Chapter 2. EXPERIMENTAL STUDIES

F. C. Hurlbut, C. R. Jih, and P. A. Witherspoon

INTRODUCTION

The underlying rationale for undertaking studies of fluid flows in porous media under rarefied gas flow conditions has been to supply the empirical basis for theory necessary to the design and understanding of a permeability probe device for *in situ* experimentation at the lunar surface. A second objective has been to provide the practical experience in such experimentation necessary to permit a sound, efficient, and workable design of such a probe.

The outlines of our attack, both theoretical and experimental, have been summarized in the 1969 Final Report, Vol. IV of IV, "Studies on Conductivity of Lunar Surface Materials," by Katz, Willis, and Witherspoon, and remain very little changed to this date. In that report our current state of knowledge was described, preliminary concepts of probe design were discussed, and directions of analysis were indicated.

The present report is confined to the description of the ongoing experimental program and to a presentation and discussion of preliminary observations. It should be understood as a record of work in progress and is to be taken, together with the 1970 Final Report, "Studies of Fluid Conductivity of Lunar Surface Materials — Theoretical Studies," by Raghuraman and Willis, as a representation of our progress in the fiscal year 1969-1970.

BACKGROUND

The flow of gases through porous media has received a moderate amount of attention over the years as, for example, the flows of low-density gases connected with the problems of catalytic beds or with those of transport phenomena at permeable barriers. The words "low density" refer here to conditions under which the Knudsen number, based on pore size, is greater than ∿ 1/100. One may note that such low-density flows might well occur at pressures above or below 1 atmosphere for rocks within the ordinary range of pore size. Above the limit of low density cited, the flow of gases in porous media may be treated by the empirical continuum methods which have been found to be successful. Prior studies of low-density flows have been confined to certain semi-continuum models or to the assumption that the flows are entirely free molecule in character. Such models imply that the density gradients are everywhere small, a constraint which cannot be applied in general to flows in porous media whose natural environment is, and has been for a very long time, a vacuum.

Related studies of the flow through capillaries have been more widely conducted, and it would be in connection with these somewhat simpler flows that one would hope to see the development of theoretical models for the transition from continuum flows to the free molecule regime. Such models would provide a valuable base for modeling the porous medium. However, we again find that nearly all theoretical work has confined itself to conditions where the density gradients are small so that the gas remains within a particular regime of flows throughout the capillary. Work relating to larger density gradients has been conducted by interpolation and fitting but without a rigorous basis in the kinetic theory. Experimental work on capillary flows has been conducted under conditions appropriate to the theory with few exceptions and in these latter cases no examination has been made of the details of the transition from continuum flow to free molecule flow.

With these limitations of available information in evidence it was determined to undertake direct measurements of porous medium permeability under low-density conditions as the most efficient route to the design and understanding of an *in situ* permeability probe for lunar materials. It was determined that initial investigations should be of one-dimensional flows through homogeneous, simulated rock samples having a range of permeabilities. Use would be made of the pumping system associated with the existing rarefied gas wind tunnel, and it was also planned that advantage would be taken of the technology and experience of the Rarefied Gas Laboratory.* The program of design, construction, and measurement has proceeded well, but not as rapidly as planned, so that to this time

*U. C. Division of Aeronautical Sciences (Mechanical Engineering) only the first phases of the measurement program have been completed. In the next sections details of the permeability apparatus are given.

DESIGN AND CONSTRUCTION OF APPARATUS

Introduction

As in the proposed permeability probe, gas from a source at moderate pressures flows into the porous specimen toward a sink at low pressures. If the Knudsen number of the flow is initially of order 1 or smaller, the flow will inevitably transform to a free molecule flow within the specimen. The character of the transition, as determined by the measured pressures at various distances from the source, will permit a calculation of permeability and possibly pore size and configuration when suitable theory becomes available. The experimental apparatus required for the investigation of one-dimensional flows within the above conceptual framework consists of a gas source and flow metering system, a specimen chamber with pressure taps distributed along its length, a pressure transducer and metering system, a high capacity vacuum pump, and the necessary valves, ancillary gauges, and pumps. A detailed description follows.

