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Stark et al.

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[54] **CAPILLARY VISCOSIMETER**

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[52] **U.S. Cl.** **73/55**
[51] **Int. Cl.** **G01n 11/06**
[58] **Field of Search** 73/55, 54, 56

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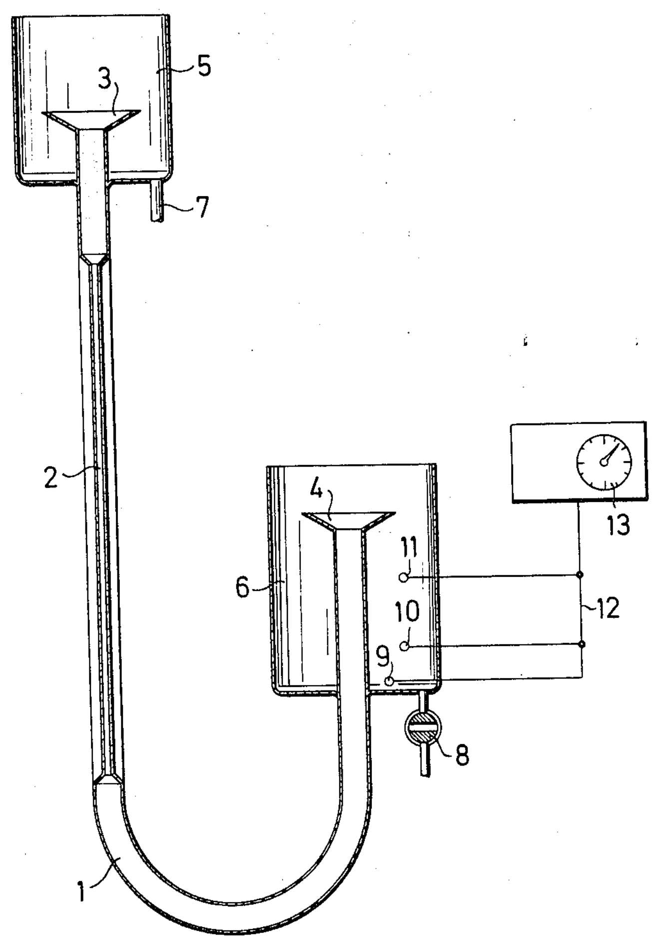
[57] **ABSTRACT**

A capillary viscosimeter of the type in which liquid is supplied in excess to an overflow vessel upstream of the capillary during a viscosity measurement and is discharged downstream of the capillary into a measuring vessel is described in which the liquid discharges into the measuring vessel from an overflow vessel which is geometrically similar to or identical with the upstream overflow vessel.

