STUDIES OF LIMESTONE AGGREGATES

BY FLUID-FLOW METHODS

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by

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PURDUE UNIVERSITY
LAFAYETTE INDIANA

Technical Paper

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TO: K. B. Woods, Director   July 1, 1959
            Joint Highway Research Project

FROM: H. L. Michael, Assistant Director   File: 4-6-6
           Joint Highway Research Project   Project: C-36-47F

        Attached is a technical paper entitled, "Studies of Limestone Aggregates by Fluid-Flow Methods", by Dr. W. L. Dolch, Research Associate on our staff. This is a copy of a paper which was presented to the 62nd Annual Meeting of the American Society of Testing Materials in Atlantic City, New Jersey, June 21-26.

        The paper will be submitted for publication by that society. It is being presented to the board for the record.

                        Respectfully submitted,

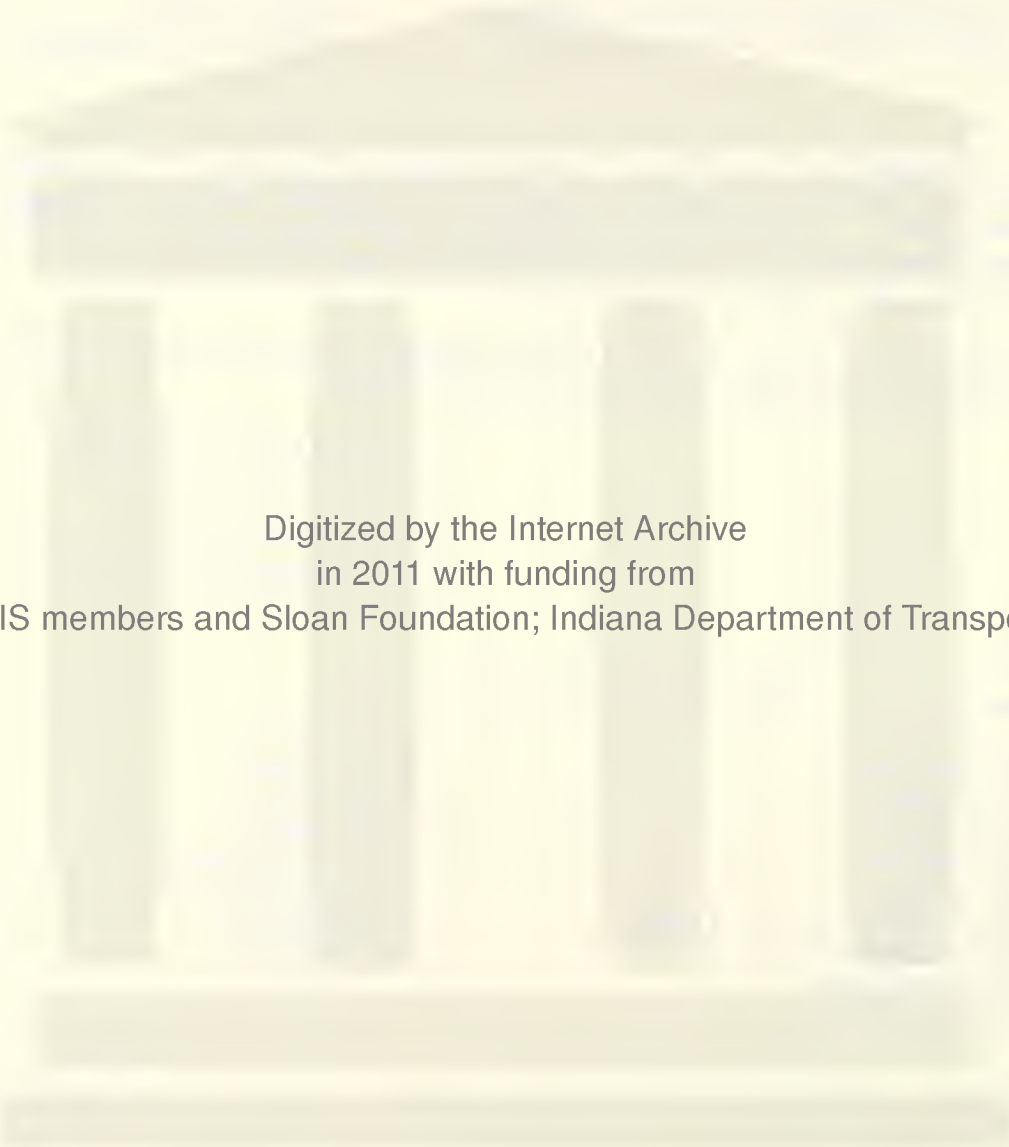   H. L. Michael, Secretary

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Attachment

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STUDIES OF LIMESTONE AGGREGATES

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W. L. Dolch
Research Associate
Joint Highway Research Project

File 4-6-6
Project C-36-47F

Purdue University
Lafayette, Indiana
July 1, 1959

Synopsis

Simple fluid-flow measurements were made in an attempt to learn more about the pore characteristics of coarse aggregates and their influence on the freezing and thawing durability of concrete. Determinations of density, porosity, absorption, degree of saturation, specific surface area, capillary absorptivity, permeability, and tortuosity factor were made on four Indiana limestones with both good and poor field and laboratory durabilities. Absorptivity was found to be better than permeability as an index of surface area. The poor stones had consistently larger values of porosity, absorption, specific surface, permeability, absorptivity, ratio of absorptivity to permeability, and coefficient of rate of saturation increase than those for the more durable materials.

