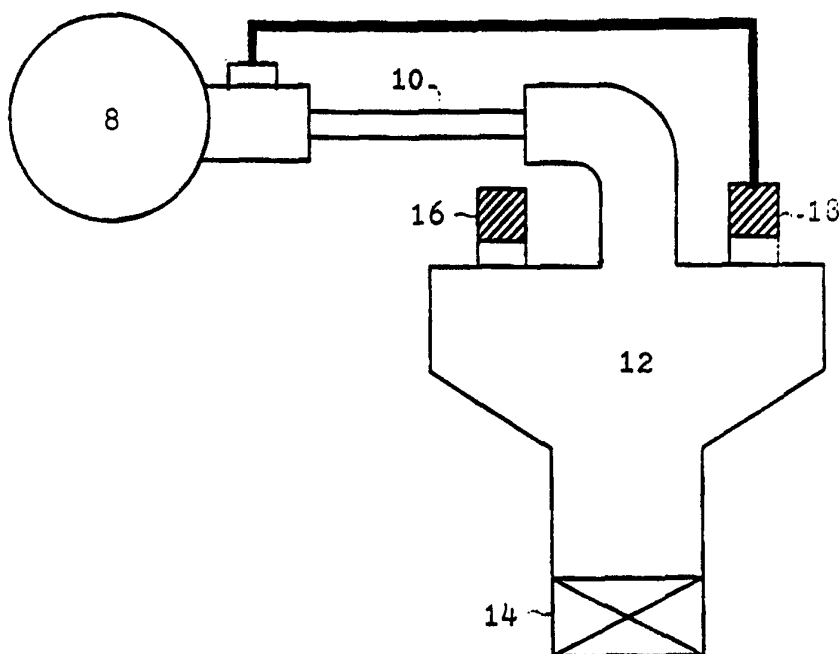




## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(54) Title:</b> METHOD AND APPARATUS FOR ON-STREAM MEASUREMENT OF RHEOLOGICAL PROPERTIES		
<b>(57) Abstract</b>  A method and apparatus for on-stream measurement of rheological properties of a sample. Sample flows through an opening of known dimensions (10) into a closed container (12) where flow is determined using the rate of change of pressure in the closed container (12). From repeated measurements during a single pass in which the flow rate starts at a maximum and approaches zero, stress vs. strain profiling can be achieved.		



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## METHOD AND APPARATUS FOR ON-STREAM MEASUREMENT OF RHEOLOGICAL PROPERTIES

The present invention relates to the characterization of flow behavior of liquids and slurries within process conditions.

5 Rheological information is important in industries such as mining and slurry processing. One objective in the slurry processing business, for example, is to maximize solid throughput while minimizing the threat of pipe plugging. Plugging may be caused by shear rates below the "yield value" of the liquid; yield value refers to the minimum force necessary to exert on a fluid to cause it to flow. Thus, information on the flow characteristics of fluids,  
10 including yield value, is important for efficient plant operation in some industries.

Newtonian liquids flow immediately upon application of force and the rate of flow is directly proportional to the force applied. Therefore, full rheological characterization is not an important concern with Newtonian liquids such as water and gasoline, whose viscosities essentially remain constant as shear rate varies. Non-Newtonian liquids, however, show non-  
15 linear flow responses when force or stress is applied; in other words, their viscosities are dependent on the rate of shear. Thus, traditional viscosity measurements, operating at single shear rates, are insufficient to determine full rheological behavior of non-Newtonian liquids.

Further, in many cases it is not possible to perform manual, off-line sampling. In such cases an on-stream analyzer would be necessary.

20 The art of rheological measurement would be improved by a more efficient method and apparatus for determining on-stream the flow behavior of such non-Newtonian fluids within process conditions.

In one respect, the present invention is a method of determining rheological properties of a sample using the rate at which said sample flows into a known volume to  
25 determine shear strain in relation to the dimensions of a conduit, and using the corresponding pressure difference across the conduit as said sample enters said known volume to determine shear stress.

The present invention is also a method of determining rheological properties of a sample comprising four steps. Step one of this embodiment of the present invention is to flow  
30 a stream of said sample through a conduit having known dimensions. Step two is to flow said stream into a sealed vessel containing a known volume of a gas and having a known initial pressure. Step three is to measure simultaneously the pressure of the gas in said sealed vessel, the difference in pressure across said conduit, and the time said stream has flowed into said sealed vessel. Step four is to calculate a shear stress from the difference in pressure across said  
35 conduit and to calculate the corresponding shear strain from the rate of change of pressure of the gas in said sealed vessel.

