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(54) **U-tube rheometer for measuring dynamic viscosity**

(57) This invention relates to a rheometer of U-tube type for measuring rheological properties of gel or other materials; and the method for the said measurement, it comprises of a U-shape channel where the test sample is contained; means for creating a desired air pressure applied to the sample from one side of the U-tube and means for monitoring the change in pressure in the en-

closed chamber at the other side of the U-tube from which the sample volume displacement can be calculated. The method allows rigidity modulus measurement implemented on a very weak gel at controllable small disturbance to the sample. The rheometer is automatic and computerized capable of both dynamic and static measurements at controlled thermostatic environment.

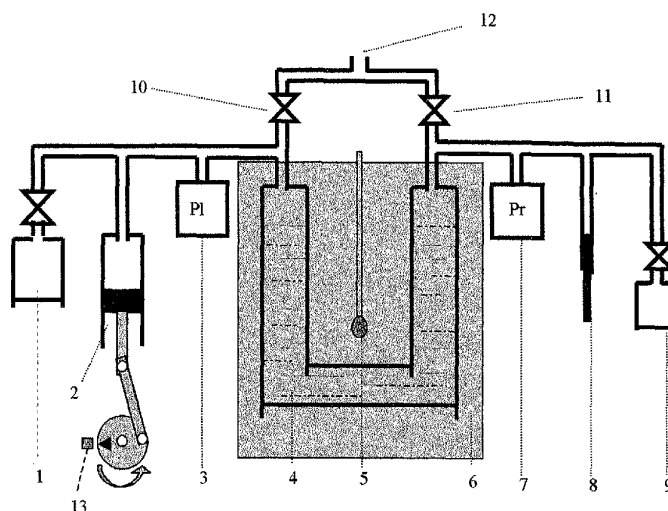


FIG. 1

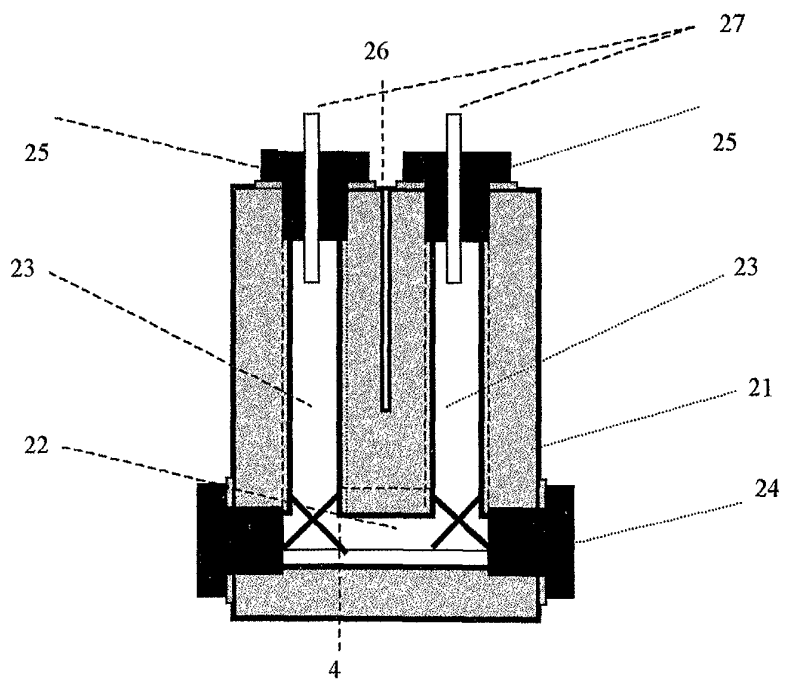


FIG. 2

## Description

### FIELD OF INVENTION

**[0001]** The present invention relates to a rheometer of U-tube type and the related measurement method for determining the complex rigidity of gels and other materials when subject to shear force introduced by applying air pressure.

### BACKGROUND OF THE INVENTION

**[0002]** Rheological properties of materials, including elasticity, viscosity etc., are often important parameters to characterize a product, or to be used as pertinent indicators for manufacturing processes. For example, regarding yogurt as a dessert product, its rigidity and viscosity are important texture parameters for consumer acceptance and they can be considered and employed as definite indicators to observe the development of the yogurt formation process- from a thin mixture of milk and rennet to a thick gel-like material. By monitoring the changes in the rheological behaviour, an investigation of the formation mechanism of yogurt gel network can be conducted. The technique can also serve as a process control method for yogurt production in industrial scale.