STUDIES OF LIMESTONE AGGREGATES BY FLUID-FLOW METHODS

Introduction

The performance of a rock as a concrete coarse aggregate is
determined to a large extent by the properties of its internal
void or pore system. Rhoades and Mielenz (1)* state "In a broad
sense, the internal pore characteristics of the particles are the
most important properties of the aggregate materials". This
influence is primarily due to the effect of these characteristics
on the absorption and retention of water by the aggregate. The
details of this influence are only imperfectly known and current
standard test methods reflect this imperfection. They measure
comparatively simple properties of the pore system and leave
considerable to be desired from the standpoint of their reliabil-
ity in performance prediction.

The properties of aggregates and specifically the influence
of pore structure on the durability of concrete have been reviewed
rather extensively (1,2,3,4,5). An important aspect of the matter
is the influence of the porosity of the aggregate on the durability
of concrete exposed to freezing and thawing. An excellent discus-
sion of the theoretical aspects of frost action on the components
of concrete has been given recently by Powers (6). He points out
that damage caused by water freezing in the aggregate pieces can

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*The numbers in parentheses refer to list of References at the end
of the paper.

be brought about in several different ways. Probably chief among these is rupture of the surrounding matrix or of the aggregate piece by the hydraulic pressure generated when the growing ice body forces still-unfrozen liquid through the porous material. The pressure established is proportional to the rate of movement of the liquid and to the distance that the liquid must traverse and inversely proportional to the permeability of the medium. Thus, for every material and condition of freezing there is a "critical size" that is the length of flow path that cannot be exceeded without the generation of stresses that will be harmful to the material.

Inherent in such a mechanism are questions of the acquisition and retention of water by the material and the degree to which it opposes movement of water within its pore system; i e. its permeability.

These properties of capillarity and permeability can also be used to give some indication of the pore structure of a porous medium. In movement through such a material a liquid experiences a viscous drag that is a function of the size of the pore through which it is traveling. In capillary absorption also the force causing ingress of the liquid is a capillary pressure that is related to the size of the pore containing the meniscus. The relationship between these parameters and pore size is, however, capable of analytical expression only for simple pore structures. The pore systems of the limestone rocks in question here are so complicated that it is necessary to postulate a model that has the

same phenomenological properties as the substance it represents but has a pore system that is simple enough so that analytical relationships can be derived that relate its pore structure to the measured properties in question. Much work has been done on a variety of porous materials and for details of this work and the theory supporting it the reader is referred to the texts by Irmay [7] and Scheidegger [8] and to various reviews [9,10,11,12].

The approach used in the work described here was the measurement of the permeability and capillary absorptivity, along with various associated properties, of four Indiana limestone aggregates, two of which have good, and two poor performance histories in pavements. These measured properties were used to compute the properties of the pore system of a model and tentative correlations are indicated between these properties and the durability histories of the materials.

Experimental Work

Test Materials. The materials used were four limestone rocks that have well-documented field histories in Indiana. These materials have been used for several other studies. Generally speaking, these previous studies [13,14,15,16,17,18,19] have shown that distress of Indiana pavements due to blowups and frost action is traceable to the coarse aggregate component of the concrete. The aggregates with poor performance usually have larger values for absorption, porosity, and vacuum degree of saturation than do those with good performance. The poor materials also have a comparatively

large amount of pore space with a "pore diameter" smaller than four
microns. These poor-performing materials have also been shown to
be generally free from alkali reactivity and from thermal incom-
patibility. The conventional test methods for frost susceptibility
are sometimes inadequate when applied to these materials but their
field performance is duplicated, in a relative sense, by the results
of freezing and thawing testsof concrete made with the aggregate in
question.

The quarries were sampled. No statistical precautions were taken;
the samples were merely picked with an eye toward similarity to the
general mass and freedom from abvious anomalies. Although the quarries
are comparatively homogeneous, this sampling procedure does necessi-
tate the specific assumption that if pavements were to be made incor-
porating the samples as coarse aggregate these pavements would then
have durability histories similar to those of the pavements that were
actually made with material from the same sources. This sampling
procedure was felt to be adequate for an exploratory study such as
this.

In Table I are listed the pavement performance histories of
the materials, their calculated compositions, and the approximate
hundred-cycle durability factors[*] (18,19) of concrete made with the
vacuum-saturated aggregates and having approximately three and one
half per cent air contents.

Sample Preparation. Small cores, approximately 1.7 cm in

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[*] See ASTM Designation : C 291-57T for a definition of the dura-
bility factor. A high factor indicates relatively durable per-
formance in laboratory freezing and thawing tests.

diameter and several centimeters long, were cut from the samples with a diamond bit. The cores so obtained were then faced off and the ends were ground by hand to be parallel and normal to the cylindrical axis. In other instances the test samples were prepared by sawing out small prisms of rock and hand-finishing the surfaces.

Density and Absorption. The values for the bulk volumes of these cylinders and prisms were obtained by measurement of their linear dimensions and calculation of the volumes. The samples were weighed oven-dry and the bulk densities were calculated. Values for 24-hr absorption were obtained in the usual way. The samples were then dried and vacuum-saturated by evacuating for one hour with an aspuator and then allowing the samples to soak an additional 23 hours. They were then surface-dried, weighed, and the vacuum absorptions were calculated.

