Feb. 27, 1962 W. C. HAMPTON 3,022,657

PENETROMETER FOR POROSIMETERS

Original Filed May 12, 1958 2 Sheets-Sheet 1

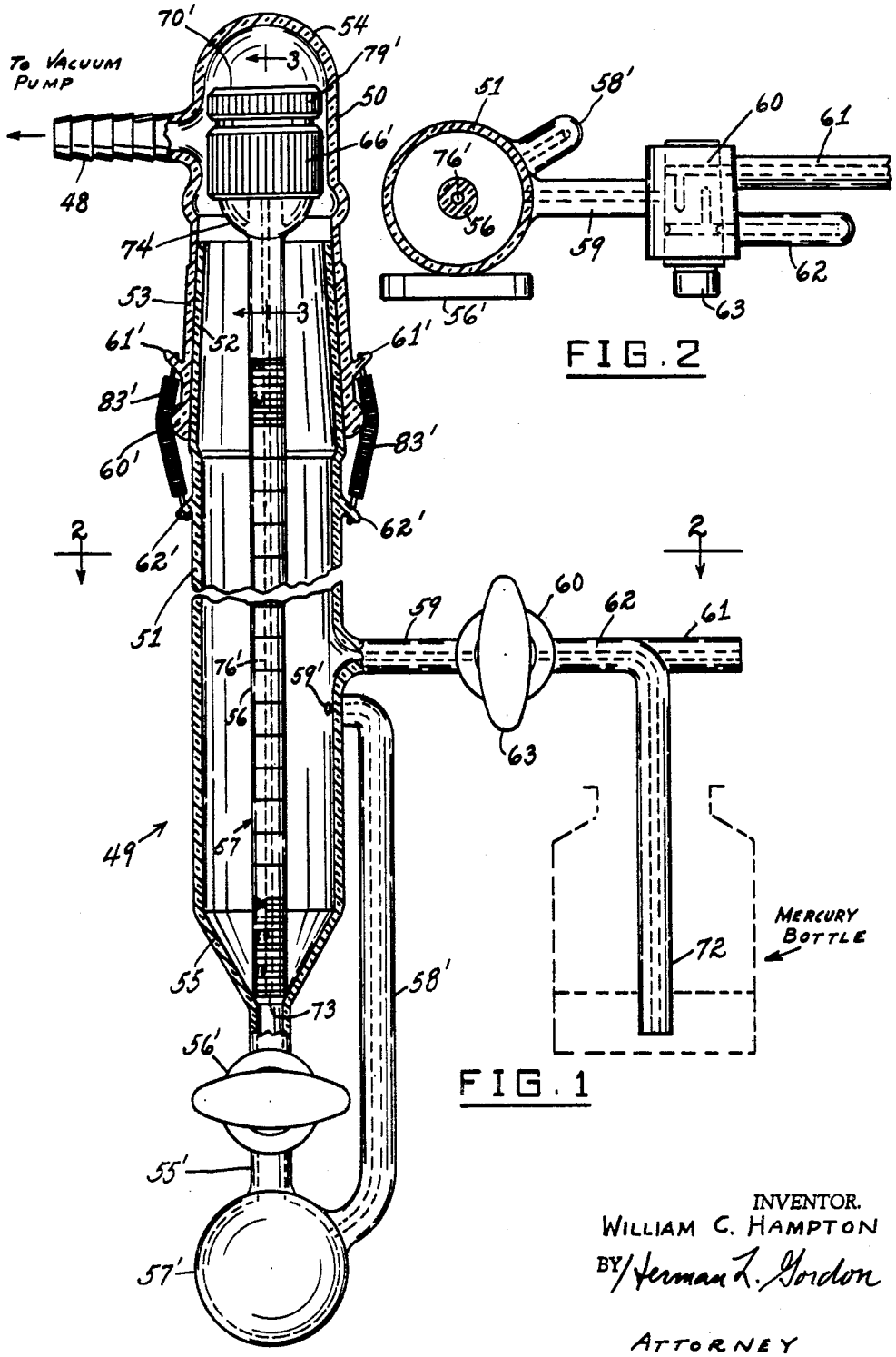


FIG. 2

FIG. 1

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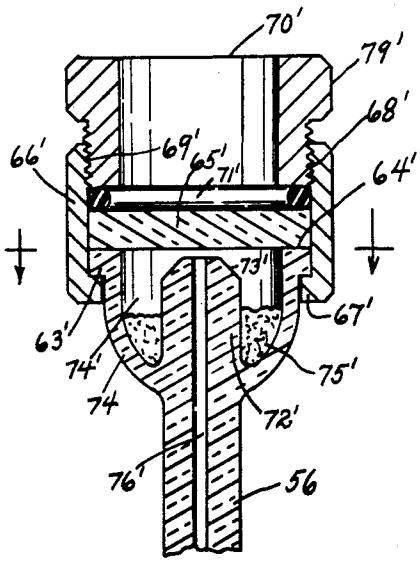


