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Remote Mercury Porosimetry

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Approved by

A.C. DEMILDT

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ABSTRACT

A commercial mercury intrusion porosimeter was modified to improve the hot cell compatibility of the instrument and to provide fast processing of the recorded values of pressure and volume.
In order to test the performance of the modified instrument, physical characterization of an unirradiated nuclear fuel pellet was carried out. Experimental procedures and results are reported.

INTRODUCTION

Ceramics when processed usually start as a highly porous compact and are frequently still porous after processing. Therefore, porosity is a highly interesting property since it is often correlated with other properties such as strength, durability, leachability, permeability, thermal conductivity, etc. ... .

Likewise, nuclear fuel pellets made from oxides can never be fabricated to reach 100% theoretical density. The fabrication of very high density pellets would be extremely difficult in the powder ceramical techniques applied. Furthermore, a certain porosity of the fuel pellets is required as a standby volume to accommodate fuel swelling during burn-up and it also improves the creep properties of the pellets. Thus, the pores did become an essential part of the texture of nuclear fuel.

On the other hand the existence of pores and their appearance (pore size distribution) play an important rôle in several detrimental effects such as the densification of the fuel at the start of the irradiation, the possible excessive fuel temperatures or clad collapse; the gas exchange behaviour in the later stages of the irradiation, possibly leading to gas release and hence pressure build-up (Xe, Kr) or the internal corrosion (Cs, I) of the cladding; etc. ... . Thus, minor differences in pore structure may greatly affect the in-pile performance and the storage behaviour of nuclear fuel rods.

Finding an adequate optimum in the pore structure is based upon the precise knowledge of interdependencies and the existence of reliable methods of measurement.

The total pore volume of a solid can be subdivided into an open fraction, i.e. with a connection to the outside of the matrix, and a closed fraction. The open porosity fraction can be determined by penetration techniques. One of these methods is mercury porosimetry. The principle of this simple and rapid experimental technique is based on the fact that mercury behaves as a non-wetting liquid towards most substances.

Consequently, it does not penetrate into the openings and cracks of these substances and one must apply pressure to make it do so. Measurement then of the volume of mercury forced into a sample under increasing pressure will reveal useful information on the open pore space characteristics of the sample.

The present paper will deal with the experimental set-up of this technique and the interpretation of the pressure-volume data obtained, especially for nuclear fuels.

APPARATUS AND OPERATING PRINCIPLE

A modified porosimeter 2020 from Carlo Erba (Milan, Italy) is used to measure the pore size distribution. Basically, the experimental set-up consists of six parts :

- a dilatometer, the container of the sample
- a mercury filling device comprising a vacuum pump
- an analytical unit comprising the pressurizing system and the mercury penetration monitoring system
- a control unit which totally automates the mercury intrusion and extrusion cycles
- a X-Y electronic recorder
- a data processing unit which records and processes the pressure-volume data.

1. The dilatometer

The dilatometer (Fig. 1) is made of glass and consists of a lower bulb inside which the weighed sample is placed and an upper removable stem with calibrated constant internal section (typical 3 mm). The capillary stem and dilatometer bulb are connected to each other through a lubricated ground joint which is secured by screwing a cap tightly.

2. The mercury filling device

The assembled dilatometer is weighed and is placed on its support on the filling device (Fig. 2) with the capillary end towards the needle of the unit to obtain a seal on the gasket.

The filling device is equipped with three valves giving access to a rotary vacuum pump, a mercury reservoir and a venting line. Opening the shut-off valve (P) connects the dilatometer with the vacuum line, which can reach a vacuum of $\sim$ 13 Pa ($\sim$ 0.1 Torr). This outgassing procedure has two functions: removing of air out of the dilatometer and removing of all foreign adsorbed species from the sample. The removal of adsorbed

species may be important in order to clean the outer surface and the pore walls of the solid, which may be decisive for obtaining the expected contact angle when it is in contact with mercury. Inadequate pumping of air from the dilatometer will leave some air in the porous sample and thus affect the intrusion of mercury into the pores. From experiments it appears not to be very critical to what pressure the sample is evacuated. Only at evacuation pressures above 1.33 kPa (10 Torr), an appreciable deviation of the pore size distribution curve caused by the compression of residual air does occur [1].

When a good vacuum has been reached, the dilatometer is filled with pure mercury by opening the valve connected to the mercury reservoir. Due to the vertical position of the dilatometer in the filling device and the special arrangement of the three valves, evacuation can be maintained during the mercury filling process, thus eliminating any residual gases. Finally, the dilatometer is vented to atmospheric pressure by actuating valve A, removed from the filling unit and weighed.

All foregoing filling operations are performed remotely, the filling device (with exception of the vacuum pump) being installed in a hot cell. Some minor modifications were necessary : the valve handles have been made master-slave manipulator compatible and the mercury reservoir which is originally placed inside the back-side of the filling unit has been displaced to the side of the unit where it is accessible for the manipulators.

3. The analytical unit

The dilatometer filled with mercury and containing the sample is placed inside the high pressure autoclave of the analytical unit (Fig. 3 and 4).

This autoclave together with its peripherals (components 1 through 7 in Fig. 3) have been removed from the original case and are reassembled in the hot cell. At the same time the two manual valves (4 and 5 in Fig. 3) and the autoclave knurled locking nut (2 in Fig. 3) have been adapted for remote handling with master-slave manipulators.

The pneumatic hydraulic system of the porosimeter analytical unit (components 8 through 15 in Fig. 3) is still located in the original case and is situated outside the hot cell.

The connection of the outer high pressure oil reservoir with the inner autoclave is made by a flexible plastic tube passing through the cell wall. In the original apparatus the autoclave is filled with high pressure oil by opening valve 4 (Fig. 3), immediately after opening valve 5 (Fig. 3) and leave it open until oil overflows. Thus oil is drawn by gravity from the high pressure oil reservoir which is located on top of the original case, above the autoclave. In the hot cell arrangement this is not the case anymore and modifications have been made. The original plastic high pressure oil reservoir is replaced by an air-tight metallic reservoir. It is connected to the compressed air circuit of the laboratory by the intermediate of a regulating valve allowing the installation of a small overpressure of about 0.1 atm in this reservoir. This overpressure serves as the driving force to fill up the autoclave in the hot cell.

When the autoclave is air free (no air bubbles appearing anymore in the overflow) valves 4 and 5 (Fig. 3) are closed. At this moment an intrusion-extrusion cycle can be started. The necessary pressure in the autoclave is produced by a pressure multiplier consisting of a differential system with two pistons, a low pressure and a high pressure piston in a ration of 1:100. The low pressure piston is actuated by oil drawn from the low pressure oil reservoir by the pump. The high pressure piston transfers the pressure to the mercury in the dilatometer through a special dielectric oil as intermediate hydraulic fluid. A pressure transducer is connected to the top part of the pressure multiplier. This transducer is calibrated for the two ranges 0 to 20 MPa and 20 to 200 MPa to improve the resolution at lower pressure. From the transducer the circuit is connected to the autoclave by means of a high pressure line (physically consisting of a thick-walled metallic capillary) passing through the cell wall.