Porosity and Degree of Saturation. The porosity (ε) is defined as the ratio of the pore volume to the bulk volume. The porosities of the samples were determined by means of an adaptation of the "McLeod Gauge" porosimeter (Figure 1) of Washburn and Bunting (20). The porosity of the rock samples in the sample chamber was measured by raising the mercury until it covered the sample and trapped the air in the pores. The level was raised to the top mark of the microburet and the upper stop cock was closed. The mercury was then lowered to expose the sample. The air came out of the pores and was trapped when the mercury level was again raised. The volume of air, which equals the volume of pores, was read directly, at atmospheric pressure, from the microburet. The degree of saturation (S_w) or, more simply,

the saturation is the ratio of the volume of water in the pores to the total pore volume. The 24-hour and vacuum saturations were calculated from the data obtained in the absorption and porosity determination.

In Table II are listed the bulk density, porosity, absorption and saturation values obtained. Each value is an average of the results of measurements on five to ten separate samples.

Permeability. The permeability of a porous medium is defined by the expression

$$\frac{V}{t\,A} = \frac{K}{\eta}\frac{\Delta P}{L} \tag{1}$$

where V is the volume of permeant traverssing the sample under steady-state conditions in time t, A is the gross area of the sample normal to the flow direction, ΔP is the pressure difference causing flow, η is the viscosity of the permeant, L is the gross length of the sample, and K is the permeability. The c.g.s. units for K are cm^2. This unit is so large that another has come into general use. This is the millidarcy (md.) and one cm^2. equals $1.013 \times 10''$ md.

The apparatus used to determine the permeabilities of the samples is shown in Figure 2. The dry sample was mounted in the holder with a hand asphalt cement. The assembled apparatus was pumped down to the higher pressure desired. The bypass stopcock was closed and the pumping was continued until the desired pressure difference was established across the sample. The system was then closed and the decay of the pressure differential was measured with a cathetometer as a function of time as the air flowed through the sample. A knowledge of thes rate of pressure change, the sample's dimensions, and certain apparatus constants permitted the calculation of the permeability of the sample. The apparatus is of the same principle as those used by Arvell (22) and Kamack (23).

When a gas is used as the permeant there are two restrictions on
the definition of permeability (equation 1). These are 1) the volume
V is that corresponding to the mean pressure across the sample and
2) K is a function of this mean pressure. This second restriction is
due to the fact that at pressures at which the mean free path of the
gas is roughly equal to size of the flow channel a gas starts to "slip"
past the channel walls and the apparent permeability is enhanced. The
influence of pressure is shown by the equation of Klinkenberg (21),

$$K = K_l \left(1 + \frac{b}{P_m}\right) \qquad (2)$$

where P_m is the mean
pressure across the sample. Klinkenberg showed theoretically and
experimentally that the intercept K_l is the permeability that would
be obtained for the flow of a liquid through the sample. The
permeabilities of these samples were, therefore, measured at several
mean pressures and plotted against reciprocal mean pressure. An
example of the results is shown in Figure 3. The values of K_l, the
intercept, obtained in this way are listed in Table III. They are
for only one sample from each source, but this sample was carefully
selected to be as representative of the group as possible.

Absorptivity. When a liquid is imbibed into a porous solid by
capillarity the volume absorbed at any time is proportioned to the
approach area of the sample and to the square root of the time
elapsed. Powers and Brownyard (24) defined the "absorptivity",
K_a by

$$\left(\frac{V}{A}\right)^2 = K_a t \qquad (3)$$

where V is the volume of capillary permeant taken up in time t through
approach area A. The c.g.s. units on K_a are cm^2./sec.

The absorptivities were increased in a simple, yet apparently
precise and accutate, way. The samples were taped with plastic
electrician's tape to prevent evaporation of water from the sides.
The sample was then placed on several filter papers that were wet
with water and a stopwatch was started at the same instant. From
time to time the watch was interrupted and the samples were removed
and weighed rapidly. They were then returned to the (essentially)
free water surface and the timing was resumed. It is assumed that a
negligible amount of flow took place within the sample during the
weighing process.

The logarithm of V/A was plotted against the logarithm of t and
K_a was calculated from the one-minute intercept. In most runs only
enough points were taken to define the line. But for two samples
from each source the process was allowed to continue until equilibrium
was reached in order to determine the degree of saturation appropriate
to the process. With the denser rodes it took several days to com-
plete a run. The filter paper was kept moist by means of a wick and
water supply.

An example of these curves is given in Figure 4. The values
obtained for K_a are listed in Table III and are averages for seven
individual samples except for 1-1S where only four were used.

The values for the capillary degree of saturation obtained in
this determination were so close to the 24-hour immersion values
that the latter were used in subsequent calculations because of the
larger number of samples tested.

Tortuosity. It seems reasonable to suppose the path of a
"particle" of fluid flowing through a porous medium to be longer than

the gross length of the specimen between inlet and outlet faces of most porous media. The square of the ratio of this "tortuous" length of flow, L_t, to the gross length, L, is called the tortuosity, k_t, of the pore system. The method used to measure the tortuosities of these samples is discussed by Wyllie and Spangler (25) and depends on the assumption of average equality between the path traversed by a fluid flowing in a porous medium and that traversed by an electrical current when the pores contain a conducting solution. The ratio of the electrical resistance of the porous medium containing the electrolyte to that of an amount of solution of the same gross dimensions as the porous medium is called the formation factor, F. If the pore space is not completely saturated with electrolyte the formation factor, F_e, will be larger than F. The ratio of F_e to F is called the resistivity index, I, and this has been found to be a simpler function of the degree of saturation,

$$\frac{F_e}{F} = I = S_w^{-n} \qquad (4)$$

The exponent n seems to be independent of the degree of saturation until comparatively low saturations are reached. Measurements on a variety of porous media have shown n to be equal to $2 \pm 1/2$. Simpler analysis, of the problem, leads to an expression for tortousity in terms of the formation factor and the porosity,

$$k_t = (F\epsilon)^2 \qquad (5)$$

The two ends of the sample prisms were coated with conducting paint. The samples were then vacuum saturated in the usual way with a five per cent sodium chloride solution. They were blotted surface dry, weighed rapidly, and their resistances were measured with a 2000-cycle

bridge using an oscilloscope as a null indicator. The exponent n in equation (4) was taken to be two. These data permitted the calculation of the tortuosity via equations (4) and (5).

