(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 235 778**
A2

(12) EUROPEAN PATENT APPLICATION

(21) Application number: **87102901.3**

(22) Date of filing: **02.03.87**

(51) Int. Cl.4: **B01L 11/00** , G01N 7/04 , B01L 3/14

(30) Priority: **03.03.86 IT 1960986**

(43) Date of publication of application: **09.09.87 Bulletin 87/37**

(84) Designated Contracting States: **AT BE CH DE ES FR GB LI NL SE**

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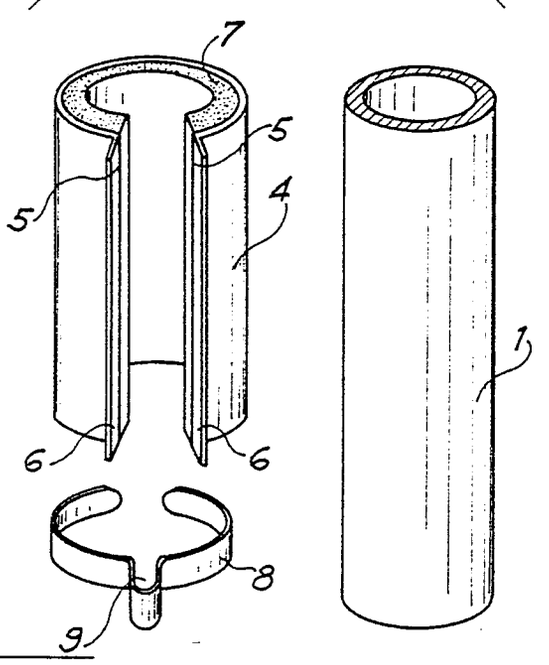
(54) A method and device for maintaining constant the wetted portion on the surface of a test-tube.

(57) This invention concerns a method for maintaining constant the wetted surface of a test-tube immersed in a thermally active liquid, as well as the device for carrying-out said method, especially suited to maintain constant and reproducible the thermal conditions of the test-tube during measurement of gas adsorption.

Such method comprises the steps of : covering at least part of the external surface of the test tube with a porous material to form a collar which can be impregnated by capillary action with the liquid; immersing the test tube at least partially so that the level of the liquid in a container is between the upper and lower levels of the porous collar; replenishing the container with the same liquid so that the level is maintained between the stated limits.

Fig. 1



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"A METHOD AND DEVICE FOR MAINTAINING CONSTANT THE WETTED PORTION ON THE SURFACE OF A TEST-TUBE"

The present invention concerns a method for maintaining constant the wetted surface of a test-tube immersed in a thermally active liquid, as well as a device for carrying-out said method. The method and device according to the invention are especially suited to the field of measurement of gas adsorption.

For such measurements a receptacle (a Dewar flask) is generally used, filled with a refrigerant liquid in which a test tube containing the solid sample is immersed in a fixed position with respect to the receptacle itself. The measurement of the gas adsorbed onto the solid sample follows the following procedure: first a control cycle is carried out in which the test tube is immersed in the liquid and the volume of gas introduced in the tube to achieve a given gas-pressure in the test tube is measured; the test tube is then substituted with an identical test tube containing the solid sample and the volume of gas needed to obtain the same given pressure is measured. The quantity of gas adsorbed can be deduced from the difference in volumes of the gas in the blank and solid sample cycles.

Since the volume of the gas in the test tube depends on the temperature of the test tube itself, the temperature of the test tube in the two cycles must be the same and be maintained rigourously constant during the whole measuring period for the test to be reliable.

It has been stated that the test tube must be immersed in a refrigerant liquid (which can be liquid nitrogen, carbonium anhydride, acetone, brine etc.) and maintained in a fixed position in relation to the receptacle; this condition makes it obvious that the temperature of the test tube will be determined by the level of the refrigerant in the receptacle, i.e. by the area of the test tube wetted by the refrigerant liquid. In the devices used up to now the level of the liquid in the receptacle is maintained constant during the measurement - (which might last for some hours) by means of a pump actuated by a float in the liquid itself.

However precise the control device for maintaining the level of the refrigerant liquid, the latter, instead of being maintained constant, fluctuates between an upper and lower limit. Inevitably, the pump tends to ripple the free surface of the refrigerant liquid, causing further disturbance of the measurement. An object of the present invention is to provide a method and device for its implementation which makes possible for each measurement and for the duration of each measurement the maintenance and reproduction of constant area wetted surfaces in a test tube immersed in a refrigerant liquid.

The above stated object is achieved by a method characterized in that it comprises the steps of : covering at least part of the external surface of a test tube with a porous material to form a collar which can be impregnated by capillary action with a thermally active liquid; immersing the test tube at least partially in a liquid receptacle so that the level of the liquid in the receptacle is between the upper and lower levels of the porous collar; replenishing the receptacle with the same liquid so that the level is maintained between the stated limits.

In particular, the collar of porous material is enclosed by a non-porous external jacket which extends at least over the range from the highest to the lowest level of refrigerant liquid with respect to the test tube.