The pneumatic hydraulic system is equipped with three independent safety mechanisms :

- an electromechanical gauge stops the pump when the oil pressure in the low pressure circuit rises above a preset value (e.g. 20 bar)

- a bursting disc is fitted to the low pressure hydraulic circuit to prevent excess pressure
- an electric command actuated by the pressure transducer when reaching a preset maximum pressure in the high pressure circuit also stops the pump.

The progress of the penetration of mercury into the sample is electrically monitored by means of a capacitance system. The capacity is measured of a conductive screen on the inner wall of the high pressure autoclave near the stem of the dilatometer. When the pressure increases, the level of mercury in the stem falls and consequently the capacitance changes. This variation is electronically processed and converted into a volume. The introduction of a conductive point in the bottom of the dilatometer-bulb (Fig. 1 : contact rod), closing the electric circuit on contact with the mercury, enables a full automation of the instrument. Thus the decrease in volume of the mercury is recorded in dependence of the pressure and a porosimetric curve is obtained indicating how large a volume penetrated into the pores of the sample at a given pressure.

4. The control unit

The porosimeter control module is a simple and easy to operate control station which totally automates the mercury intrusion and extrusion cycles. The analytical parameters are set and displayed on the front panel of the control unit (Fig. 5) :

- the pressure limit : to set the maximum pressure which has to be obtained in the analysis (max. 1990 bar)
- the decrease : to set the decreasing rate up to a maximum of 19 bar.s^{-1}. This control is actuated after the preset maximum pressure has been obtained at which point the decreasing cycle is automatically initiated.
- the pump speed : to set the pump speed, 1 being the lowest speed and 5 the highest one. For research purposes a pump speed of 1 to 2 is recommended. The rate of pressure build-up may not be too high because a certain time is needed for the transport of mercury

through the porous system and thus to reach intrusion equilibrium. As pointed out by L. Moscou and S. Lub [1] , real intrusion equilibrium is reached in the Carlo Erba porosimeters. At a speed adjustment of 1, the pressure is built up gradually to a final pressure of 199 MPa in about 45 minutes.

- a pressure and volume visual display : to monitor the pressure and the volume. The autozero push-button and the zero volume fine knob are used to adjust zero volume at the beginning of an analysis. The zero pressure fine knob allows the adjustment of the starting atmospheric pressure.
- the start, stop and decrease push-buttons : to initiate or stop the intrusion and extrusion cycles. LED's are foreseen to monitor the working state of the pump and hysteresis-valve and to indicate the end of an analysis.

5. The X-Y recorder

Analogous signals of the volume of sample intruded by mercury and of the logarithm of the applied pressure are fed by the control unit to a X-Y recorder. This gives an immediate fingerprint spectrum on paper of the sample measured.

6. The data processing unit

The heart of the data processing unit (Fig. 6) is an IBM PC micro computer. The PC configuration is composed of :

- 640 Kbytes of RAM
- an Azerty keyboard
- a monitor : Hercules (720x 348)
- two DS/DD floppy disks : drive A contains the program disk while drive B is reserved for data disks
- one serial I/O port for the HP 7475 A plotter
- one parallel printer port for the proprinter
- a battery backup clock/calendar

The porosimeter has two BCD output ports (pressure and volume) and one start/stop flag. The two BCD output ports have each 15 bits. The highest bit from each port is the status-flag and this flag indicates when the datum of the port is ready. The 14th bit is an indication where the decimal point must be placed.

When the start signal on the porosimeter control unit is given, the start/stop flag is set active. Thus the computer, checking this flag, will start data processing.

The communication between the PC and the porosimeter is made by an I/O chart (DIAN DMS 541) equipped with two 16 bit input modules (DMS 211) and one 8 bit I/O module (DMS 201) for checking the start/stop flag.

The software is menu driven and written in basic. There are two blocks of programmes :

- measure : the data given by the porosimeter are transformed by the software from BCD to linear format and saved on floppy disk ;
- analyse : the saved results can be processed and written to the monitor screen, the printer or the plotter.

DATA PRESENTATION AND CALCULATIONS

The experimental data obtained with the porosimeter are pressure-volume pairs as shown in the tabulated print-out of the results acquired during the analysis of an unirradiated fuel pellet (Table 1).

To know how large a volume of mercury penetrated into the pores of a sample at a given pressure, corrections have to be made for the compressibility of the mercury.

This effect is eliminated by doing a blank run without a sample in the dilatometer (Fig. 7). The blank pressure-volume data can be described by a polynomial. This mathematical description is depicted by the solid line in Fig. 7 and fits very well the experimental data which are represented by the interrupted line pattern in the same Fig. 7.

Based on this polynomial and taking into account the proportion of mercury present into the dilatometer in the sample run relative to the blank run, the blank signal is subtracted from the sample signal. The result is illustrated by the solid line in Fig. 8 where the interrupted line pattern is corresponding to the raw data obtained during the analysis of the unirradiated fuel pellet (as tabulated in Table 1). Thus the total open porosity of this sample (4.887 g ; d = 10.43 $g.cm^{-3}$; V = 468.6 mm^3) amounts to 2.76 mm^3 or 0.59 %.

Under the assumption that the pores have openings of circular cross-section, the radius r of the pore into which mercury can be forced at a pressure p is given by Washburn's relationship :

$$r = \frac{-2\,\sigma \cos\Theta}{p} \qquad (1)$$

where r = pore radius

σ = surface tension of the mercury

Θ = contact angle of mercury with the solid

p = pressure

Although in most any porous body there are no cylindrical pores, equation (1) is nevertheless used almost universally to convert pressure data to pore sizes : the mercury forced into the pores of a solid under

the pressure p does not completely fill the pores, but approximates the shapes of the pores to a surface characterized by the minimum radius r of the mercury meniscus necessary for penetration to occur.
Values as different as 0.410 and 0.515 $N.m^{-1}$ have been quoted for σ and a range of 112 - 180° is often quoted for θ, the commonest reported angles being situated between 130° and 142° ([2] - [5]). For the purpose of daily routine, a surface tension value of 0.480 $N.m^{-1}$ and a contact angle of 141.3° are used, which bring the Washburn equation into a very simple form :

$$r(\text{Å}) = \frac{75{,}000}{p} \qquad (2)$$

with r(Å) = pore radius in Angstrom (= 10^{-10}m)
p = pressure in bar (= $10^{5}N.m^{-2}$)

These assumed values for surface tension and contact angle allow comparison of the pore size distribution of samples with the same nature, leaving some uncertainties in the accuracy of the absolute pore size.
It is apparent from the Washburn equation that at initial pressures of 1 bar, pores having radii of 7.5 µm will fill with mercury. The lower pore size limit is determined by the maximum pressure achievable in the porosimeter and is 38 Å at 1990 bar.
An important question is whether the porous solid will resist the high pressure during the mercury intrusion procedure. This was investigated experimentally by measuring the sample volume before and after the porosity measurement by means of the mercury immersion technique (mechanical vacuum pycnometer : [6]).
It was found that up to the maximum pressure of the porosimeter, no damage of the nuclear fuel could be observed.
Fig. 9 shows the diagram of cumulative pore volume (normalized to 100 %) and of the pore size distribution (graphically presented as the proportional change in pore volume per logarithmic unit interval of pore radius), both versus the logarithm of the pore radius r as derived from the pressure-volume data obtained for the nuclear fuel pellet sample. In this example, there is a marked rise of the pore volume accessible from the outside in the region between 0.4 and 2.6 µm.