Another embodiment of the present invention is a method of determining the average shear strain of a sample comprising six steps. Step one is to flow a stream of the

sample through an opening of known dimensions into a sealed vessel having a known volume and containing a steady number of moles of a gas, such that as the sample flows into the sealed vessel the gas is compressed. Step two is to measure the pressure of the gas at a first time. Step three is to measure the pressure of the gas at a second time. Step four is to measure the interval between the first time and the second time. Step five is to use the difference in pressure of the gas between the first time and the second time to determine the volume of sample which entered the sealed vessel between the first time and the second time. Step six is to determine the average shear strain of the sample, in relation to the dimensions of the opening, from the volume of sample which flowed into the sealed vessel in the interval between the first time and the second time.

Another embodiment of the present invention is an apparatus suitable for determining rheological properties of a sample comprising a conduit, a closable receiving vessel, a means for measuring the change in pressure across said conduit, and a means for measuring pressure in said receiving vessel; said conduit is in fluid communication with said receiving vessel.

Fig. 1 is a schematic diagram of an embodiment of the apparatus of the present invention in which hydrostatic pressure will not oppose flow.

Fig. 2 is a schematic diagram of an embodiment of the apparatus of the present invention in which hydrostatic pressure will oppose flow.

In one respect, the present invention is a method of determining rheological properties of a sample using the rate at which said sample flows into a known volume to determine shear strain in relation to the dimensions of a conduit, and using the corresponding pressure difference across the conduit as said sample enters said known volume to determine shear stress.

The present invention is also a method of determining rheological properties of a sample comprising four steps. Step one is to flow a stream of said sample through a conduit having known dimensions. Step two is to flow said stream into a sealed vessel in which pressure, temperature, and volume can be determined simultaneously. Step three is to measure simultaneously the difference in pressure across said conduit, and the rate at which said stream of sample flows into said sealed vessel. Step four is to calculate a shear stress from the difference in pressure across said conduit and to calculate the corresponding shear strain from the rate at which sample flows into said sealed vessel.

Another embodiment of the present invention is a method of determining rheological properties of a sample comprising the following steps. Step one of this embodiment of the present invention is to flow a stream of said sample through a conduit having known dimensions. Step two is to flow said stream into a sealed vessel containing a known volume of a gas and having a known initial pressure. Step three is to measure simultaneously the pressure of the gas in said sealed vessel, the corresponding difference in

pressure across said conduit, and the time said stream of sample has flowed into said sealed vessel. Step four is to calculate a shear stress from the difference in pressure across said conduit and to calculate the corresponding shear strain from the rate of change of pressure of the gas in said sealed vessel.

5                   The word "conduit" as used in this specification means any configuration through which fluid may flow to a receiving area and which obeys the laws underlying the equations that are described herein. For example, a suitable conduit may be a tube. Preferably, the known dimensions of the conduit include the inner diameter and length.

                  The term "sealed" means substantially air tight such that pressure measurements  
10   may be obtained. This result may be achieved in various ways, such as deliberately operating a vessel with a known rate of leakage provided that one compensates for the leakage in the calculations. However, the preferred mode is to use a vessel from which any loss of gas is negligible when obtaining pressure measurements. A vessel may be permanently sealed, or, preferably, sealed periodically such as by closing a valve.

15                   The vessel receiving the sample may be any type of container suitable to hold sample.

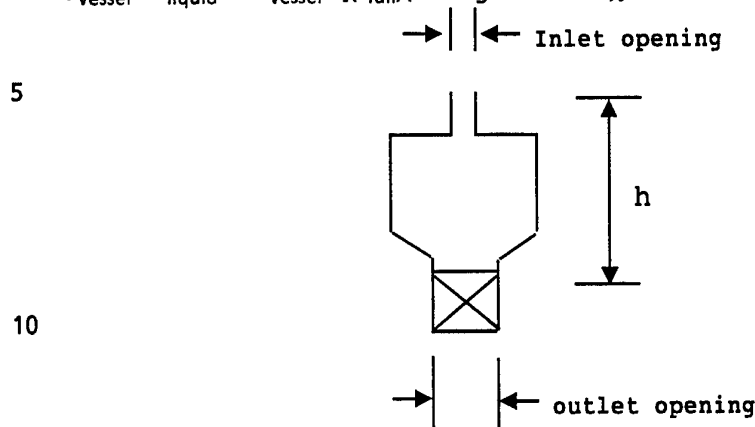
                  One way to determine the volume of gas in the vessel is by subtracting the volume of liquid in the sealed vessel ( $V_{\text{liquid}}$ ) from the volume of the sealed vessel itself ( $V_{\text{vessel}}$ ). The volume of the sealed vessel itself may be determined by filling the vessel with water and  
20   then removing and measuring the volume of this water. Next, the volume of liquid in the vessel when the test is begun should be calculated in order to accurately determine the initial volume of gas in the vessel. However, the relative importance of correcting for the volume of liquid in the vessel at the beginning of the test diminishes as the volume of the liquid stream is decreased in relation to the volume of the vessel itself, for example, by using a smaller inlet  
25   opening.