The device for carrying-out the method according to the invention is characterized in having an essentially cylindrical jacket in porous material, adapted to be completely impregnated with a thermally active liquid by capillary action, the internal surface of the collar being shaped so as to adhere to at least part of the external lateral surface of the test tube.

The method and device according to the invention allow the object of the invention to be achieved because by their use an extension of the test tube wetted surface is obtained independently of the level of the liquid itself in the receptacle. It is sufficient that the end of the collar of porous material touches the refrigerant liquid so that the liquid can be transported by capillary action to the interior of the collar until the collar is completely soaked. Consequently the surface of the test tube in contact with the porous material is wetted by the refrigerant liquid indepently of the level of the liquid itself in the receptacle. Further, replenishment of the refrigerant liquid to maintain the level between maximum and minimum values do not in the least disturb the conditions in which the measurement takes place, since the free surface of the liquid is not in contact with the poruos collar or with the test tube itself. In fact, given that the temperature of the test tube is determined by the extent of the surface wetted by the refrigerant liquid, maintaining this surface constant also maintains the temperature constant.

The method and related device according to the invention shall be described in detail with reference to the enclosed drawing illustrating a possible embodiment of the device in which :

-Fig. 1 is a three-dimensional view of the device according to the invention and a part of the test tube;

-Fig. 2 is a three-dimensional view of the device according to the invention fitted to a part of the test tube;

-Fig. 3 is a transverse section of the test tube-device assembly of Fig. 2;

-Fig. 4 is a longitudinal section of the receptacle with the test tube and the device in the measurement position.

The invention is particularly applicable to the measurement of adsorption of gas onto a solid or other measurements requiring the repeatability and the constancy of the same thermal conditions of the test tube or the like and therefore the constancy of the area of surface wetted by a thermally active liquid. With reference to the first measurement, a test tube 1 is generally used, connected to a source of the gas in question and a receptacle 2 (Dewar flask) containing a refrigerant liquid 3. This refrigerant liquid can be liquid nitrogen, carbonium anhydride, acetone, brine etc. The test tube is placed in the receptacle so that it is partially immersed in the refrigerant liquid.

As stated above, a blank cycle is first carried out in which the volume of gas needed to fill the empty test tube to reach a given internal gas-pressure is measured. The solid is then introduced to the test tube and the volume of gas needed to reach the same internal gas-pressure is measured. The quantity of gas adsorbed onto the solid can be deduced from the difference between the volumes of gas required to give the same gas-pressure in the blank and sample cycles. To maintain the temperature constant and therefore the area wetted by the refrigerant liquid, the device according to the invention comprises a jacket 4 in aluminium or other non-porous material, essentially cylindrical in form and presenting an opening 5 along a generator of the cylinder. The edges of this opening form wings 6 turned outwards and forming an angle of 90 with the edges themselves. On the internal surface of the jacket 4 is fixed a collar 7 of porous material of constant thickness capable of transporting the refrigerant liquid by capillary action. The porous material can be felt, paper, carbon or any other porous material with interconnected pores. The collar 7 itself has a vertical opening corresponding to the opening in the aluminium jacket 4 and has an internal diameter slightly smaller than that of the test tube. In this way, separating the wings 6 slightly it is possible to increase the internal diameter of the collar of porous material to facilitate the introduction of the test tube into the collar. Once the test tube is inserted, the collar is locked around the test tube by spring-clips 8 embracing the outer surface of the jacket 4. Such spring-clips maintain the external surface of the test tube in close contact with the porous material. In particular, the spring-clips themselves have a slot 9 extending radially outwards and shaped to house the wings 6 of the jacket 4; in this way the collar remains locked since the wings 6, once engaged in the slot 9, are not able to separate.

When the collar is fixed to the test tube, the latter is located in the receptacle. The refrigerant liquid in the receptacle partially washes the outer walls of the aluminium jacket and the lower surface of the porous material of the collar is thus below the level of the surface of the refrigerant liquid. The refrigerant liquid soaks into the porous material of the collar by capillary action until it reaches the upper surface of the same.

In this way, it is sufficient that the lower surface of the porous material of the collar is immersed in the refrigerant liquid for the outer surface of the test tube to be wetted by the refrigerant liquid, independently of the level of the refrigerant liquid in the receptacle. The inevitable variations of the level of the refrigerant liquid with respect to the test tube, e.g. during replenishment, are not communicated as such by the latter whose outer surface remains constantly in contact with the collar of porous material and so remains constantly wetted in an unvarying way by the refrigerant liquid. Consequently the pressure inside the test tube changes isothermically.

Naturally, the invention is applicable to other measurements where it is necessary to maintain a constant and repeatable area of wetted surface on a test tube or similar container and thus maintain constant and repeatable thermal conditions.

