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The pore size distribution of several battery separators were determined experimentally by mercury penetration method using the Aminco-Winslow porosimeter. The performances of the separators under electrolyte penetration were also studied. The results of electrolyte penetration revealed that the degree of penetration was affected by the anions of the entrained salts only.

ELECTROLYTE PENETRATION OF BATTERY SEPARATORS USING A MERCURY POROSIMETER

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ELECTROLYTE PENETRATION OF BATTERY SEPARATORS USING A MERCURY POROSIMETER

INTRODUCTION

The use of mercury penetration to study both the size and the volume of voids in porous materials was first proposed by Washburn in 1921. The feasibility of this idea stems from the non-wetting properties of liquid mercury. In any system consisting of a porous solid and a non-wetting liquid, there is a repulsion of the liquid from the surface of the solid. Pressure, therefore, is required to force the mercury to enter the pore, and the required pressure is inversely proportional to the pore size because energy is expended to break the surface tension of the liquid mercury.

Within some 35 years after its introduction, this method, which was considered to be an elegant method, was used in research investigations only occasionally because the factors such as the cost, the availability, and the difficulty of manipulating the apparatus placed a practical limitation of the more general use of this method for the purpose of measuring the pore size. About 1950, Winslow devised a practical prototype instrument, which subsequently evolved into the porosimeter introduced by the American Instrument Company about 1958. Thereafter, this instrument has been made available commercially. The application of this technique to analyze the pore structure of the porous materials has spread into many areas such as plastics, ceramics, catalysts and sorbants. From the data reported in the literature, this method has been proven reliable when compared with other methods and in one instance when confirmed by the penetration of an orfice of known size (5).

Statement of the Problem

The working voltage of a lead-acid storage battery is lower than its electromotive force by an amount equal to the voltage drop within the cell itself. The magnitude of the voltage drop depends upon the internal resistance of the battery. In order to minimize the internal resistance of the battery, it is desirable to keep the plates of the battery as close as possible. However, practical limits to the plate separation are set by (a) the tendency for the dendritic lead to deposit on the negative plate during the charging process causing possibly short-circuited plates, (b) the buckling of persistently undercharged or long-discharged plates by the pressure of deposited lead sulfate and (c) the distortion of plates by mechanical and thermal shock to their supports. These limitations are largely overcome by the use of insulating and porous separators which fill almost all the space between the plates. The primary object of the separators is to prevent metalic conduction. Considering the porous separators, the size of the pores affects the strength and electrical resistance of the battery. The resistance for electrolyte conduction is inversely proportional to the pore size of the porous materials. It is also easier to have short circuits with those separators with larger pores because scaling of the active materials on the negative plates would invade the pores and make a metalic connection between plates of opposite polarity.

A variety of materials have been used as separators, such as alkali treated wood and paper, porous rubber, glass mats, and microporous polyvinyl chloride. Since the resistance to the passage of the electric current through the separators is an important parameter, varying with the kind of materials of which the separators is made and the treatment it has received, the object of this work is to study the performance of the separators by inspecting their pore-size distribution curves and examining the effects of entrained salts on porosimeter measurements.

THEORY

A liquid introduced into a porous solid or compacted powder will form a meniscus of curvature determined by the shape and size of the pores. Washburn has pointed out the fact that surface tension opposes the entrance into a small pore of any liquid having an angle of contact greater than 90°; therefore, a positive pressure will be needed to force the liquid into the pores. For contact angles less than 90°, the liquid enters spontaneously, and a retarding pressure will be needed to prevent entrance. The pressure, P, required to maintain equilibrium is given by the equation of Young and Laplace,

$$P = -\gamma_{LV}\left(\frac{1}{r_1} + \frac{1}{r_2}\right) \cos \theta_{LS} \qquad (1)$$

where θ_{LS} is the contact angle between the solid and liquid, γ_{LV} is the surface tension between the liquid and its vapor, and r_1 and r_2 are the radii of curvature (not necessary the principal radii of curvature) of any point on the surface boundary of the liquid in contact with its vapor in the pore space. For thermodynamic equilibrium to exist in a two-phase, gas liquid system, separated by an interface possessing surface tension, it is required that the surface free energy of the system be a minimum. Since the magnitude of the surface free energy is proportional to the surface area of the system, the total surface would be a minimum. From geometrical criterion, for minimum surface, the sum $\left(\frac{1}{r_1} + \frac{1}{r_2}\right)$ is a constant; therefore, the pressure does not depend upon the manner in which r_1 and r_2 are chosen.

Generally, the simplifying assumption is made that the pores can be treated in terms of some equivalent pore of circular cross section. If the cross section is not too large in radius, the meniscus will be approximately hemispherical. The two radii of curvature are thus equal to each other and to the radius of the cross section. Then Eq. 1 is reduced to

$$P\, r = -2\gamma_{LV} \cos \theta_{LS} \qquad (2)$$

or

$$P\, D = -4\gamma_{LV} \cos \theta_{LS} \qquad (3)$$

where D is the diameter of the equivalent cylindrical pores.

From the stated relationship, it appears that a porous material under zero pressure will absorb no non-wetting liquid in which it is immersed. When the pressure is raised to some finite value, the liquid will penetrate and fill all the pores having radii greater than the radii calculated from equation 2. When the pressure is increased, the amount of liquid absorbed increases continuously and monotonically at a rate proportional to the differential pore volume. Thus, a given pore size distribution will give a unique penetration volume and pressure curve; and, conversely, a given penetration volume and pressure curve will afford a unique determination of the pore size distribution.

The question of the correct contact angle between liquid mercury and solid is uncertain. Ritter and Drake (4) measured the contact angle of mercury on a wide variety of substances and found that it varied between 135° and 142°. They selected 140° as a reasonable average. They also quoted that these values may be a little high, because of the method of measurement they used. It was confirmed that nickel has a wetting angle of 130°, which is considered the most acceptable value for a wide range of materials, and was used by American Instrument Company in the above equation. Since the effect of this discrepancy in the interpretation of the data has been relatively small, the contact angle of 130° has been selected for simplicity. Substituting 473 dynes per centimeter for γ_{LV}, the equation for equivalent cyclindrical pores reduces the empirical approximation:

$$D = \frac{175}{P} \tag{4}$$

where D is obtained in microns if P, the absolute pressure, is expressed in pound force per square inch.

Eq. 4 gives the smallest pore diameter entered by the mercury under pressure. If large openings within the sample are connected to the surface by narrow pores, i.e., an ink bottle pore, the pore volume will be indicated at the diameter of the narrow pores.

EXPERIMENTAL PROCEDURE

The instrument used for the experimental work was the 5,000 p.s.i. hand operated model Aminco-Winslow porosimeter of the American Instrument Company. The separators used were commercially available; they were (1) Evans STD paper, (2) Evans N-P paper, (3) Dewey-Almy Armor Coated paper, (4) Texon paper, (5) Evans PVC, (6) Porvic II.

Mercury Penetration

A sample of appropriate size was weighed on a balance, placed in a penetrometer and then, in turn, placed stem down in a filling device, which was evacuated to a pressure of 50 microns or less by an oil pump. The penetrometer was next filled with mercury. The filling device was tilted until the tip of the penetrometer immersed in the mercury pool, and the stopcock of the filling was then opened slowly until the 0-15 p.s.i. gauge registered 6.8 p.s.i. Pore sizes of seventeen microns or larger were determined in suitable increments with the penetrometer in a vertical position.

For subsequent determinations, the penetrometer was then transferred to the pressure chamber in which a pressure from 0 to 5,000 p.s.i.g. can be applied, hence, pore sizes down to 0.035 microns can be measured. As the pressure was raised, mercury was forced into smaller and smaller pores, and the volume of the pores was continuously indicated by the mercury as the level dropped in the stem of the penetrometer. The graduated stem of the penetrometer was visible at all times through a window in the pressure chamber.

Electrolyte Penetration

When electrolyte penetration was desired, the sample was boiled for 30 minutes in a boiling electrolytic solution after its weight has been noted. This boiling procedure is intended to force the electrolyte to penetrate into the voids of the sample. The concentration of the electrolytes studied was 2N, which is approximately the concentration of the electrolyte in a storage battery.

After the boiling, the sample was left immersed in the electrolyte, and subsequently cooled down to the room temperature. This procedure was very important because it allowed more solution to penetrate into the pore spaces of the sample and gave a stable penetration volume reading. The wet sample was next placed into the penetrometer which was, in turn, placed in the filling device. The pressure in the filling device was then pumped down to the vapor pressure of the electrolyte. This could be done by evacuating the filling device in approximately 15 seconds, then close the vacuum toggle valve and let the electrolyte vaporize. One must exercise great care not to pump too much below the solution vapor pressure for all the penetrated electrolyte would vaporize if the pressure was much below the vapor pressure. Then the experimental procedure following became the same as for mercury penetration.

Although almost all the pore space was filled by the electrolyte, there were still some pores remaining which were subject to mercury penetration. Since the surface tension of each electrolyte is different, the degree of penetration or the amount of electrolyte which had penetrated into the voids of the sample was different. The pore size distribution curve obtained was not the real pore size distribution of the sample, it was only the distribution of those pores unfilled by the electrolyte.

EXPERIMENTAL RESULTS

During the experiment, the penetration volumes are read directly from the penetrometer stem. The smallest pore diameter entered by the mercury under pressure is given in Eq. 4. The pressure, P, in that equation is the absolute pressure on the sample; therefore, the head of mercury must be included in calculating absolute pressure for measurement of diameters of larger pores. Table 1 gives the head of mercury.

Table 1: Head of Mercury

Penetrometer Reading	Hg. Head Pressure in PSI
0.00	4.53
0.01	4.40
0.02	4.25
0.03	4.15
0.04	4.00
0.05	3.85
0.06	3.75
0.07	3.60
0.08	3.45
0.09	3.30
0.10	3.15

Absolute pressure at the sample is equal to the reading of the 0-15 PSI gauge minus mercury head pressure plus the reading on the pressure gauge.

In the process involving the diffusion rates and the availability of internal surfaces, a knowledge of total pore volume is less important than a knowledge of the fraction of total pore volume contributed by pores in a given size range. Using the penetration volume at 5,000 p.s.i. as the total pore volume of the sample, the fraction of total pore volume contributed by the pores of a given pore diameter is simply the penetration volume corresponding to that pore diameter divided by the total pore volume of the sample. Sample calculation is shown in Fig. 1. Figures 2-4 give the pore size distribution curves of the separators studied.