17 Claims, 4 Drawing Figures



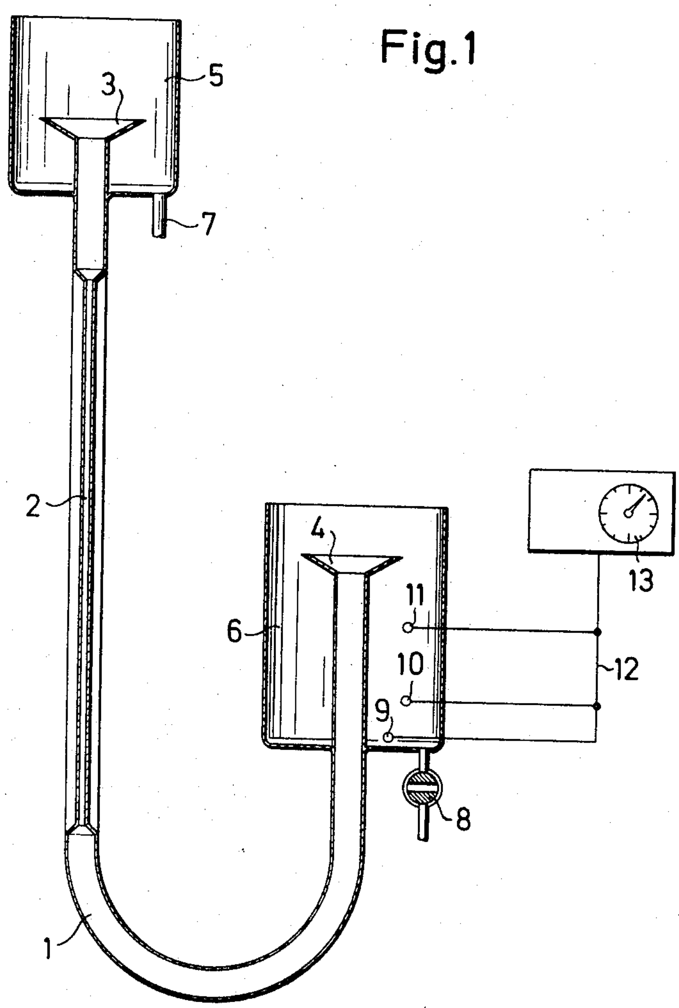


Fig.1