Description of the Apparatus

It was a basic objective of the design that it should permit the detailed examination of pressures as a function of position along a one dimensional flow through a porous specimen. It was determined that the specimen should consist of up to 10 cylindrical slabs, each of thickness to 1 inch and diameter 2.5 inches, permitting pressure measurements to be made at discrete intervals by sampling the space between slabs. The arrangement is shown in Figure 2-1, a dimensioned assembly drawing of the equipment. The specimen chambers, shown in greater detail (Figure 2-2), consist of 2 flanged cylinders of stainless steel each with provisions for 5 segments of specimen. Each specimen segment consists of a plexiglass ring within which is cast the porous material. A seal between the plexiglass ring and the inner wall of the specimen chamber is arranged by an "O" ring set in a groove in the chamber wall. Pressure taps with

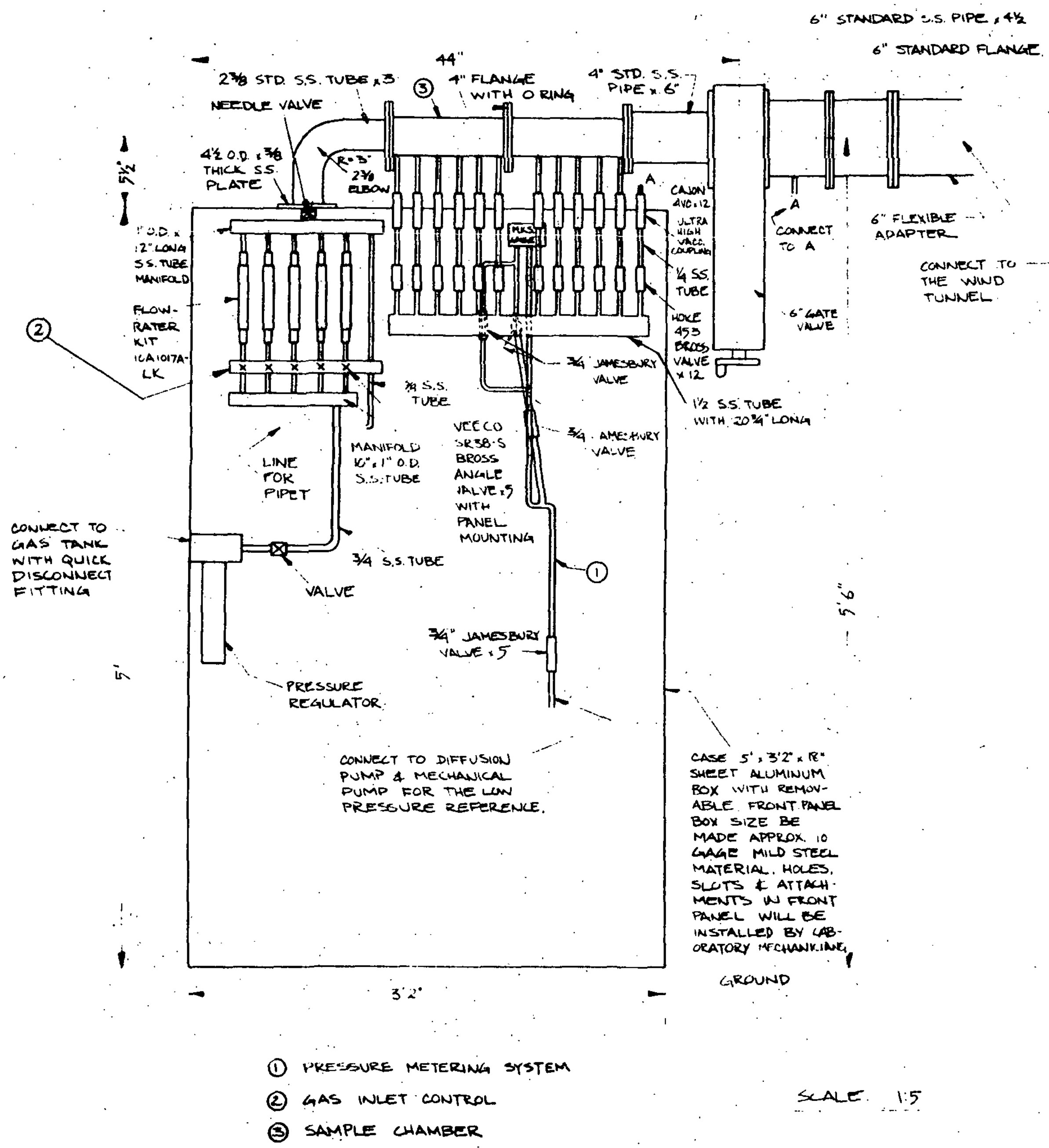

Fig. 2-1. System assembly.