Figures 5-8 are the pore size distribution curves of the separators by electrolyte penetration. Since the surface tensions of the electrolytes are different, the penetration curves are different. A comparison of the effects of the electrolytes on penetration are shown in Figures 8-14.

Hg. Head Pressure (psi)	0-15 Gauge Reading (psi)	Pressure Gauge Reading (psi)	Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (microns)	Volume (%)
4.53	6.8	0	2.27	0	77.00	0
4.15	9.55	0	5.40	0.037	32.41	60.66
4.00	12.50	0	8.50	0.042	20.59	68.85
4.00	14.70	0	10.70	0.044	16.36	72.13
4.00	0	10	20.70	0.048	8.454	78.69
4.00	0	100	110.70	0.049	1.581	80.33
3.85	0	300	310.85	0.050	0.563	81.97
3.85	0	600	610.85	0.051	0.286	83.61
3.85	0	1200	1200	0.053	0.146	86.89
3.85	0	2500	2500	0.056	0.070	91.80
3.85	0	3400	3400	0.058	0.052	95.08
3.75	0	4400	4400	0.060	0.040	98.36
3.75	0	5000	5000	0.061	0.035	100.0

Figure 1.

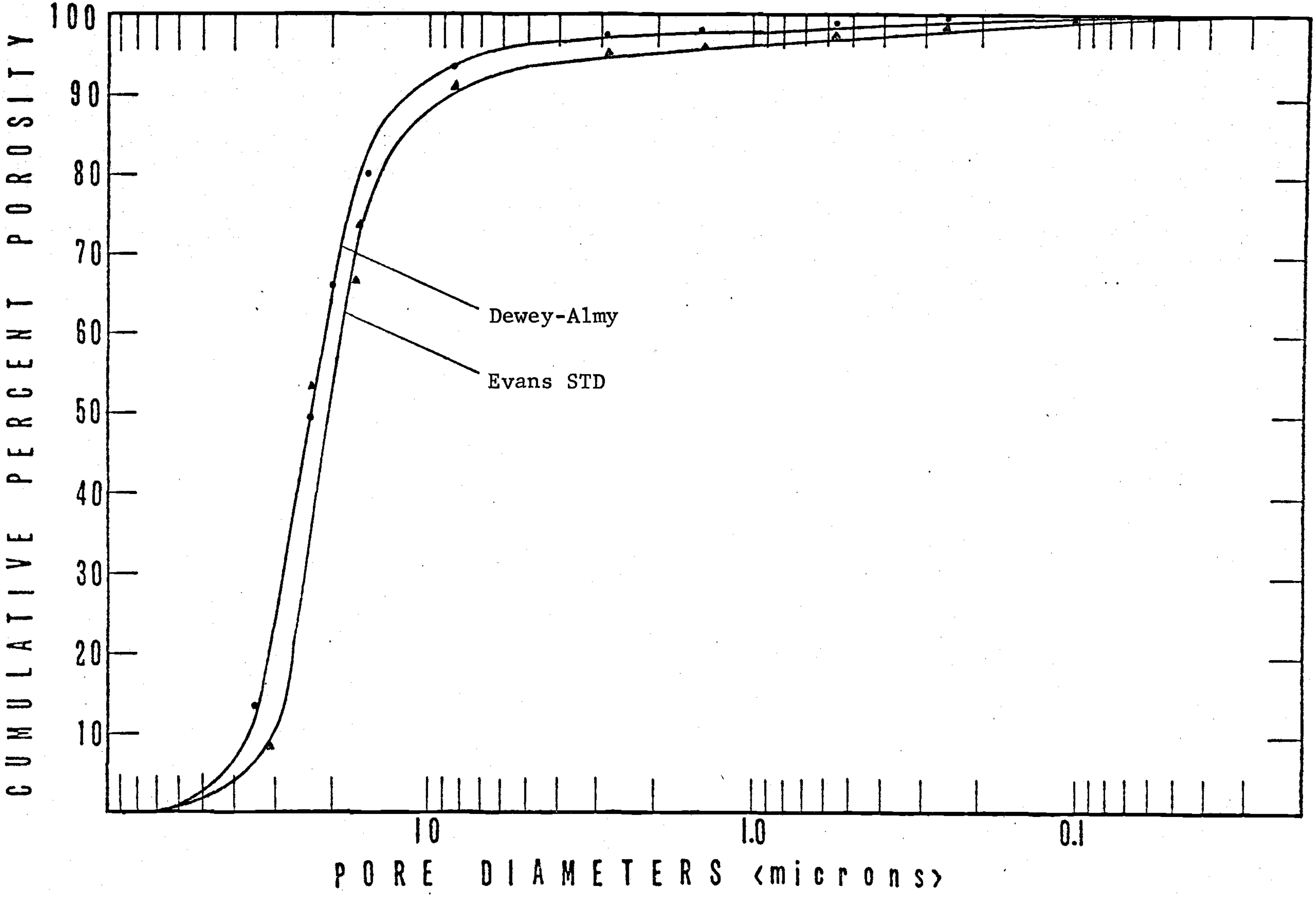

Figure 2. Pore size distribution curves of Evans STD and Dewey-Almy Armor coated paper.

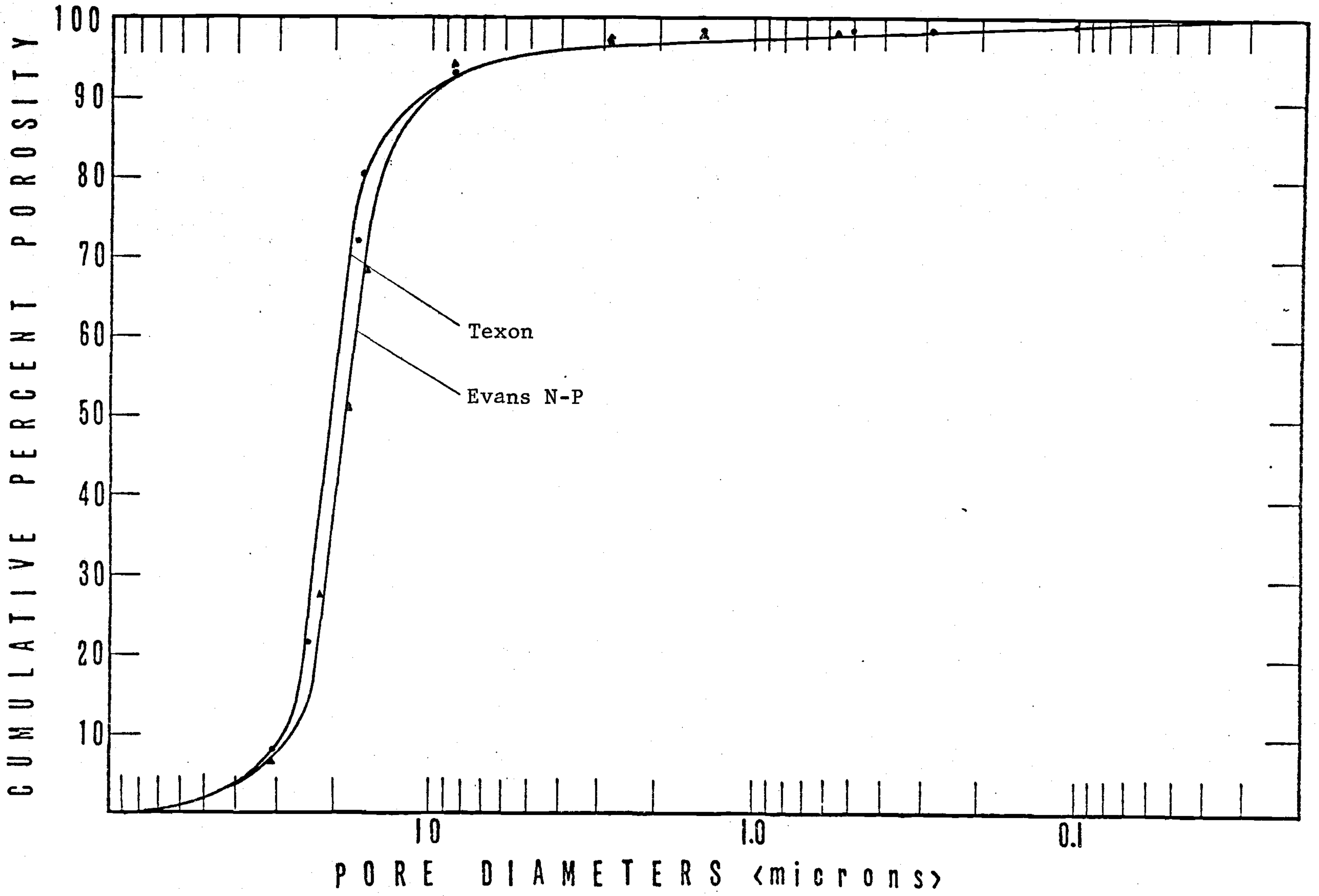

Figure 3. Pore size distribution curves of Texon and Evans N-P.