FIG. 3

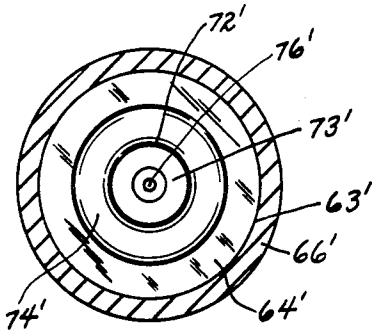


FIG. 4

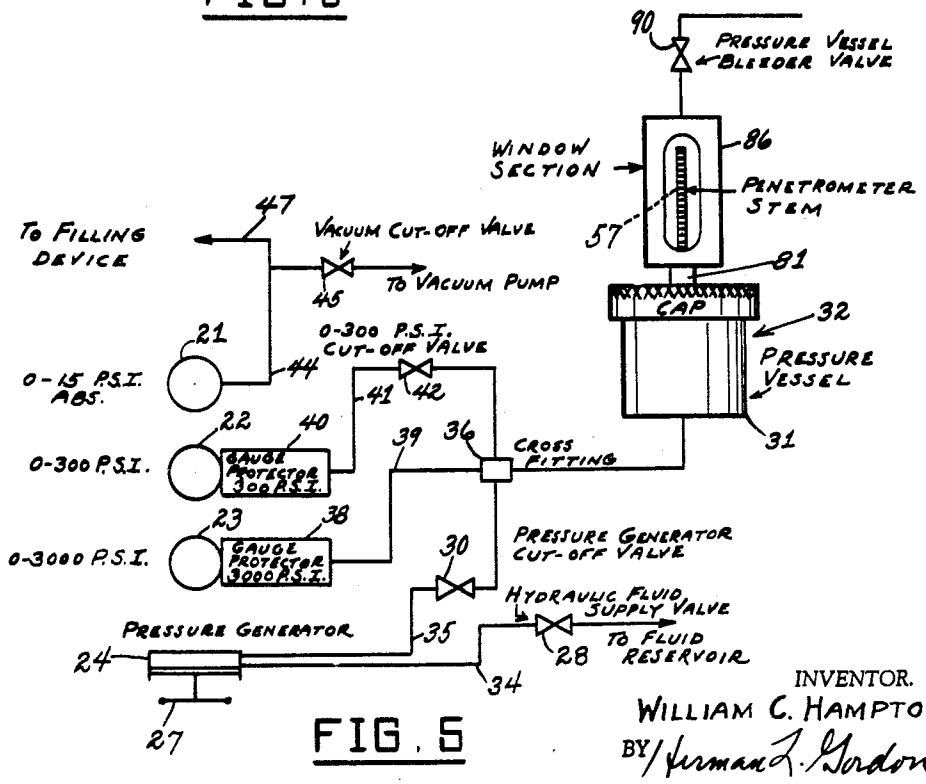


FIG. 5

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PENETROMETER FOR POROSIMETERS

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Original application May 12, 1958, Ser. No. 734,778. Divided and this application Feb. 21, 1961, Ser. No. 90,709

5 Claims. (Cl. 73—38)

This is a division of Serial No. 734,778, filed May 12, 1958, entitled "Penetrometer and Filling Device Therefor for Porosimeters."

This invention relates to porosimeters, and more particularly to a penetrometer for use with a porosimeter of the type employing the mercury intrusion method.

A main object of the invention is to provide a novel and improved penetrometer for use in a porosimeter of the type employing the mercury intrusion method to measure the sizes and volumes of the pores of porous materials.

A further object of the invention is to provide an improved penetrometer for use in a porosimeter, said penetrometer being simple in construction, being easy to fill, and being especially suitable for use in testing powdered samples of material, the penetrometer being formed and arranged so that it is not necessary to employ means to plug up its capillary stem to prevent the powdered material from entering the capillary passage thereof and so that the inconvenience and inaccuracy involved in using such means is avoided.

A still further object of the invention is to provide an improved penetrometer for use in a porosimeter of the type disclosed in U.S. Patent No. 2,886,964, to Justin J. Shapiro and Nathaniel M. Winslow.