**[0003]** Regarding the rigidity measurement for gel-like materials E. Kinkel and E. Sauer proposed a method in Z. Angew. Chem., 1925, 38, 413, in which the gelatin solution was allowed to set as a cylindrical column in a glass tube. In measurement, an air pressure was applied to one end of the tube causing a deformation to the gelatin and the rigidity modulus can be calculated from the air pressure and the resulting displacement of the center of the gelatin meniscus. This method was not adopted due to the difficulty in accurate measurement of the displacement. Later a modification to the method was reported by P.R.Saunders and A.G.Ward in "An absolute method for the rigidity modulus of gelatin gels" in Proceedings of the Second International Congress on Rheology pp284-290 (V.G.W.Harrison, ed) Academic Press (1954). The proposed method, widely known as U-tube method, has thereafter stimulated an increasing research on rheological behaviour of gel-like materials. In this method the volume displacement of the gelatin gel was measured instead of displacement of the meniscus. The U-tube apparatus comprises joined together two vertical glass tubes of different radii, a wide limb and a capillary limb. In the capillary limb, and the lower part of the wide limb, mercury is contained as an index liquid. On top of the mercury in the wide limb a column of gel is allowed to set. Small air pressures were applied above the gel and the corresponding volume displacements were calculated from the rise of the mercury in the vertical capillary limb of the U-tube. Due to the remarkable radius difference between the two limbs a very small deformation to the gel gave a large movement of

mercury meniscus so that the volume displacement can be accurately measured. From the effective pressure, i. e., the applied air pressure corrected for any back pressure due to the displacement of the index liquid, and the corresponding volume deformation the absolute rigidity of the gel can be calculated with a modified Poiseuille's equation.

**[0004]** It was found that the U-tube method of Saunders and Ward (1954) can be used conveniently for studying gelatin gel having rigidities from about 0.1 to  $50 \times 10^4$  dynes/cm<sup>2</sup>. Gelatin gel produces no syneresis, i.e., a film of exuded liquid formed at the surface which prevents adhesion between the gel and a measuring device. If a gel studied is syneresing, it slips, as a whole under the applied air pressure. In an attempt to overcome this problem H.Komatsu and P.Sherman introduced many small protuberances on the inside wall of the wide bore tube, reported in Journal of Texture studies 5 (1974) 97-103, and this enabled W/O emulsions to be studied successfully with the U-tube method. Atkin and Sherman (1980) later examined the viscoelastic properties through creep compliance-time measurement of some weak food gels, including natural yogurt, yogurt containing fruit pieces, jams containing fruit, and mayonnaise and from rheological analysis more meaningful information than conventional quality control methods were obtained.

**[0005]** G.Stainsby, S.G.Ring and G.R.Chivers in their paper "A Static Method for Determining the Absolute Shear Modulus of a Syneresing Gel", J. of Texture Studies, Vol.15 pp23-32(1984) reported a modification of U-tube which has allowed the determination of shear modulus of food gels made from amylose, starch, K-carrageenan and egg white, all of which exhibit extensive syneresis. To prevent slippage the inside of the wide bore tube is coated with a single layer of anti-bumping granules of approximately 1 mm diameter. The apparatus uses a modular design in which gels were prepared and aged in the wide bore tubes, and connected to the capillary only during rheological measurement. In order to avoid the development of back pressure during measurement the capillary and wide bore tube are in line. The apparatus was subsequently modified to permit investigation of the viscoelastic properties of rennet- milk gels, which have a much weaker internal structure.

**[0006]** In the paper "An Apparatus for measuring the elastic properties of very soft gels", Laboratory Practice, Oct. 1957. G.W.Scott Blair described his U-tube device and with it he was able to measure very weak gels having rigidity from about 0.1 to  $5.0 \times 10^2$  dynes/cm<sup>2</sup>. His U-tube apparatus consists of a U-tube of two identical arms in which some 50ml. of rennet- milk are set To reduce the syneresis and movement as a whole compressed air is applied to either surface of the sample alternately, by means of a 4-way tap, and the other side of the U-tube to which pressure is not being applied is connected to the nearly horizontal capillary. Another advantage of the design is no direct contact between the

sample and index liquid. With this device he was able to follow the increasing rigidity of rennet- milk gels in terms of generalized modulus and in terms of creep, recovery, relaxation and elastic "memory".