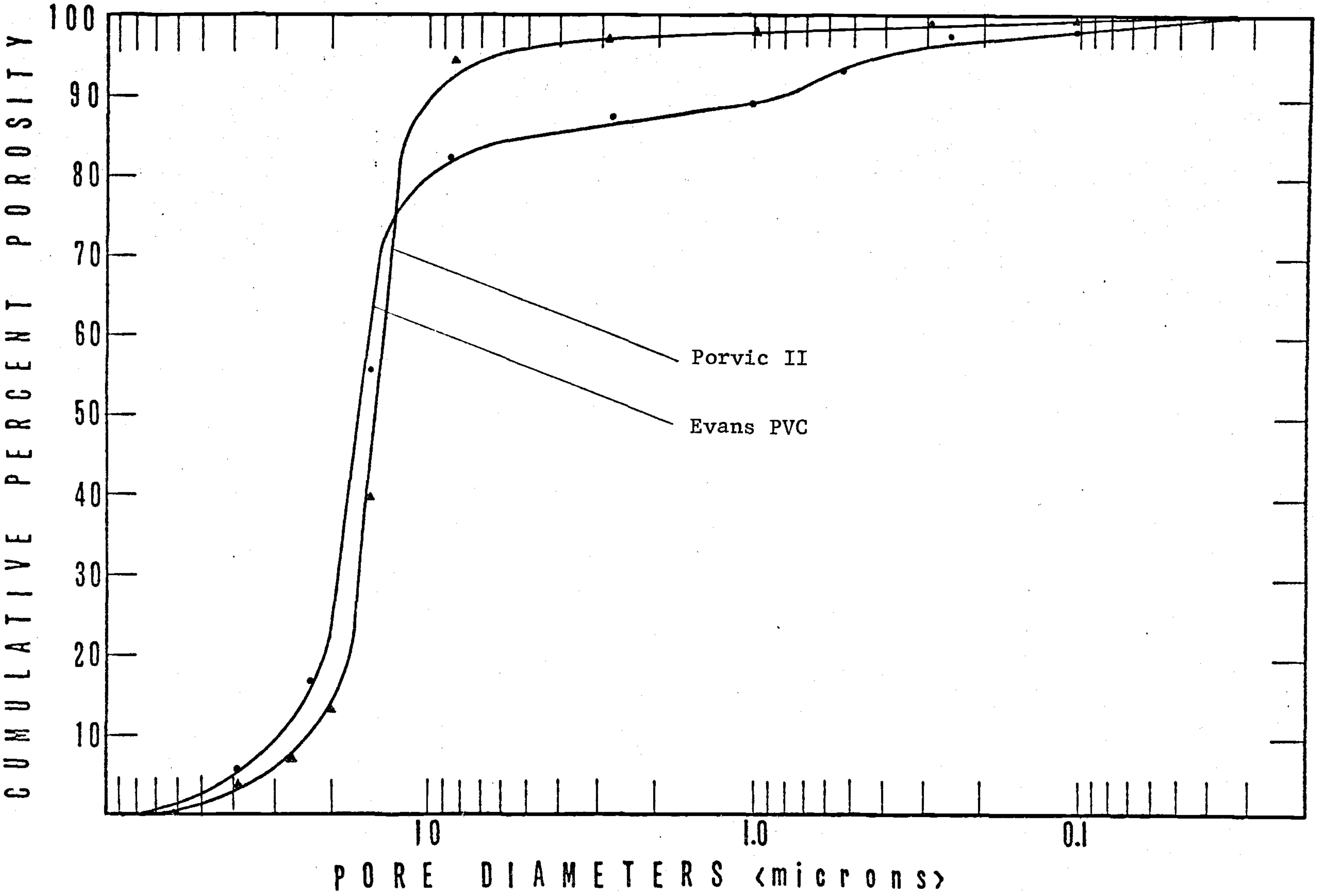

Figure 4. Pore size distribution curves of Porvic II and Evans PVC.

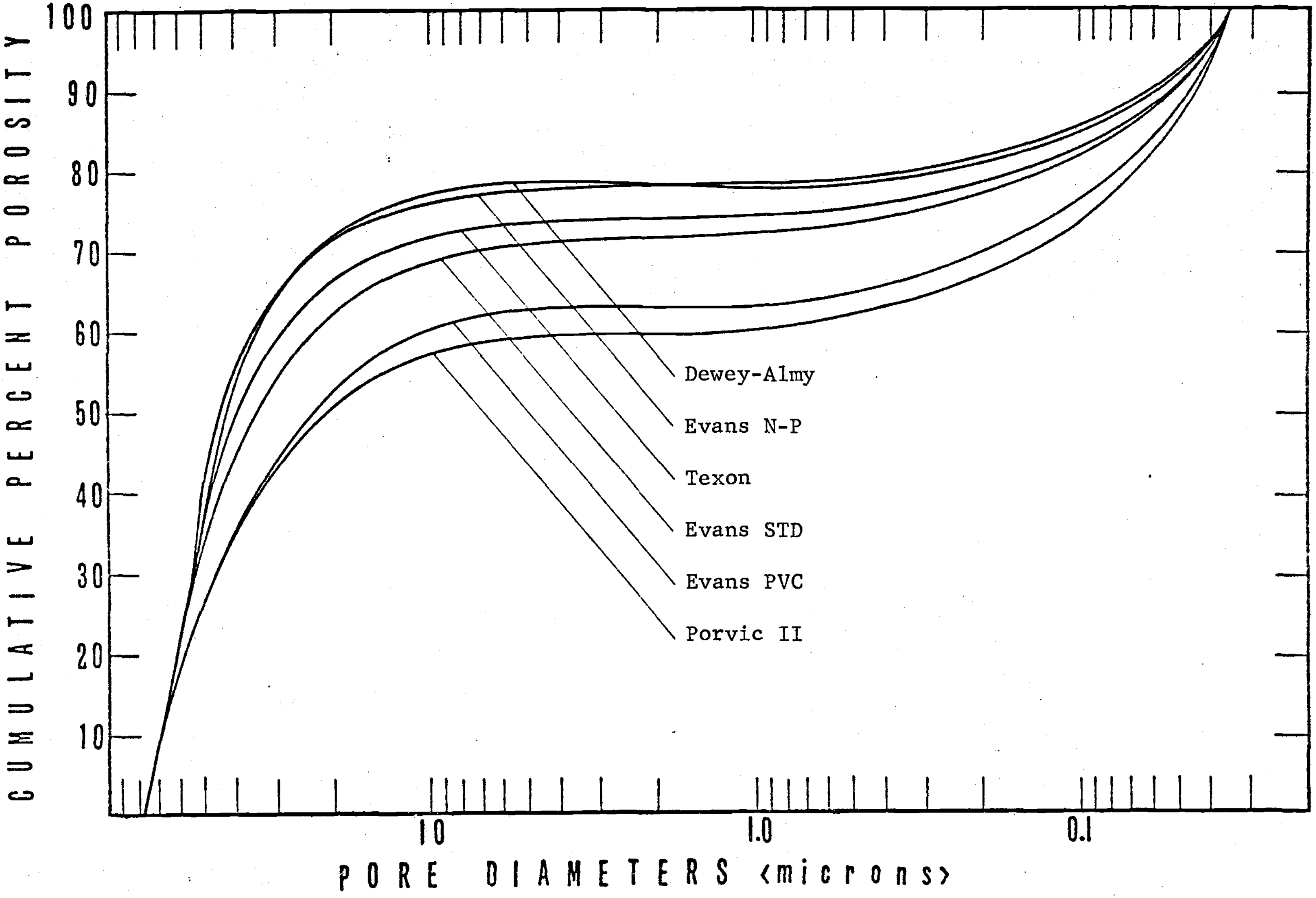

Figure 5. Electrolyte $Zn(NO_3)_2$ penetration curves of the separators.

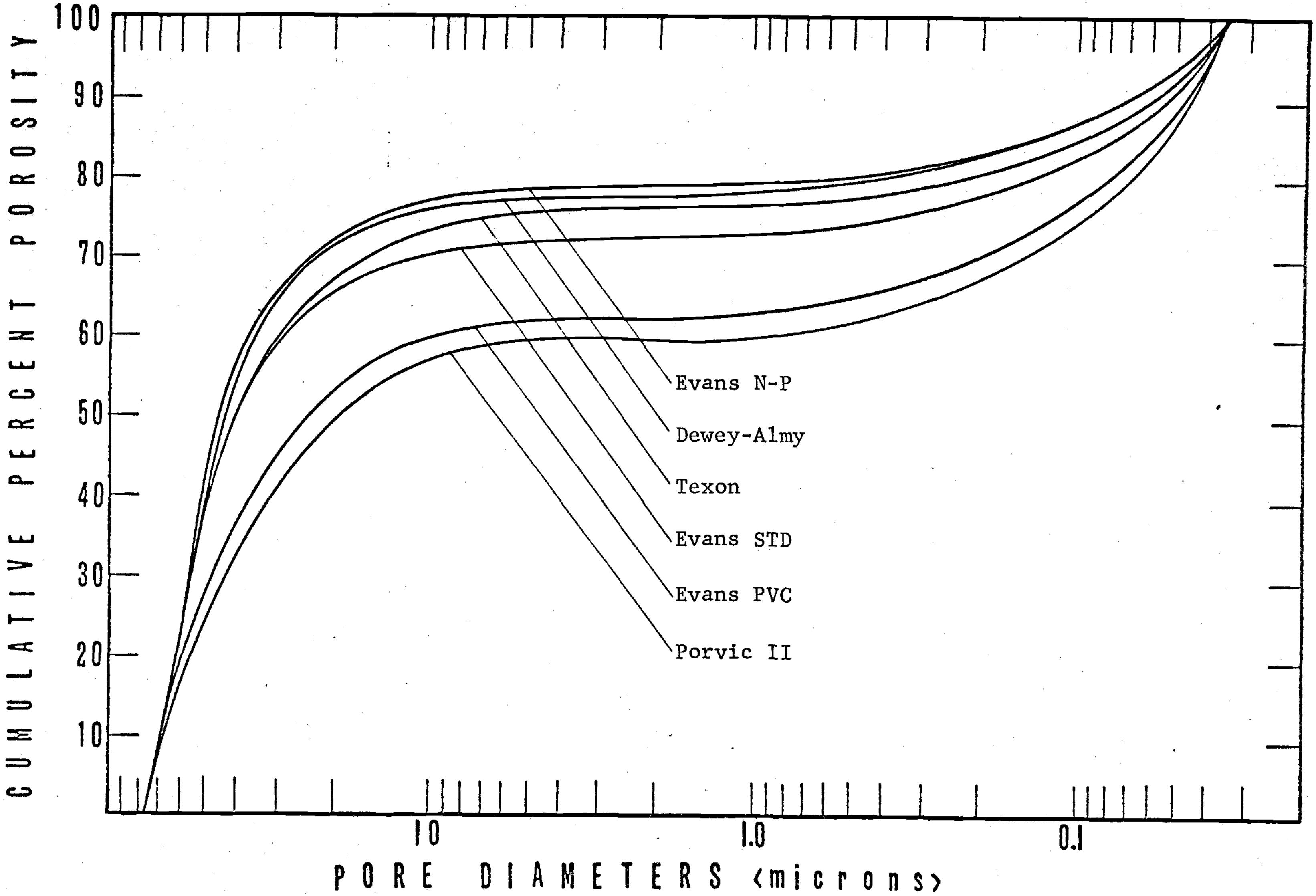

Figure 6. Electrolyte NH_4NO_3 penetration curves of the spearators.