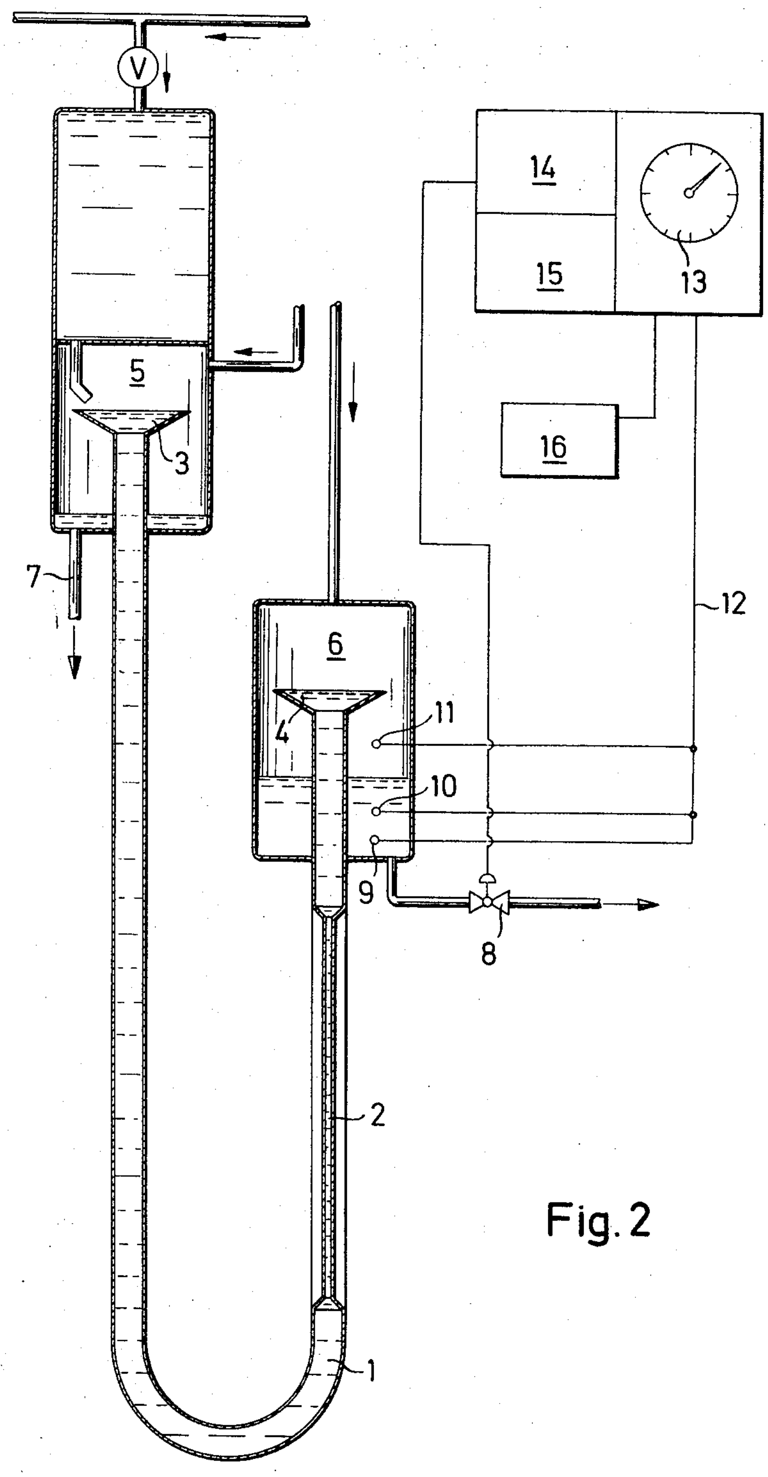
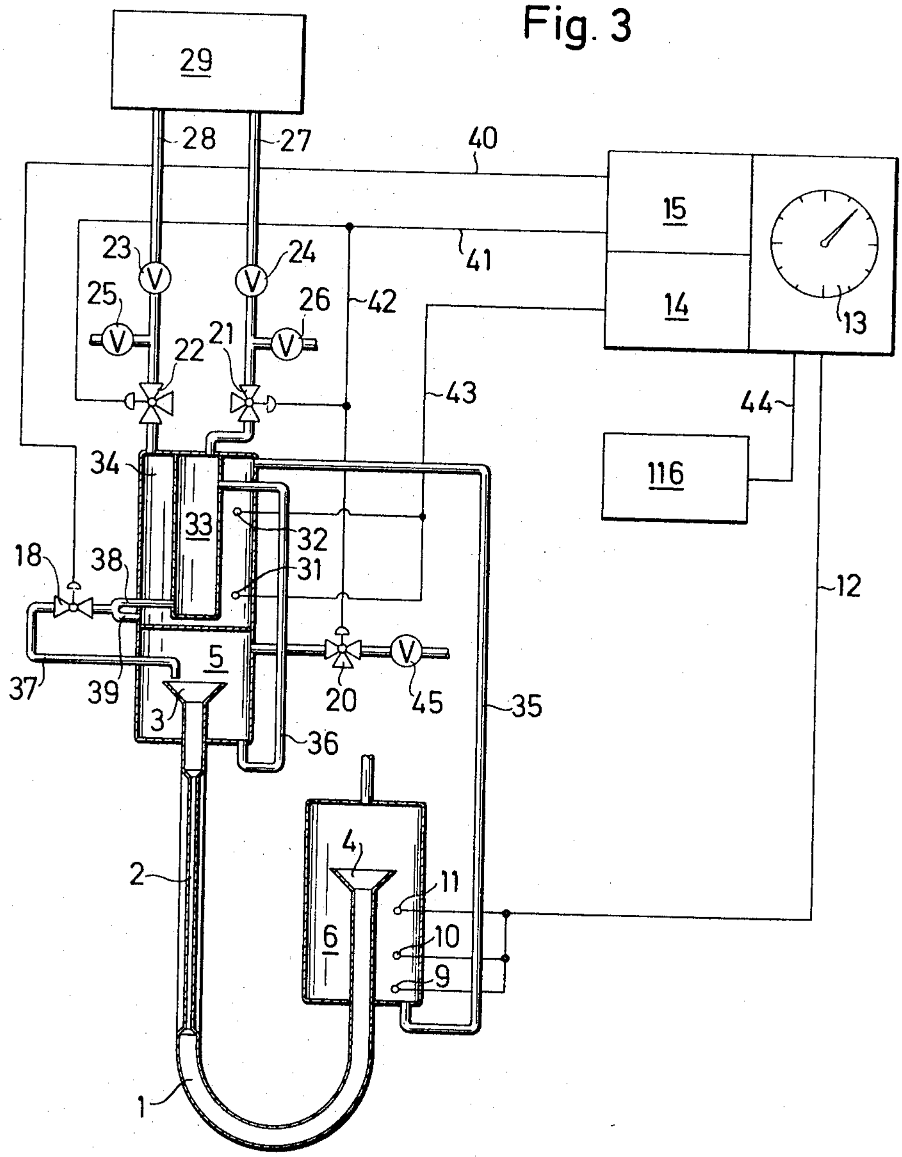