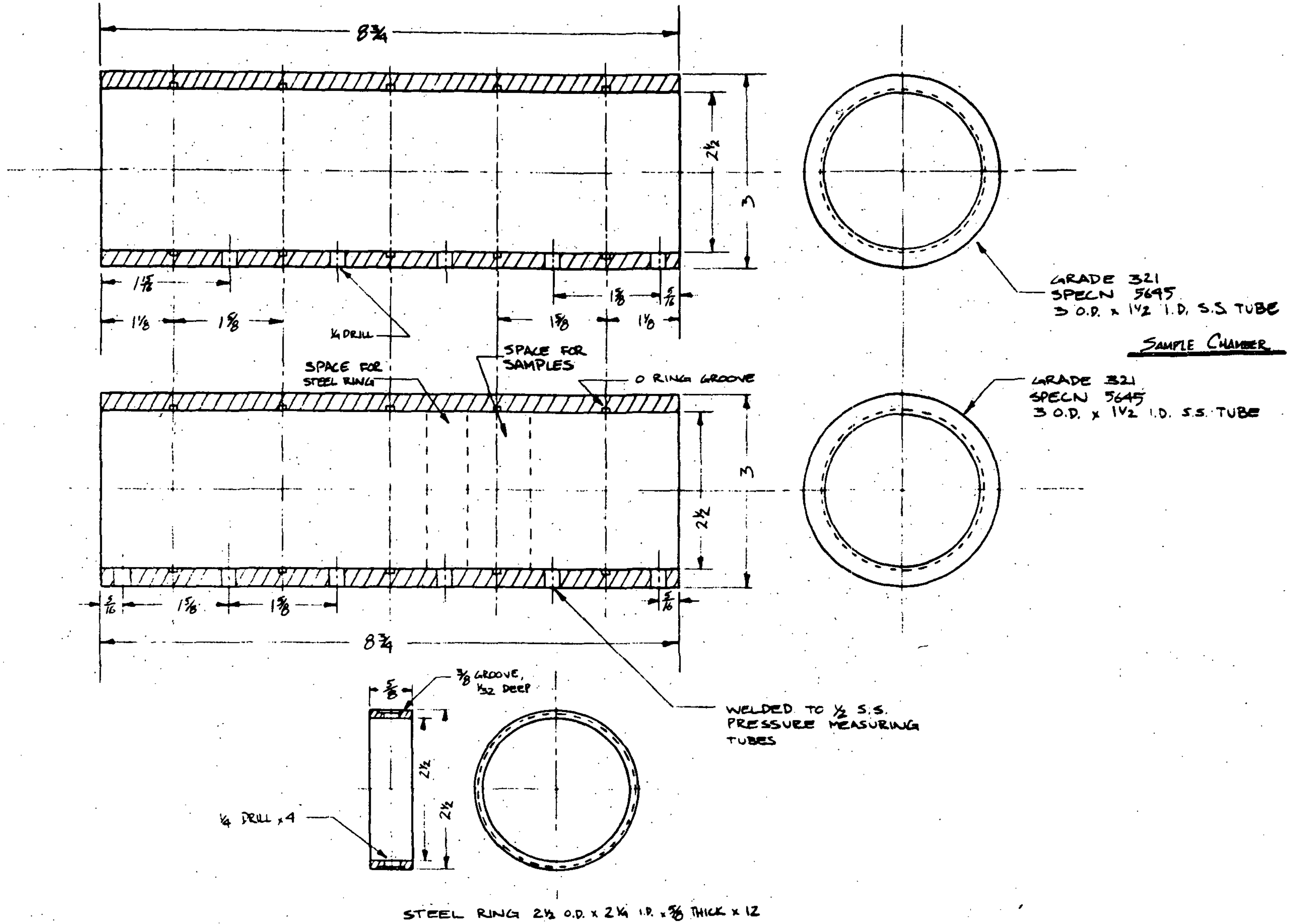

Fig. 2-2. Sample chamber.

pressure leads of 1/4" diameter stainless tubing welded in place are provided between each specimen position. Spacer rings between each specimen maintain the correct position.

Uniform entry conditions over each slab face are established by virtue of the high flow conductance of the large gap between slabs as compared with the lower conductance of the slab material. Thus a segmented ideal one-dimensional flow is permitted. Note that the number of slabs may be varied from 1 to 10 and that the thickness of each slab may be arbitrarily determined up to 1 inch.

Each pressure tap is connected via a valve to a central manifold and that manifold is connected through 3/4-inch copper tubing and a quarter swing valve to a pressure transducer. Ample conductance is provided to permit degassing the specimens and to make possible a sufficiently short gauge response time. The pressure transducer is an MKS diaphragm gauge having a maximum differential pressure range of 30 Torr. This device was selected for its well-known accuracy, stability, and insensitivity to gas composition. Since it is a differential pressure gauge it must be connected to a reference vacuum system, details of which are shown in Figures 2-1 and 2-3. Note that a bypass valve is provided for establishing the zero of the gauge and to permit evacuation of the manifold.

At the downstream end of the specimen chambers is a 6-inch vacuum gate valve and beyond that, the main manifold of the rarefied gas wind tunnel. The pumps associated with the wind tunnel flow system have the capacity to maintain the downstream end of the permeability apparatus at 1 to 2 microns Hg for any realistic flow within the porous samples.

The gas supply system is also shown in Figure 2-1. This system consists of high and intermediate pressure regulators, appropriate shut-off valves, and a gas-service regulator followed by a system of 5 viscosity type flow raters covering a range of flow rates to $\sim 3 \times 10^{-3}$ cc/sec. For lower rates of gas flow the film-capillary, positive displacement method will be used. A needle valve between the flow metering system and the first specimen chamber serves to regulate the flow rate.