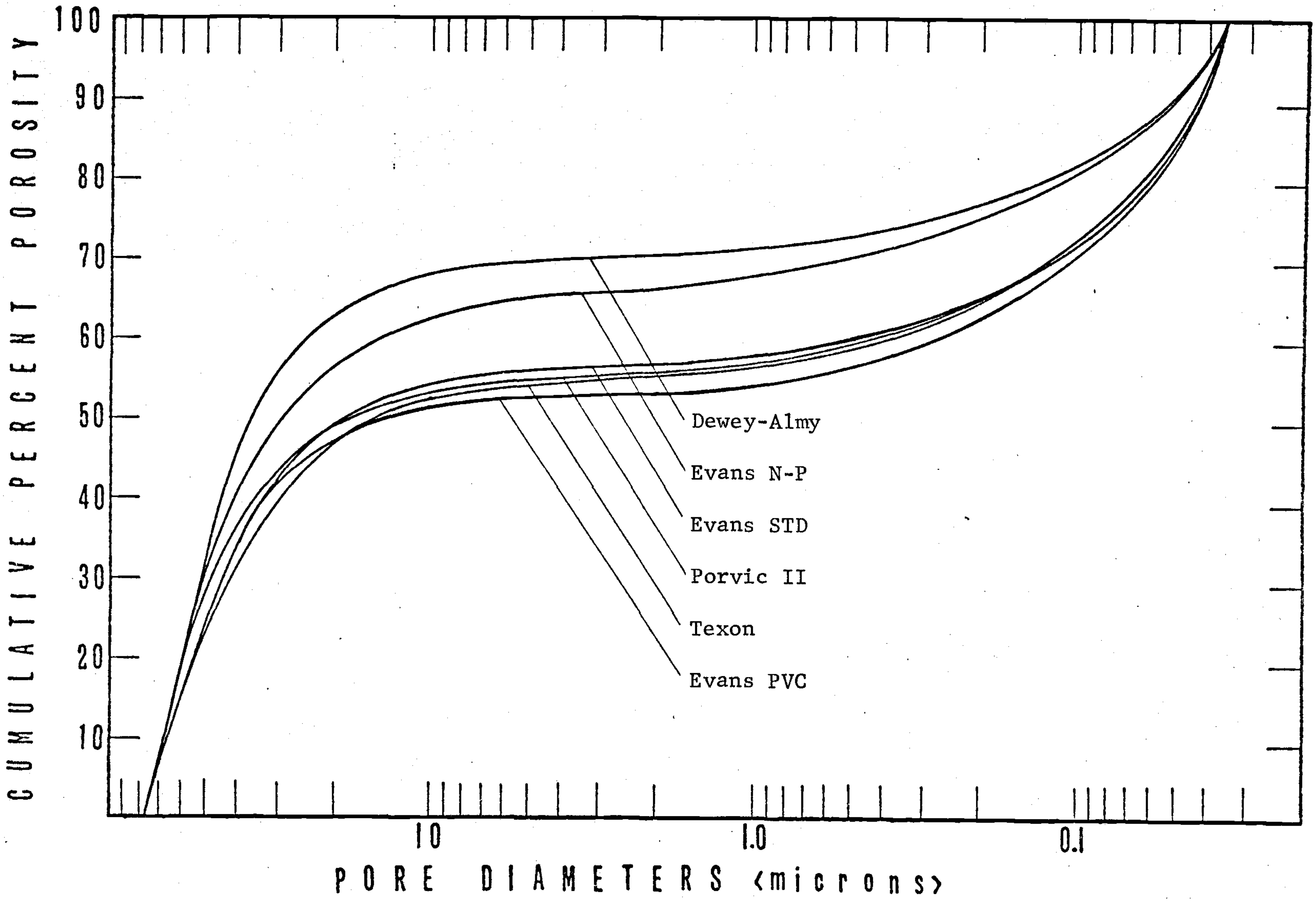

Figure 7. Electrolyte $(NH_4)_2SO_4$ penetration curves of the separators.

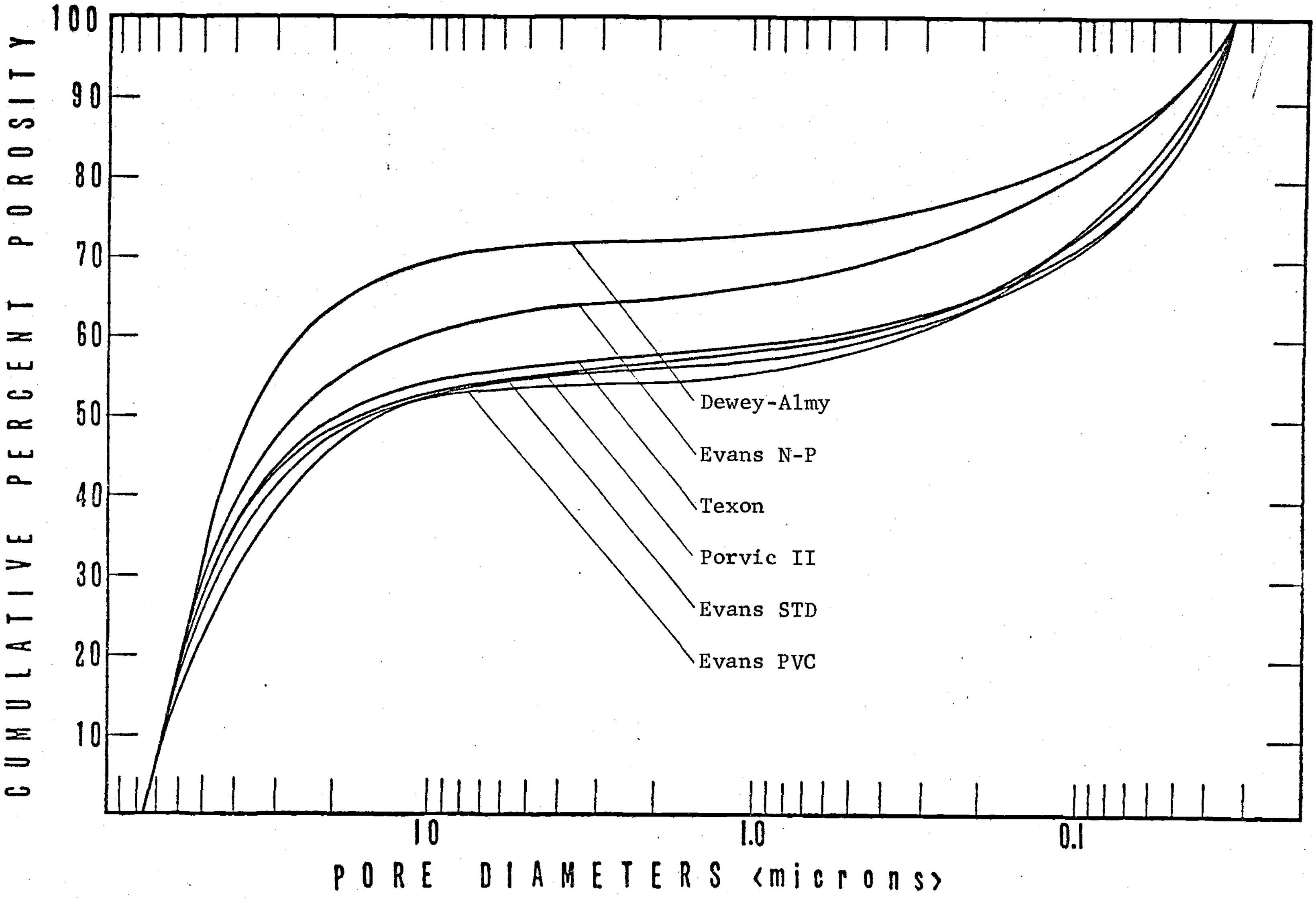

Figure 8. Electrolyte $ZnSO_4$ penetration curves of the separators.

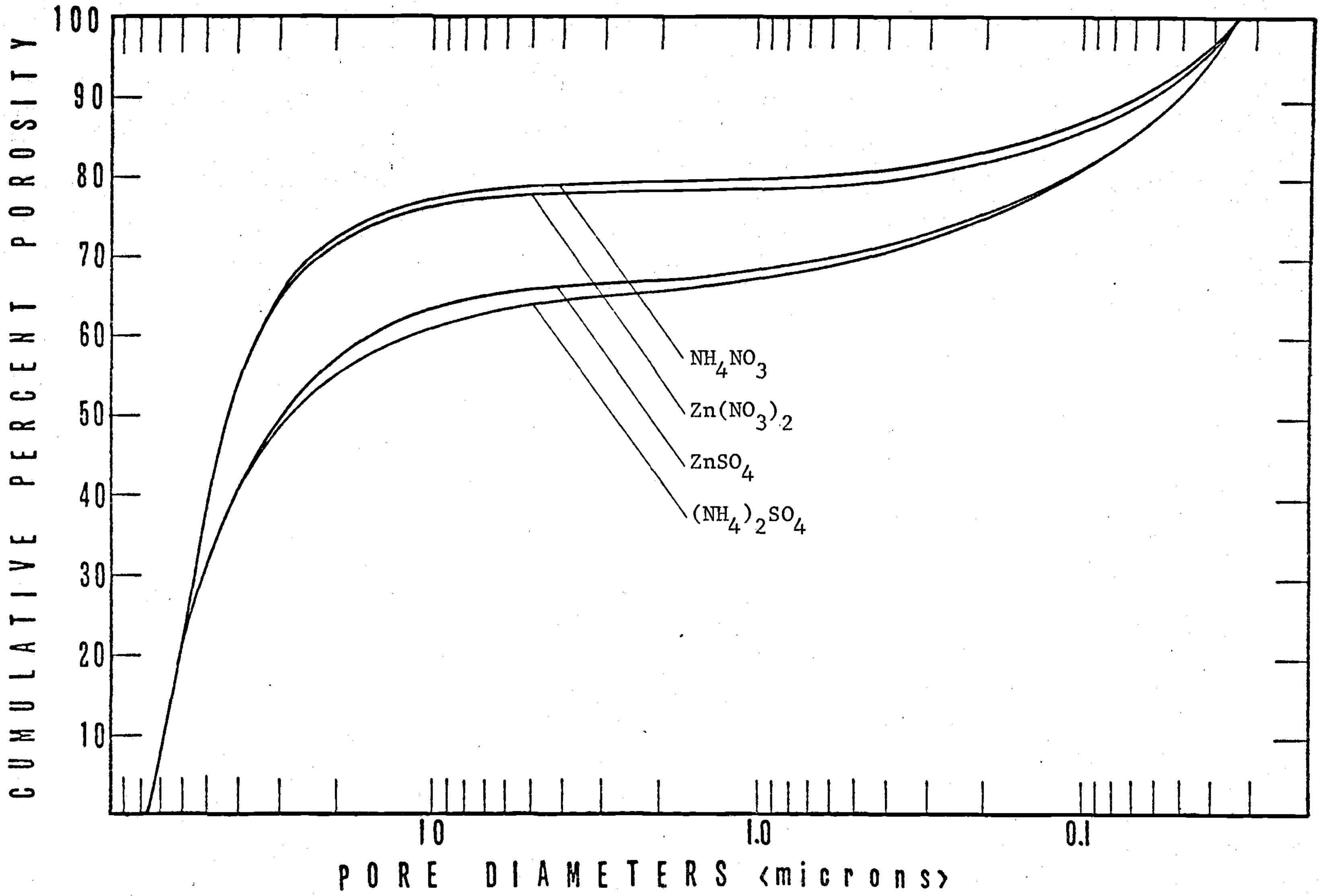

Figure 9. Comparison of the effects of the electrolytes on penetration. (Sample: Texon)