Claims

1. A method for maintaining a constant area of the external surface of a test tube wetted by a thermally active liquid contained in a receptacle, characterized in that it comprises the steps of: covering at least part of the external surface of the test tube with a porous material to form a collar which can be impregnated with the liquid by capillary action; immersing the test tube at least partially so that the level of the liquid in the receptacle is between the upper and lower levels of the porous collar; replenishing the receptacle with the same liquid so that the level is maintained between the stated limits.

2. A method according to Claim 1, characterized in that the collar of porous material is jacketed by a non-porous material extending at least over the range of the maximum and minimum levels of the liquid with repect to the test tube.

3. A method according to Claim 2, characterized in that the test tube is in the form of a sample carrier for the measurement of the adsorption of gas, the liquid is a refrigerant and the receptacle is a Dewar flask.

4. A device for carrying-out the method according to one of claims 1,2 or 3, characterized in that it comprises an essentially cylindrical collar of porous material that can be impregnated completely with the liquid by capillary action, the internal surface of the collar being shaped in such a way as to adhere at least in part to the external lateral surface of the test tube.

5. A device according to claim 4, characterized in that said porous material has pores of interconnected type.

6. A device according to claim 4 or 5, characterized in . that the outer surface of said collar is at least partially covered by a coating of non-porous material.

7. A device according to claim 6, characterized in that said non-porous material is a sheet which jackets the outer lateral surface of said collar of porous material.

8. A device according to claim 6 or 7, characterized in that the collar of porous material and the jacket of non-porous material can open to allow the test tube to be inserted, and in that said collar and jacket are locked by means of spring means so as to maintain the collar itself in close contact with the test tube.

Fig.1

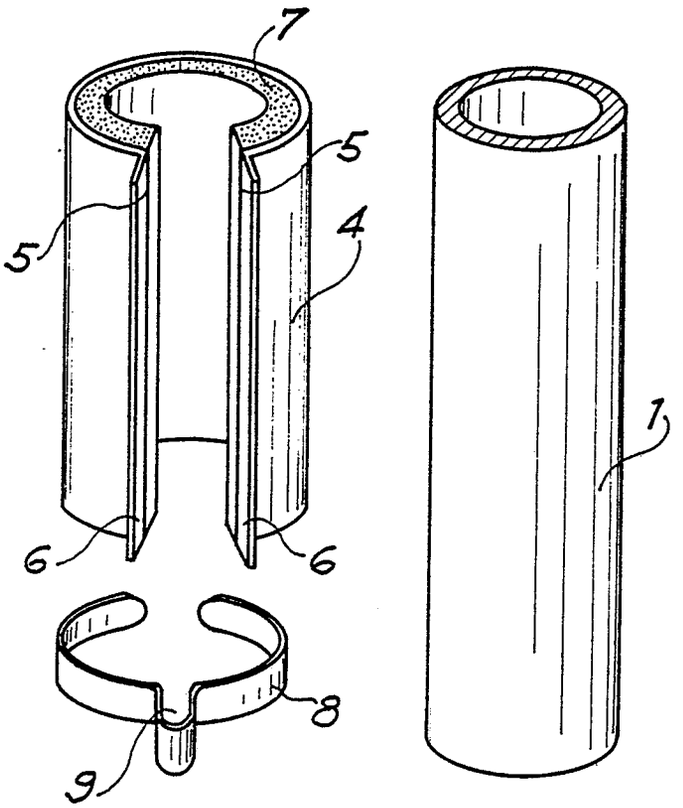

Fig.2

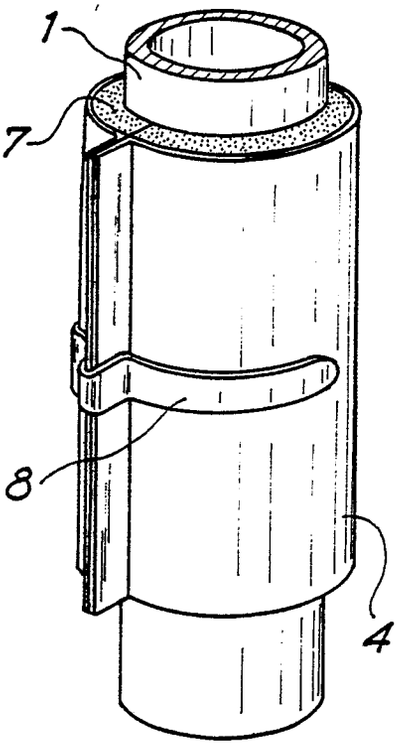

Fig.3

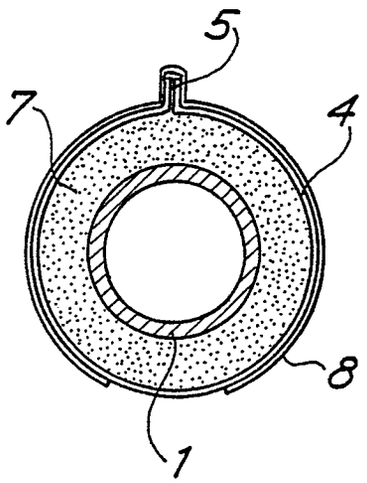

Fig.4