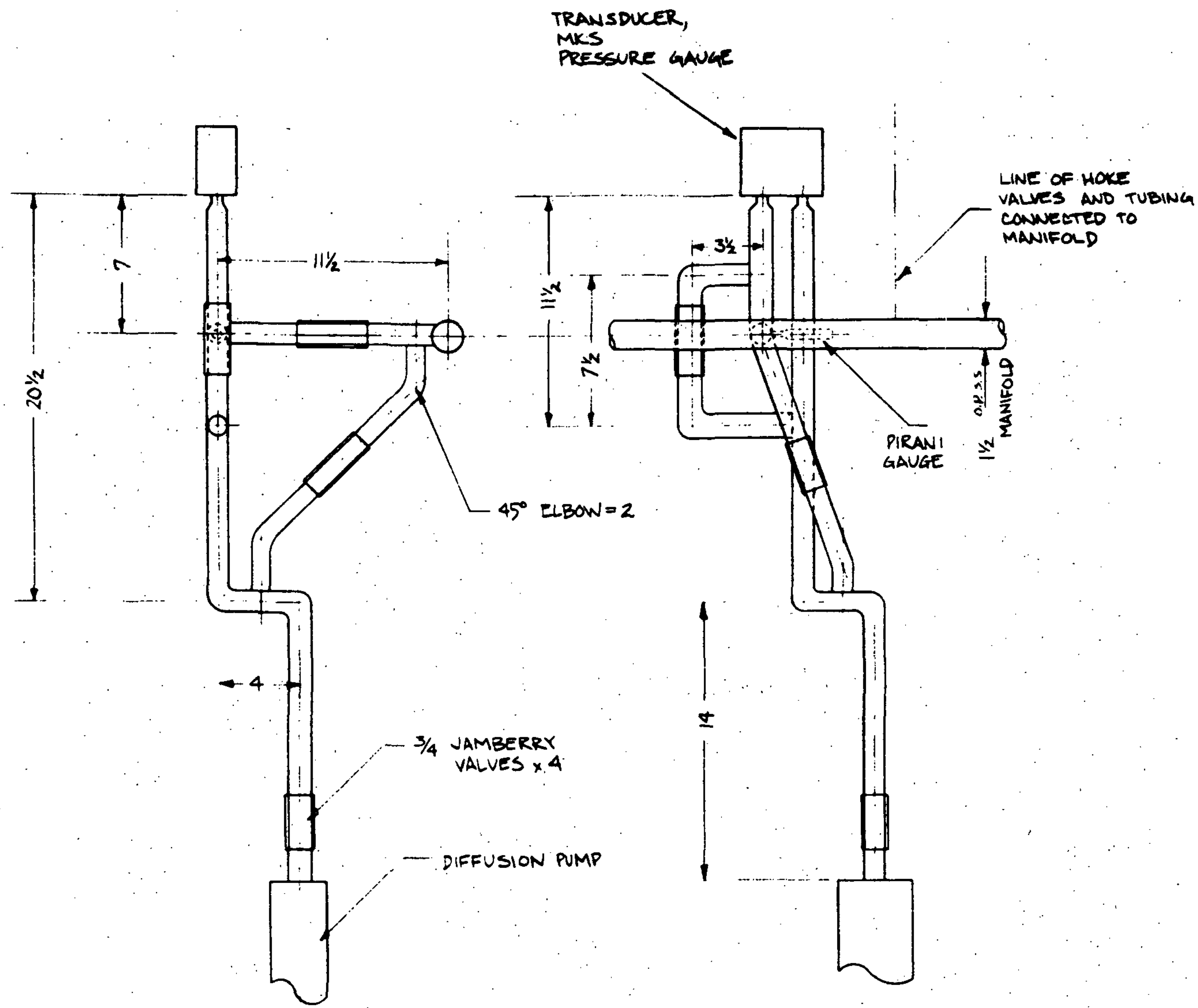

Fig. 2-3. Pressure metering system.

An overall view of the permeability apparatus is shown in the photograph, Figure 2-4.

SPECIMEN PREPARATION

A number of options exist for the preparation of porous samples. Among these are the aggregation of sand particles using wax or resin binders; the casting of concretes and artificial stones; the sintering of metallic beads, chips, filings, or fibers; the sintering of ceramic materials in fibre, bead, or rod form; and the cutting of natural rocks, particularly those of volcanic origin. To be suitable for low pressure measurements, the porous media must be free of organic materials having vapor pressures in the micron range. In order to insure an extended region of transition flow under pressure conditions appropriate to this experiment, the samples should have much greater permeability than ordinarily found in natural rock.