CONCLUSION

A commercial mercury intrusion porosimeter has been successfully adapted for remote operation in a hot cell environment.
At the same time the porosimeter has been equipped with a computer-based data processing unit, so that the work-time to obtain an analysis is reduced to a minimum.
It has been demonstrated that the open porosity of high density nuclear fuel can be measured and that more detailed information on the pore size distribution of this open porosity can be obtained.

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Table 1 : Raw Pressure-Volume data for an unirradiated nuclear fuel pellet

Analysis number: 37
Date of performance: 15-01-88
Name of sample: PRIMO 4319/5 6/40 B2411

Raw data

No.	Pressure(bar)	Volume(mm3)	No.	Pressure(bar)	Volume(mm3)
1	1.00	0.00	2	1.07	0.00
3	1.17	0.00	4	1.32	0.00
5	1.55	0.06	6	1.79	0.10
7	2.08	0.10	8	2.49	0.19
9	3.08	0.20	10	3.89	0.28
11	4.80	0.30	12	5.76	0.30
13	6.45	0.38	14	6.92	0.40
15	7.48	0.40	16	8.09	0.40
17	8.79	0.40	18	9.61	0.40
19	10.41	0.47	20	11.28	0.50
21	12.28	0.50	22	13.28	0.50
23	14.34	0.52	24	15.42	0.39
25	16.52	0.60	26	17.72	0.60
27	18.91	0.62	28	20.09	0.70
29	21.27	0.70	30	22.48	0.70
31	23.83	0.72	32	25.16	0.79
33	26.51	0.80	34	27.89	0.80
35	29.25	0.87	36	30.72	0.90
37	32.20	0.92	38	32.68	1.00
39	35.13	1.02	40	36.62	1.10
41	38.19	1.12	42	38.78	1.20
43	41.32	1.23	44	42.83	1.30
45	44.42	1.35	46	46.10	1.40
47	47.77	1.49	48	49.43	1.55
49	51.03	1.60	50	52.69	1.70
51	54.50	1.75	52	56.18	1.82
53	57.88	1.90	54	59.60	1.98
55	61.33	2.02	56	63.15	2.12
57	64.93	2.20	58	66.65	2.26
59	68.34	2.34	60	70.19	2.41
61	72.00	2.50	62	73.79	2.55
63	75.58	2.65	64	77.34	2.71
65	79.22	2.79	66	81.12	2.85
67	82.92	2.95	68	84.78	3.00
69	86.61	3.08	70	88.48	3.15
71	90.39	3.20	72	92.23	3.30
73	94.04	3.37	74	95.91	3.42
75	97.88	3.50	76	99.79	3.57
77	101.62	3.60	78	105.51	3.75
79	111.37	3.93	80	117.22	4.10
81	123.11	4.28	82	129.03	4.45
83	135.01	4.59	84	141.01	4.74
85	147.10	4.85	86	153.16	4.98
87	159.28	5.10	88	165.36	5.21
89	171.59	5.32	90	177.81	5.43
91	184.08	5.50	92	190.37	5.61
93	193.87	5.71	94	199.13	5.81
95	205.45	5.91	96	211.75	6.02
97	218.18	6.11	98	224.45	6.21
99	230.85	6.32	100	237.20	6.41

101	243.80	6.52	102	250.28	6.62
103	256.73	6.72	104	263.23	6.82
105	269.90	6.93	106	276.40	7.04
107	282.93	7.16	108	289.60	7.27
109	296.30	7.36	110	302.93	7.48
111	309.60	7.58	112	316.38	7.69
113	323.05	7.81	114	329.88	7.92
115	336.83	8.03	116	343.58	8.15
117	350.53	8.25	118	357.13	8.36
119	364.30	8.47	120	371.28	8.58
121	378.13	8.69	122	385.18	8.80
123	392.15	8.91	124	399.05	9.02
125	406.23	9.14	126	413.20	9.25
127	420.65	9.35	128	427.80	9.47
129	434.75	9.59	130	442.03	9.70
131	449.30	9.81	132	456.53	9.93
133	463.90	10.04	134	471.23	10.15
135	478.53	10.28	136	485.80	10.42
137	493.45	10.54	138	500.98	10.65
139	508.20	10.76	140	515.75	10.88
141	523.20	11.01	142	530.95	11.12
143	538.50	11.26	144	546.05	11.38
145	553.83	11.51	146	561.38	11.63
147	569.20	11.75	148	577.00	11.87
149	584.73	11.99	150	592.60	12.14
151	600.40	12.24	152	608.15	12.36
153	616.30	12.48	154	624.10	12.61
155	632.08	12.75	156	640.05	12.86
157	648.03	12.99	158	656.05	13.13
159	664.18	13.24	160	672.10	13.36
161	680.25	13.48	162	688.48	13.61
163	696.65	13.75	164	705.00	13.85
165	712.90	13.99	166	721.28	14.13
167	729.88	14.24	168	738.23	14.36
169	746.60	14.49	170	755.05	14.63
171	763.35	14.75	172	771.85	14.86
173	780.40	15.01	174	788.85	15.14
175	797.25	15.26	176	805.85	15.38
177	814.63	15.53	178	823.08	15.65
179	831.78	15.77	180	840.58	15.92
181	849.30	16.05	182	858.18	16.18
183	866.88	16.31	184	875.75	16.45
185	884.58	16.57	186	893.60	16.70
187	902.35	16.83	188	911.40	16.95
189	920.33	17.08	190	929.45	17.23
191	938.73	17.35	192	947.75	17.47
193	956.85	17.61	194	966.18	17.74
195	975.30	17.86	196	984.48	17.98
197	993.95	18.11	198	1003.18	18.24
199	1012.50	18.36	200	1021.95	18.51
201	1031.20	18.63	202	1040.65	18.75
203	1050.25	18.87	204	1059.63	19.02
205	1069.10	19.15	206	1078.70	19.27
207	1088.15	19.41	208	1097.85	19.54
209	1107.15	19.67	210	1116.83	19.79
211	1126.48	19.92	212	1136.23	20.04
213	1145.98	20.17	214	1155.80	20.31