                  If the test is begun immediately after sealing the vessel, the only liquid in the vessel is from the stream of sample between the inlet to the vessel and the valve because the sample has not had time to accumulate in the vessel. Assuming the inlet opening letting  
30   sample into the vessel is positioned directly above the valve as in the figure below, the volume of sample in the stream between the inlet opening and the valve can be determined from the rate at which the sample flows into the vessel and the time taken for the liquid to fall through the distance between the inlet opening and the valve. The flow rate for this purpose may be determined by placing a beaker under the drainage valve and dividing the collected volume of the sample by the time over which the sample was collected. The time taken ( $t_{\text{fall}}$ ) for the liquid  
35   to fall through the distance from the inlet opening to the valve (height) can be calculated from the formula:

$$\text{height} = (1/2)g(t_{\text{fall}})^2$$

where  $g$  is the acceleration due to gravity ( $9.81 \text{ m/sec}^2$ ). Therefore, the volume of gas at the beginning of the test =  $V_o =$

$$V_{\text{vessel}} - V_{\text{liquid}} = V_{\text{vessel}} - [(t_{\text{fall}})(\text{average flow rate})].$$



With this established, the operation of the apparatus proceeds as follows. From Boyle's Law,  $P_o V_o = P_f V_f$  at constant temperature. Thus, by determining  $V_o$  as described above, one can calculate the volume of gas in the vessel ( $V_f$ ) and hence the volume of sample in the vessel at any time during the test by measuring the pressure in the vessel ( $P_f$ ).

Preferably, the pressure difference across the conduit and the rate the stream of sample flows into the receiving vessel are simultaneously and repeatedly determined such that shear stress and shear strain can be calculated over a wide range of values and a stress vs. strain profile can be obtained. In other words, preferably, steps three and four of this embodiment of the present invention are repeated over time. Most preferably, the pressure difference across the conduit and the pressure in the vessel are measured repeatedly from the time the sample begins to flow into the sealed vessel until the flow of sample into the vessel ceases as indicated by the pressure difference across the conduit approaching a minimum value within an acceptable time frame. The minimum pressure difference is usually zero.

For example, to employ the method of the present invention one could perform the following steps. Referring now to Fig. 1, a small volume of sample is continuously tapped from the main process line 8 which passes through the rheometer while the drainage valve 14 is left open. The drainage valve is then closed and the pressure readings from the differential transducer 18 and the absolute pressure transducer 16 are collected. Both pressure readings are obtained simultaneously and repeatedly until the reading of the differential pressure transducer 18 approaches a minimum value. The drainage valve 14 may then be opened to drain the vessel in preparation for the next test.

Four rheological parameters can be determined from this test: (1) shear stress, (2) shear rate or shear strain, (3) yield stress, and (4) apparent viscosity.

The first parameter, shear stress, is given by the equation:

$$\text{shear stress} = \Delta PD/4L$$

where  $\Delta P$  is the flow pressure  
 i.e., the difference in pressure across the conduit;  
 $D$  is the inner diameter of the conduit; and  
 $L$  is the length of the conduit.

#### 5 Example 1

To determine the rheological properties of a Carbopol™-941 solution at 0.2% concentration, using a 0.425 cm diameter and 45.6 cm length stainless steel tube, the following data were obtained:

Initial volume of the vessel,  $V_o = 1672.77 \text{ cm}^3$

10 Initial pressure of air in vessel with valve open,  $P_o = 14.16 \text{ psig}$  (97,660 Pa)

Vessel pressure at time = 40 sec,  $P_{t=40} = 14.92 \text{ psig}$  (102,900 Pa)

Differential pressure at  $t = 40 \text{ sec}$ ,  $\Delta P_{t=40} = 3.18 \text{ psi}$  (21,930 Pa)

Number of differentiation points,  $N = 30$

Slope of pressure vs. time curve,

15  $dP/dt$  at  $t = 40 \text{ sec} = 0.0155 \text{ psi/sec}$  (107 Pa/sec)

Thus, Boyle's law constant,  $K = V_o P_o = (1672.77 \text{ cm}^3)(14.16 \text{ psig}) = 23686.42 \text{ cm}^3 \cdot \text{psig}$ ,

and flow rate,  $Q_{t=40} = \frac{K}{(P_{t=40})^2} \frac{dP}{dt} =$

$\frac{(23686.42 \text{ cm}^3 \cdot \text{psig})(0.0155 \text{ psi/sec})}{(14.92 \text{ psig})^2} = 1.649277 \text{ cm}^3/\text{sec}$ .