Fig. 2

Fig. 3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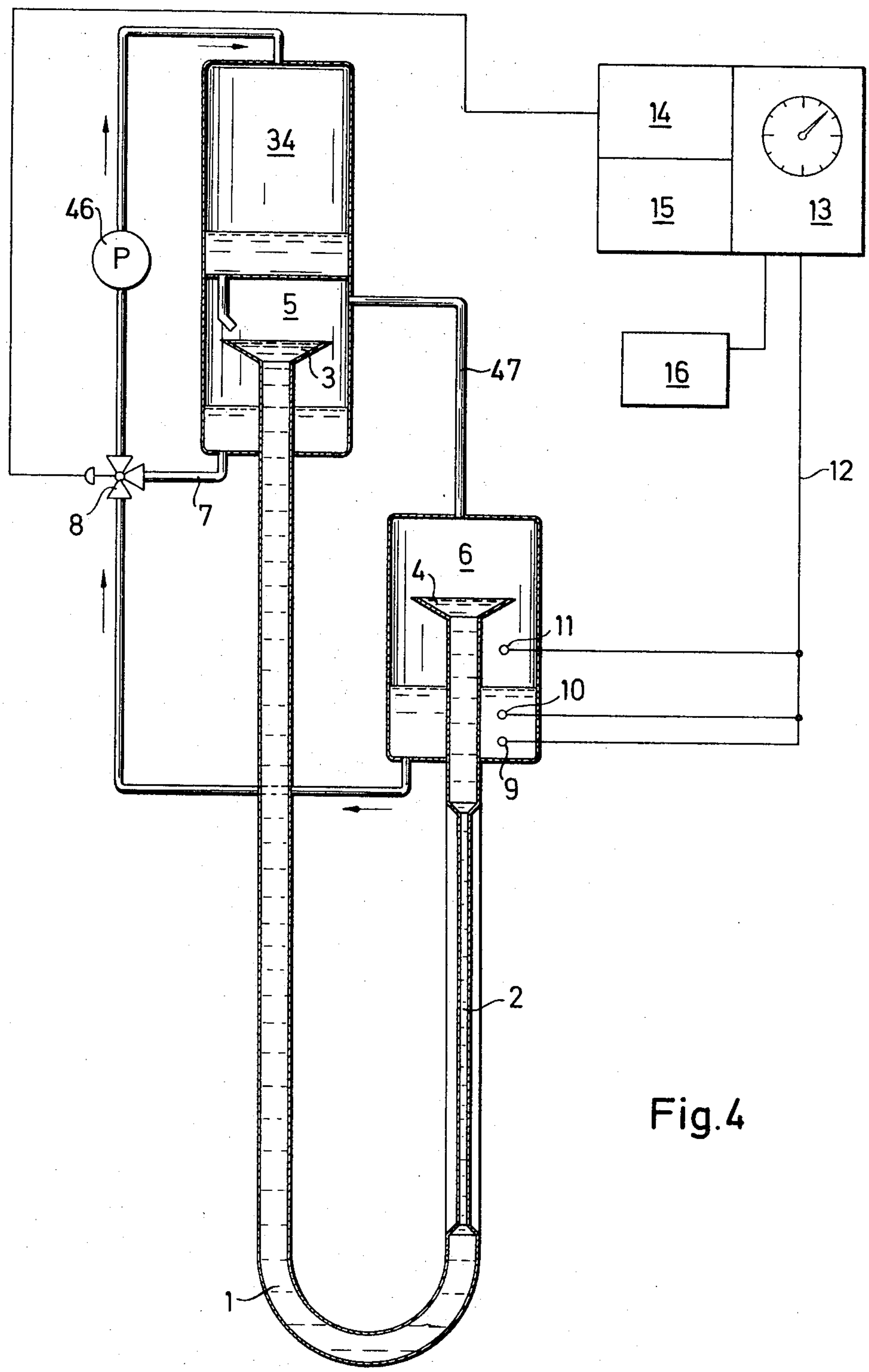

Fig.4

CAPILLARY VISCOSIMETER

FIELD OF THE INVENTION

The present invention relates to a capillary viscosimeter of the type in which an overflow vessel is connected upstream of the capillary and is adapted to be supplied with excess liquid during a viscosity measurement, and in which a measuring vessel is provided for collecting liquid which has passed through the capillary.