215	1165.85	20.44	216	1175.43	20.56
217	1185.78	20.70	218	1195.43	20.84
219	1205.40	20.96	220	1215.33	21.09
221	1225.35	21.24	222	1235.40	21.36
223	1245.48	21.48	224	1255.73	21.62
225	1265.68	21.75	226	1275.75	21.87
227	1286.20	21.99	228	1296.30	22.14
229	1306.65	22.25	230	1316.53	22.38
231	1327.08	22.53	232	1337.55	22.66
233	1347.50	22.79	234	1358.05	22.93
235	1368.38	23.05	236	1378.80	23.20
237	1389.35	23.33	238	1399.70	23.46
239	1410.25	23.60	240	1420.93	23.73
241	1431.43	23.85	242	1442.18	23.98
243	1452.63	24.12	244	1463.20	24.25
245	1474.08	24.38	246	1484.93	24.51
247	1495.50	24.64	248	1506.30	24.77
249	1516.93	24.89	250	1527.80	25.03
251	1538.58	25.17	252	1549.45	25.28
253	1560.43	25.43	254	1571.35	25.56
255	1582.38	25.69	256	1593.28	25.82
257	1604.03	25.94	258	1615.20	26.07
259	1626.28	26.21	260	1637.15	26.34
261	1648.20	26.46	262	1659.40	26.58
263	1670.75	26.73	264	1681.95	26.85
265	1693.10	26.98	266	1704.08	27.13
267	1715.48	27.25	268	1726.90	27.39
269	1738.03	27.53	270	1749.08	27.65
271	1760.88	27.78	272	1771.93	27.90
273	1783.13	28.03	274	1794.95	28.15
275	1806.35	28.28	276	1817.70	28.42
277	1829.15	28.54	278	1840.73	28.67
279	1851.95	28.80	280	1863.83	28.94
281	1875.20	29.06	282	1886.85	29.19
283	1898.75	29.33	284	1910.33	29.46
285	1921.98	29.58	286	1933.83	29.73
287	1945.65	29.84	288	1957.30	29.97
289	1969.23	30.10			

