(54) Title: PROCESS FOR CONTINUOUS DETERMINATION OF THE VISCOSITY OF LIQUIDS AND A DEVICE FOR CARRYING OUT THE PROCESS

(57) Abstract

The viscosity of a liquid is determined according to the capillary method, by establishing a constant liquid level within or above the capillary, and determining the liquid flow velocity through the capillary. A device for carrying out this process includes a capillary fitted with an inlet tube for introducing the liquor in the upper part (4) of the capillary, and an extraction tube (6) for drawing off excess of added liquid, as well as a device (9) for the determination of the amount of liquid leaving the lower end (7) of the capillary.
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Process for Continuous Determination of the Viscosity of Liquids and a Device for Carrying out the Process.

The present invention relates to a process for determination of the viscosity of liquids and more specifically the continuous process for this determination, as well as a device for carrying out this process.

The viscosity of liquids is normally determined by means of one of the following main types of device:

1. Rotation viscometer
2. Falling ball
3. Capillary viscometer

Capillary viscometry is based on measurement of the flow velocity for a certain known volume of the liquid, flowing freely through a given capillary under well defined, standardized conditions. In practice this is given as the volume present between two given marked levels in the inlet part of the capillary. The time used for the surface of the liquid to pass between these two levels is thereby at the same time identical with the efflux time for an equivalent liquid volume passing through the capillary. Traditionally such capillary viscometry is based on the use of a stop watch and visual judgement of the liquid surface passing the level marks.

Automation of the method has comprised substituting this visually/manually determined time measurement by the use of optical or electrical (conductance) sensors. Such automatic viscometer devices are commercially available.

A further description of various viscometers, as well as the underlying theory, can be found in Kirk-Othmer, Encyclopedia of Chemical Technology, Volume 21, pages 460-484.

Viscosity is an important product parameter within the pulp industry. Samples of chemical pulp (bleached cellulose) is
then dissolved in for example cupriethylene diamine (CED) or

cuprammonium, and the viscosity of the resulting liquid will
be among the qualities used to characterize the pulp.

Within the Nordic pulp industry the method SCAN-C 15:62 is
used for this analysis. Later this method is mainly
accepted and used by ISO as ISO 5351/1-1981. A few years ago
SCA Control Systems AB, Sundsvall, Sweden, introduced a
viscosity analyzer "Viscomat 320 20" for this quality
control analysis. Even this advanced analytical equipment,
reported to be the result of considerable research and
development, is based on the samples being sucked up
into/flowing out of capillaries with fused-in platinum
electrodes.

It is found that the well known process for determination of
viscosity, especially for determination of cellulose
viscosity, can be simplified if liquid to be studied pass
under constant pressure down through the capillary, and the
amount of liquid passing per unit of time is determined.

This last determination is most easily carried out if the
drops leaving the lower part of the capillary are registered
and the number of drops per unit of time is determined.

The invention is to be described further with reference to
the drawings where

Fig. 1 shows a device for carrying out the process,

Fig. 2 and 3 show graphically a comparison of results
obtained by following the present process with results
according to the use of standard method.
Fig. 4 shows another modification of the device shown in Figure 1.

In Fig. 1 is shown a device for carrying out the process, and this comprises a capillary shown inside a jacket 1 through which for example water at a certain temperature can be added through the pipe stub 2 and let out through the pipe stub 3.

The capillary has an extended part 4 to which the liquid is introduced by means of a pump and by a tube 1. The liquid is introduced into the expanded part 4 in an amount exceeding the amount of liquid flowing out through the capillary. Excess of liquor is extracted by means of a pump through tube 6 in such a way that a constant liquid level is maintained within the expanded part of the capillary. The liquid flows out through the lower end 7 of the capillary leaving in the form of drops 8. The lower end of the capillary is located within a drop counting device generally named 9 containing a device to show or register the drops. In the example shown here to demonstrate this, the device 9 comprises a light source, which can be a photo diode, as well as a photo detector which is connected to an electronic counting device or a time measuring device.

When the sample liquid is introduced through the tube 5, liquid will build up in the capillary itself and in the expanded part of it and reach the extraction tube 6, and drops will start falling from the lower end 7 of the capillary. The falling drops will pass through the light beam from the photo diode in such a way that the passing of the drops will be detected by the photo detector and registered by the electronic counting device. The liquid flow out from the capillary depends upon the viscosity of the liquid, and consequently the number of drops per unit of time will be a measure for the viscosity of the liquid. The
time between the drops can be calculated consecutively, preferably by counting drops and measuring the time for a certain number of drops, followed by the calculation of an average for the time between two succeeding drops. Optionally one can consider counting of the number of drops falling within a given period of time, and calculate the average for such measurements.

In Figure 4 a modified form of the device according to Figure 1, is shown. The numbers 1-9 refer to the same parts as in Figure 1.

The device according to Figure 4 is equipped with a Mariotte flask, in such a way that the pressure can be varied. The additional device comprises a vessel 10, a connection tube 11 and a close cap 12 through which the tubes 5 and 6 are mounted in an airtight way. Down into the vessel 10 a sliding tube 13 is introduced, the position of which, below the liquid surface 14, can be adjusted. By adjusting the tube 13 into the liquid in this manner, the pressure in the system can be reduced by h mm liquid column. This is an advantage because when shifting to another viscosity range, one will be able to operate in approximately the same range of linear flow through the capillary. As an example can be indicated, that a gradual decrease in the pressure over the liquid surface in the top of the viscometer, brought about a linear increase in the dropping rate for a 20% glycerol solution, from 2.79 sec/drop (atmospheric pressure) to 6.10 sec/drops (atmospheric pressure - 100 mm water column). The correlation coefficient was found to be 0.982.