BACKGROUND TO THE INVENTION

A capillary viscosimeter of this general kind is disclosed in German Patent Specification No. 713,990. Such viscosimeters differ from other capillary viscosimeters (for example Ubbelohde viscosimeters and variations thereof such as that described by Cannon in U.S. Pat. No. 2,805,570) by virtue of the fact that it is not necessary for the liquid to flow twice through the measuring capillary in performing a complete measuring cycle.

The known capillary viscosimeter of German Patent No. Specification No. 713,990 comprises a vertical strand in which, as seen from top to bottom, a storage vessel for the liquid to be measured is followed by a drip capillary which in turn extends into a spherical overflow vessel which is disposed upstream of the measuring capillary proper. An overflow duct extends laterally from the overflow vessel and the measuring capillary extends from the lowest point of the overflow vessel. The lower end of the measuring capillary directly opens into a zone of increased cross-section which forms the upper end of a graduated measuring vessel. The said upper end of the aforementioned measuring vessel is connected through a rising pipeline to a manifold duct into which the overflow duct from the overflow vessel also extends, the lower end of the aforementioned manifold duct extending into a collecting vessel.

The hydrostatic pressure which acts on the liquid column in the measuring capillary of this arrangement is practically constant because the difference of head between the (constant) level in the overflow vessel and the meniscus which forms at the lower end of the measuring capillary is constant. However, the hydrostatic pressure is not the only force which acts on the liquid column and is therefore not the only factor which influences the rate of flow of the liquid through the measuring capillary per unit time. A further force acts as a result of the surface tension of the liquid itself particularly at the meniscus formed at the lower end of the measuring capillary so that an additional correction is necessary when working with the capillary viscosimeter of German Patent Specification No. 713,990.

It is an object of the present invention to provide a capillary viscosimeter in which the hydrostatic pressure across the capillary may be maintained constant whilst avoiding surface tension effects at the lower end of the capillary due to the formation of a meniscus.

It is another object of this invention to provide a simple accurate and reliable capillary viscosimeter.

These and other objects of the invention will become apparent from the following detailed description when taken together with the accompanying drawings.

SUMMARY OF THE INVENTION

The objects of this invention are obtained by a capillary viscosimeter, comprising:

- a capillary through which liquid is arranged to flow during a viscosity measurement;
- an overflow vessel connected upstream of said capillary and adapted to be supplied with an excess of said liquid during a viscosity measurement;
- a discharge overflow vessel connected downstream of said capillary and arranged to discharge liquid which has passed through said capillary by overflow; and
- a measuring vessel positioned to receive the overflow from said discharge overflow vessel.

Preferably the two overflow vessels are geometrically similar or better still geometrically identical. It is convenient if both overflow vessels are funnel-shaped. In this case any forces acting on the liquid column in the capillary and resulting from the surface tension of the liquid will be cancelled.

The capillary is advantageously provided as part of a U-shaped tube, the overflow vessels being connected to opposite ends thereof. In this case, it is advantageous if the discharge overflow vessel is connected directly downstream of the capillary. In other words, this means that when seen in the flow direction, the capillary is disposed in the rising branch of the U-shaped tube. This enables any air bubbles that may be formed to rise freely into the discharge overflow vessel without impairing the flow through the capillary.