**[0007]** U-tube method has numerous advantages including the simplicity in construction, very little disturbance to sample, which is important for some bioproducts such as rennet- milk, and tolerance to wide range of samples, some of which can not be handled by conventional cone and plate rheometers, such as yogurt with fruit pieces.

**[0008]** It is an object of the present invention to develop a U-tube rheometer, capable of determining, in both static and dynamic (oscillatory) modes, the rheological behavior of gel-like or other materials over wide range of rigidity values, including very low rigidity. It is also an object of the present invention to design an automatic and computerized rheometer based on the U-tube method in which means of pressure measurement, instead of displacement of index fluid in capillary, is used for the determination of volume displacement.

#### SUMMARY OF THE INVENTION

**[0009]** According to the present invention a U-tube type rheometer for measuring the rheological properties of a gel or other materials comprises a U-tube unit where the sample is contained in a U-shape channel, to which at one side an air pressure, delivered from a moving piston in cylinder, is applied to the test sample, causing a sample volume displacement along the U-shape channel and consequently a change in pressure in the enclosed chamber above the sample at the other side. The counterbalancing effect of the pressure in the said enclosed chamber against the said applied pressure results in a diminishing effective driving pressure, hence a limited volume displacement even for a sample of very low rigidity, allowing measurements applied to weak gels at controllable small disturbance to the sample. The rigidity of the sample is calculated from the applied pressure and the corresponding volume displacement, derived from the changing pressure in the said enclosed chamber, with a modified Poiseuille's equation. Thermostatic controls for the U-tube unit are provided.

#### BRIEF DESCRIPTION OF FIGURES

**[0010]** The invention will now be described in detail, reference being made to the accompanying drawings which illustrate a preferred embodiment and in which

FIG. 1 is a schematic of the rheometer which shows the consisting elements and the configuration;  
FIG. 2 is a schematic of the U-tube unit; and  
FIG. 3 is a schematic of the assembly for temperature control in which the U-tube unit was inserted in

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

**[0011]** According to FIG. 1 the embodiment of the U-tube rheometer of the present invention comprises the U-tube unit 4, which has a U-shape tunnel wherein the test sample is contained; The two identical limbs of the U-tube, for the sake of convenience they are referred to as left side and right side of the U-tube in the following description, were connected to other elements of the rheometer. To its left side including the valves 10; the pressure transmitter 3; The air pressure generating unit 2; and one or more dead volume-valve assembly 1. To the right side of U-tube connected are the valve 11; The pressure transmitter 7; The scaled mini piston-cylinder unit 8; and one or more dead volume-valve assembly 9; According to Fig. 3 the U-tube unit is inserted in between two thermostat units 6 which, in conjunction with the temperature sensor 5 mounted in the central region of the U-tube unit, provide a thermostatic environment for the sample. The said valves are preferably solenoid valves communicating on one side to atmosphere through opening 12. The operation of the rheometer is fully automatic and computerized.

**[0012]** The working principle of the rheometer can be described briefly as follows: The test sample is allowed to set in the U-tube 4 at a temperature set by the thermostat unit 6 and with the valves 10 and 11 being open to atmosphere. In measurement both sides of the sample are enclosed from atmosphere by turning off valves 10 and 11, followed by the application, to the left side of the sample, of a controlled air pressure created from the air pressure generating unit 2. The applied air pressure is recorded in the computer through the pressure transmitter 3. The range of the applied air pressure can be selected by including a number of dead volume element 1 through its control valve. The volume displacement of the sample under the applied air pressure causes a change in pressure in the enclosed chamber on the right side of the U-tube which is also recorded in the computer through pressure transmitter 7. The pressure on the right side on the U-tube is called back pressure, which counterbalances the applied air pressure on the left side resulting in a reduced effective pressure. The magnitude of the back pressure can be influenced by including a number of dead volume elements 9 through its control valve. The utilization of the back pressure in the present invention allows rigidity measurement applied to very weak gel at a predictable volume displacement limit. The scaled mini piston-cylinder 8, for which a syringe can be used, provides a means of determining the dead volume existed above the right side of the sample. The volume displacement can then be calculated from the back pressure. The rigidity modulus of the sample is calculated from the effective pressure and the resulting volume displacement.

