

PATENT SPECIFICATION

(11) 1 487 289

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- (21) Application No. 37859/75 (22) Filed 15 Sept. 1975
(31) Convention Application No. 2444148
(32) Filed 16 Sept. 1974 in
(33) Federal Republic of Germany (DT)
(44) Complete Specification published 28 Sept. 1977
(51) INT CL² G01N 11/08
(52) Index at acceptance
GIS 1M



(54) VISCOMETER

(71) We, DR. KARL THOMAE G.M.B.H., a Body Corporate organised under the laws of the Federal Republic of Germany of Biberach an der Riss, Federal Republic of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to an apparatus for use in capillary-viscosimetric measurements of liquids, particularly homogeneous non-Newtonian and inhomogeneous (both Newtonian and non-Newtonian) liquids and more particularly sputum samples and a process for carrying out such measurements.

Mucous membrane secretions are inhomogeneous solutions of macromolecules with long chains. The inhomogeneous character of the mucous membrane secretions, which may often be non-uniformly mixed with very viscous clumps, makes it impossible to measure with the usual capillary viscometers. Free flow, resulting from the specific weight and the difference in height of the vertical capillaries, is blocked as soon as viscous clumps of mucus enter the tubes; measurement of the time of through flow therefore becomes impossible.

British Patent Specification No. 263,781 describes an apparatus for measuring the viscosity of a liquid which comprises regulating means for causing the liquid to flow at a definite constant velocity through a passage wherein its flow is retarded and means for measuring the pressure thereof. However the regulating means described therein comprise devices driven by the liquid such as turbines, water wheels and the like. Such an apparatus is thus unsuitable for measuring inhomogeneous liquids and particularly for measuring liquids containing clumps since the liquid is either wholly or partially homogenised during passage through the apparatus. Moreover, particularly when measuring

inhomogeneous liquids, such regulating means lead to heating of the liquid and thus to a change in viscosity. A further disadvantage of the apparatus of British Patent Specification No. 263,781 for use in measuring inhomogeneous liquids arises in that liquids containing viscous clumps would lead to repellant forces, which would effect the flow to an extent such that the regulating means would be unable to compensate, or even to blockages.

M. W. Arveson has constructed a capillary viscometer in which highly viscous fats are pressed through a capillary by means of a piston, wherein the pressure needed to do this is measured. However, this apparatus is not suitable for measuring small, inhomogeneous quantities of sputum such as those obtained in clinical examinations. The apparatus is, moreover, very expensive to build and extremely unwieldy. Therefore, S. R. Hirsch and R. O. Kory (J. Allergy 39, 265 (1967)) later developed a sputum viscometer wherein the sputum is pressed through a perforated injection piston, while the pressure built up is measured. This apparatus, however, gives insufficiently accurate values since no genuine capillary action is achieved and thus laminar flow is not assured; in addition, it is impossible, or only possible to a limited extent, to determine all the various quantities important when evaluating the sputum or a therapeutic acting on it. When evaluating a sputum sample, it is first necessary to measure the overall viscosity of this sputum sample and secondly, the maximum and minimum viscosities of this sputum sample should be determined. It has become evident that the conventional viscometers, e.g. capillary viscometers, falling sphere viscometers, rotation viscometers, permit only inadequate determination of these values in inhomogeneous sputum samples of restricted volume which *per se* represent a non-Newtonian solution.

According to one feature of the present invention there is provided apparatus for

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use in determining the viscosity of liquids which comprises a capillary; a first tube having a bore cross-sectional area at all points along the tube greater than the bore cross-sectional area of the capillary and adapted at one end for liquid-tight connection to one end of the capillary, said first tube having projecting laterally therefrom a second tube adapted for connection in use to means for measuring the pressure in the first tube, the bores of the said tubes being in communication; and a cylinder fitted with a piston and adapted at one end for liquid-tight connection to the end of the first tube remote from the end adapted for connection to the capillary; said first and second tubes in combination having a substantially lesser bore volume than the total internal volume of the cylinder, the apparatus being constructed such that in use liquid may be forced through the cylinder by the piston and thence, via the first tube through the capillary.