Further objects and advantages of the invention will become apparent from the following description and claims, and from the accompanying drawings, wherein:

FIGURE 1 is a side elevational view, partly in vertical cross-section, of an improved penetrometer and a filling device therefor, constructed in accordance with the present invention, the penetrometer being shown in its inverted position and arranged for filling.

FIGURE 2 is a horizontal cross-sectional view taken on line 2—2 of FIGURE 1.

FIGURE 3 is an enlarged vertical cross-sectional view taken through the bulb end of the penetrometer on the line 3—3 of FIGURE 1.

FIGURE 4 is a horizontal cross-sectional view taken on the line 4—4 of FIGURE 3.

FIGURE 5 is a schematic view showing the conduit connections of a porosimeter such as that disclosed in U.S. Patent No. 2,886,964, wherein a penetrometer according to the present invention may be employed, and illustrating how said penetrometer is utilized.

In a porosimeter employing the mercury intrusion method, as disclosed in U.S. Patent No. 2,886,964, a gas-free hydraulic system is used to transmit pressure from a pressure generator 24 (FIGURE 5) to a pressure vessel 32 containing a calibrated transparent chamber, such as a penetrometer 57, exposed to the pressure in said pressure vessel and containing the porous material under test. The volume of penetration by mercury after application of pressure, at each pressure, is read on the calibrated stem of the penetrometer, which is visible through a transparent window section of the pressure vessel.

The porosimeter includes respective pressure gauges 21, 22 and 23. Gauge 21 measures pressure from zero to 15 pounds per square inch absolute, gauge 22 measures pressure from zero to 300 pounds per square inch, and gauge 23 measures pressure from zero to 3000 pounds per square inch.

The outlet of the pressure generator 24 is connected through a conduit 35 and a manually operated control valve 30 to the bottom member 31 of the pressure vessel 32 through a cross fitting 36. A hydraulic fluid supply conduit 34, provided with a manually controlled valve 28 is connected between a fluid reservoir and the pressure generator 24. Gauge 23 is connected through a conventional gauge protector 38 and a conduit 39 to cross fitting 36.

Gauge 22 is connected through a conventional gauge protector 40 and a conduit 41, including a manually controlled cut-off valve 42 to the cross fitting 36.

Gauge 21 is connected through a conduit 44 and a vacuum cut-off valve 45 to a conventional external vacuum pump. Also connected to the conduit 44 is a conduit 47 connected to the upper conduit-connection tube 48 of a filling device 49, shown in detail in FIGURES 1 and 2.

Filling device 49 comprises an upper chamber element 50 and a tubular lower chamber element 51 having a tapered top end portion 52 adapted to be sealingly and detachably secured in the downwardly flaring lower end portion 53 of upper chamber element 50. Chamber element 50 has the rounded top end wall 54.

Chamber element 51 has the conical bottom end portion 55 adapted to supportingly receive the end of the calibrated tube portion 56 of the penetrometer 57, as shown in FIGURE 1.

Integrally formed with the bottom of the conical member 55 and depending axially therefrom is a conduit 55′ provided with a manually operated cut-off valve 56′, leading to a spherical reservoir 57′, integrally connected to the end of the conduit 55′. A pressure-equalizing conduit 58′ connects reservoir 57′ to a portion of tubular chamber 51 spaced a substantial distance above the conical lower end 55 thereof, as shown at 59′.

Chamber 51 is provided with a horizontally extending conduit element 59 provided with a three-way stop cock 60 formed with an atmospheric vent conduit 61 and another conduit 62, said stop cock being of conventional construction. The stop cock 60 is provided with an operating element 63 which may be rotated to respective different positions in which the stop cock (1) seals off conduit 59, (2) connects conduit 59 to conduit 62, or (3) connects conduit 59 to atmospheric vent conduit 61.

As shown in FIGURE 1, the downwardly flaring skirt member 53 is formed at its lower end with a peripheral bead 60′. Above said bead, the skirt member 53 is provided iwth respective upwardly and outwardly projecting lugs 61′, 61′. The upper portion of chamber 51 is provided with respective downwardly and outwardly extending lugs 62′, 62′ located below the lugs 61′, 61′. Respective coiled fastening springs 83′ are detachably fastened at their ends to the pairs of lugs 61′, 62′ and engage on the bead 60′, as shown in FIGURE 1, whereby the tension of the springs acts to retain the skirt member 53 in sealing engagement with the upwardly tapering member 52.