As will be readily understood, the time taken for a predetermined volume of liquid to pass through the capillary provides a relative indication of viscosity. Alternatively if the geometry of the capillary is known, an absolute determination is possible.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic representation of a capillary viscosimeter constructed in accordance with the present invention;

FIG. 2 shows schematically a variation of the capillary viscosimeter adapted to periodically measure the viscosity of a liquid which is obtained continuously or at regular intervals from a storage vessel or a duct;

FIG. 3 schematically illustrates a modified capillary viscosimeter adapted for periodic measurement of the viscosity of a measured liquid specimen; and

FIG. 4 shows a further variation in schematic form.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring again to FIG. 1, there is shown diagrammatically an embodiment of capillary viscosimeter comprising a U-shaped tube **1** which may be constructed of glass or some other material and is disposed in a thermostatic bath which is not shown in the drawing. A measuring capillary is shown at **2**, and the upper end of that branch of the U-shaped tube which contains the said measuring capillary is provided with an overflow funnel **3** which opens into a collecting vessel **5** here shown open-topped. The lower end of the said collecting vessel is provided with a discharge connection **7**. The other branch of the U-shaped tube **1** is provided with a similarly formed overflow funnel **4** which opens into a measuring vessel **6**, here shown open at the top. The lower end of the said measuring vessel is provided with a discharge connection having a closable tap **8**. Contacts **9, 10, 11,** shown in FIG. **1**, are connected via a line **12** of control conductors to an electric clock. The contact **9** is arranged to function as a common terminal, the contact **10** to initiate timing and the contact **11** to cause the timing to cease. In operation liquid is supplied to funnel **3** continuously and in excess through the open top of vessel **5** so that it overflows into the said vessel over the rim of funnel **3** and is discharged through connection **7**. The timing procedure may be repeated at will by opening the tap **8**, discharging the measuring vessel and then closing the tap **8** again. Timing then occurs automatically.

FIG. **2** shows a variation of the capillary viscosimeter adapted to periodically measure the viscosity of a liquid which is obtained continuously or at regular intervals from a storage vessel or a duct. In this embodiment the capillary is incorporated into the shorter branch of the U-tube so that any bubbles occurring will automatically escape through the capillary. The timing apparatus and the discharge tap **8** are connected to control apparatus **14, 15** which is arranged to first open the discharge tap **8**, then to close the discharge tap, and periodically to initiate and complete the timing operation in accordance with a preset cyclic timing programme. A recorder **16** may be provided as shown for automatically recording the measured times. The liquid whose viscosity is to be measured is supplied to funnel **3** in excess from an inlet vessel supplied in turn through a valve from the duct. The excess liquid overflows from the rim of funnel **3** into vessel **5** as before and is discharged through connection **7**. The gas spaces in the closed top vessels **5** and **6** are maintained at the same pressure.

The modified capillary viscosimeter schematically illustrated in FIG. **3** is adapted for periodic measurement of the viscosity of a measured liquid specimen which is returned into the storage vessel after measurement is completed. To this end, the viscosimeter is provided with two separate inlet vessels **34, 33** which may be connected by ducts **38** and **39** to a remotely controlled valve **18** and thence through a duct **37** to the vessel **5**. In operation, a measured liquid volume is first filled into one of the two vessels **33** or **34**, the valve **18** remaining closed for the time being. The said valve is then opened and liquid begins to flow through the duct **37** into the overflow funnel **3**. The liquid flow through the duct **37** should exceed the amount which is discharged through the capillary **2** so that the funnel **3** is completely filled and overflows along its edge into the vessel **5**. The viscosimeter becomes filled with liquid to the edge of the funnel **4** and liquid will then also overflow from this funnel into the measuring vessel **6**. The filling process is completed when the entire supplied liquid volume has been discharged from the vessel **33** or **34**.

Each of the vessels **33, 34** is connected by a respective duct **27, 28** to a vacuum chamber **29**. Each duct **27, 28** includes a respective control valve **21, 22** and a pressure regulating valve **24, 23**. Further regulating valves **25** and **26** provide for regulation of the pressure in ducts **28** and **27** relative to atmospheric. After the filling process is complete valves **21** and **22** are opened under control of the unit **15** to which they are connected by respective lines such as **41** while valve **18** is automatically closed under control of unit **15** via a line **40**. A valve **20** is opened by control of unit **15** via a line **42** to connect the interior of vessel **5** to atmosphere via a pressure regulating valve **45**. The liquid disposed in the measuring vessel **6** is drawn back through a duct **35** into the vessel **34** as a result of the negative pressure in the vessels **33** and **34** and the liquid which is disposed in the vessel **5** is drawn through a duct **36** into the vessel **33**. To this end, the valves **23, 24, 25, 26** and **45** are adjusted so that the correct pressure conditions required for this operation are obtained in the vessels **33, 34** and **5**. Due to the negative pressure which prevails in the vessel **5** the liquid disposed in the right-hand branch of the viscosimeter is simultaneously drawn back towards the capillary **2** so that any air bubbles which may be present in the instrument escape through the overflow funnel **3**. The back-drawing operation is automatically complete as soon as the liquid level in the vessel **34** reaches a contact **32** provided for this purpose therein.