According to a further feature of the present invention there is provided a process for determining the viscosity of a liquid which comprises drawing the liquid into a cylinder by means of a piston; forcing the liquid by means of the piston, at a constant rate of flow through a liquid-tight connection into a first tube and then via a further liquid-tight connection through a capillary; and, while the liquid is being forced through the capillary, measuring the pressure in the first tube by means of a pressure sensing device connected to a second tube projecting laterally from the first tube and being in communication therewith.

As a cylinder with a piston, preferably a commercial medical syringe is used. The use of a syringe is of particular advantage in that it may be disconnected from the apparatus, directly used to take a sample and then be reconnected. Thus it is possible to use a relatively small quantity of sample and the risk of contamination is reduced, since the sample may be incorporated directly into the apparatus. By adapting the apparatus to incorporate a commercial syringe a further advantage is that the syringe, once used, may be discarded and a new syringe may be used to take a fresh sample.

After intake of the sample, the cylinder and the first tube are conveniently connected by means of two interfitting conical surfaces but alternative means such as a threaded collar or interlocking threads may be used if desired. Similar means may also be used to connect the capillary to the first tube, the use of interfitting conical surface again being preferred. The capillary is preferably a stainless steel capillary. A

pressure sensing device is used to measure the pressure in the first tube. Suitable devices include, for example, a device with a wire strain gauge or a piezo-electric or inductive pressure sensor. The pressure sensing device is conveniently connected to the second tube by means of two interfitting conical surfaces but alternative means, such as those mentioned above, may be used if desired. The readings from the pressure sensing device are preferably fed to a pen recorder.

In order to obtain a constant rate of flow the apparatus conveniently includes a means, preferably continuously regulatable, for evenly moving the piston. An infusion pump, for example, commonly used in clinics, is suitable for this purpose.

The second tube preferably projects substantially perpendicularly from the first tube. It is preferred to mount the second tube substantially half way along the first tube but it may be mounted in alternative positions, if desired. A particularly preferred configuration for the first and second tubes is that of a T-piece.

In the process according to the present invention, after the cylinder containing the liquid has been connected to the first tube, any air present in the cylinder between the piston and the first tube is preferably first compressed. The pressure sensing device measures the pressure in the first tube immediately in front of the capillary, therefore when the air has been compressed the position of the piston is conveniently held constant while the "zero" pressure, now present after the pressure exchange, is noted by, for example, if a chart recorder is used, setting this "zero" pressure at the desired datum line on the recorder. The piston is then used to force the liquid through the first tube and the capillary and actual measuring begins by recording the pressure variation in the first tube.

The connection between the cylinder and the first tube preferably has a small internal volume so that when subsequently measuring another liquid, the small volume of liquid in the connection (0.05 ml, for example when using a syringe as the cylinder and piston) is quickly forced through and replaced by the liquid to be measured.

If the readout from the pressure sensing device is fed to a chart recorder, the length (abscissa) of the curve recorded is a measurement of the quantity of liquid measured, when the rate of flow is known and constant.

The height of the deviations (ordinate) is a measurement of the viscosity. The "average viscosity" can be obtained by integration and subsequent division by the

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length of the curve. Maximum and minimum values of the viscosity can be read off directly.

The accuracy of the measurement is determined by the evenness of the movement of the piston since on this depends the constancy of the rate of flow. It is also important in carrying out capillary viscometric determination on sputum that the measurements are not taken at too slow a rate of flow, owing to the thixotropic properties of human sputum, for which otherwise an allowance must be made.

The principal advantage of the apparatus according to the invention lies in the fact that large variations in viscosity within one sample can be determined, the quantity of the sample is not limited and that the apparatus can be used as a genuine capillary viscometer for series examinations, without needing attention each time. The bore volume of the first and second tubes in combination is preferably small since this permits the measurement of smaller quantities e.g. of about 0.2 ml. The most appropriate bore diameter for the capillary is from 0.3 to 0.8 mm; it is advantageous to work with a rate of flow of approx. 0.1 to 0.6 ml/minute. There is also a considerable advantage in the fact that standardised one-way syringes can be used, which, according to our tests, have only insignificant tolerances, with the result that it is not necessary to clean the cylinder and piston.