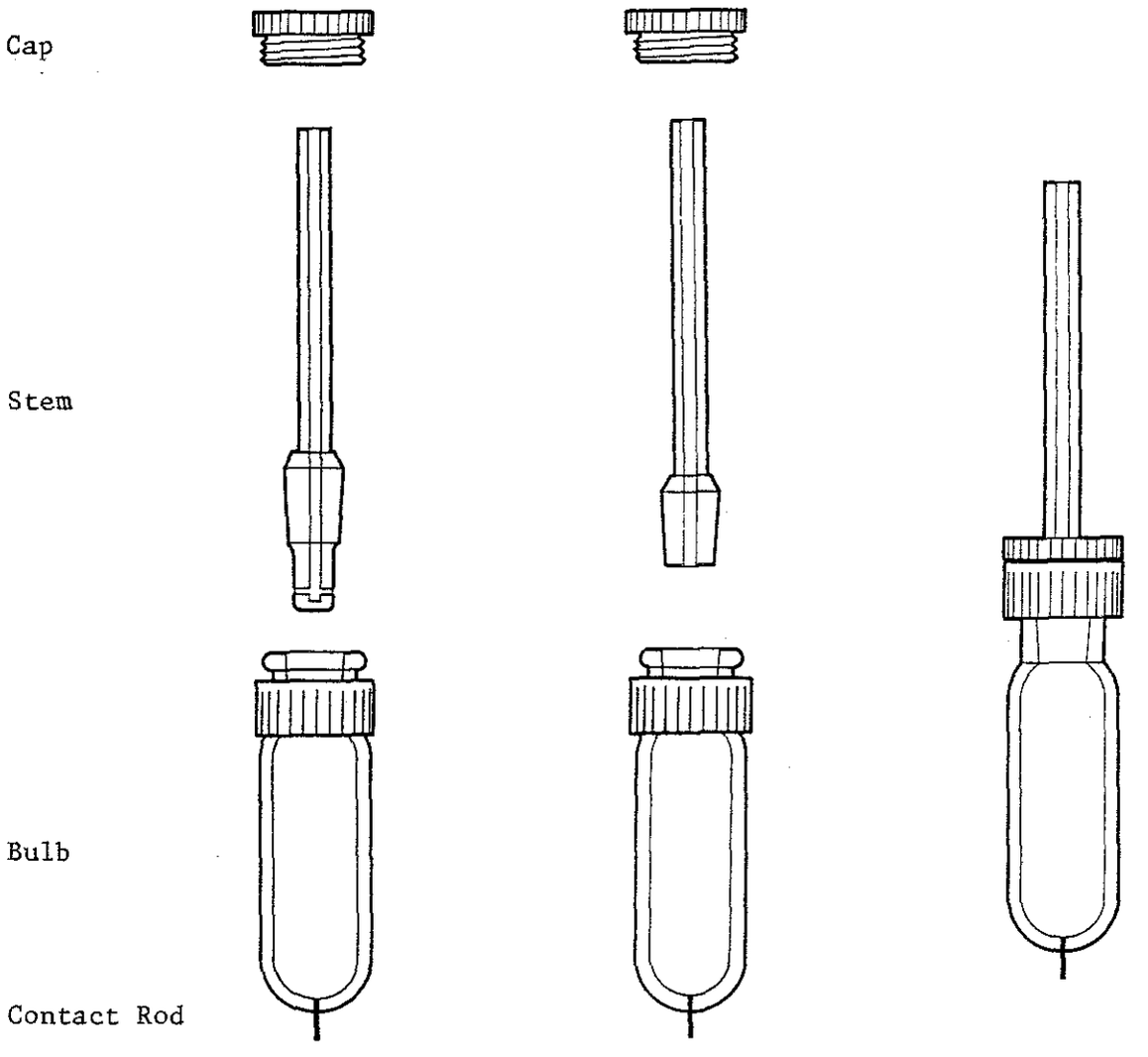


Fig. 1 : Dilatometer

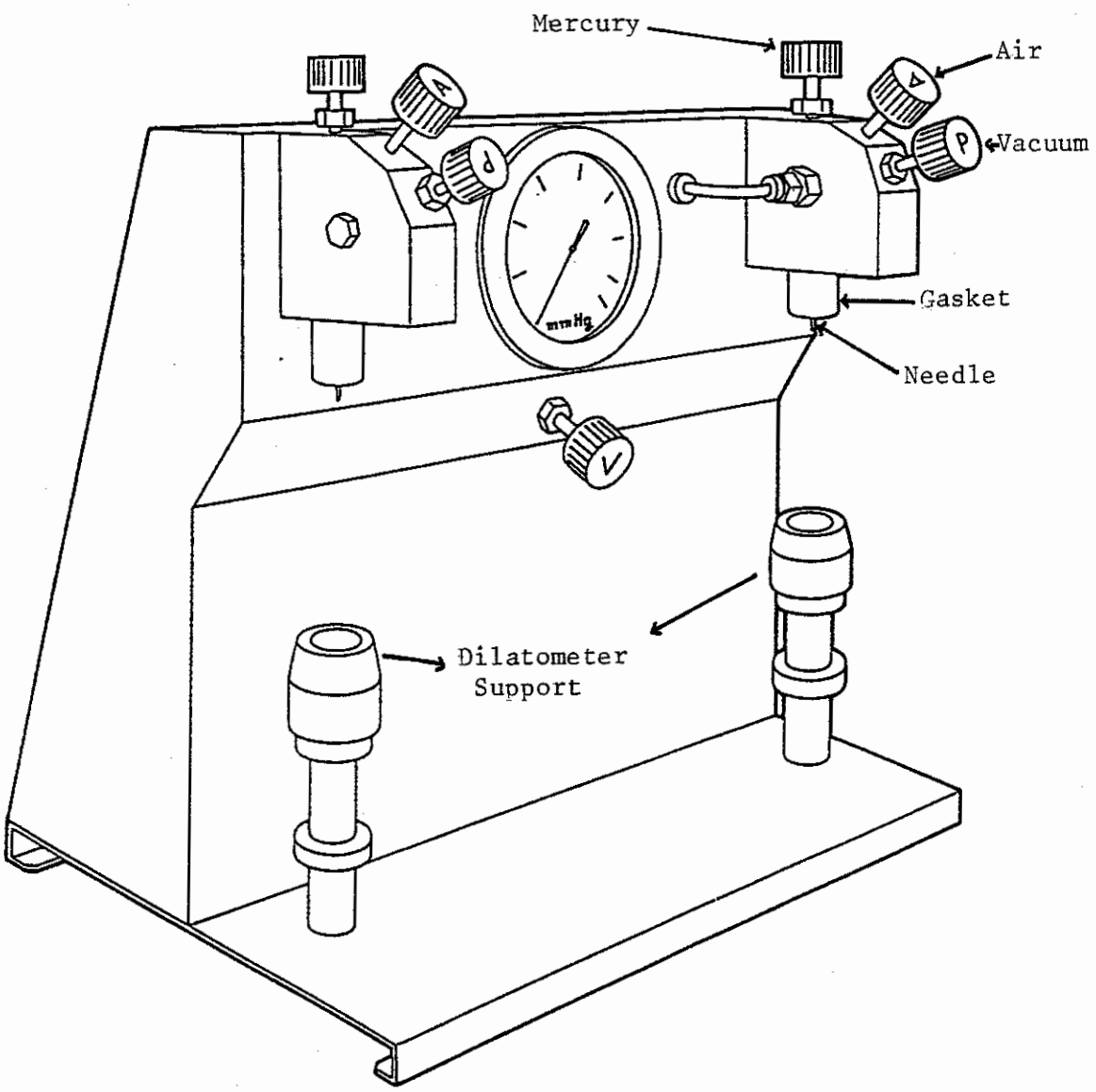
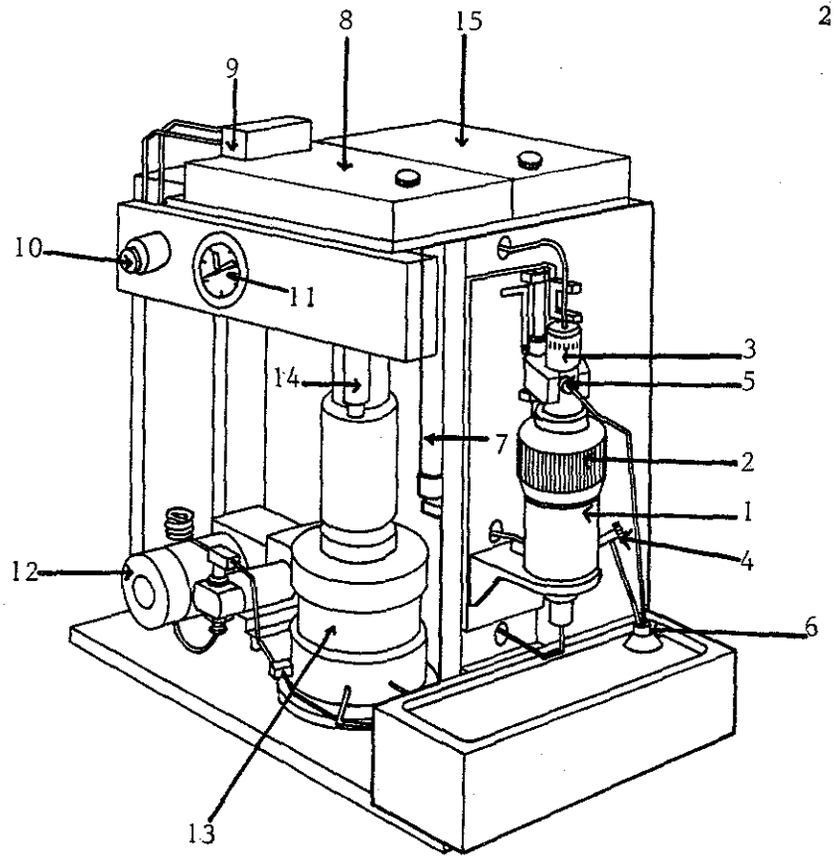


Fig. 2 : Duplex dilatometer filling unit



1. High pressure autoclave
2. Autoclave knurled locking nut
3. Capacitance detector
4. Three-way manual valve for filling and draining
5. Manual valve for oil overflow
6. Oil overflow and draining waste bottle
7. Autoclave head hydraulic damping system

8. Low pressure oil reservoir
9. Bursting disc for pressure safety
10. Programmable valve for hysteresis
11. Safety pressure gauge
12. Pressure pump
13. Pressure multiplier
14. Pressure transducer
15. High pressure oil reservoir