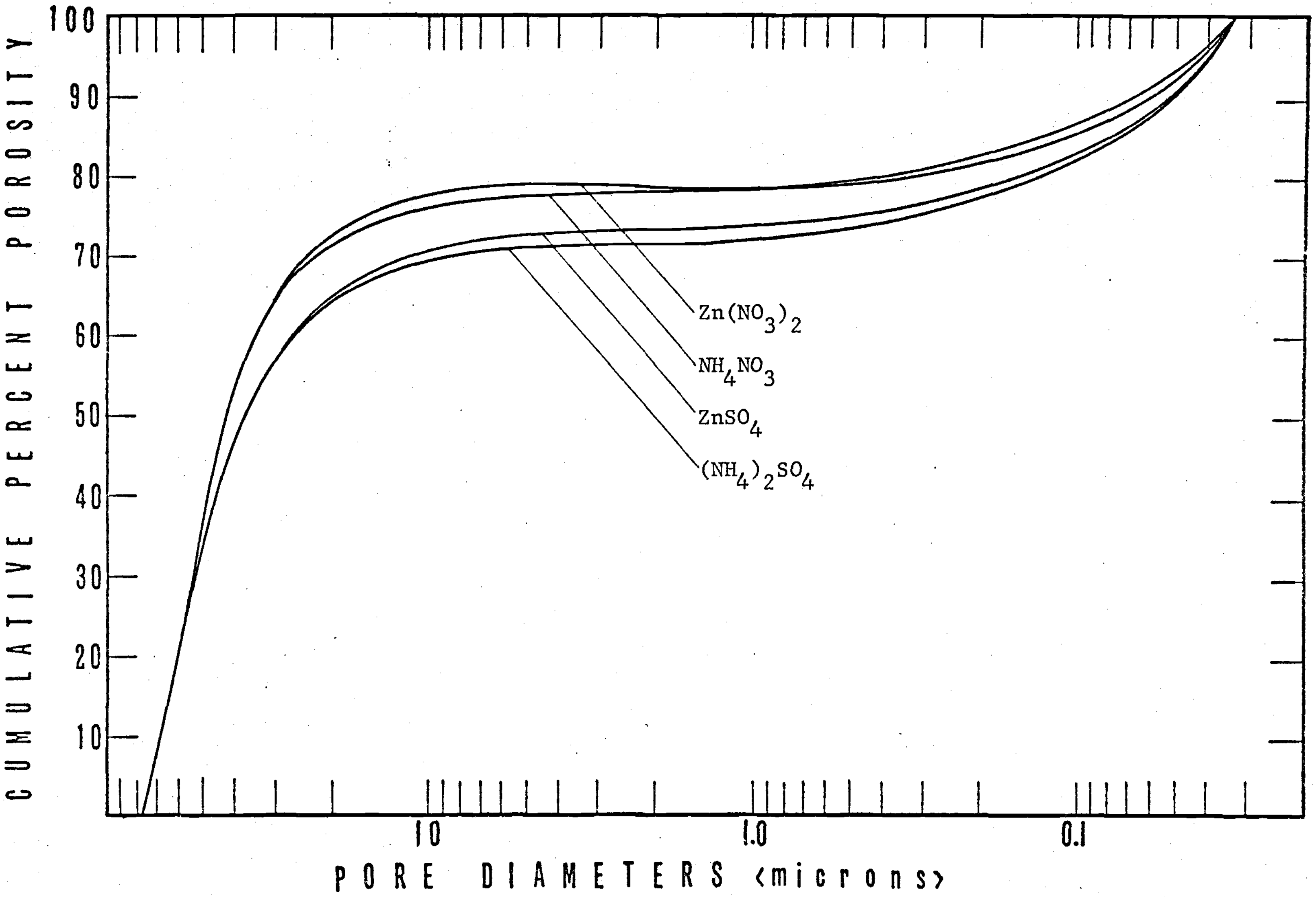

Figure 10. Comparison of the effects of the electrolytes on penetration. (Sample: Dewey-Almy)

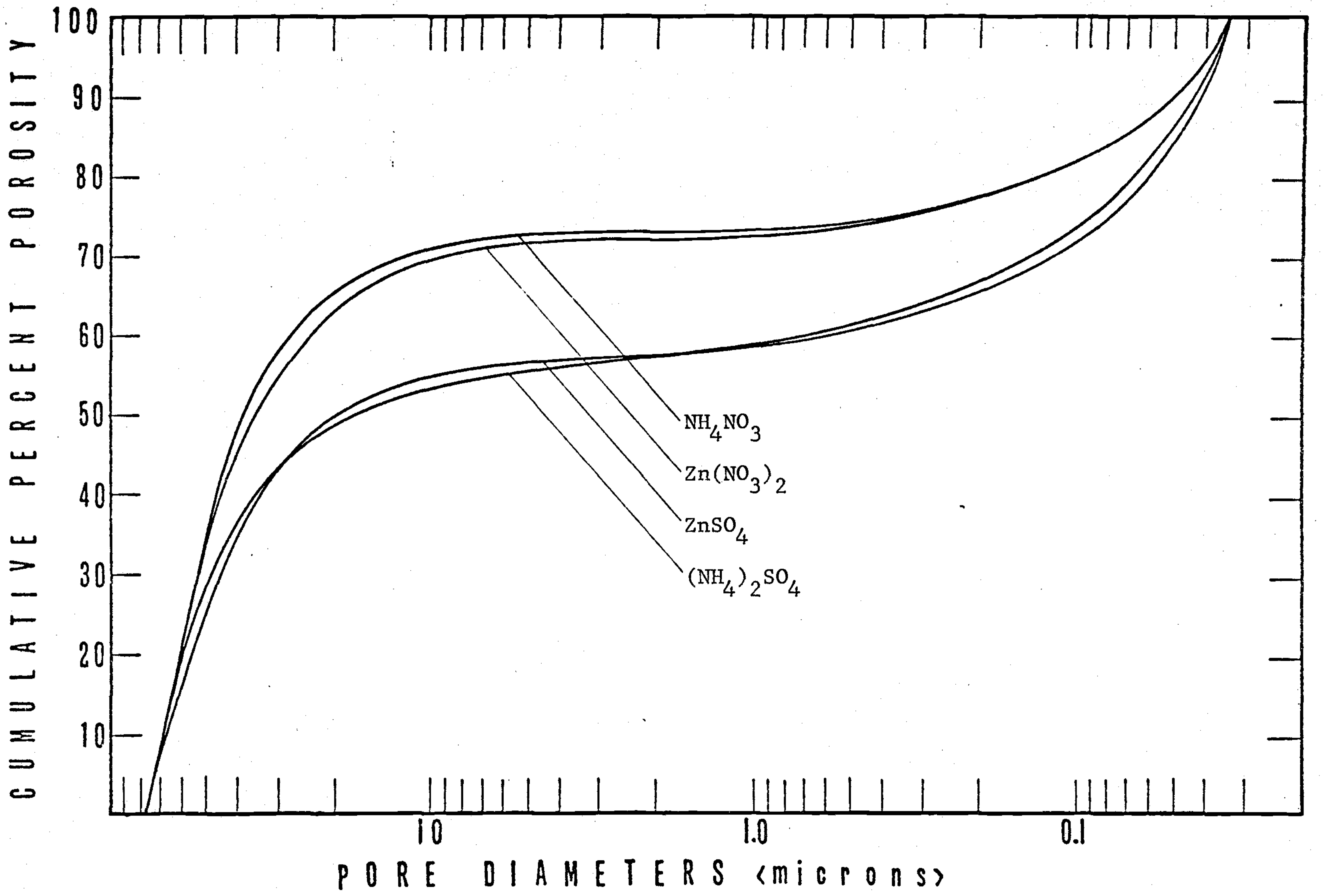

Figure 11. Comparison of the effects of the electrolytes on penetration. (Sample: Evans N-P)