Because of the uncertainties with respect to various aspects of this determination, including those inherent in the basic analogy on which the method depends, the tortuosity values reported in Table III are the most unreliable data reported here. Also, only one sample from each source was used to obtain these values.

If the pore space is only partially saturated the tortuosity of the liquid phase will be higher than k_t.

The column in Table III headed "Tortuosity at 24-hours saturation" are the values calculated by means of

$$k_{t\alpha} = k_t \; S_w^{2(1-n)} \qquad (6)$$

where $k_{t\alpha}$ is the tortuosity considered to be applicable to the water phase during capillary absorption, i.e. with one 24-hour saturation values being used for S_w and n equal to two.

Water Vapor Adsorption. The specific surface areas of the rocks were measured by means of the water vapor adsorption technique and the theory of Brunauer, Emmet, and Teller (26). The isotherms were determined using 16-30 mesh samples and the ordinary gravimetric technique with a vacuum dessicator and saturated salt solutions for the establishment of constant vapor pressure. The sorptions were measured up to approximately thirty per cent humidity. The values obtained for the specific surfaces of the samples are listed in Table IV.

Discussion

The relationship between the permeability and pore characteristics that will be used here is the Kozeny-Carman equation. This relationship involves a model that has two attributes of most real porous media -1) a tortuous length of flow path greater than the gross length of the specimen and an irrigular cross-section of the flow channels. The derivation of the equation is given by Carman (7,20) and the final result is

$$K_1 = \frac{\varepsilon\, m^2}{k_o\, k_t} \tag{7}$$

where k_o is a factor related to the cross-sectional shape of the pores and m is the hydraulic radius of the pores, i.e. the ratio of the cross-sectional area to the perimeter. By means of the relationship $m = \varepsilon / S_g \rho_B$ where ρ_B is the bulk density of the porous solid and S_g is its specific surface, i.e. the surface area of one gram of the solid, equation (7) can be transformed to

$$K_1 = \frac{\varepsilon^3}{k_o\, k_t\, S_g^2\, \rho_B^2} \tag{8}$$

The shape factor, k_o, is equal to two for a round cross-section and changes only a little for other shapes. For consolidated porous media Wyllie and Spangler (25) gave evidence in favor of 2.7 and that value is used here. For a model of round tubes of radius r equation (7) becomes

$$K_1 = \frac{\varepsilon\, r^2}{8\, k_t} \tag{9}$$

The values for specific surface, calculated by means of equation (8), are given in Table IV and those for the radii, calculated from equation (9), are given in Table V both under the heading "Permeability".

When water enters a capillary tube and progresses through it, the equation describing the flow can be simplified by the assumption that the only forces of consequence are the capillary force causing ingress of the water and the viscous drag of the liquid that retards permeation. With the further assumption that the contact angle of water on these rock surfaces is zero degrees, the final result of equating these faces for dynamic equilibrium is

$$\gamma m = k_o \eta \, x \left(\frac{dx}{dt} \right) \tag{10}$$

where x is the tortuous distance the liquid has progressed in time t, η is the viscosity and γ the surface tension of the liquid, and the other symbols are as previously defined. If the area of the tube normal to the tortuous flow direction is a, the volume of liquid in it at time t will be ax and the rate of change of this volume will be

$$\frac{dV}{dt} = \frac{a \, dx}{dt} \tag{11}$$

If the tube is considered to consist of the entire interconnected pore space then the fraction of the bulk volume that is penetrated by capillarity is defined as the capillary porisity, α, i.e.

$$\alpha = \frac{a \, L_t}{A \, L} = \frac{a \, k_{t\alpha}^{\frac{1}{2}}}{A} \tag{12}$$

Elimination of x and a from equations (10), (11) and (12) gives

$$\frac{V \, dV}{dt} = \frac{\gamma m \, A^2 \alpha^2}{k_o \, \eta \, k_{t\alpha}} \tag{13}$$

When this equation is solved, the initial condition of V = 0 for t = 0 is imposed, and the definition of K_a (equation 3) is inserted the final result is

$$K_a = \frac{2 \, \gamma_m \alpha^2}{k_o \, k_{t\alpha} \, \eta} \tag{14}$$

which becomes, for round tubes of radius r

$$K_a = \frac{r}{2} \frac{\gamma \alpha^2}{\eta \, k_{t\alpha}} \tag{15}$$

If, finally, it is postulated that the hydraulic radius for the non-capillary - penetrable pore space is equal to that for the capillary pore space, then $\rho_B \, S_g \, m = \epsilon$ and elimination of m from equation (14) gives

$$S_g = \frac{2 \gamma \epsilon \alpha^2}{k_o \, k_{t\alpha} \, K_a \, \rho_B \, \eta} \tag{16}$$

The values for specific surface calculated from equation (16) are given in Table IV and those for the radii calculated from equation (15) are given in Table V both under the heading of "Absorptivity".