The instrument is then ready for measurement. The measurement is started by first opening the valves **20, 21** and **22** to atmosphere. The valve **18** is then opened and liquid begins to flow from the two vessels **33** and **34** through the duct **37** into the funnel **3**. The measuring vessel **6** begins to fill after the overflow funnel **4** is filled. Timing starts automatically as before when the contact **10** is reached and is completed when contact **11** is reached after which the instrument is ready for a fresh cycle.

The valve **18** should be constructed so as to open its entire cross-section when the valve is in the open state. This prevents any rheological changes resulting from increased shear action on the liquid as it flows through a restriction, since such changes could falsify subsequent measurement. A magnetically operated tube squeezing valve or a cock whose opening in the open state corresponds to the full cross-section of the supply duct **37** are suitable to this end.

The operation of the cycle, closing and opening of the various valves and resetting of the timer to zero after a cycle has elapsed are controlled by the control unit **15** while the time sequence of the individual measuring cycles is controlled by a timer **116**.

The valves **20, 21** and **22** may be three-way valves, and instead of connecting them to atmosphere during the timing procedure, they may be connected to a further chamber, not shown in the illustration, but having a controlled pressure higher or lower than atmospheric. The force acting on the liquid and conveying it through the measuring capillary may thus be altered at will and measurements may be performed at different rates of shear which may be freely selected. This may also be achieved in another way by connecting the two branches of the viscosimeter with a flexible tube by means of which the difference of head between the upper edges of the two overflow funnels **3** and **4** may be altered as desired.

FIG. **4** shows a further variation, in which a sample of the liquid whose viscosity is being measured may be returned by means of the pump **46** into inlet vessel **34**. The system is completely enclosed so as to avoid any loss of liquid due to evaporation. The pump operates continuously and the measuring process is subdivided into two operating cycles by periodic resetting of the valve **8** which here controls discharge both from vessel **6** and from vessel **5**.

Cycle **1** (measuring cycle)

The vessel **34** is filled with liquid to a specific level; the liquid level in the vessel **5** is slightly below the edge of the inlet funnel **3**. The valve **8** is open in the direction towards the vessel **5** but closed in the direction towards the vessel **6**. The liquid disposed in the vessel **5** is pumped back into the vessel **34** from which it flows downwardly into the inlet funnel **3** in excess. From funnel **3** the liquid flows through the U-tube **1** and the capillary **2** to the overflow funnel **4** so that the measuring vessel **6** is gradually filled and the contacts **10** and **11** for controlling timing are successively actuated as before. In the meantime the vessel **5** is emptied by the pump because the amount of liquid which overflows from the funnel **3** into vessel **5** is less than the delivery capacity of the pump. After timing is completed a control device **14** opens the valve **8** towards the vessel **6** and closes it in the direction towards the vessel **5**.

Cycle 2 (return)

The action of the pump returns the liquid from the measuring vessel **6** into the inlet vessel **34**. Liquid continues to be discharged from the vessel **34** to the inlet funnel **3**. Since the valve **8** to the duct **7** is closed the vessel **5** is filled by the liquid which overflows from the funnel **3**. The measuring vessel **6** must continue to be emptied for as long as the liquid level in vessel **5** is still below the edge of the funnel **3**. The time measuring device **13** is reset to zero by a control device **16**. The control device **14** is arranged to switch the valve **8** into the other operating position and the measuring cycle is then restarted.

The two vessels **5** and **6** remain connected by a duct **47** during both operating cycles so that there is a constant pressure compensation between both vessels.