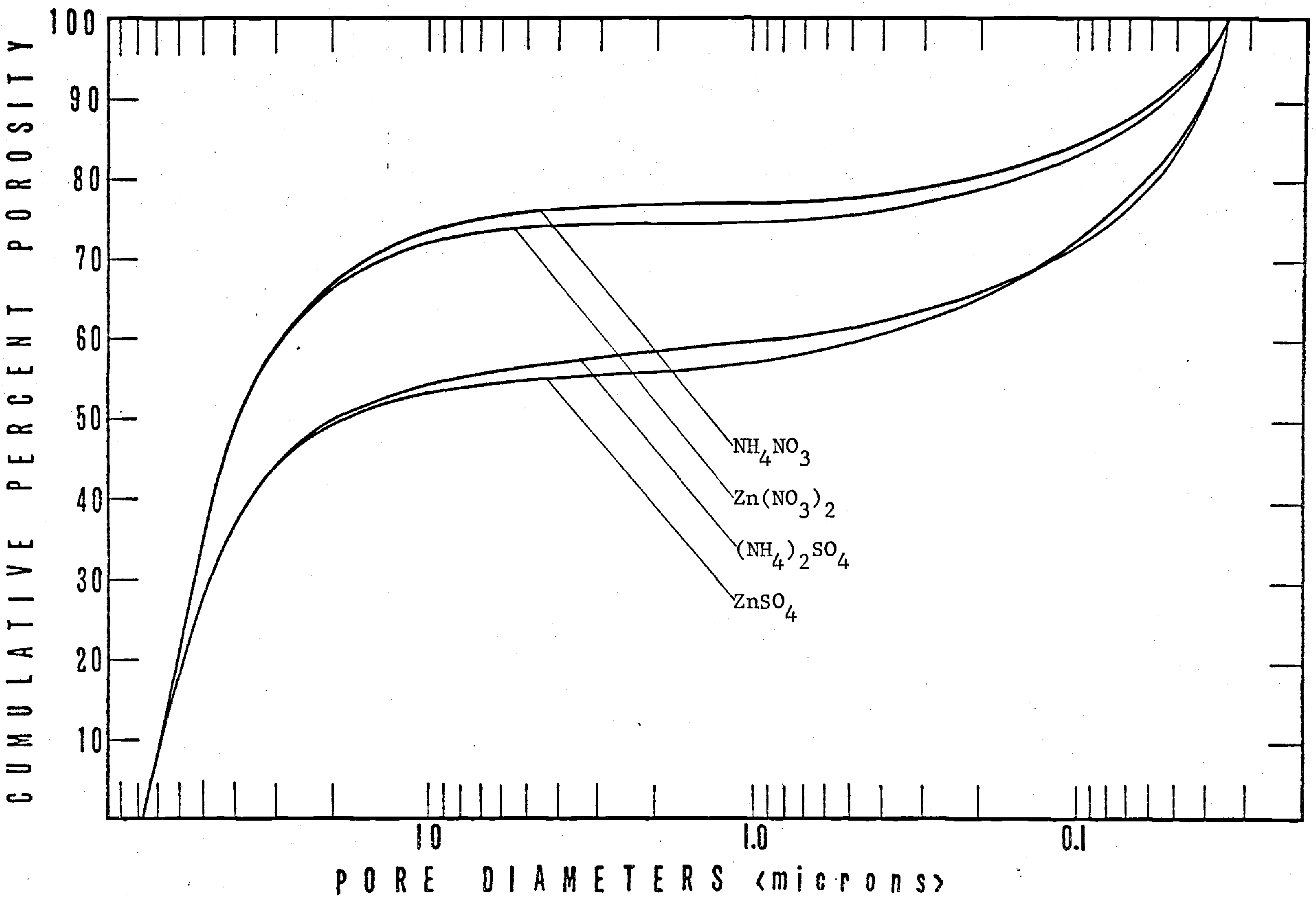

Figure 12. Comparison of the effects of the electrolytes on penetration. (Sample: Evans STD)

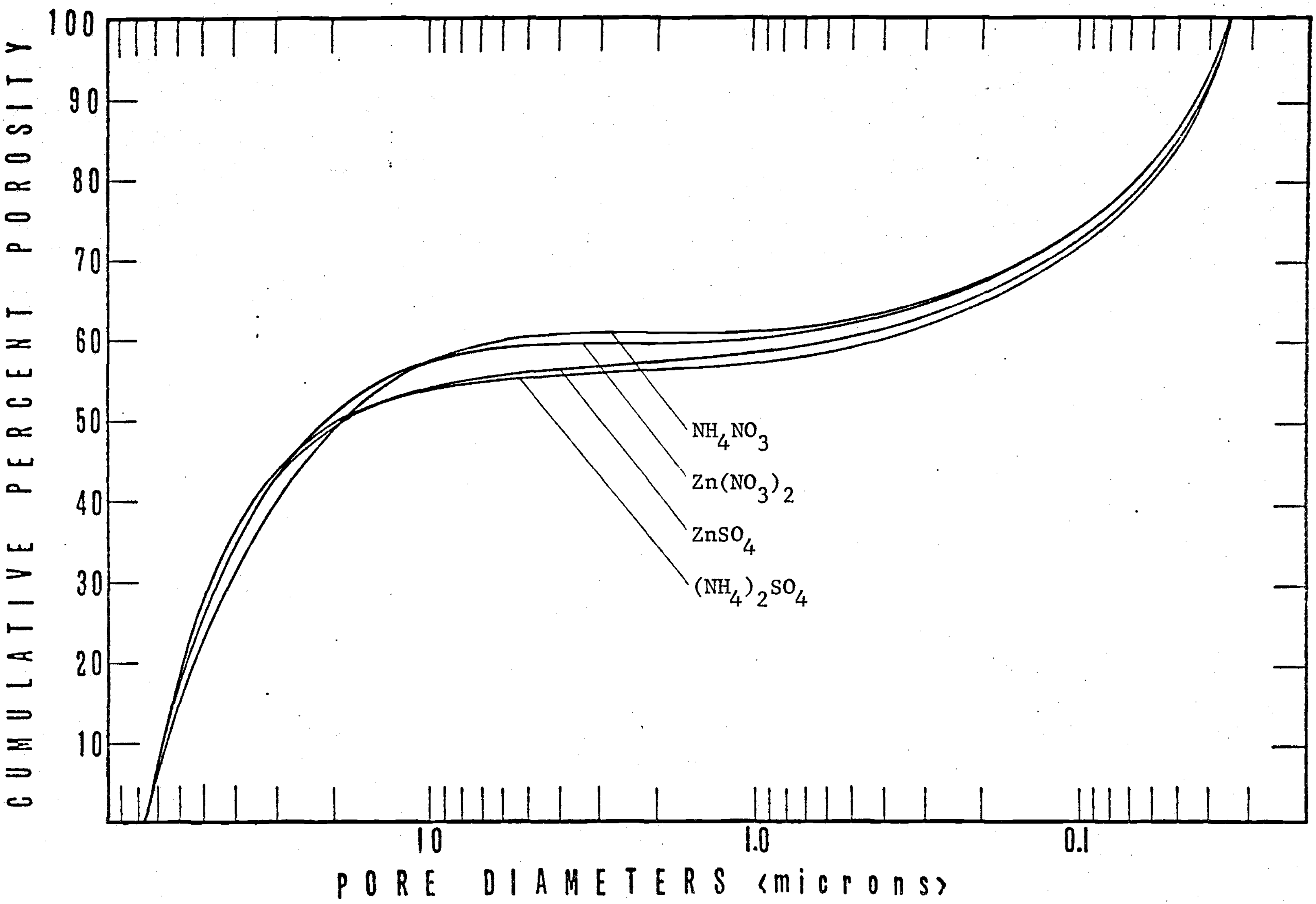

Figure 13. Comparison of the effects of the electrolytes on penetration. (Sample: Porvic II)

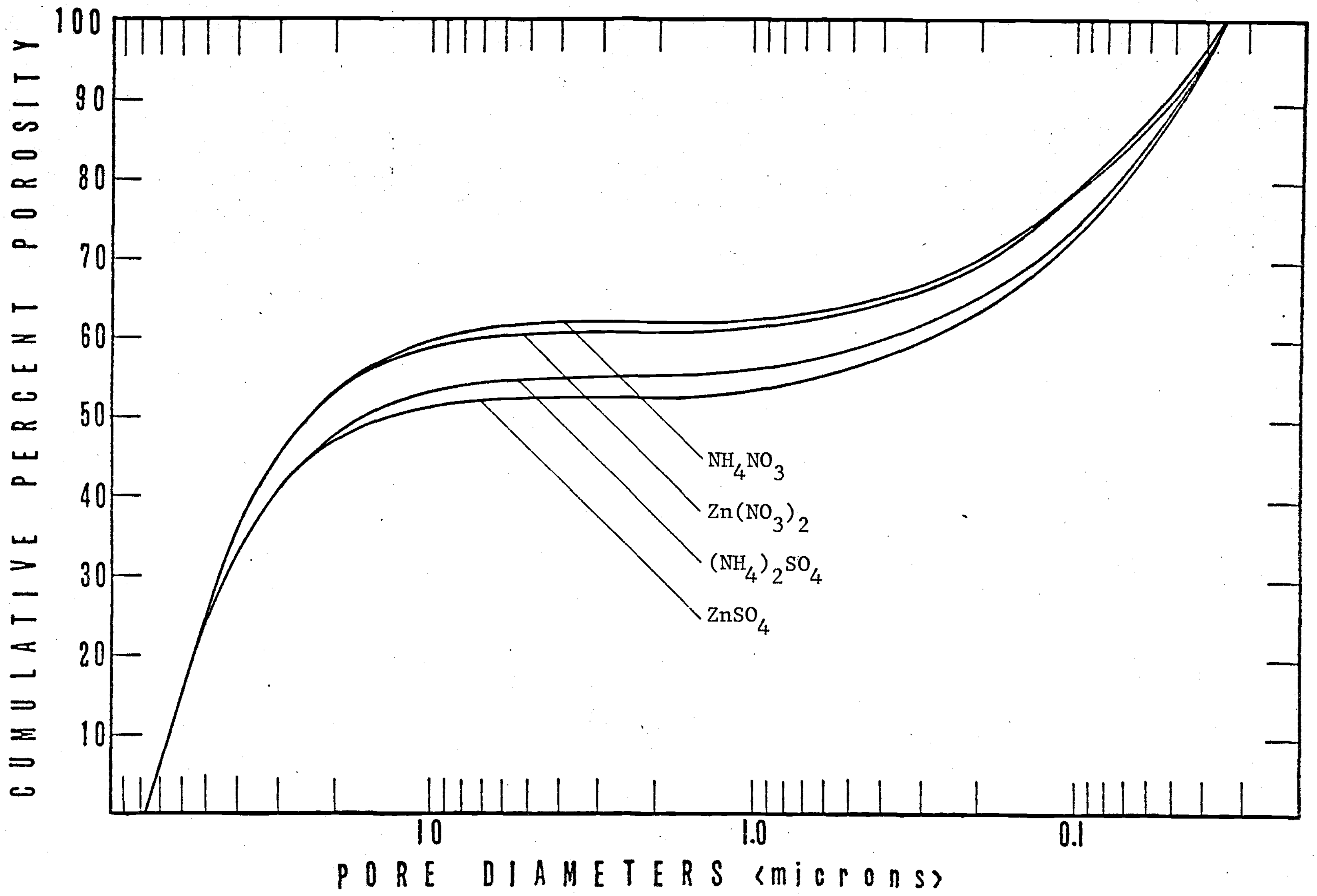

Figure 14. Comparison of the effects of the electrolytes on penetration. (Sample: Evans PVC)

DISCUSSION OF RESULTS

To select a suitable separator for a certain kind of storage battery, several characteristic factors must be considered. Some of these are the total porosity, the average pore size of the separator and the separator's resistance to electrolytic conduction. All of the separators used in this study are porous to the extent of 65% to 77% by volume (Table 2).