In stead of using a light source and a photo detector, the drops can for example be detected and counted by hitting a small microphone, passing between two electronic tips or any device that can register a falling drop.
In Figure 2 the intrinsic viscosity, determined and calculated as cm$^3$/g according to SCAN-C 15, is shown versus sec/drop according to the device in Figure 1, for a series of cellulose pulps under the following conditions:

x-axis: The pulp dissolved in CED, adapted to 0.6 mm capillary with drop counting and time measurement according to Fig. 1.

y-axis: The pulp dissolved in CED according to SCAN-C 15:62. 0.5 mm capillary. Intrinsic viscosity calculated according to SCAN-C 15. r = 0.989.

A comparison between conventional, discontinuous measurement of the efflux time, and a measurement carried out according to the present process and device was performed for a mixture of glycerol and water in various ratios. For 20-55% glycerol solutions a good linear relationship was found. The discontinuous viscosiy measurement was carried out according to SCAN-C 15. The correlation coefficient was calculated to 0.9995.

Even though the present way of carrying out the process and the device of doing this, primarily is meant for viscosity determinations of viscose solutions, it is obvious that the process and the device can be used in all cases where the viscosity is a property one want to watch and determine continuously.
Claims

1. Process for continuous determination of the viscosity of a liquid by use of the capillary method, characterized by that it within or above the capillary is established a constant liquid level and that the flow velocity through the capillary is determined.

2. Process according to claim 1, characterized by the liquid being added to the capillary with a flow exceeding the outlet flow and that the excess liquor is sucked off in such a way that a constant liquid level is obtained.

3. Process according to any of the preceding claims, characterized by that the drops leaving the lower end of the capillary are registered and the time between each drop is used as an expression for the viscosity of the liquid.

4. Device for carrying out the process according to claims 1-3, including a capillary, optionally fitted with a water jacket 1 with inlet stub (2) and outlet stub (3) for leading thermostatically controlled water through the jacket, characterized by an inlet tube (5) for introducing liquid to the upper part (4) of the capillary and an extraction tube (6) for drawing off excess of the liquid added, and a drop counting device (9) mounted at the lower end (7) of the capillary.

5. Device according to claim 4, characterized by measures (10, 11, 12, 13) to adjust the pressure above the liquid surface in the upper part 4 of the capillary.
6. Device according to claim 5, characterized by that the measures for the pressure regulation includes a liquor vessel (10) that via a tube (11) is connected to a close fitting cap (12), and where it in the vessel (10) is mounted a closely fitted, sliding tube (13).
AMENDED CLAIMS

[received by the International Bureau on 19 November 1986 (19.11.86); original claims 1-6 replaced by amended claims 1-4 (2 pages)]

1. A method for continuous determination of the viscosity of a liquid using the capillary method, by establishing a constant liquid level above a capillary tube and determine the rate of flow through the capillary tube, characterized in that the drops of liquid emerging from the lower part of the capillary tube are recorded, and the time period between each drop is used as an expression for the viscosity of the liquid.

2. A device for carrying out the method in accordance with claim 1 comprising a capillary tube optionally provided with a water mantel (1) with an inlet (2) and outlet (3) for passing of thermostated water, characterized in an inlet (5) for introduction of the liquid in the upper part (4) of the capillary tube and a suction tube (6) for withdrawal of excess of added liquid and a drop counting device (9) arranged at the lower part of the capillary tube.

3. A device in accordance with claim 2, characterized in means (10, 11, 12, 13) to adjust the head of the liquid level in the upper part (4) of the capillary tube.

4. A device in accordance with claim 3, characterized in that the device for regulating the head comprises a container (10) for the liquid which via a
tube (11) is connected with an airtight hood (12), and that in the container (10) an adjustable tube (13) is arranged in an airtight manner.
Fig. 1

Light source (photo diode)

Electronic counting and time measurement.

Photo detector (pindiode)
Sample liquid in

Excess of liquid
To drain

Liquid level

Capillary

Atmos. pressure
- h mm H₂O

Light source (photo diode)

Photo detector (pindiode)

Electronic counting and time measurement.

Fig. 4
# INTERNATIONAL SEARCH REPORT

**International Application No:** PCT/NO86/00043

## I. CLASSIFICATION OF SUBJECT MATTER

If several classification symbols apply, indicate all:

According to International Patent Classification (IPC) or to both National Classification and IPC:

**G 01 N 11/06**

## II. FIELDS SEARCHED

### Minimum Documentation Searched

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Documentation searched other than Minimum Documentation to the extent that such documents are included in the fields searched:

SE, NO, DK, FI classes as above

## III. DOCUMENTS CONSIDERED TO BE RELEVANT

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<td>FR, A, 2 559 906 (ETUDES ET FABRICATION DOWELL SCHLUMBERGER) 23 August 1985</td>
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<td>X</td>
<td>GB, A, 768 610 (LIEB, OTTO) 20 February 1957</td>
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## IV. CERTIFICATION

Date of the Actual Completion of the International Search: 1986-08-25

Date of Mailing of this International Search Report: 1986-09-08

International Searching Authority: Swedish Patent Office

Signature of Authorized Officer: Inger Lottgren

Form PCT/ISA/210 (second sheet) (January 1985)