**[0013]** The detailed configuration of the embodiment U-tube unit is shown in FIG.2. The unit was made from

a rectangular block of aluminum 21, through whose central plane were drilled three communicating bores, namely through the lower part a horizontal bore 22 and to which intersect the two vertical bores 23. The three bores have the same diameter and were all tapped on the inner wall with screw thread. A hole 26 in the middle of the unit is provided for placing the temperature sensor. The horizontal bore 22 can be sealed on both sides with the threaded stopper-spacer assemblies 24 to form the U-tube, i.e. a U-shape channel, in conjunction with the two vertical bores 23. Connecting tube 27 has one end fixed to the top stopper-spacer assemblies 25, leaving the other end for communicating with other parts of the rheometer. Advantages of the design include a) Good temperature uniformity throughout the U-tube unit due to the high thermal conductivity of aluminum; b) The screw thread made on the inner wall of the bores serves both purposes of fastening the stoppers and preventing slippage of test samples against the U-tube wall; and c) By removing the stopper-spacer assemblies the U-tube channel can be readily cleaned.

**[0014]** Thermostatic condition is essential to rheological measurement. Thermostatic condition for the U-tube unit in this rheometer was provided by the two thermostats 6, shown in detail in FIG.3. These thermostats were made from rectangular aluminum blocks and, through whose central planes continuous zigzag channels were made. A flexible tube 35 joined together the zigzag channels of the two thermostats 6 at their lower openings, while nozzle 36 are fixed to the two upper openings allowing use with a circulation bath for temperature above or below ambient. Besides, mounted on the outer surface of each thermostat 6 there is an electric heater 31 which, in conjunction with the temperature sensor 5, can provide temperature control above room temperature. The two thermostats 6 were connected with plate springs 33 leaving a gap in between into which the U-tube unit is inserted giving a good thermal contact. The sandwich assembly was embraced with thermal insulator 32 for enhanced temperature uniformity throughout the U-tube - thermostat assembly.

**[0015]** The air pressure generating unit 2 is based on an engine mechanism comprising a piston in cylinder and a crank-connecting rod. The rotating crank driven by an electric motor, preferable a stepping motor or a DC servo motor, reciprocates the piston. A simple harmonic movement can be achieved by making the connecting rod much longer than the crank. A Scotch yoke mechanism has also been tried to deliver a simple harmonic movement. The crank reference position detector 13 is a switch of mechanical or optical type and changes its status when the crank moves to the reference position. This status can be read by the computer for a phase reference in producing a desired air pressure. In normal measurement, valves 10 and 11 are switched off for enclosing both sides of the U-tube from atmosphere while the crank is at the reference position, so that a forward/backward piston movement will generate a positive/negative

negative air pressure in reference to atmosphere. Various forms of pressure versus time can be produced through motor rotation control, such as sinusoidal, ramp, step or other sophisticated forms. By applying alternate positive and negative pressure the tendency of sample shifting toward one side in the U-tube can be eliminated.

**[0016]** The two pressure transmitters 3 and 7 provide the means for monitoring the air pressures applied on the left side and right side of the sample, respectively. These pressure transmitters preferably comprise silicon piezoresistive type pressure transducer and the related signal processing circuits capable of sending air pressure signal to the computer.

**[0017]** The dead volume - control valve units 1 and 9 are incorporated into the left side and right side of the U-tube respectively. When their control valves are turned on, the corresponding volumes are included into the system, altering the total dead volume on the related side of the U-tube.

**[0018]** The scaled mini piston-cylinder unit 8, which can be a scaled syringe, is connected to the right side of the U-tube for delivering a known volume displacement in determining the dead volume on that side of the U-tube.

**[0019]** Theoretically the rheometer works on a well-known ideal gas law stating that for air in an enclosed chamber the product of pressure P and the volume V remains constant in an isothermal process, expressed by the basic equation  $P_1 V_1 = P_0 V_0$ , where the subscript 0 represents a reference status and the subscript 1 represents any other status. We can apply this basic equation to the present U-tube rheometer. When the valves 10 and 11 are open to atmosphere allowing pressure equilibrium achieved on both side of the U-tube, therefore  $P_0 = 1 \text{ atm}$ , this status is designated as the reference status at which the crank is normally driven to the reference position, and the piston rests at approximately the mid stroke. By turning off these valves certain amount of air is enclosed on each side of the U-tube and the following status can be investigated with the basic equation. Applying the basic equation to the enclosed volume on the left side of the U-tube we obtain an equation

$$P_1 V_1 = (P_{10} + \Delta P_1) (V_{10} + \Delta V_1) = P_{10} V_{10},$$

where we use 1 to denote the left side and we also expressed a new status in terms of an increment with respect to the reference status, i.e.  $\Delta P_1$  and  $\Delta V_1$ . It is evident that the magnitude of the pressure increment  $\Delta P_1$  created from a given volume increment  $\Delta V_1$  by a piston displacement is dependent on  $V_{10}$ , namely the initial volume enclosed in the left side. By means of including/excluding dead volume element 1,  $V_{10}$  can then be adjusted for delivering a desired magnitude of air pressure, namely a smaller  $V_{10}$  for higher pressure and a bigger  $V_{10}$  for lower pressure. Besides, in a static mode a re-

duced air pressure can also be obtained by a smaller rotation angle of the driving motor, consequently the piston displacement. Both approaches are used in practice through programming.