Table 2: Total Porosity of Separators

Sample	Real Density (gm/cc)	Apparent Density (gm/cc)	Total Porosity (%)
Evans STD	1.44	0.342	76.7
Evans N-P	1.50	0.460	67.5
Dewey-Almy	1.46	0.400	72.6
Texon	1.42	0.375	70.5
Evans PVC	2.372	0.800	66.4
Porvic II	2.364	0.815	65.4

Figures 2-4 show the pore size distribution of the separators. They indicate the pore sizes of Dewey-Almy Armor Coated paper are the largest for the sample having a mean pore diameter of twenty-four microns. The average pore diameter of Porvic II plastic is fifteen microns, which is the smallest among the studied separators. The ability of smaller pores to oppose the intrusion of the scaling of active materials on the negative plates is better than for large pores.

The resistance of electrolytic conduction of a separator is affected by the materials of which the separator is made, the treatment it receives, and by the extent of electrolytic penetration, i.e., the amount of electrolytes held in the pore spaces of the separator. Figures 9-13 compare the amount of penetration of different electrolytes into the separators. Among the studied electrolytes, these figures reveal that for two electrolytes with the same cations but different anions, there are differences in the degree of penetration. However, for electrolytes of the same anions but different cations, the penetration curves almost coincide. Therefore, the penetration depends solely on the anions and is negligible with the cations. These figures also indicate that the penetration curves of the electrolytes containing sulfate ions are lower than the curves of electrolytes containing nitrate ions. From Table 3, we know the percentage of the total pore volume filled by electrolytes containing sulfate is larger than that for the nitrate ions. Therefore, a lower position of the curves in the proceeding figures means more electrolyte has penetrated into the voids of the separator. For this reason, sulfate ions can enter the pores more readily than nitrate ions; thus one might conclude that the resistance of separators in electrolytes containing sulfate ions would be less than that in electrolytes containing nitrate ions; considering all else equal.

Table 3: Extent of Electrolytic Penetration

Sample	Void Volume (c.c./gm)	% filled up by $(NH_4)_2SO_4$	% filled up by $ZnSO_4$	% filled up by NH_4NO_3	% filled up by $Zn(NO_3)_2$
Evans STD	1.313	93.14	92.50	89.30	90.50
Evans N-P	1.113	93.70	94.17	89.50	89.70
Dewey-Almy	1.357	94.30	94.55	91.70	92.30
Texon	1.692	96.30	96.10	92.80	93.60
Evans PVC	0.345	92.60	94.02	91.90	90.40
Porvic II	0.448	94.10	93.20	91.70	92.10

Figures 5-8 show the performances of the separators in electrolytes. They indicate that the penetration curves of Dewey-Almy Armor Coated paper, Evans STD, Evans N-P, and Texon in NH_4NO_3 and $Zn(NO_3)$ solutions are close to each other, but the curves of Evans PVC and Porvic II are much lower than that of the above four. However, when they are in $(NH_4)_2SO_4$ and $ZnSO_4$ solutions, the penetration curves of Dewey-Almy and Evans N-P are much higher than those of Evans STD, Texon, Evans PVC, and Porvic II. In general, the position of the curves is affected by the pore size distribution of the separator and the degree of penetration by electrolyte. Referring to Table 3 and the curves, when the percentages of total pore volume filled up by the electrolyte are equal, the position of the curves depends upon the total pore volume remaining unpenetrated. When a separator is subjected to electrolyte penetration, all the pores will be filled if their pore diameters are greater than the diameter in Eq. 3. Only those pores having diameters smaller than given by Eq. 3 remain for mercury penetration. The total volume of mercury, which penetrates into the remaining pore spaces, is proportional to the remaining void volume. If we let V_o equal to the total volume of mercury that penetrated into the remaining pores and V equal to the penetration volume at a certain pressure, then the ratio of V/V_o is smaller for a separator with high distribution in smaller pores, and thus, the position of the penetration curve is lower than that of the separator with high distribution in larger pores.

When the percentage of total pore volume filled by the electrolytes is not equal, the position of the penetration curves depend on both pore sizes and the degree of penetration by electrolytic solution. If the pore sizes are nearly equal, then the position of curves are inversely proportional to the degree of penetration. However, if the pore sizes vary a great deal, then either one of the above factors could dominate.

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APPENDIX

APPENDIX

1. Description of some terms.

a) Displacement volume.

The displacement volume of a sample is defined as the volume enclosed by the outside surface of the sample minus the volume of those pores penetrated by mercury at a hydrostatic pressure of 1.8 p.s.i.a. It can be calculated by the following equation:

$$V_s = V_p - \frac{W-(W_p + W_s)}{Hg}$$

where V_s: the displacement of the sample.

V_p: the internal volume of the empty penetrometer.

W_p: weight of the empty penetrometer.

W_s: weight of penetrometer with sample placed in it.

W : weight of the penetrometer with sample placed in it and filled with mercury.

Hg: density of mercury.

b) Apparent density.

Apparent density of a sample is its mass divided by the volume enclosed by the outside surface of the sample. It may be measured, for example, by cutting a cube of material and dividing the measured mass of the cube by the length of one edge raised to the 3rd power.

c) Real density.

Real density of the sample is its mass divided by the volume occupied by the solid material only.

d) Total porosity.

Total porosity of a sample is the fraction of total volume of the sample occupied by the voids of the sample.

2. An example of total porosity determination.

1. Sample Dewey-Almy
2. Wt. of penetrometer, empty g. 68.7755
3. Wt. of penetrometer, filled with mercury, g. 159.7517
4. Wt. of sample, g. 0.127
5. Wt. of penetrometer with sample and filled with mercury, g. 156.2082
6. Displacement volume of sample (3 plus 4 minus 5)/13.55, c.c. 0.271 at absolute pressure of, psi 1.8
7. Apparent density g./cc. 0.400
8. Real density, g./cc. 1.46
9. Apparent volume of sample, (4/7), cc. 0.3175
10. Real volume of sample, (4/8), cc. 0.087
11. Total pore volume of sample, (9 minus 10), cc. 0.2305
12. Total porosity of sample, 100x11/9, % 72.6

III. Selected Experimental Data

A. Mercury Penetration

1) Sample: Porvic II. Sample Weight: 0.240 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
4.47	0.004	39.2	3.39
6.47	0.008	27.1	6.78
8.60	0.016	20.3	13.6
10.70	0.047	16.35	40.2
20.55	0.1105	8.52	94.2
60.55	0.114	2.89	97.1
180.55	0.115	0.97	98
610.55	0.116	0.287	99
1600	0.116	0.1089	99
3200	0.1165	0.0546	99.5
5000	0.1173	0.035	100

2) Sample: Evans PVC. Sample Weight: 0.215 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
4.47	0.004	39.2	5.4
7.60	0.013	23.05	17.6
10.70	0.042	16.35	56.7
20.95	0.062	8.67	83.8
60.95	0.0642	2.87	86.7
160.95	0.066	1.085	89.2
311.10	0.070	0.562	94.6
600	0.072	0.287	97.2
1600	0.073	0.1089	98.6
3200	0.0735	0.0546	99.3
5000	0.074	0.035	100

3) Sample: Texon. Sample Weight: 0.117 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.60	0.016	31.3	8.08
7.10	0.083	24.7	41.9
9.85	0.14	17.25	70.7
10.55	0.158	16.6	80
20.55	0.185	8.52	93.5
60.55	0.192	2.89	97
110.55	0.195	1.58	98.5
310.55	0.196	0.564	99
600	0.1965	0.287	99.2
1600	0.197	0.1089	99.5
3200	0.197	0.0546	99.5
5000	0.198	0.035	100

4) Sample: Evans N-P. Sample Weight: 0.1275 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.60	0.01	31.3	7.05
7.50	0.04	23.3	28.2
9.30	0.074	18.8	52.2
10.55	0.10	16.6	70.3
20.55	0.134	8.52	94.4
60.55	0.1395	2.89	98.2
110.55	0.1395	1.58	98.2
310.55	0.14	0.564	98.5
600	0.14	0.287	98.5
1600	0.141	0.1089	99.2
3200	0.1415	0.0546	99.5
5000	0.142	0.035	100

5) Sample: Evans STD. Sample Weight: 0.113 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.60	0.012	31.2	8.08
7.55	0.082	23.2	55.2
9.85	0.101	17.75	68
10.55	0.112	16.6	75.5
20.55	0.136	8.52	91.5
60.55	0.142	2.89	95.6
110.55	0.1445	1.58	97.3
310.55	0.146	0.564	98.2
600	0.147	0.287	99
1600	0.148	0.1089	99.6
3200	0.148	0.0546	99.6
5000	0.1485	0.035	100

6) Sample: Dewey. Sample Weight: 0.140 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
4.75	0.028	36.9	14.75
7.20	0.097	24.3	51.1
8.85	0.128	19.8	67.4
10.55	0.152	16.6	80
20.55	0.178	8.52	93.7
60.55	0.185	2.89	97.5
110.55	0.187	1.58	98.5
300	0.1885	0.564	99
600	0.189	0.287	99.5
1600	0.190	1.095	100
3200	0.190	0.0545	100
5000	0.190	0.035	100

B. Electrolyte Penetration

1. $(NH_4)_2SO_4$

a) Sample: Dewey-Almy. Sample Weight: 0.518 gm

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.30	0.0235	33.02	55.95
8.30	0.026	22.1	61.90
10.45	0.027	16.75	64.29
20.55	0.030	8.516	71.43
110.55	0.0305	1.583	72.62
310.55	0.031	0.563	73.81
610.55	0.032	0.287	76.19
1210.55	0.034	0.1446	80.95
2000	0.036	0.0875	85.71
3100	0.038	0.0564	90.48
4100	0.040	0.0427	95.24
5000	0.042	0.035	100

b) Sample: Evans N-P. Sample Weight: 0.530 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.10	0.017	34.31	52.3
7.85	0.0185	22.29	56
10.45	0.0192	16.75	59.1
20.45	0.021	8.557	64.62
110.45	0.0215	1.584	66.15
310.45	0.0225	0.564	69.2
610.45	0.0235	0.287	72.31
1400	0.026	0.125	80
2400	0.028	0.0729	86.15
3400	0.030	0.0515	92.30
5000	0.0325	0.035	100

c) Sample: Porvic II. Sample Weight: 0.997 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.10	0.012	34.3	40.7
8.10	0.014	21.6	47.4
10.30	0.015	16.99	50.8
20.30	0.016	8.62	54.2
110.30	0.017	1.585	57.6
310.30	0.018	0.564	61
610.30	0.0185	0.287	62.7
1200	0.020	0.1458	67.8
2000	0.022	0.0875	74.6
3000	0.024	0.0583	81.4
5000	0.0295	0.035	100

d) Sample: Texon. Sample Weight: 0.456 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.20	0.013	33.65	44.8
7.90	0.0145	22.15	50
10.30	0.015	16.99	51.7
20.30	0.016	8.62	55.2
110.30	0.0165	1.584	56.85
310.30	0.017	0.565	58.7
610.30	0.018	0.287	62.1
1200	0.020	0.1458	69
2000	0.022	0.0875	75.8
3400	0.025	0.0515	86.2
5000	0.029	0.035	100

e) Sample: Evans STD. Sample Weight: 0.242 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.25	0.016	33.29	44.44
7.80	0.0178	22.44	49.44
10.30	0.018	16.99	50
20.45	0.020	8.56	55.56
110.45	0.021	1.584	58.33
310.45	0.021	0.564	58.33
610.45	0.0225	0.287	62.5
1200	0.0245	0.1458	68.06
2300	0.028	0.0761	77.78
3500	0.032	0.05	88.89
5000	0.036	0.035	100

f) Sample: Evans PVC. Sample Weight: 0.4525 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.15	0.010	33.98	37.04
7.85	0.012	22.29	44.44
10.30	0.0125	16.99	46.30
20.30	0.014	8.62	51.85
110.30	0.0145	1.585	53.70
310.30	0.0153	0.565	56.67
610.30	0.016	0.287	59.25
1300	0.018	0.1346	66.67
2300	0.020	0.0760	74.07
3100	0.022	0.0564	81.48
3900	0.024	0.449	88.89
5000	0.027	0.035	100