Fig. 3 : Original analytical unit

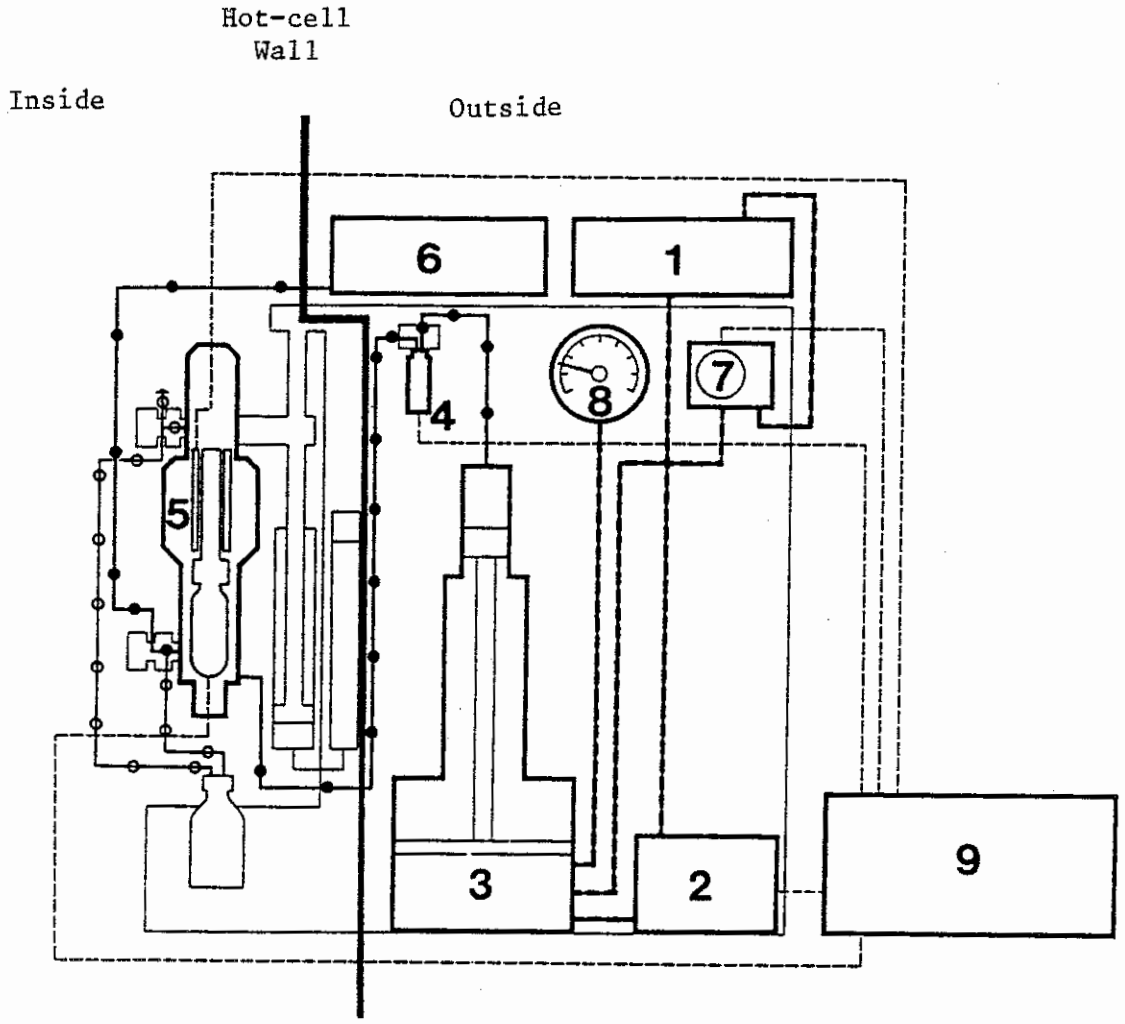


Fig. 4 : Schematic performance of the Analytical Unit

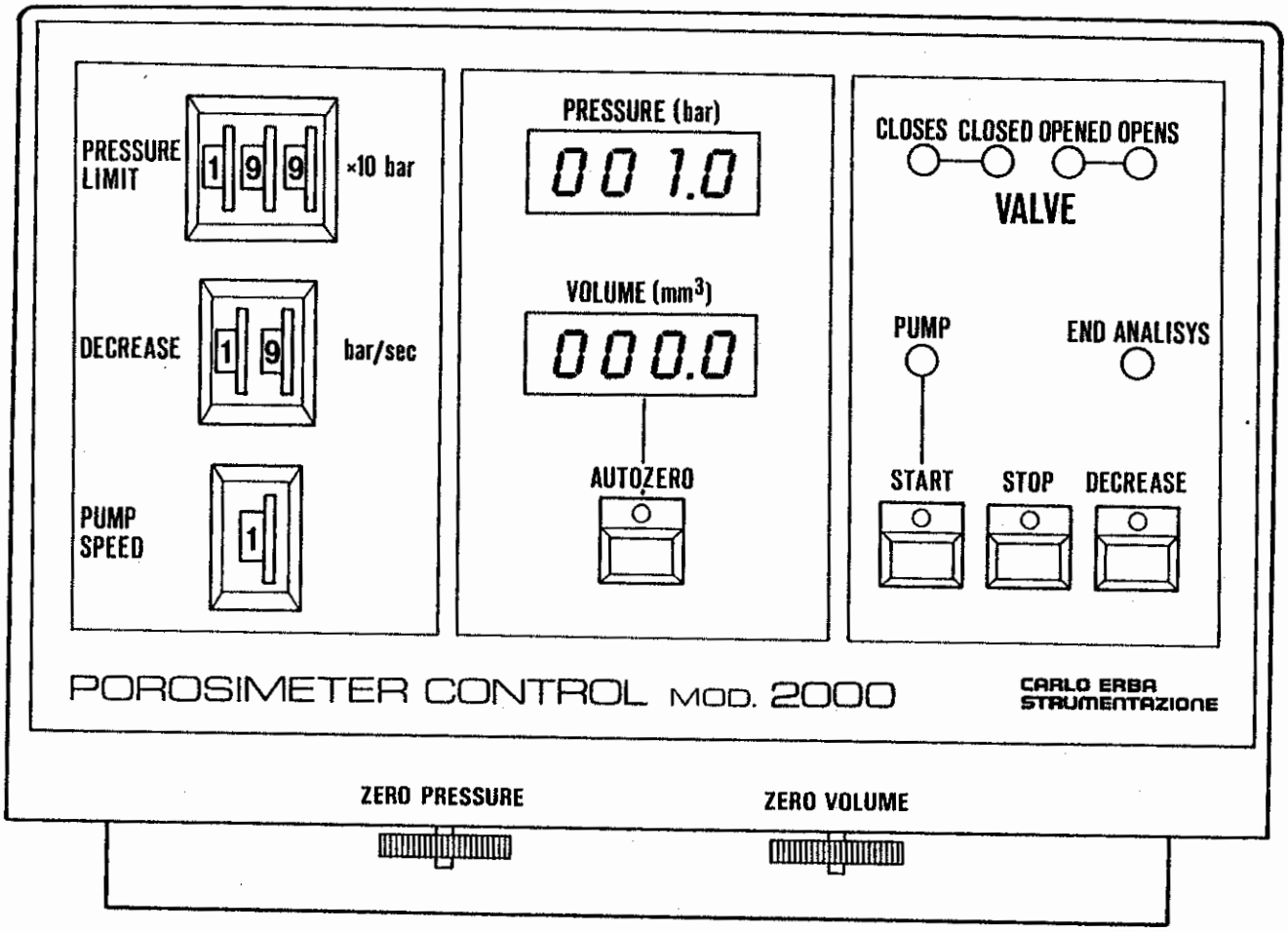


Fig. 5 : Porosimeter Control Unit

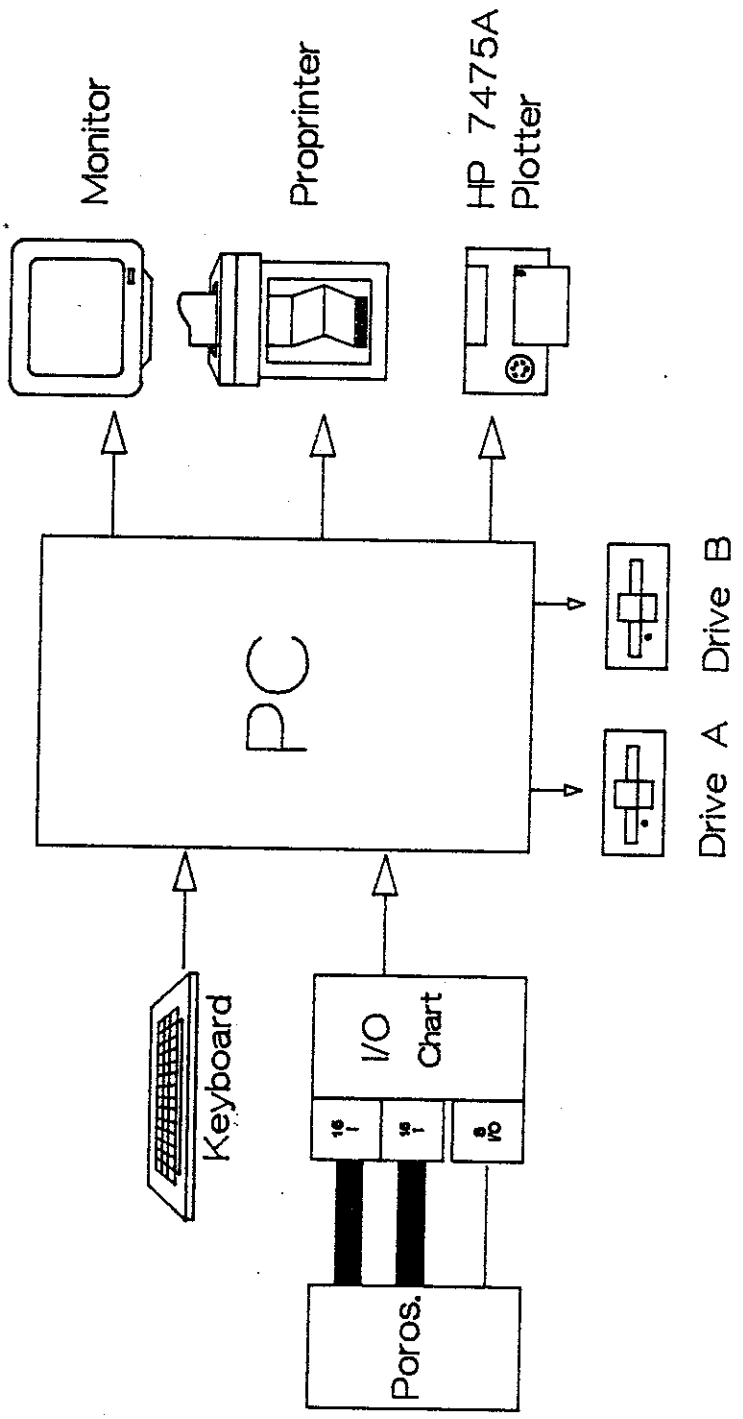


Fig. 6 : Data processing unit