**[0020]** The amplitude of volume displacement of a reciprocating piston and the amplitudes of the resulting air pressure for all the combinations of dead volume element 1 can be calculated from the relevant dimensions of the rheometer with the basic equation mentioned above, and can be checked by experiment as well. An air pressure created from a given crank angular displacement with respect to the reference position can be calculated based on the amplitude and a sine relation. Amplitudes at various dead volume combinations were stored in computer for lookup in creating a desired air pressure.

**[0021]** Similarly, applying the basic equation to the enclosed volume on the right side of the U-tube, we obtain an equation analogous to that for the left side

$$P_r V_r = (P_{r0} + \Delta P_r) (V_{r0} + \Delta V_r) = P_{r0} V_{r0}$$

Where we use  $r$  to denote the right side. It is obvious that the volume displacement  $\Delta V_r$  causes a pressure increment  $\Delta P_r$  whose magnitude is influenced by the initial enclosed volume  $V_{r0}$ . Therefore, by including/excluding dead volume element 9,  $V_{r0}$ , hence  $\Delta P_r$ , can be adjusted.  $\Delta P_r$ , can be called back pressure, results a diminishing effective pressure which is the air pressure on the left side subtracts the one on the right side. This auto adaptive nature allows measurement being implemented on a sample of very low rigidity, namely, a small air pressure applied to the sample causes a  $\Delta V_r$  which further causes a  $\Delta P_r$  comparable to the applied driving pressure, resulting in a significantly reduced effective air pressure, hence a limited volume displacement. The extent of the volume displacement can be adjusted by including various dead volume elements 9.

**[0022]** It is also evident from the equation that if  $V_{r0}$  is known the volume displacement of the sample can be calculated from pressure increment  $\Delta P_r$ .  $V_{r0}$ , the initial dead volume enclosed above the right side of the sample, can be determined from a known volume increment,  $\Delta V_r$ , delivered by the scaled mini piston-cylinder 8, and the resulting pressure increment,  $\Delta P_r$ , based on the above equation. In practice  $V_{r0}$  was divided into two parts which were determined separately. First the U-tube unit was detached from the assembly connected to its right side whose dead volume was determined using the mini piston-cylinder 8 as aforementioned in order to avoid errors caused by sample deformation; while the dead volume existing in the U-tube unit is determined from its dimensions. The value of  $V_{r0}$  is then obtained by summing up the two parts. For a given construction, it is obvious that the values of the first part of  $V_{r0}$  for all the possible combinations of dead volume element 9, are rheometer constants, therefore can be stored in

computer for lookup, while the second part of  $V_{r0}$  should be determined for each sample. The value of  $V_{r0}$  was determined and input to computer before test started.

**[0023]** For an entirely elastic sample the shear modulus measured with U-tube method can be calculated from the following equation analogous to that of Poiseuille's :

$$G = \pi R^4 (P_1 - P_r) / 8 L \Delta V_r$$

Where  $P_1$  and  $P_r$  are the pressures measured from the left side and right side above the sample, respectively;  $L$  is the total length of the sample;  $R$  is the effective radius of the U-tube; and  $\Delta V_r$  is the volume displacement of the sample which can be calculated from resulting  $\Delta P_r$ , then we have the following equation for  $G$  calculated from pressure measurements,

$$G = (\pi R^4 / 8 L V_{r0}) (P_1 - P_r) (1 + P_{r0} / \Delta P_r)$$

**[0024]** Assuming  $P_{r0} / \Delta P_r \gg 1$ , this is often the case in practice, then  $G$  can be approximated by the following equation

$$G = (\pi R^4 P_{r0} / 8 L V_{r0}) (\Delta P_1 / \Delta P_r - 1)$$

From which the working essence of the present invention becomes even more apparent, i.e.  $G$  is measured by the ratio of the gauge pressures measured from both sides of the sample, namely  $\Delta P_1 / \Delta P_r$ . For a sample of very low modulus the ratio of  $\Delta P_1 / \Delta P_r$  is reduced to about 1, indicating a back pressure  $\Delta P_r$  nearly equal to the driving pressure  $\Delta P_1$ . Such a measurement can be realized at a limited sample volume displacement with the present invention by adequate selection of the driving pressure  $\Delta P_1$  and the dead volume  $V_{r0}$ .