The column in Table V labelled "Sorption" is the radius of round pores that would give a specific surface equal to that measured in the water vapor sorption determinations.

A limitation, which was emphasized by Carman (10), of the Kozeny equation, is that the pore size should be reasonably uniform since a few large pores contribute to the permeability all out of proportion to their surface area. Inspection of Tables IV and V shows this effect. The radii calculated from permeability are all comparatively large and the specific surfaces are small, indicating the reflection of the larger pore size by this method of measurement. The same situation has been described by others for consolidated porous media (7,27).

On the other hand, the absorptivities should reflect a smaller pore size and a larger surface area because the liquid will tend to accumulate in these smaller pores as a result of their higher capillary potential. The values in Tables IV and V seem to show this effect also.

The calculated radii are smaller and the surfaces larger than for the values derived from the permeabilities. The values presented can be considered only as indications of the trend and should probably not be taken too literally because of the comparative inaccuracies of the tortuosity determinations, among other things. This is amply evidenced by two of the four absorptivity surface values being higher than the sorption surface, which is the theoretical maximum. The trend, however, does seem to be there and the absorptivity method is probably better than the permeability method as an interpretation of specific surface - at least for the kind of materials considered here.

With respect to problems of frost action in aggregate particles, the data in Tables IV and V show no obvious distinctions between the good and bad materials. Blaine, Hunt, and Tomes (28) concluded that materials with poor freezing and thawing durability have comparatively high specific surfaces and the data reported here lend some support to this conclusion.

Inspection of Table II shows the poor materials to have distinctly higher porosities and absorptions than do the good ones. It also shows that a low value for vacuum saturation usually means a good material while a high value, especially one greater than about 91 per cent, means a poor material, but that a border line region exists where the distinction between good and poor cannot be made on this basis. All this is, however, nothing new and has been reported many times before.

The permeability and absorptivity values in Table III show order of magnitude differences between the good and poor stones. These differences may be indicative but, on the other hand, they are what would be expected for materials whose porosities had the variations of these.

Two new parameters have been developed that seem to distinguish between the good and poor materials and have, furthermore, a logical support from the theoretical concepts of frost action.

Powers (6) states " a principal difference among different rock particles is the rate at which they become saturated when given free access to water". Wray and Lunsfield[(24)] also showed the importance of this characteristic. A simple derivation shows that the total rate of change of the saturation can be expressed as

$$\frac{dS_w}{dt} = \frac{K_a}{2S^2}\left(\frac{1}{S_w}\right)$$ (7)

Values of $K_a / 2c^2$ for these materials are given in Table VI and do show a separation between the good and poor aggregates. Indeed, 9-18, is given the stigma that it deserves with respect to its field history.

The ratio of K_a / K_1 should, on grounds of logic, be a measure of the susceptibility of a particle to frost action. A high absorptivity means a comparatively high rate of absorption while a low permeability means a comparative reluctance of the material to permit the passage of water and therefore a lower critical size. Both effects should tend toward an increased frost susceptibility and so the higher the ratio the worse. Similarly a rock with large bulges in the pore system that can act to improve frost durability in the same way as those in an air-entrained cement paste do should show a comparativity low value of this ratio. These ratios are given in Table VI and do indeed show the separation in the logical direction between the good and poor materials.

Conclusions

Based on the materials studied and the tests performed the following tentative conclusions seem reasonable:

1. The rate of increase of the degree of saturation of a material when exposed to free water, as measured by the ratio of $K_a / 2\varepsilon^2$, and the ratio of the absorptivity to the permeability are two possibly-useful indexes of frost susceptibility when the material is used as a concrete coarse aggregate. The more susceptible materials can be expected to have comparatively large values of both these ratios.

2. The absorptivity is superior to the permeability as an index of surface area of a porous soild when a simple model is used to analyze the results. Its ease of measurement, compared to the permeability, should also be kept in mind.

Acknowledgements

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TABLE I

Source No.	Calcite,%	Dolomite, %	Field Performance	Approx. 100-Cycle DF (18,19
1-1S	3	93	Good	98
9-1S	5	89	Poor	35
47-2S	77	17	Poor	40
67-2S	97	2	Good	98

TABLE II

Source No.	Bulk Density, g/cm³	Porosity, %	Absorption, % 24-hr	Absorption, % Vacuum	Saturation, % 24-hr	Saturation, % Vacuum
1-1S	2.73	2.31	0.72	1.17	58	89
9-1S	2.20	24.20	7.58	10.28	69	93
47-2S	2.42	10.30	2.51	3.75	59	87
57-2S	2.67	2.46	0.55	0.61	59	67

TABLE III

Source No.	Permeability md	Absorptivity cm²/sec	Tortuosity k_t	Tortuosity at 24-hr. saturation $k_{t\alpha}$
1-1S	0.00205	4.59×10^{-9}	145.	420.
9-1S	4.30	3.24×10^{-5}	4.54	9.52
47-2S	0.032	5.00×10^{-7}	29.9	85.0
67-2S	0.0092	6.10×10^{-9}	29.3	85.6

TABLE IV

Specific Surfaces, m^2/g

Source No.	Sorption	Permeability	Absorptivity
1-1S	0.67	0.046	0.46
9-1S	2.04	0.238	5.85
47-2S	2.54	0.271	2.22
67-2S	1.09	0.061	2.22

TABLE V

Pore Radii,* microns

Source No.	Sorption	Permeability	Absorptivity
1-1S	0.03	0.32	0.03
9-1S	0.11	0.79	0.03
47-2S	0.03	0.23	0.03
67-2S	0.02	0.26	0.01

*The values in this table have been rounded to two decimal places to
facilitate comparisons.