Brief reference should also be made to the physical basis of this measurement of viscosity; from the equation

$$\eta = \frac{\Delta p \cdot \pi \cdot r^4}{8 \cdot l \cdot I}$$

wherein

η represents the viscosity in dyne. sec. cm^{-2} ,

Δp represents the differential pressure in microbars,

r is the radius of the bore of the capillary in cm,

l is the length of the capillary in cm and

I is the rate of flow, expressed by the relationship volume (ml) divided by the time of flow (sec),

It can be seen that the viscosity can be determined by measuring the varying differential pressure Δp , if all the other values are kept constant. This differential pressure Δp is necessary to press the liquid through the capillary at a constant rate of flow I . The value of the other parameters which are kept constant may be obtained

by calibration with a liquid of known viscosity.

For a better understanding of the present invention reference is made to the accompanying drawings, wherein:—

Figure 1 shows schematically an exploded cross-section through the vertical plane of one embodiment of an apparatus according to the invention; and

Figure 2 shows a typical readout from the apparatus of Figure 1 as recorded by a tape printer.

Referring to Figure 1 of the drawings, the apparatus shown comprises a replaceable measuring capillary 1 and a first tube 2 which are connected by means of an internal conical surface 3 mounted on the end of the capillary 1 and a corresponding external conical surface 4 on the appropriate end of the first tube 2. The first tube 2 has projecting at substantially 90° therefrom a second tube 5, the first and second tubes forming together a T-piece. The free end of the second tube 5 is provided with an internal conical surface 6. A corresponding external conical surface 7 connects a pressure sensing device 8 to the second tube 5.

The end of the first tube 2 remote from the capillary 1 is also provided with an internal conical surface 9 which, by interlocking with a corresponding external conical surface 10 provides a liquid-tight connection from the first tube 2 to a syringe 11. The syringe 11 is provided with a plunger 12 which may be evenly moved by a movement control device 13 incorporating an infusion pump by means of a movable carrier 14 mounted on the movement control device 13. The syringe 11 is supported by a fixed support 15 also mounted on the movement control device 13.

In use, the sample is first drawn into the syringe 11. The syringe is then connected to the tube 2 by means of the conical surfaces 9 and 10 and the tubes 2 and 5 are connected to the capillary 1 and pressure-sensing device 8 respectively. The plunger 12 is attached to the movable carrier 14 and the syringe 11 is attached to the fixed support 15. Sample is forced through the apparatus by advancing the plunger 12 and the pressure in the tube 2 is continuously monitored by a pen recorder (not shown) connected to the pressure-sensing device 8. The viscosity of the sample may then be determined by analysis of the pen-recorder readout as hereinbefore described.

The invention is not restricted to a process and an apparatus for determining the viscosity of sputum samples but is applicable for any liquid. The advantages of the present invention are, however, most apparent in determinations on

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homogeneous non-Newtonian liquids and inhomogeneous (both Newtonian and non-Newtonian) liquids, where up till now, accurate measurements have not been possible. Other liquids to which the present invention is advantageously applicable include, for example, biological liquids other than sputum, inhomogeneous oils or liquid inhomogeneous plastics or solutions or suspensions containing plastics or pigments of various shape and chemical constitution.

WHAT WE CLAIM IS:—

1. Apparatus for use in determining the viscosity of liquids which comprises a capillary; a first tube having a bore cross-sectional area at all points along the tube greater than the bore cross-sectional area of the capillary and adapted at one end for liquid-tight connection to one end of the capillary, said first tube having projecting laterally therefrom a second tube adapted for connection in use to means for measuring the pressure in the first tube, the bores of the said tubes being in communication; and a cylinder fitted with a piston and adapted at one end for liquid-tight connection to the end of the first tube remote from the end adapted for connection to the capillary; said first and second tubes in combination having a substantially lesser bore volume than the total internal volume of the cylinder, the apparatus being constructed such that in use liquid may be forced through the cylinder by the piston and thence via the first tube through the capillary.