The filling device 49 is adapted to be supported in a vertical position by conventional supporting clamps on a vertical bracket on the porosimeter, as described in U.S. Patent No. 2,886,964. Also adjustably supported on this bracket is a bottle containing suitable hydraulic liquid, such as alcohol, or the like, the conduit 34 being connected to the lower portion of this bottle through the supply valve 28. Also supported on this bracket is another bottle adapted to contain mercury, said last-named bottle being located below the filling device 49 and being so arranged that the depending vertical portion 72 of the conduit element 62 may be at times engaged through the neck of the bottle into the mercury contained therein,

whereby mercury may be admitted into conduit 59 and into the filling device, as will be presently described.

The chamber elements 50 and 51 are formed of suitable transparent material, such as glass, or the like.

The penetrometer 57 comprises the calibrated glass stem 56, open at one end 73 and formed at its other end with a flaring bell-shaped member 74 having an outwardly projecting top flange 63' provided with a ground flat annular top surface 64'. A ground flat disc 65' is engaged on the top surface 64', the contacting surfaces being preferably coated with a light film of sealing material, such as silicon grease. A cylindrical metal ring member 66' surrounds flange 63' and disc 65' and is formed with an inturned bottom flange 67' which underlies flange 63'. The upper portion of ring member 66' is formed with internal threads 68' with which are threadedly engaged the external threads 69' of annular clamping ring 70'. An O-ring 71' of resilient deformable material, such as rubber, or the like, is engaged between the bottom end of ring 70' and the peripheral margin of disc 65'. Ring 70' is provided with a serrated top flange 79' which is substantially equal in outside diameter to the ring member 66', whose outside surface is similarly serrated.

The penetrometer stem is extended upwardly inside member 74, as shown at 72', so that it terminates a very short distance below the cover disc 65', and is bevelled at its top end, as shown at 73'. Thus, there is defined an annular space 74' around the extension 72', adapted to receive the material under test, for example, powdered material 75'. The open end of extension 72' is above the surface of the sample 75' when the penetrometer is held in its customary upright position during loading of the test sample in annular space 74', thus preventing the sample from sliding down the capillary bore 76'. When the mercury rises in this annular space 74' it floats the sample up to the top of the space, but since the mercury is flowing into the space via the open end of extension 72', the sample cannot escape down the bore.

As is fully described in U.S. Patent No. 2,886,964, the bottom member 31 of pressure vessel 32 is formed with an internal cavity adapted to receive the sample-containing bulb portion of the penetrometer when the penetrometer is in its working position (inverted as compared with the position thereof shown in FIGURE 1), and the pressure vessel 32 is provided with a neck portion 81 having an internal bore of sufficient size to freely receive and vertically support the penetrometer stem 56 therein. The calibrated stem of the penetrometer is visible through a vertical transparent window provided in the observation chamber 86.

In determining the pore sizes and volumes of a material, a weighed sample 75', of known volume or density, of the material is placed in the penetrometer sample space 74' and said space is sealed by fastening the cover disc 65' thereon and securing same on the sealing surface 64' by means of the elements 66', 70' and 71', as shown in FIGURE 3. The penetrometer 57 is then placed, stem down, in the filling device 49, which is then closed, as shown in FIGURE 1, and is connected to the vacuum pump by the conduit 47 engaged on the tube element 48, as above described.

The mercury bottle is then clamped in filling position with the mercury filling tube 72 well below the surface of the mercury.

With vacuum valve 45 open and the filling device stop cock 60 closed to both tube 61 and conduit 62, the vacuum pump is started. The stop cock 60 is then carefully manipulated by means of its valve handle 63 to draw mercury just beyond the stop cock into conduit 59, but not into the filling chamber.

The filling chamber is then evacuated to a pressure below 100 microns, after which vacuum valve 45 is closed.

Sufficient mercury to fill the penetrometer 57 is then admitted into the filling device by means of stop cock 60. At this time the valve 56' is closed, so that the mercury accumulates around the bottom end of stem 56 in conical portion 55.

The stop cock 60 is then carefully manipulated to admit air into the filling device, forcing the mercury into the penetrometer, the stop cock 60 being closed at an air pressure in the filling chamber sufficient to completely fill the penetrometer, the value of said pressure being indicated on gauge 21.

Following this, valve 56' may be opened to allow the excess mercury to drain into bulb 57', whereby the calibrations on the end of stem 56 may be observed for subsequent changes in the air pressure in the filling chamber, with the apparatus in the position of FIGURE 1.

Thus, stop cock 60 may be further manipulated to allow the air pressure in the filling chamber to increase, whereby the mercury in the penetrometer bore will be forced upwardly in accordance with the penetration of the mercury into the sample 75' as the air pressure increases.