20  $(14.92 \text{ psig})^2$

The purpose of this example is to demonstrate how shear stress may be determined. As stated above, shear stress is calculated from the equation:

$$\text{shear stress} = \frac{\Delta P D}{4 L}$$

25 Thus, using the data obtained, shear stress =

$$\frac{(3.18 \text{ psi})(0.425 \text{ cm})(68947.5729 \text{ dynes/cm}^2 \cdot \text{psi})}{4(45.6 \text{ cm})} = 510.87 \text{ dynes/cm}^2$$

The method shown in this example is useful to calculate a value for shear stress of a sample, however, other methods of calculation may also be used.

30 The second parameter, shear strain or shear rate, is calculated using the equation:

$$\text{shear strain} = 8Q/DA$$

where  $Q$  is the flow rate of the stream of sample through a conduit;

$D$  is the inner diameter of the conduit; and

$A$  is the cross-sectional area of the conduit.

35 In the equation above, the flow rate,  $Q$ , may be calculated from the rate of change of gas volume in the vessel. Referring to Fig. 1, as soon as the drainage valve 14 is closed, sample begins to accumulate in the vessel 12 such that the gas in the vessel is compressed and the gas volume decreases. The change in gas volume is determined from

Boyle's law, which states that, at constant temperature, the volume of a sample of gas is directly proportional to its pressure. This is expressed as  $PV = K$  where  $P$  is pressure,  $V$  is volume and  $K$  is a constant. Therefore, given an initial volume and pressure at the beginning of the test, one can calculate the volume of the gas inside the vessel at any time during the test from a pressure measurement. Constant temperature should be maintained throughout the test for ease of calculation when applying Boyle's law. This may be achieved by means such as steam or electrical tracing with temperature control. In the laboratory, constant temperature may be maintained by means such as a water bath.

The flow rate may be calculated using the equation:  $Q = (V_t - V_o)/t$

wherein

$t$  = the time the stream has flowed into the sealed vessel;

$V_t = P_o V_o / P_t$  = the volume of gas in the sealed vessel after some time,  $t$ ;

$V_o$  = the initial volume of gas in the sealed vessel,

$P_o$  = the initial pressure of gas in the sealed vessel; and

$P_t$  = the pressure of gas in the sealed vessel after some time,  $t$ .

Another way to express the flow rate,  $Q$ , is by the equation:  $Q = dV/dt$ .

Because  $V = K/P$ ,  $Q = \frac{dV}{dt} = \frac{d(K/P)}{dt} = \frac{K}{P^2} \frac{dP}{dt}$

Therefore, flow rate at any time,  $t$ , is calculated by measuring the vessel pressure, ( $P_t$ ), and the slope of the pressure vs. time curve at time  $t$ , ( $dP_t/dt$ ). This slope may be calculated using the following formula:

$$\frac{dP}{dt} = \frac{\sum XY - [(\sum X)(\sum Y)/N]}{\sum (X^2) - [(\sum X)^2/N]}$$

where  $X$  = time,  $t$

$Y$  = vessel pressure,  $P_t$

and  $N$  = number of differentiation points.

#### Example 2

This example uses the data obtained in Example 1, to demonstrate how shear rate can be calculated from the flow rate,  $Q$ .

$$\text{Shear rate} = \frac{8Q}{DA}$$

where  $A$  = cross-sectional area =  $\pi r^2 = \pi D^2/4$ .

Thus, shear rate =  $\frac{32Q}{\pi D^3} = \frac{32(1.649277 \text{ cm}^3/\text{sec})}{\pi(0.425 \text{ cm})^3} = 218.84 \text{ sec}^{-1}$

for a 0.2% solution of Carbopol™-941 as described in Example 1.

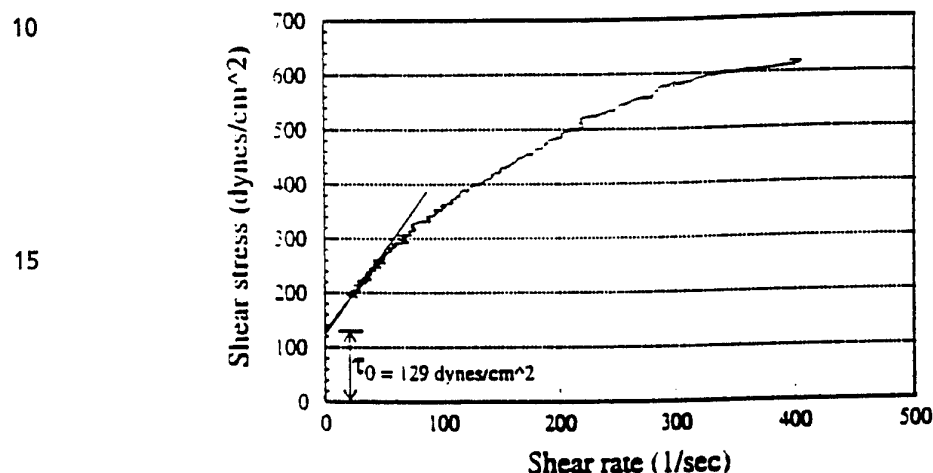
The third rheological parameter, yield value, which is also called yield point or yield stress, can be obtained by plotting a graph of shear stress vs. shear rate as those



parameters are described above. The yield value is the value at the intersection of the shear stress vs. shear rate curve with the Y-axis, which is the shear stress axis in this case. If necessary, the curve obtained from the data collected can be extended to intersect with the Y-axis. For example, the curve may be extended by a line tangential to the curve at low shear rates.