2. Apparatus as claimed in claim 1 further including a pressure sensing device as a means for measuring the pressure in the first tube.

3. Apparatus as claimed in claim 2 wherein the pressure sensing device is a device with a wire strain gauge or a piezo-electric or inductive pressure sensor.

4. Apparatus as claimed in claim 2 or claim 3 wherein the pressure sensing device is provided with a conical surface capable, in use, of interfitting with a mating conical surface on the free end of the second tube.

5. Apparatus as claimed in claim 4 wherein the pressure sensing device is provided with an external conical surface and the second tube with an internal conical surface.

6. Apparatus as claimed in any of claims 2 to 5 wherein the pressure sensing device is connected to a pen recorder.

7. Apparatus as claimed in any of the preceding claims wherein the piston is supported on a movable carrier.

8. Apparatus as claimed in claim 7 wherein the movable carrier is such that the piston can be advanced evenly.

9. Apparatus as claimed in claim 8 wherein the movement of the carrier is continuously regulatable, whereby in use a constant rate of flow of the liquid is obtainable.

10. Apparatus as claimed in claim 8 or claim 9 further including a movement control device incorporating an infusion pump which in use may evenly advance the movable carrier.

11. Apparatus as claimed in any of the preceding claims wherein the cylinder is supported on a fixed support.

12. Apparatus as claimed in any of the preceding claims wherein the cylinder is provided with a conical surface capable, in use, of interfitting with a corresponding conical surface on the appropriate end of the first tube.

13. Apparatus as claimed in claim 12 wherein the cylinder is provided with an external conical surface and the corresponding conical surface on the first tube is an internal conical surface.

14. Apparatus as claimed in any of the preceding claims wherein the cylinder is fitted with a piston is a syringe.

15. Apparatus as claimed in any of the preceding claims wherein the second tube projects substantially perpendicularly from the first tube.

16. Apparatus as claimed in any of the preceding claims when the second tube is mounted substantially half way along the first tube.

17. Apparatus as claimed in any of the preceding claims wherein the first and second tubes together form a T-piece.

18. Apparatus as claimed in any of the preceding claims wherein the capillary is provided with a conical surface at one end capable, in use, of interfitting with a corresponding conical surface on the appropriate end of the first tube.

19. Apparatus as claimed in claim 18 wherein the capillary is provided with an internal conical surface and the corresponding conical surface on the first tube is an external conical surface.

20. Apparatus as claimed in any of the preceding claims wherein the capillary is of stainless steel.

21. Apparatus as claimed in any of the preceding claims wherein the capillary has a bore diameter of from 0.3 to 0.8 mm.

22. Apparatus as claimed in claim 1 substantially as herein described.

23. Apparatus substantially as herein described with reference to Figure 1 of the accompanying drawings.

24. A process for determining the viscosity of a liquid which comprises drawing the liquid into a cylinder by means of a piston; forcing the liquid by means of the piston, at a constant rate of flow



homogeneous non-Newtonian liquids and inhomogeneous (both Newtonian and non-Newtonian) liquids, where up till now, accurate measurements have not been possible. Other liquids to which the present invention is advantageously applicable include, for example, biological liquids other than sputum, inhomogeneous oils or liquid inhomogeneous plastics or solutions or suspensions containing plastics or pigments of various shape and chemical constitution.

WHAT WE CLAIM IS:—

1. Apparatus for use in determining the viscosity of liquids which comprises a capillary; a first tube having a bore cross-sectional area at all points along the tube greater than the bore cross-sectional area of the capillary and adapted at one end for liquid-tight connection to one end of the capillary, said first tube having projecting laterally therefrom a second tube adapted for connection in use to means for measuring the pressure in the first tube, the bores of the said tubes being in communication; and a cylinder fitted with a piston and adapted at one end for liquid-tight connection to the end of the first tube remote from the end adapted for connection to the capillary: said first and second tubes in combination having a substantially lesser bore volume than the total internal volume of the cylinder, the apparatus being constructed such that in use liquid may be forced through the cylinder by the piston and thence via the first tube through the capillary.