2. $ZnSO_4$

a) Sample: Evans N-P. Sample Weight: 0.536 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.15	0.012	33.98	42.1
7.95	0.145	22.01	50.88
10.30	0.015	16.99	52.63
20.30	0.0175	8.564	61.40
110.30	0.018	1.586	63.16
310.30	0.019	0.564	66.67
610.30	0.020	0.287	70.18
1400	0.022	0.125	77.19
2400	0.024	0.0729	84.21
3400	0.026	0.0515	91.23
5000	0.0285	0.035	100

b) Sample: Texon. Sample Weight: 0.445 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
6.02	0.0065	29.2	46.4
10.17	0.007	17.2	50
20.17	0.008	8.67	57.2
60.17	0.008	2.91	57.2
310.17	0.0083	0.564	59.3
610.17	0.0087	0.287	62.15
1400	0.010	0.125	71.4
3000	0.012	0.0585	85.7
4000	0.013	0.0437	92.8
5000	0.014	0.035	100

c) Sample: Dewey-Almy. Sample Weight: 0.285 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.10	0.0112	34.3	56
8.05	0.0132	21.75	65.5
10.30	0.0135	16.99	67.5
20.30	0.014	8.62	70
110.30	0.014	1.585	70
310.30	0.0143	0.564	71.5
610.30	0.015	0.287	75
1200	0.016	0.1458	80
2300	0.017	0.0762	85
3300	0.018	0.0531	90
5000	0.020	0.035	100

d) Sample: Texon. Sample Weight: 0.249 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.02	0.006	34.86	37.5
10.17	0.0065	17.21	40.63
20.17	0.009	8.676	56.25
110.17	0.009	1.588	56.25
310.17	0.0095	0.564	59.38
610.17	0.010	0.287	62.5
1400	0.011	0.125	68.75
2900	0.013	0.0603	81.25
3700	0.014	0.0473	87.5
5000	0.016	0.035	100

e) Sample: Evans PVC. Sample Weight: 0.937 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
4.85	0.008	36.31	42.11
7.67	0.009	22.82	47.37
10.30	0.010	16.99	52.63
30.30	0.010	5.78	52.63
310.30	0.0105	0.564	55.26
610.30	0.011	0.287	57.89
1210.30	0.012	0.145	63.16
2000	0.014	0.0875	73.68
3300	0.016	0.053	84.21
4200	0.018	0.0417	94.74
5000	0.019	0.035	100

f) Sample: Evans STD. Sample Weight: 0.192 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.07	0.0065	34.5	41.2
10.17	0.008	17.15	50.7
20.17	0.008	8.67	50.7
310.17	0.0085	0.564	53.8
610.17	0.009	0.287	56.9
1310.3	0.010	0.1345	63.3
2400	0.012	0.0673	76
3600	0.014	0.0487	88.6
5000	0.0158	0.035	100

3. $Zn(NO_3)_2$

a) Sample: Evans N-P. Sample Weight: 0.541 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.5	0.043	31.82	66.15
8.0	0.048	21.87	73.85
10.85	0.050	16.13	76.92
20.85	0.0505	8.393	77.69
110.85	0.051	1.578	78.46
310.85	0.0522	0.563	80.31
610.85	0.054	0.287	83.08
1410.85	0.056	0.124	86.15
2200	0.058	0.0795	89.23
3000	0.060	0.0583	92.31
3800	0.062	0.046	95.38
5000	0.065	0.035	100

b) Sample: Texon. Sample Weight: 0.445 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.30	0.033	32.95	62.26
7.85	0.036	22.31	67.92
10.55	0.038	16.59	71.70
30.7	0.040	5.70	75.47
110.7	0.040	1.581	75.47
310.7	0.0405	0.563	76.42
610.7	0.041	0.286	77.36
1210	0.043	0.145	81.13
2410	0.046	0.0726	86.80
3200	0.048	0.0547	90.57
4000	0.050	0.0437	94.34
5000	0.053	0.035	100

c) Sample: Dewey. Sample Weight: 0.565 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.30	0.032	32.95	59.26
7.85	0.039	22.31	72.22
10.70	0.0405	16.30	75
20.70	0.042	8.45	77.78
110.70	0.042	1.581	77.78
610.70	0.044	0.286	81.48
1210.70	0.0455	0.145	84.26
2200	0.048	0.795	88.89
3000	0.050	0.0583	92.6
4000	0.052	0.0437	96.3
5000	0.054	0.035	100

d) Sample: Porvic II. Sample Weight: 0.902 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
4.90	0.012	35.72	41.38
7.25	0.014	24.17	48.28
10.30	0.0145	16.99	50.00
20.30	0.017	8.621	58.62
110.30	0.018	1.587	62.07
310.30	0.018	0.564	62.07
610.30	0.019	0.287	65.52
1400	0.021	0.125	72.41
2000	0.022	0.0875	75.86
2800	0.024	0.0625	82.76
3600	0.026	0.0486	89.66
5000	0.029	0.035	100

e) Sample: Evans STD. Sample Weight: 0.1985 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
6.1	0.014	28.9	53.3
10.30	0.0158	17	62.9
60.30	0.018	2.90	71.1
310.30	0.0183	0.565	72.3
610.30	0.019	0.287	75.1
1300	0.020	0.1345	79
2500	0.022	0.07	86.9
3900	0.024	0.045	94.8
5000	0.0253	0.035	100

f) Sample: Evans PVC. Sample Weight: 0.562 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.07	0.006	35.45	41.3
7.57	0.0065	23.1	44.8
10.17	0.007	17.2	48.3
20.17	0.008	8.58	55.2
110.17	0.0085	1.59	58.6
310.17	0.009	0.563	62
610.17	0.0092	0.287	63.5
1200	0.010	0.1458	69
2000	0.011	0.0875	75.8
3000	0.012	0.0583	82.75
3800	0.013	0.046	89.7
5000	0.0145	0.035	100

4. NH_4NO_3

a) Sample: PorvicII. Sample Weight: 0.664 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
4.47	0.008	39.18	30.77
6.7	0.012	26.11	46.15
10.3	0.014	16.99	53.85
20.3	0.016	8.62	61.54
110.3	0.016	1.586	61.54
310.3	0.017	0.564	65.38
610.3	0.017	0.287	65.38
1410.3	0.019	0.124	73.08
2400	0.021	0.0729	80.77
3600	0.023	0.0486	88.46
5000	0.026	0.035	100

b) Sample: Evans N-P. Sample Weight: 0.530 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.40	0.0365	32.41	64.04
8.55	0.04	20.47	70.18
10.7	0.042	16.36	73.68
20.7	0.045	8.454	78.94
110.7	0.046	1.581	80.70
310.7	0.047	0.563	82.46
610.7	0.048	0.287	84.21
1400	0.050	0.125	87.72
2600	0.053	0.0673	92.98
3600	0.055	0.0486	96.49
5000	0.057	0.035	100

c) Sample: Texon. Sample Weight: 0.466 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.15	0.027	33.98	55.10
8.30	0.032	21.08	65.31
10.55	0.033	16.59	67.35
20.55	0.0365	8.516	74.49
110.55	0.037	1.583	75.51
310.55	0.038	0.564	77.55
610.55	0.0382	0.287	77.96
1300	0.040	0.1346	81.63
2400	0.043	0.0729	87.76
3400	0.046	0.0515	93.88
5000	0.049	0.035	100

d) Sample: Dewey-Almy. Sample Weight: 0.582 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.50	0.045	31.82	62.32
8.60	0.0515	20.35	71.33
10.85	0.054	16.13	74.79
20.85	0.0575	8.393	79.64
110.85	0.0585	1.579	81.02
310.85	0.0595	0.563	82.41
610.95	0.0605	0.286	83.79
1200	0.062	0.1458	87.26
2600	0.066	0.0673	91.41
3400	0.068	0.0515	94.18
4000	0.070	0.0437	96.95
5000	0.0722	0.035	100

e) Sample: Evans STD. Sample Weight: 0.426 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
5.40	0.037	32.41	57.8
8.45	0.042	20.71	65.7
10.7	0.0435	16.36	68.0
20.7	0.048	8.454	73.5
110.7	0.048	1.581	73.5
310.7	0.049	0.563	76.6
610.85	0.050	0.286	78.1
1200	0.051	0.1458	79.7
2000	0.054	0.0878 5	84.4
3200	0.058	0.0547	90.6
5000	0.064	0.035	100

f) Sample: Evans PVC. Sample Weight: 0.936 gm.

Total Absolute Pressure (psi)	Penetrometer Stem Reading (c.c.)	Diameter (micron)	Cumulated Percentage (%)
2.27	0	77	0
4.70	0.010	36.45	41.7
7.00	0.012	25.0	50
10.30	0.0125	1.697	52.1
20.30	0.014	8.61	58.3
110.30	0.015	1.58	62.5
310.30	0.016	0.564	66.6
610.30	0.016	0.287	66.6
1600	0.018	0.1092	75
3000	0.020	0.0582	95.8
5000	0.024	0.035	100