The above procedure allows the porosity data for the sample material to be obtained at the large pore end of the spectrum, namely, the data derived from the displacement of the end of the mercury column relative to the calibrations on stem 56 as air is admitted to increase the pressure in the filling device in steps up to and including atmospheric pressure.

After this preliminary data has been obtained, the filling device 49 is carefully unfastened from its clamps and inverted, being refastened to its clamps in inverted position. The drop in level of the mercury column in stem 56, corresponding to the volume of pores entered at this pressure, is then noted, said pressure being the sum of the pressure due to the height of mercury above the sample (1 inch of mercury=0.492 pound per square inch gauge) and the indicated absolute pressure (atmospheric pressure).

The penetrometer is then carefully removed from the filling device 49 and is placed in the pressure vessel 32 for the continuation of the test, as decribed in U. S. Patent No. 2,886,964. Thus, the penetrometer is inserted in the pressure vessel with its bulb portion housed in the member 31 and the calibrations on the stem 56 facing outwardly and visible through the transparent window of observation section 86. The hydraulic fluid bottle (connected to conduit 34 through valve 28) is then raised to an elevated position such that liquid emerges from the bleeder valve 90, and is secured in said position, the bleeder valve 90 being then closed. Valve 28 is then closed and the pressure in chamber 32 is then raised by rotating the handle 27 of the pressure generator 24, the pore volumes and pore diameters (as related to gauge pressures) being noted from the mercury column in stem 56 at appropriate intervals. Since stem 56 is open at its top end, the pressure in chamber 32 is applied to the mercury column in said stem and to the mercury in space 74', causing the height of said mercury column to change with the variations in pressure.

When a pressure of about 250 pounds per square inch is reached, valve 42 is closed, the higher pressures being then read on the gauge 23.

When the pore size spectrum readings have been obtained over the desired pressure range, the pressure is relieved by backing off the pressure generator operating screw slightly. Valve 28, bleeder valve 90 and valve 42 are then opened. The hydraulic fluid supply bott'e is then lowered to a position below the top rim of member 31, allowing the instrument to drain. The pressure vessel 32 is then opened and the penetrometer 57 is removed, whereby the apparatus is ready for another determination.

While a specific embodiment of an improved apparatus for measuring the pore sizes and volumes of pores in material has been disclosed in the foregoing description, it will be understood that various modifications within the spirit of the invention may occur to those skilled in the art. Therefore it is intended that no limita-

tions be placed on the invention except as defined by the scope of the appended claims.

What is claimed is:

1. In a porosimeter, a penetrometer comprising a bell-shaped element, a sealing plate on said bell-shaped element, means clampingly securing said plate to said bell-shaped element, an elongated, tubular, open-ended stem member rigidly and communicatively connected to said bell-shaped element, and an extension of said tubular stem member in said bell-shaped element extending toward and terminating a relatively short distance from said sealing plate, said extension defining an annular sample-retaining space around the extension in said bell-shaped element.

2. A penetrometer comprising a bell-shaped member, a peripheral flange on said bell-shaped member, a cover disc sealingly engaged on said flange, a ring member surrounding said flange and said cover disc, means on said ring member engaging beneath said flange, a clamping member threadedly engaged in the upper portion of said ring member and clampingly engaging said cover disc, an elongated, tubular, open-ended stem element rigidly and communicatively connected substantially axially to said bell-shaped member, and an extension of said tubular stem element in said bell-shaped member extending toward and terminating a relatively short distance from said cover disc, said extension defining an annular sample-retaining space around the extension in said bell-shaped member.

3. A penetrometer comprising a bell-shaped element,

sealing means secured on said bell-shaped element, an elongated, tubular, open-ended stem member rigidly and communicatively connected to said bell-shaped element, and an extension of said tubular stem member in said bell-shaped element extending toward and terminating a relatively short distance from said sealing means, said extension defining an annular sample-retaining space around the extension in said bell-shaped element.

4. A penetrometer comprising a bell-shaped element, a sealing plate secured on said bell-shaped element, an elongated, tubular, open-ended stem member rigidly and communicatively connected to said bell-shaped element, and an extension of said tubular stem member in said bell-shaped element extending toward and terminating a relatively short distance from said sealing plate, said extension defining an annular sample-retaining space around the extension in said bell-shaped element.

5. A penetrometer comprising a bell-shaped element, a sealing plate secured on said bell-shaped element, an elongated, tubular, open-ended stem member rigidly and communicatively connected to said bell-shaped element, and an extension of said tubular stem member in said bell-shaped element extending toward and terminating a relatively short distance from said sealing plate, said extension being bevelled at its top end and defining an annular sample-retaining space around the extension in said bell-shaped element.

No references cited.