SCK/CEN Mol
Sample : BLANK

Anal.No. : 43
Date : 18-03-88

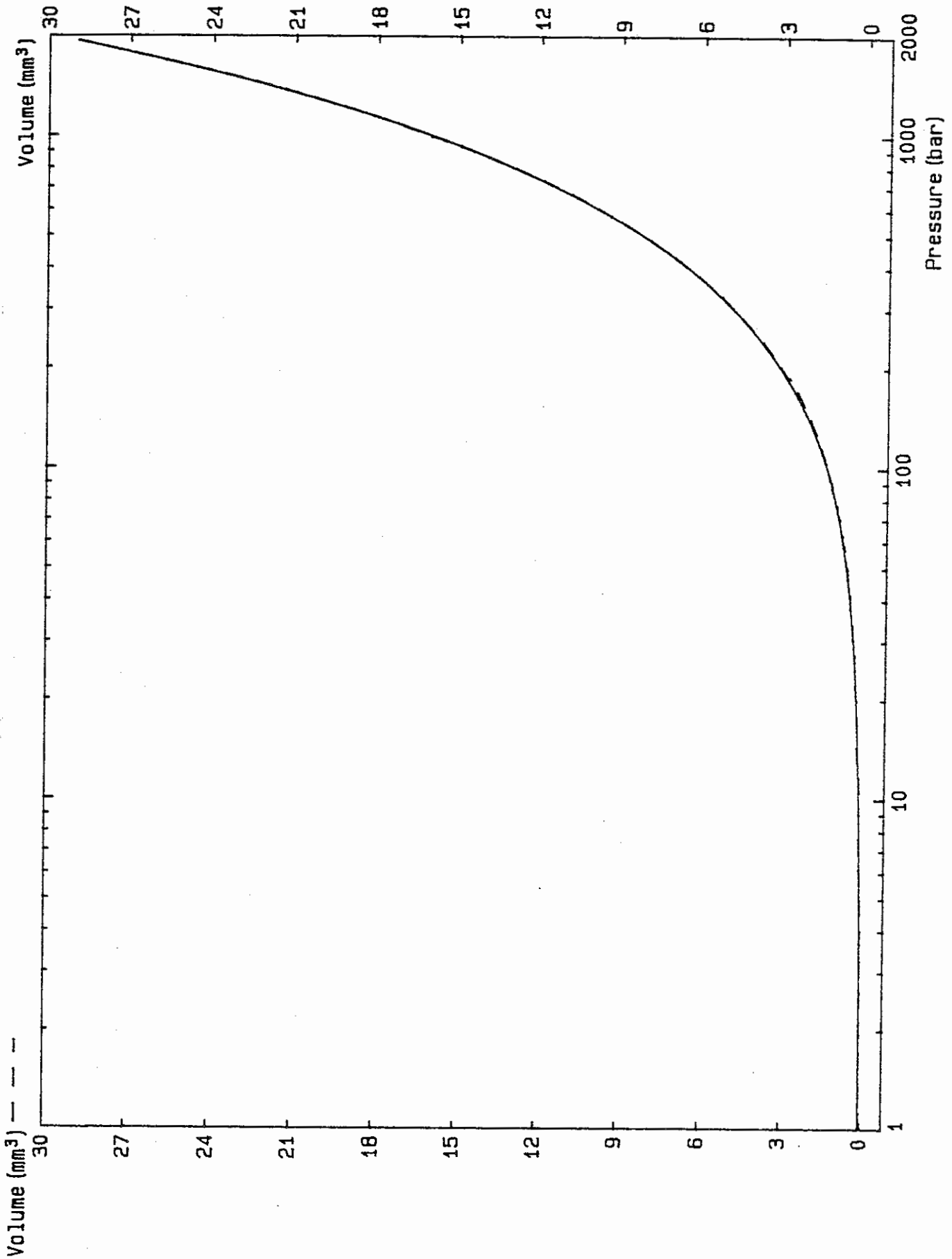


Fig. 7 : Porosimetric curve for a blank run

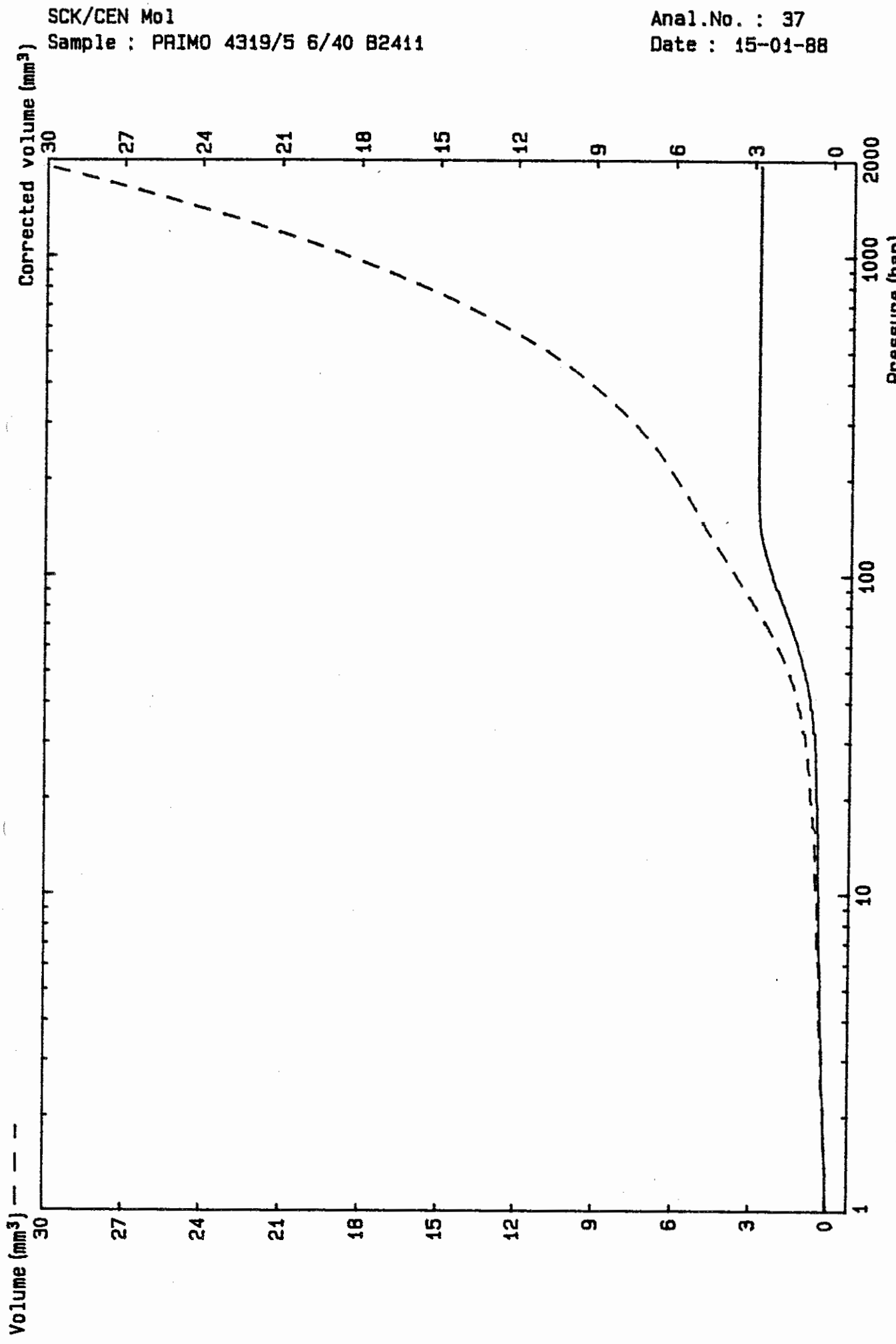


Fig. 8 : Raw Pressure-Volume data (---) and Volume of mercury intruded in