### 5 Example 3

This example demonstrates a way to determine yield stress or yield value using data obtained following the method of the present invention. The following shear stress vs. shear rate curve was obtained for a 0.2% solution of Carbopol™-941 as described in Example 1.



20

From this graph the yield stress,  $T_0$ , was found to be 129 dynes/cm<sup>2</sup>.

The final parameter, apparent viscosity, is obtained by dividing shear stress by shear rate.

### Example 4

25 The apparent viscosity of a 0.2% solution of Carbopol™-941 may be obtained using the shear stress and shear rate calculated in Examples 1 and 2. The apparent viscosity at the shear rate calculated in Example 2 =  $\frac{\text{shear stress}}{\text{shear rate}}$

(510.87 dynes/cm<sup>2</sup>)

30 (218.84 sec<sup>-1</sup>) = 2.3344 poise = 233.44 centipoise.

In addition to being a method of obtaining a stress vs. strain profile of a sample, another embodiment of the present invention is a method of determining the average shear strain of a sample comprising six steps. Step one is to flow a stream of the sample through an opening of known dimensions into a sealed vessel having a known volume and containing a steady number of moles of a gas, such that as the sample flows into the sealed vessel the gas is compressed. Step two is to measure the pressure of the gas at a first time. Step three is to measure the pressure of the gas at a second time. Step four is to measure the interval between the first time and the second time. Step five is to use the difference in pressure of the gas

between the first time and the second time to determine the volume of sample entering the sealed vessel during the interval between the first time and the second time. Step six is to determine the average shear strain of the sample, in relation to the dimensions of the opening, from the volume of sample which flowed into the sealed vessel in the interval between the first  
5 time and the second time. As described above, the shear strain may be calculated by the equation  $\text{shear strain} = 8Q/DA$  where Q is the flow rate, A is the cross-sectional area of the opening, and D is the inner diameter of the opening. Determination of average shear strain is useful if shear strain changes over time.

The phrase "steady number of moles of a gas" means any change in the number  
10 of moles of gas in the vessel is negligible, for example, due to leakage.

Another embodiment of the present invention is an apparatus suitable for determining rheological properties of a sample comprising a conduit, a closable receiving vessel, a means for measuring the change in pressure across said conduit, and a means for measuring absolute pressure in said receiving vessel; said conduit is in fluid communication  
15 with said receiving vessel.

Referring now to Fig. 1, a conduit 10 is shown through which sample flows into a receiving vessel 12. The receiving vessel 12 is in turn in fluid communication with a release valve 14 through which sample can be removed from the receiving vessel 12 and accumulated pressure can be released. A pressure transducer 16 is connected to the receiving vessel 12 to  
20 measure gas pressure in the receiving vessel. Another pressure transducer 18 measures the pressure difference across the conduit 10.

Preferably, the conduit is detachable such that conduits of different length to diameter ratio may be used.

Preferably, the conduit is in fluid communication with the receiving vessel such  
25 that the hydrostatic pressure of the sample in the receiving vessel will not oppose the flow of the sample from the tube. In other words, the sample freely falls into the receiving vessel.

Alternatively, the conduit is in fluid communication with the receiving vessel such that the hydrostatic pressure of the sample in the receiving vessel will oppose flow. This may occur if the flow of sample rises within the receiving vessel relative to the opening through  
30 which the sample enters the receiving vessel. For example, hydrostatic pressure of the sample may oppose flow if the receiving vessel were shaped like a U-tube and sample entered one closed vertical leg of the U-tube from the bottom.

Referring now to Fig. 2, therein is shown an embodiment of the apparatus of the present invention in which hydrostatic pressure of the sample will oppose flow. This apparatus  
35 uses controlled gas flow to manipulate sample in a controlled-geometry chamber or a receiving vessel. The analysis mechanism is to isolate a fresh sample from the main process line 38 by using an automatic valved bypass. The isolated sample is then manipulated within a "U-tube" system in which each side is equipped with a precision gas mass flow controller 30 and 32, a

pressure transducer 34 and 36, and a vent valve 26 and 28 to manipulate and monitor the sample. Suitable gas mass flow controllers include common, proven Matheson or Brooks type. After analysis, the sample is discharged and the system is blown clean.