What is claimed is:

1. A capillary viscosimeter, comprising:

a capillary through which liquid is arranged to flow during a viscosity measurement;

an overflow vessel connected upstream of said capillary and adapted to be supplied with an excess of said liquid during said viscosity measurement;

a discharge overflow vessel connected downstream of said capillary and arranged to discharge liquid which has passed through said capillary by overflow;

a measuring vessel positioned to receive the overflow from said discharge overflow vessel;

means within said measuring vessel for defining and sensing a lower and a higher liquid level within said measuring vessel; and

time measuring means connected to said level defining and sensing means for measuring the time interval required for the liquid level in said measuring vessel to rise from said lower to said higher level.

2. A capillary viscosimeter according to claim **1**, wherein said capillary forms part of a U-shaped tube, one end of which is connected to the first mentioned overflow vessel, and the other end of which is connected to said discharge overflow vessel.

3. A capillary viscosimeter according to claim **2**, wherein said discharge overflow vessel is directly connected to the downstream side of said capillary.

4. The capillary viscosimeter as defined in claim **2**, wherein said U-shaped tube comprises two straight legs and a flexible tube connecting the lower ends of said two straight legs, said two straight legs being arranged to be adjustable in height relative to each other.

5. A capillary viscosimeter as claimed in claim **1**, wherein said discharge overflow vessel is geometrically similar to said first mentioned overflow vessel.

6. A capillary viscosimeter as claimed in claim **1**, wherein saiddischarge overflow vessel is geometrically identical to said first mentioned overflow vessel.

7. A capillary viscosimeter according to claim **6**, wherein both said first mentioned overflow vessel and said discharge overflow vessel consists of a funnel.

8. A capillary viscosimeter according to claim **6**, wherein said capillary forms part of a U-shaped tube, one end of which is connected to the first mentioned overflow vessel, and the other end of which is connected to said discharge overflow vessel.

9. A capillary viscosimeter according to claim **8**, wherein said discharge overflow vessel is directly connected to the downstream side of said capillary.

10. The capillary viscosimeter as defined in claim **1**, wherein each of said liquid level defining and sensing means comprise an electrode having one end communicating with the inside of said measuring vessel and cooperating with a common electrode disposed below said lower liquid level defining and sensing electrode, each of said two electrodes forming with said common electrode and the liquid therebetween a switch-on and a switch-off means, respectively, for said time measuring means.

11. The capillary viscosimeter as defined in claim **10**, wherein a control and reset unit is associated to said time measuring means and wherein said control and reset unit is controlled by a signal derived from said switch-off means to reset said time measuring means, to discharge said measuring vessel and to initiate a further viscosity measurement.

12. A capillary viscosimeter, comprising:

a capillary through which liquid is arranged to flow during a viscosity measurement;

an overflow vessel connected upstream of said capillary;

means for supplying excess of said liquid to said overflow vessel whereby liquid overflows therefrom during a viscosity measurement;

a discharge overflow vessel connected downstream of said capillary and arranged to discharge liquid which has passed through said capillary by overflow;

a measuring vessel positioned to receive the overflow from said discharge overflow vessel;

means defining a lower and a higher liquid level in said measuring vessel; and

time measuring means for measuring the time interval for the liquid level in said measuring vessel to rise from said lower to said higher level.

13. A capillary viscosimeter according to claim **12**, wherein said capillary forms part of a U-shaped tube, one end of which is connected to the first mentioned overflow vessel, and the other end of which is connected to said discharge overflow vessel.

14. A capillary viscosimeter according to claim **13**, wherein said discharge overflow vessel is directly connected to the downstream side of said capillary.

15. The capillary viscosimeter as defined in claim **13**, wherein said discharge overflow vessel is connected directly to the downstream end of said capillary.

16. The capillary viscosimeter as defined in claim **13**, wherein said U-shaped tube comprises two straight legs and a flexible tube connecting the lower ends of said two straight legs, said two straight legs being arranged to be adjustable in height relative to each other.

17. The capillary viscosimeter as defined in claim **12** wherein said discharge overflow vessel is geometrically similar to said first mentioned overflow vessel.

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