TABLE VI

Source No.	$K_2/2\epsilon^2$, cm^2/sec	K_2/K_1, sec^{-1}
1-1S	0.4×10^{-5}	2.3×10^5
9-1S	27.6	7.6
47-2S	2.4	15.8
67-2S	0.5	0.9

5 ML. MICROBURET

INDENTATIONS

SAMPLE
CHAMBER

35/25

8 MM. O.D.

MERCURY

1/4 X 1/16 IN.
TYGON

FIG. I POROSIMETER

TO PUMP

TO ATMOSPHERE

1/4 X 1/16
TYGON

8 MM. O.D.

25 MM. O.D.

50/30

TO PUMP

ASPHALT

SAMPLE

1/4 X 1/16
TYGON

35/25

MERCURY

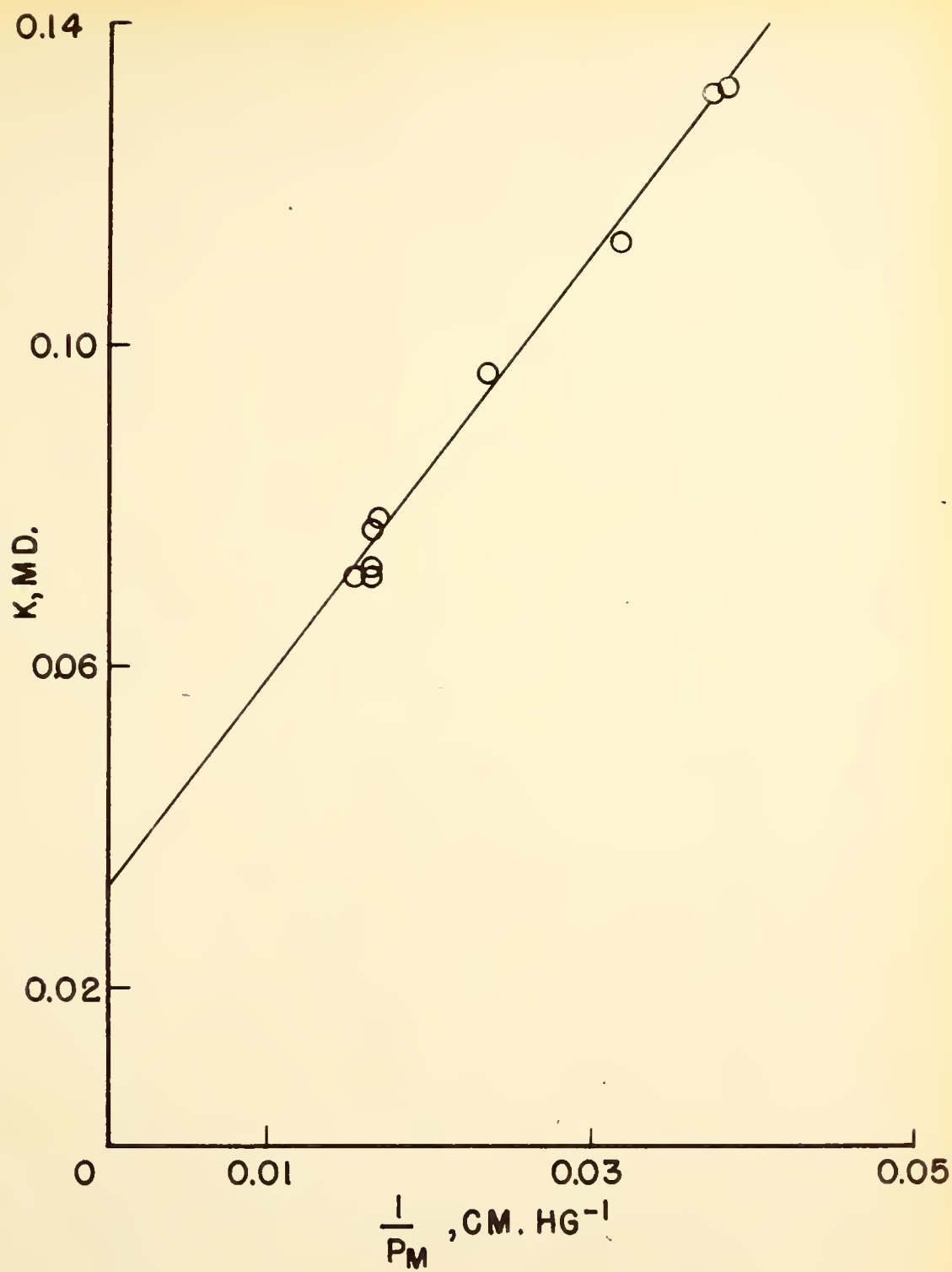2000 ML.