The conduit in the center of the "U-tube" 20 acts as the tube viscometer to isolate the analysis of the sample in the system. Calculations based on the gas laws and the Hagen-Poiseuille equation reveal the motion of the sample and the corresponding forces. Essentially the "U-tube" can be divided into two sides when a sample is present in the system. The center of the conduit 20 will be the dividing point in the system. The operation of the analyzing model is based on Boyle's Law which can be written for both sides of the "U-tube";  $P_1V_1 = K_1$  and  $P_2V_2 = K_2$ . Due to the symmetrical design of the system,  $P_1V_1 = P_2V_2$  at the moment the ball valves 22 and 24 are closed and before any forced gas flow is introduced to either side of the system. This equation describes static equilibrium in the system.

As gas flow is introduced to one side of the system to move the sample through the conduit, the system becomes dynamic. The following equation describes the dynamic condition in the system:

$$P_q = P_1 - P_2 - pgh$$

where

$P_q$  = flow pressure, i.e., Hagen-Poiseuille related pressure due to the resistance to flow through the conduit;

$P_1$  = pressure on the forcing side from which gas flow is introduced into the system;

$P_2$  = pressure on the reacting side which is opposite the gas flow side;

$pgh$  = pressure due to the head on the reacting side;

$p$  = density of material;

$g$  = force due to gravity; and

$h$  = height of head.

For the tube viscometer to produce valid readings, the length-to-diameter ratio of the conduit is preferably greater than fifty. When this condition is met, the flow inside the tube becomes laminar and the entrance and exit effects of the conduit become negligible. The entrance and exit area of the conduit is a gradual slope and not a square surface. This slope also helps to eliminate potential entrance and exit effects and eliminates dead spots which might be seen at the corners in a square surface.

The flow rate within the system is calculated by repeatedly using Boyle's Law to monitor the changing volume over a period of time. This may be achieved by noting when the system is at static equilibrium, the initial pressure and initial volume are known for each side of the "U-tube". Thus, using Boyle's law, constant  $k$  can be determined for both sides by using standard atmospheric pressure as the initial pressure, and the air space only, not including sample, as the initial volume. When dynamic conditions occur, constant  $k$  is known and the

pressure at time (t) may be monitored; therefore, the volume can be calculated during the dynamic conditions. Using a differential method, the change in volume on the reacting side can be calculated to determine the flow rate in units of volume change/second:  $V(\text{change}) = V_{(t+1)} - V_{(t)}$ . Only the reacting side is used in the calculations because the forcing side has a gas flow introduced to it.

From the flow pressure ( $P_q$ ) the shear stress can be calculated using the dimensions of the conduit; and from the flow rate, the shear rate can also be calculated using the dimensions of the conduit by applying the equations discussed above with respect to the method of the present invention.

Preferably, the receiving vessel is of fixed volume such that the volume of the vessel does not change even though the volume of sample and gas inside the vessel does change. Further, the vessel should be closed to the outside atmosphere during testing so the gas inside the vessel may be compressed.

Preferably, the means for measuring the change in pressure across the conduit and the means for measuring pressure in the receiving vessel are pressure transducers. Suitable pressure transducers may be purchased commercially from Rosemount, Inc. at 12001 Technology Drive, Eden Prairie, MN 55344 or Foxboro, Inc. For example, a suitable absolute pressure transducer to measure pressure in the receiving vessel is Rosemount Model 3051CA2A22A1A; and a suitable differential pressure transducer to measure pressure across the conduit is Rosemount Model 3051CG4A22A1A.

Preferably, the receiving vessel is in fluid communication with a release valve which may be used periodically to release accumulated pressure and to drain sample from the receiving vessel.

One may also be able to achieve the same results using an apparatus held at constant pressure and volume while measuring change in temperature or using an apparatus held at constant pressure and temperature while measuring change in volume by applying the equation  $PV = nRT$ .

30

35

## WHAT IS CLAIMED IS:

1. A method of determining rheological properties of a sample comprising:
  - (a) using the rate at which said sample flows into a known volume which is at a pressure of at least ambient pressure, and using the dimensions of a conduit through which
 

5 said sample flows into the known volume to determine shear strain; and
  - (b) using the corresponding pressure difference across the conduit as said sample enters said known volume to determine shear stress.
2. A method of determining rheological properties of a sample comprising:
  - (a) flowing a stream of said sample through a conduit having known
 

10 dimensions;
  - (b) flowing said stream into a sealed vessel which is at a pressure of at least ambient pressure, and in which pressure, and temperature can be determined simultaneously;
  - (c) simultaneously measuring the difference in pressure across said conduit, and determining the rate said stream of sample flows into said sealed vessel; and
  - (d) calculating a shear stress from the difference in pressure across said conduit
 