**[0025]** Dynamic or creep measurements from this apparatus are available for rheological modeling and viscoelastic analysis.

**[0026]** The main operation procedures include a) Prepare test sample in the U-tube at controlled temperature leaving valves 10 and 11 open. b) Bring the crank to the reference position. c) Turn off the valves 10 and 11. d) Activate the air pressure generating unit for a desired driving air pressure and record the pressures on both side of the sample. e) Repeat the measurement at an interval to monitor the sample. To avoid possible sample shifting toward one side in the U-tube, which might happen to a syneresing gel, it is advantageous that balanced driving pressures, namely alternate positive and negative driving pressures are applied.

## Claims

1. The present invention relates to rheometer and rheological method for the complex rigidity evaluation of gels and other materials consisting a U-tube unit having a U-shape channel of two identical limbs denoted as left limb and right limb respectively; communicating elements to the left limb are : a control valve communicating with atmosphere when turned on; means for measuring air pressure; an air pressure generating unit; and one or more dead volume-control valve assembly; communicating elements to the right limb are : a control valve communicating with atmosphere when turned on; means for measuring air pressure; means for creating a known volume displacement; and one or more dead volume-control valve assembly; means for creating a thermostatic environment for the U-tube unit; and means for automation and computerization of the rheometer.
 

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2. According to claim 1 wherein the U-tube unit is preferably made from a rectangular block of aluminum, through its central plane three bores of equal diameter were perforated, namely two vertical bores intersect with a horizontal bore, furthermore these bores are all tapped with screw thread in the inner walls along the whole length.
 

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3. According to claims 1 and 2 wherein in the middle through the central plane of the U-tube unit a vertical hole is provided for installing a temperature sensor.
 

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4. According to claims 1 and 2 wherein two stopper-spacer assemblies are provided for hermetically sealing the two openings of the horizontal bore in the said U-tube unit by virtue of the engaging thread so that the three bores form a U-shape channel wherein the test sample is loaded.
 

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5. According to claims 1 and 2 wherein two stopper-spacer assemblies are provided for hermetically sealing the two openings of the vertical bores in the said U-tube unit by virtue of engaging thread to facilitate the U-tube unit being connected to other elements of the rheometer through the connecting tubes fixed at one end to the said stopper.
 

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6. According to claims 1,2 and 5 wherein control valves are connected to each side of the U-tube through the said connecting tubes allowing either side open to atmosphere when the valves are turned on; or enclosed from atmosphere when the valves are turned off.
 

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7. According to claims 1 and 5 wherein connected to both sides of the U-tube through the said connect-
 

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- ing tubes there are means for measuring air pressure which preferably are silicon piezoresistive pressure transducers and the related signal processing circuits capable of sending air pressure signal to the computer.
 

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8. According to claims 1 and 5 wherein connected to the left side of the U-tube through the said connecting tube there is an air pressure generating unit, preferably based on an engine mechanism, comprises a piston-in-cylinder driven by a electric motor through a crank-connecting rod mechanism..
 

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9. According to claims 1 and 8 wherein the said crank-connecting rod mechanism consists of a connecting rod significantly longer than the crank so that a good approximate simple harmonic piston movement, hence a volume displacement of that kind can be delivered from a continuous motor rotation of constant speed..
 

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10. According to claims 1, 8 and 9 wherein the said driving electric motor, having its output shaft directly coupled to the said crank, is preferably a DC servo motor or a stepper motor whose rotation, including the rotation speed and the angular position with respect to the reference position, can be controlled through computer to deliver a desired piston displacement, consequently a desired air pressure versus time, such as simple harmonic, ramp, step or sophisticated forms of air pressure.
 

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11. According to claims 10 wherein the reference crank position, which preferably corresponds to a mid stroke piston position, is indicated by a crank reference position detector which can be a switch of mechanical, optical or other type, capable of changing status when the crank is in the said reference position.
 

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12. According to claims 1 and 5 wherein connected through the said connecting tubes to each side of the U-tube unit there is one or more dead volume - control valve elements which preferably are solenoid valves, by turning on or turning off a control valve the related dead volume can be added to or withdraw from one side of the U-tube, thereby altering the total dead volume on that side of the U-tube.
 