2. Apparatus as claimed in claim 1 further including a pressure sensing device as a means for measuring the pressure in the first tube.

3. Apparatus as claimed in claim 2 wherein the pressure sensing device is a device with a wire strain gauge or a piezo-electric or inductive pressure sensor.

4. Apparatus as claimed in claim 2 or claim 3 wherein the pressure sensing device is provided with a conical surface capable, in use, of interfitting with a mating conical surface on the free end of the second tube.

5. Apparatus as claimed in claim 4 wherein the pressure sensing device is provided with an external conical surface and the second tube with an internal conical surface.

6. Apparatus as claimed in any of claims 2 to 5 wherein the pressure sensing device is connected to a pen recorder.

7. Apparatus as claimed in any of the preceding claims wherein the piston is supported on a movable carrier.

8. Apparatus as claimed in claim 7 wherein the movable carrier is such that the piston can be advanced evenly.

9. Apparatus as claimed in claim 8 wherein the movement of the carrier is continuously regulatable, whereby in use a constant rate of flow of the liquid is obtainable.

10. Apparatus as claimed in claim 8 or claim 9 further including a movement control device incorporating an infusion pump which in use may evenly advance the movable carrier.

11. Apparatus as claimed in any of the preceding claims wherein the cylinder is supported on a fixed support.

12. Apparatus as claimed in any of the preceding claims wherein the cylinder is provided with a conical surface capable, in use, of interfitting with a corresponding conical surface on the appropriate end of the first tube.

13. Apparatus as claimed in claim 12 wherein the cylinder is provided with an external conical surface and the corresponding conical surface on the first tube is an internal conical surface.

14. Apparatus as claimed in any of the preceding claims wherein the cylinder is fitted with a piston is a syringe.

15. Apparatus as claimed in any of the preceding claims wherein the second tube projects substantially perpendicularly from the first tube.

16. Apparatus as claimed in any of the preceding claims when the second tube is mounted substantially half way along the first tube.

17. Apparatus as claimed in any of the preceding claims wherein the first and second tubes together form a T-piece.

18. Apparatus as claimed in any of the preceding claims wherein the capillary is provided with a conical surface at one end capable, in use, of interfitting with a corresponding conical surface on the appropriate end of the first tube.

19. Apparatus as claimed in claim 18 wherein the capillary is provided with an internal conical surface and the corresponding conical surface on the first tube is an external conical surface.

20. Apparatus as claimed in any of the preceding claims wherein the capillary is of stainless steel.

21. Apparatus as claimed in any of the preceding claims wherein the capillary has a bore diameter of from 0.3 to 0.8 mm.

22. Apparatus as claimed in claim 1 substantially as herein described.

23. Apparatus substantially as herein described with reference to Figure 1 of the accompanying drawings.

24. A process for determining the viscosity of a liquid which comprises drawing the liquid into a cylinder by means of a piston; forcing the liquid by means of the piston, at a constant rate of flow

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- through a liquid-tight connection into a first tube and then via a further liquid-tight connection through a capillary; and, while the liquid is being forced through the capillary, measuring the pressure in the first tube by means of a pressure sensing device connected to a second tube projecting laterally from the first tube and being in communication therewith.
25. A process as claimed in claim 24 wherein air in the cylinder between the piston and the first tube, if present, is first compressed and the pressure in the first tube noted before the liquid is forced through the first tube.
26. A process as claimed in claim 24 or claim 25 wherein the rate of flow is from 0.1 to 0.6 ml/ minute.
27. A process as claimed in any of claims 24 to 26 wherein the liquid is a homogeneous non-Newtonian liquid or an inhomogeneous liquid.
28. A process as claimed in claim 27 wherein the liquid is sputum.
29. A process as claimed in any of claims 24 to 28 when carried out using apparatus as claimed in any of claims 1 to 23.
30. A process for carrying out a capillary-viscosimetric determination of a liquid as claimed in claim 24 substantially as herein described.

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Printed for Her Majesty's Stationery Office, by the Courier Press, Leamington Spa, 1977
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from
which copies may be obtained.



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