15 and calculating the corresponding shear strain from the rate at which sample flows into said sealed vessel.
3. A method of determining rheological properties of a sample comprising:
  - (a) flowing a stream of said sample through a conduit having known
 

20 dimensions;
  - (b) flowing said stream into a sealed vessel containing a known volume of a gas and having a known initial pressure which is at least ambient pressure;
  - (c) simultaneously measuring the pressure of the gas in said sealed vessel, the corresponding difference in pressure across said conduit, and the length of time said stream of
 

25 sample has been flowing into said sealed vessel; and
  - (d) calculating a shear stress from the difference in pressure across said conduit and calculating the corresponding shear strain from the rate of change of pressure of the gas in said sealed vessel.
4. The method of Claim 3 wherein steps (c) and (d) are repeated.
5. The method of Claim 3 wherein step (d) is accomplished by
  - (a) calculating the shear stress using the equation:
 

shear stress =  $\Delta PD/4L$

wherein

$\Delta P$  is the difference in pressure across the conduit;

D is the diameter of the conduit; and

L is the length of the conduit; and

  - (b) calculating the shear strain using the equation: shear strain =  $8Q/DA$

wherein

Q is the flow rate of the stream of sample through the conduit;

D is the diameter of the conduit; and

A is the cross-sectional area of the conduit.

- 5           6.     The method of Claim 5 wherein the flow rate, Q, is calculated using the equation:

$$Q = (V_t - V_o)/t$$

wherein

t = the length of time the stream has been flowing into the sealed vessel;

10            $V_t = P_o V_o / P_t$ ;

$V_o$  = the initial volume of gas in the sealed vessel;

$P_o$  = the initial pressure of gas in the sealed vessel; and

$P_t$  = the pressure of gas in the sealed vessel after said time, t.

- 15           7.     A method of determining the average shear strain of a sample comprising:

- (a)     flowing a stream of the sample through an opening of known dimensions into a sealed vessel having a known volume, which is at a pressure of at least ambient pressure, and containing a fixed number of moles of a gas such that as the sample flows into the sealed vessel the gas is compressed;

- 20           (b)     measuring the pressure of the gas at a first time;
- (c)     measuring the pressure of the gas at a second time;
- (d)     measuring the interval between the first time and the second time;
- (e)     using the difference in pressure of the gas between the first time and the second time to determine the volume of sample which entered the sealed vessel between the first time and the second time; and

- 25           (f)     determining the average shear strain of the sample from the volume of sample which flowed into the sealed vessel in the interval between the first time and the second time.

8.     An apparatus suitable for determining rheological properties of a sample comprising a conduit through which the sample is to flow, a closable receiving vessel containing a gas which is at least at ambient pressure, a means for determining the difference in pressure across said conduit, and a means for measuring pressure of the gas in said receiving vessel; said conduit is in fluid communication with said receiving vessel.
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9.     The apparatus of Claim 8 wherein said receiving vessel is of fixed volume.

10.    The apparatus of Claim 8 wherein said conduit is in fluid communication with said receiving vessel which receives said sample such that hydrostatic pressure of said sample in said receiving vessel will not oppose the flow of said sample from said conduit.
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11. The apparatus of Claim 8 wherein said conduit is in fluid communication with said receiving vessel which receives said sample such that hydrostatic pressure of said sample in said receiving vessel will oppose flow.

12. The apparatus of Claim 8 wherein said means for determining the  
5 difference in pressure across the conduit and said means for measuring pressure of a gas in said receiving vessel are pressure transducers.

13. The apparatus of Claim 8 wherein said receiving vessel is in fluid communication with means for releasing the sample from the receiving vessel.