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13. According to claims 1 and 5 wherein connected through the said connecting tube to the right side of the U-tube there is a means for delivering a known volume displacement, preferably a scaled mini piston-cylinder unit, for which an accurate scaled syringe can be used, used for determining the dead volume enclosed in the right side of the U-tube.
 

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14. According to claim 1, wherein the preferable ther-
 

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mostat unit consists of two thermostats made from rectangular aluminum blocks in which zigzag channels were perforated for use with circulation thermostat.

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15. According to claims 1 and 14 wherein the two said thermostats are joined together through plate springs to form a gap in between where the U-tube unit is inserted for good thermal contact.

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16. According to claims 1,3, 14 and 15 wherein the mounted to the outer surfaces of the said thermostats there are electric heaters as well, which in conjunction with the said temperature sensor provide temperature control for the U-tube unit, as an alternative to the circulating bath

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17. According to claim 1, wherein means for automation and computerization of the rheometer consists of various signal processing circuits and a computer and related programs.

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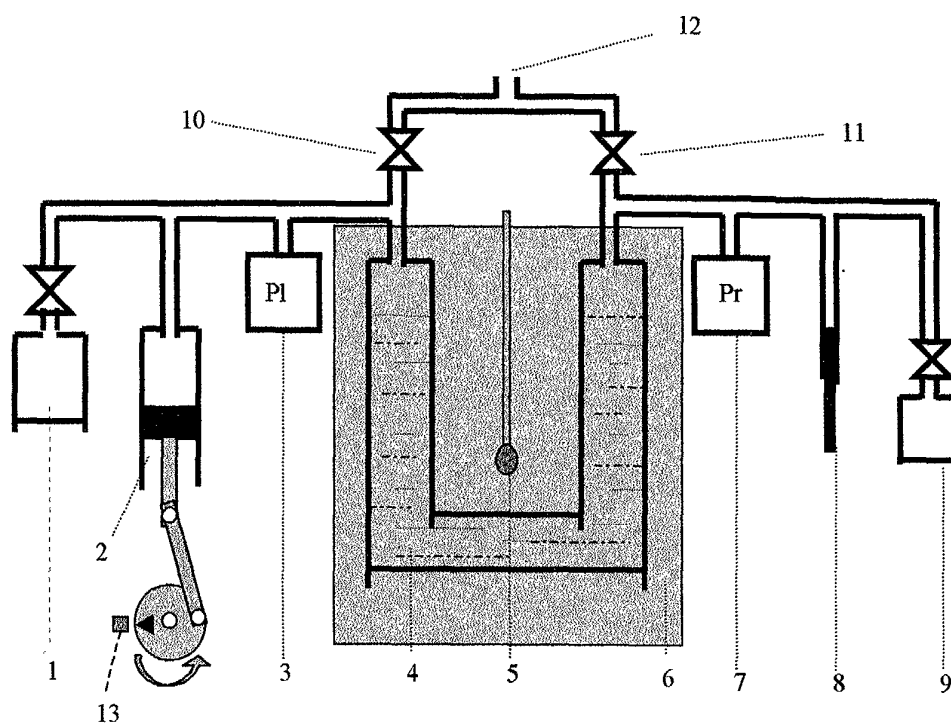