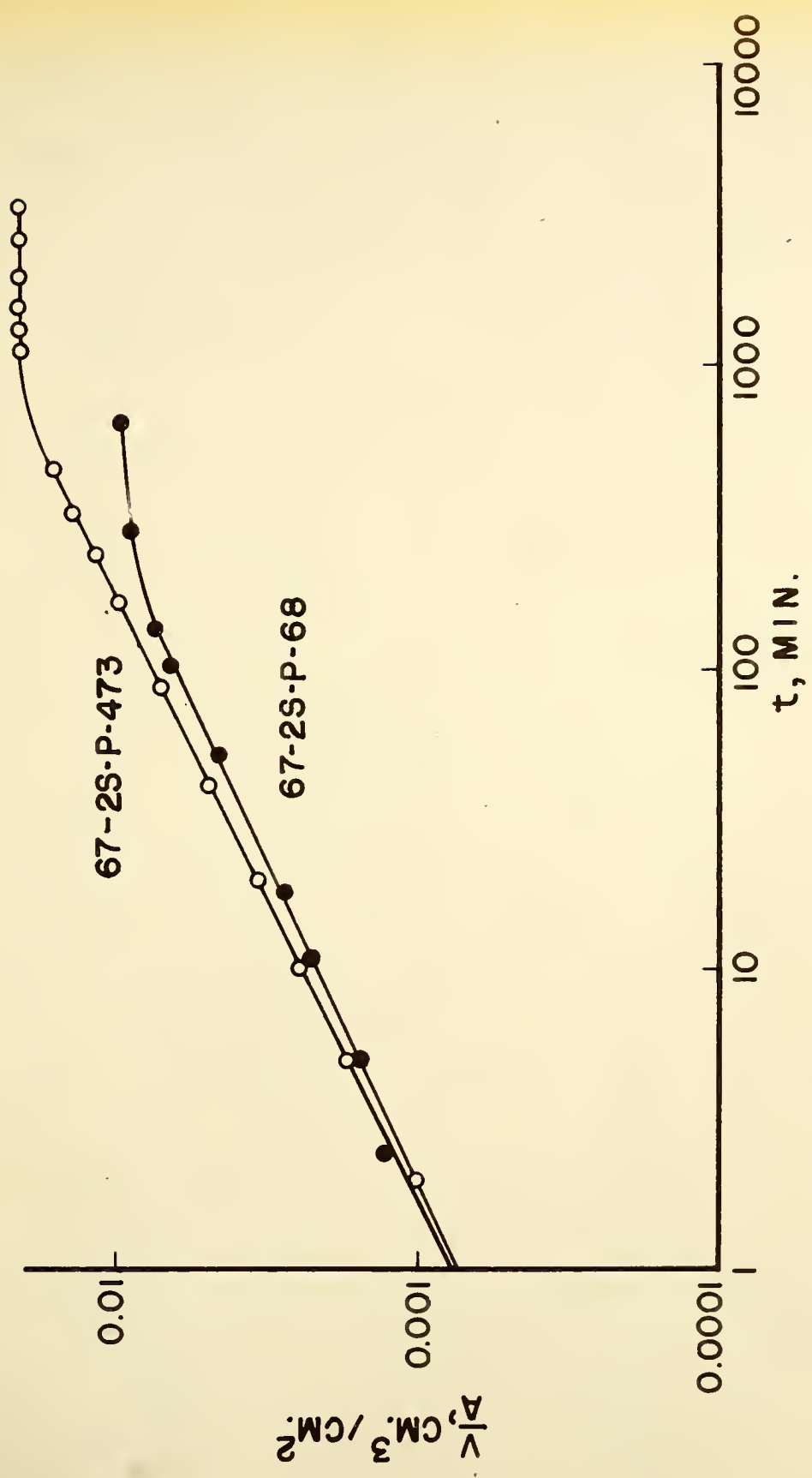SURGE FLASK
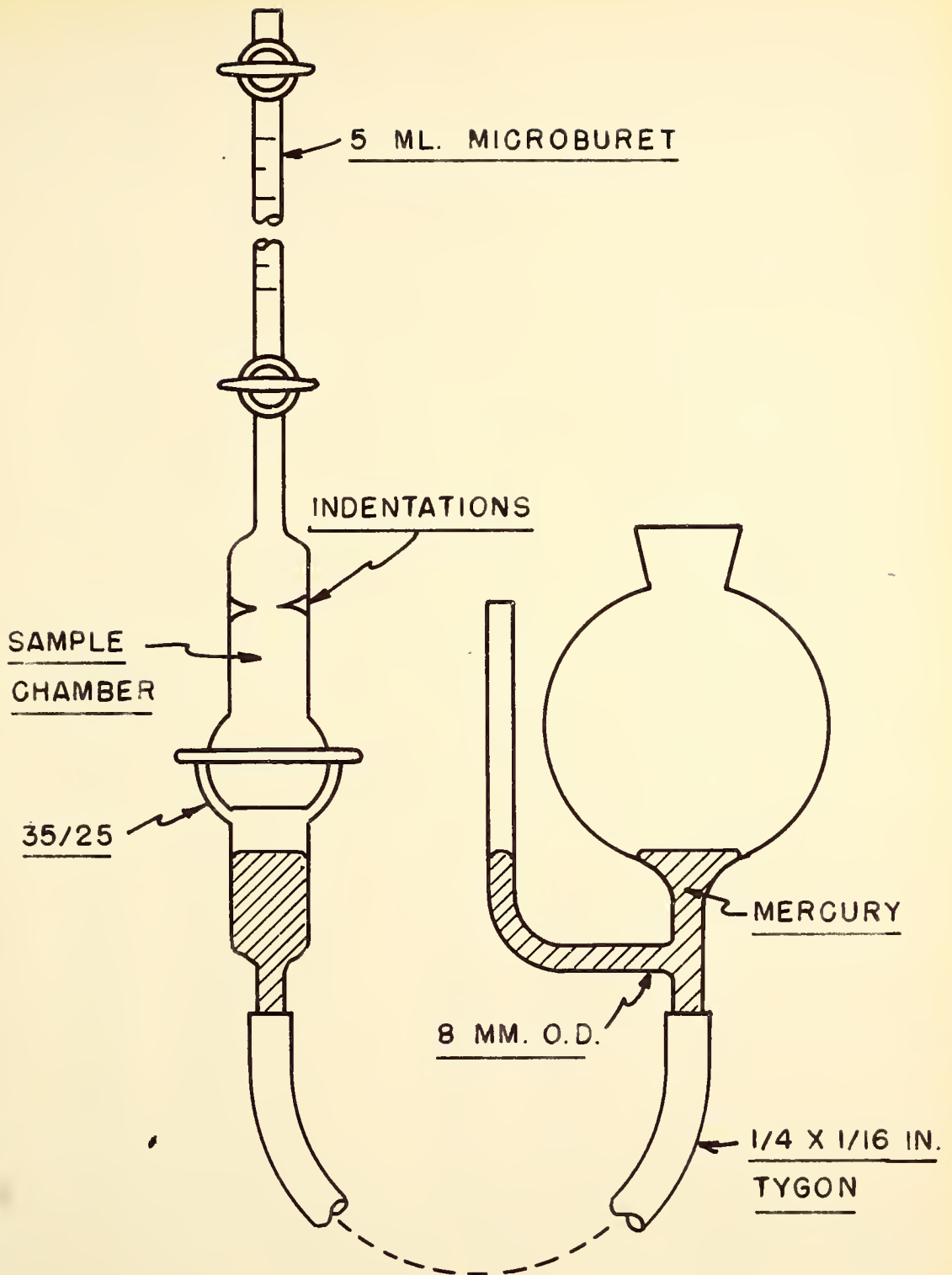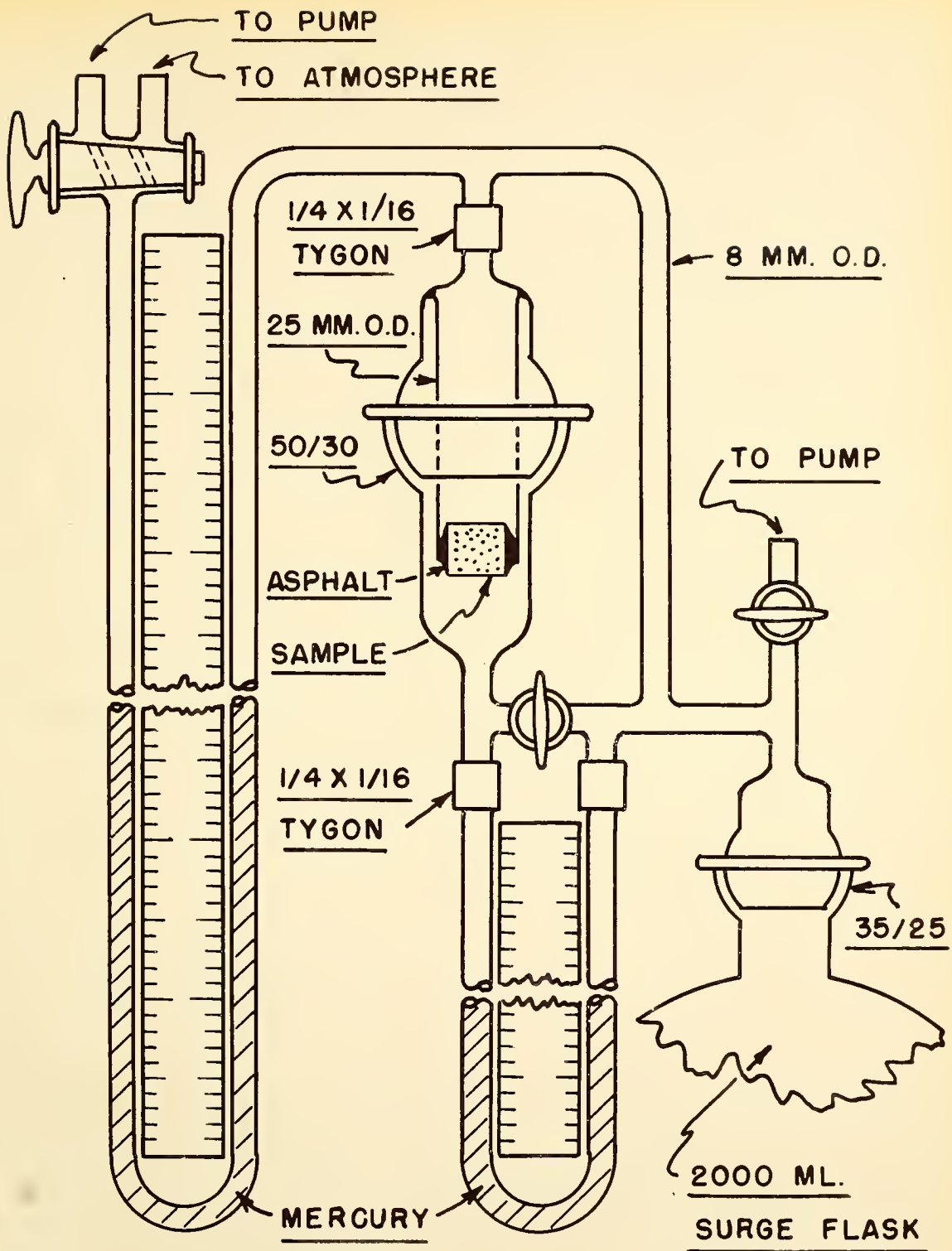
FIG. 2   PERMEAMETER

FIG. 3   PERMEABILITY   OF   47-2S-P-98

FIG. 4   ABSORPTIVITY OF 67-2S