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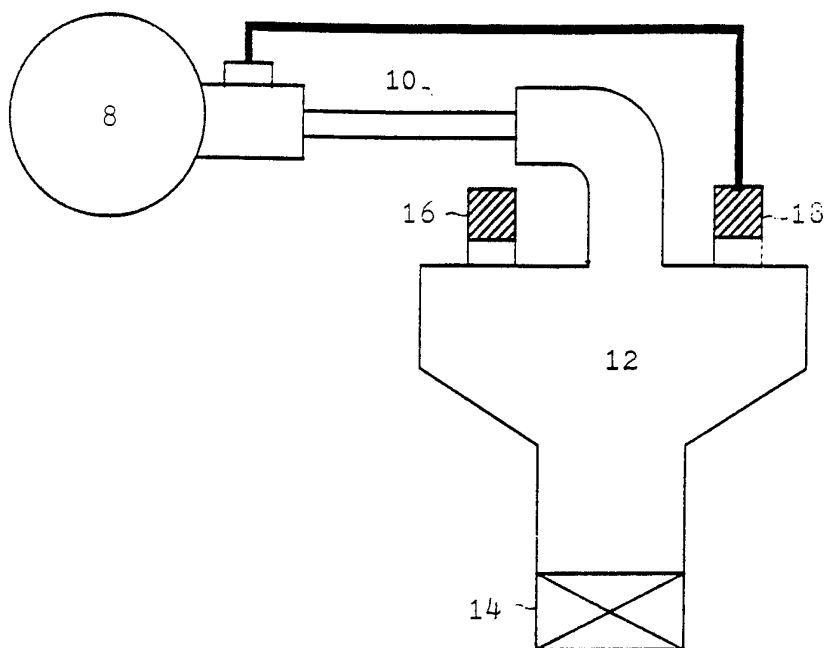
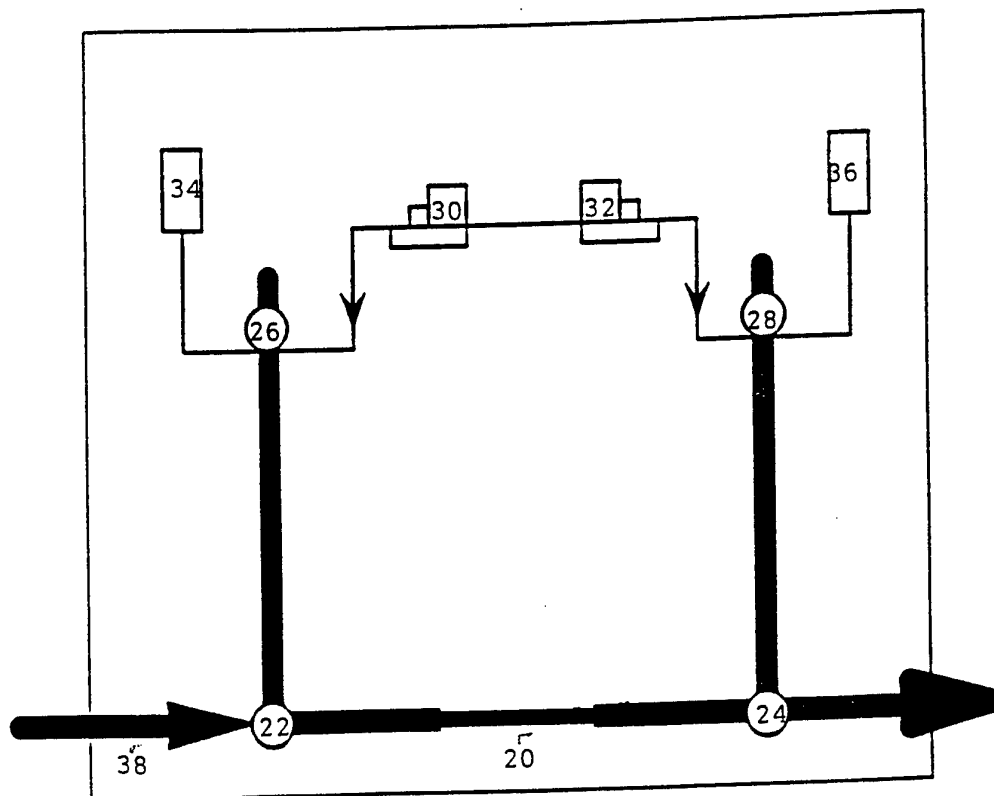


FIG. 1

FIG. 2





## INTERNATIONAL SEARCH REPORT

 International Application No  
 PCT/US 95/07945

 A. CLASSIFICATION OF SUBJECT MATTER  
 IPC 6 G01N11/06

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

 Minimum documentation searched (classification system followed by classification symbols)  
 IPC 6 G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y A	US,A,3 677 069 (RUBIN) 18 July 1972 see column 1, line 70 - column 2, line 10 see column 4, line 70 - column 5, line 52; figures ---	1 2-6 10
Y A	EP,A,0 492 664 (NISSHO CORPORATION) 1 July 1992 see page 3, line 4 - line 40 see page 5, line 4 - page 6, line 35 see figures ---	2-6 8,11
A	US,A,4 495 798 (EHRGOTT) 29 January 1985 see column 3, line 7 - line 28; figure 2 see column 5, line 18 - line 58; figure 4 --- -/--	1

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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"&amp;" document member of the same patent family

Date of the actual completion of the international search

13 October 1995

Date of mailing of the international search report

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# INTERNATIONAL SEARCH REPORT

International Application No  
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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A	US,A,3 952 577 (CANADIAN PATENTS AND DEVELOPMENT LTD) 27 April 1976 see column 9, line 43 - line 54; figure 2 see column 18, line 55 - line 68; figure 9 -----	1

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Information on patent family members

International Application No

PCT/US 95/07945

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