FIG. 1

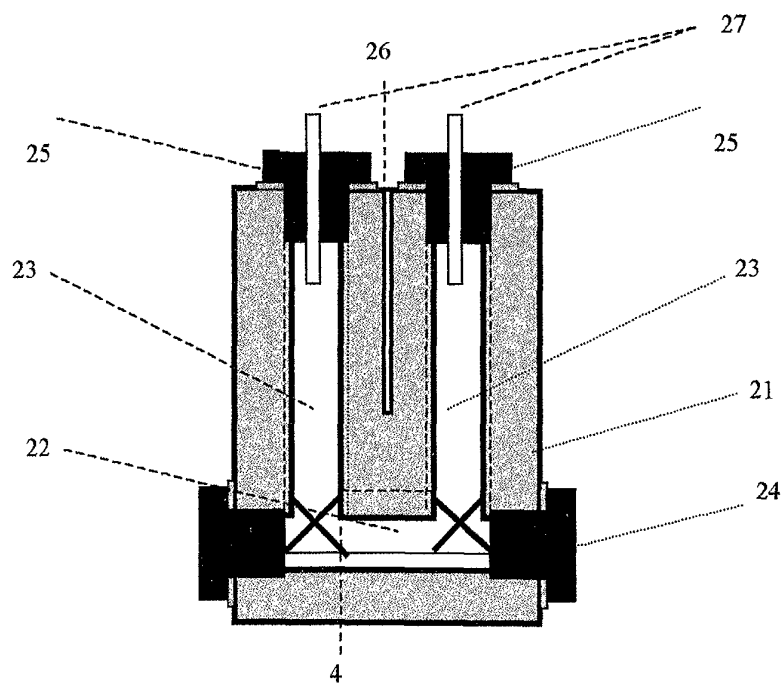


FIG. 2

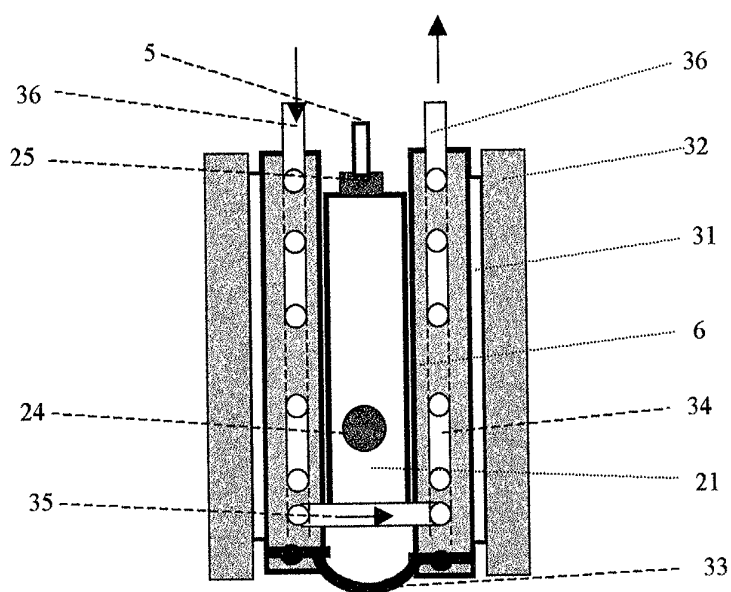


FIG. 3



European Patent Office

EUROPEAN SEARCH REPORT

Application Number  
EP 03 38 6003

| DOCUMENTS CONSIDERED TO BE RELEVANT                                                                                                                                                                                                                    |                                                                                                                                                                                                                                                                                                                                                                                                                                          |                                                                                                                                                                                                                                                                                  |                                              |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------|
| Category                                                                                                                                                                                                                                               | Citation of document with indication, where appropriate, of relevant passages                                                                                                                                                                                                                                                                                                                                                            | Relevant to claim                                                                                                                                                                                                                                                                | CLASSIFICATION OF THE APPLICATION (Int.Cl.7) |
| X                                                                                                                                                                                                                                                      | US 4 691 558 A (SMITH WILLIAM H ET AL)<br>8 September 1987 (1987-09-08)                                                                                                                                                                                                                                                                                                                                                                  | 1,5-13,<br>16,17                                                                                                                                                                                                                                                                 | G01N11/08                                    |
| Y                                                                                                                                                                                                                                                      | * column 2, line 18 - column 2, line 65 *<br><br>* column 2, line 18 - column 2, line 65 *<br>* column 3, line 57 - column 3, line 63 *<br>* column 4, line 11 - column 4, line 62 *<br>* column 5, line 1 - column 5, line 37 *<br>* column 6, line 28 - column 7, line 29 *<br>* column 8, line 1 - column 8, line 62 *<br>* column 9, line 42 - column 9, line 49 *<br>* column 12, line 21 - column 12, line 34 *<br>* figures 1-3 * | 1,5-13,<br>16,17                                                                                                                                                                                                                                                                 |                                              |
| Y                                                                                                                                                                                                                                                      | GB 434 994 A (RICHARD VYNNE<br>SOUTHWELL;ANDREW GEMANT)<br>12 September 1935 (1935-09-12)<br>* page 2, line 45 - page 3, line 15 *<br>* page 3, line 29 - page 3, line 68 *<br>* figure 1 *                                                                                                                                                                                                                                              | 8-10                                                                                                                                                                                                                                                                             |                                              |
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| The present search report has been drawn up for all claims                                                                                                                                                                                             |                                                                                                                                                                                                                                                                                                                                                                                                                                          |                                                                                                                                                                                                                                                                                  |                                              |
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