagents flow from said storing means.

9. In fluid processing apparatus, the combination comprising:

a reservoir of gas releasing reagents under pressure,

a storage chamber for storing a quantity of the gas releasing reagents under pressure,

a closed reaction capsule,

a sampling valve having a fixed valve block, a rotor valve member on said block and a top valve member on said rotor valve member,

said valve block having a bore communicating with a drain, a pair of bores communicating with said storage chamber and a bore communicating with said reservoir,

said top valve member being an inlet bore receiving a fluid sample and a bore communicating with said reaction capsule,

said rotor valve member having a plurality of controlling positions and a plurality of communication bores, one of said communication bores permitting a flow of the fluid sample from said inlet bore to said drain bore and another of said communication bores permitting a flow of the gas releasing reagents from said reservoir through one of said pair of bores to said storage chamber when said rotor valve member is in one controlling position,

the said one communication bore trapping a volume of the fluid sample therein as said rotor valve member is rotated from said one controlling position to another controlling position, the said one communication bore permitting the gas releasing reagents from said storage chamber to force said fluid sample into said reaction capsule when said rotor valve member is in said another controlling position.

10. The combination as recited in claim **9** wherein said valve block has a purge bore, said rotor valve member has a purge communication bore, and said top valve member is rotatable to a purge position in which said capsule bore communicates with said purge bore and said purge communication bore when said rotor valve member is in said one controlling position.

11. The combination as recited in claim **10** wherein said valve block, said rotor valve member and said top valve member are assembled in stacked relation and spring means biases said top and rotor valve members toward said valve block.

12. The combination as recited in claim **11** wherein detent means are operatively connected between said valve block and said top and rotor valve members to retain said top and rotor valve members in selected controlling positions.

13. In a fluid sampling apparatus the combination comprising:

a reservoir of gas releasing agents,

a metering valve defining a storage chamber,

actuating means for reducing the volume of said storage chamber when the fluid pressure in said chamber is less than a predetermined value,

a source of gas having a pressure exceeding said predetermined value,

conduit means between said source and said reservoir for pressurizing the releasing reagents above said predetermined value,

a reaction capsule,

sampling valve means controlling fluid flow to said reaction capsule and having a chamber,

a pair of valve members in said sampling valve means movable between a plurality of controlling positions, said valve members having first positions permitting a flow of gas from said source to said reaction capsules for purging the same, second positions for permitting a flow of fluid sample through said sampling chamber to a drain and permitting a flow of pressurized releasing reagents from said reservoir to said storage chamber, and third positions permitting a flow of fluid sample in said sampling chamber and gas releasing reagents from said storage chamber to said reaction capsule.

14. The combination as recited in claim **13** wherein said metering valve includes means to adjust the volume of said storage chamber to a predetermined volume.

15. The combination as recited in claim **13** wherein said sampling valve means includes detent means operatively connected to said pair of valve members for retaining in same in the first, second and third positions.

16. In blood gas measuring apparatus the combination comprising:

a storage chamber for storing a predetermined quantity of gas releasing reagents,

a reaction chamber,

a sampling valve having a fixed valve block, a rotor valve member on said block and a top valve member on said rotor valve member,

said valve block having a bore communicating with a drain and a pair of bores communicating with said storage chamber,

said top valve member having an inlet bore for receiving a blood sample and a bore communicating with said reaction chamber,

said rotor valve member having a plurality of controlling positions and a plurality of communication bores, one of said communication bores permitting a flow of the blood sample from said inlet bore to said drain bore when said rotary valve member is in one controlling position, the said one communication bore sealing a volume of the blood sample therein as said rotary valve is rotated from said one controlling position to another controlling position,

the said one communication bore permitting the transfer of a volume of the gas releasing reagents from said storage chamber to said reaction chamber together with the volume of the blood sample when said rotary valve member is in said another controlling position.

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