an unirradiated nuclear fuel pellet as a function of the pressure applied (——)

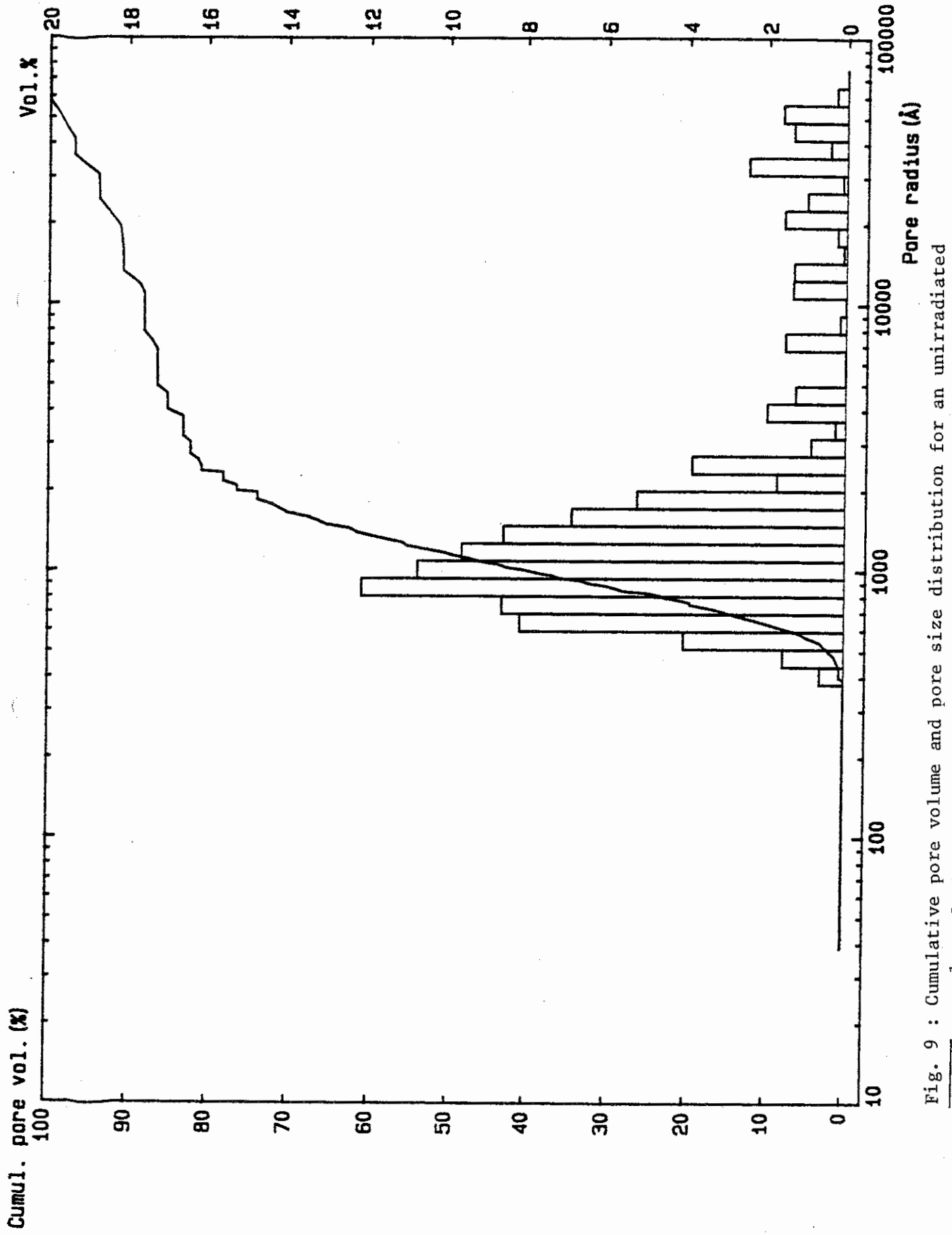


Fig. 9 : Cumulative pore volume and pore size distribution for an unirradiated nuclear fuel pellet

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