The various constraints of the present program favored the construction of sintered materials, preferably ceramics. However, for initial performance testing it was decided that cast concrete specimens would serve and that these could be constructed using materials and technology readily available in the Civil Engineering Laboratories. Accordingly, three sets of cast concrete samples were prepared, careful attention being paid to mixing and uniformity of casting procedure. The composition of these samples is shown in Table 2-1.

Table 2-1

Sample	Sand (gm)	Cement (gm)	Water (gm)
1	2000	100	100
2	2000	500	250
3	2000	800	400

In each set the concrete was cast into 12 to 16 plexiglass ring forms of 1-inch depth. Upon curing and drying the samples, the permeability for air was measured at atmospheric pressure and above using

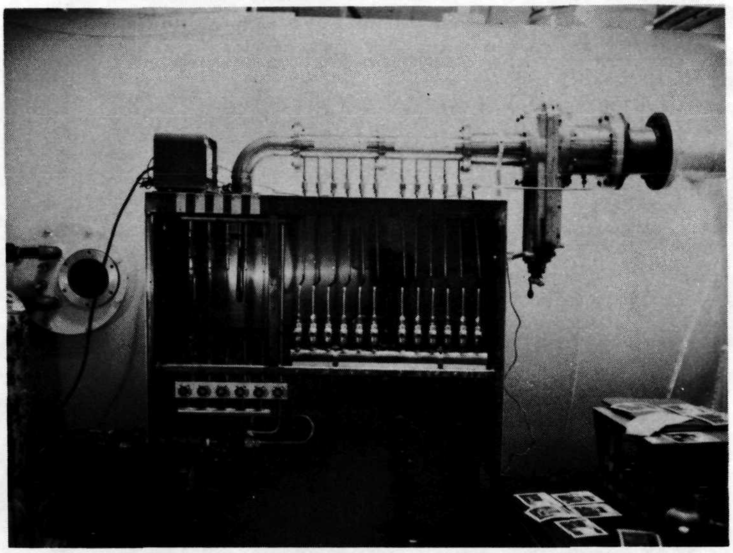

Fig. 2-4. View of the permeability apparatus.

a standard permeability apparatus. Set 3 was rejected immediately as too impermeable, and the 10 slabs most uniform in permeability were selected from each of sets 1 and 2. The results of these tests are shown in Table 2-2.

Table 2-2

Slab No.	Permeability K(cm^2) ($\times 10^{-11}$)	Slab No.	Permeability K(cm^2) ($\times 10^{-12}$)
1-1	9.3	2-2	4.5
1-2	9.3	2-3	6.8
1-3	9.3	2-5	4.5
1-4	9.3	2-7	4.8
1-5	9.1	2-8	7.5
1-6	9.1	2-9	4.8
1-7	9.1	2-10	5.2
1-8	9.1	2-11	5.8
1-9	9.5	2-13	5.2
1-10	9.5	2-16	5.4

It may be noted that Set No. 1 is both more uniform and more permeable than Set No. 2. The permeabilities are within the range of the more porous natural rocks.

PRELIMINARY OBSERVATIONS

Preliminary measurements were made on a set of 4 slabs (No.'s 2-2, 2-3, 2-10 and 2-11) to gain operational experience with the instruments and to develop a physical sense for the appropriate permeability ranges of the next generation of specimens. The gas was nitrogen. The tunnel downstream pressure and the M.K.S. gauge reference pressure were at 1 micron or below. Operational experience was obtained, although no useful quantitative information has resulted to this point, owing to the low permeability of the present specimens. Certain conclusions may be drawn which are summarized as follows:

1. It must be recalled that the objective of the system design is to permit the study of the regime of transition flows in porous media. This is accomplished by extending the region of transition in physical space over slabs which are somewhat less in thickness than that region. It is implied that specimens of very high permeability must be employed.

2. The flow conductance of the specimens must be sufficiently great that the pressure taps open into, effectively, an unlimited reservoir of gas at the measured pressure. Thinner samples and greater permeabilities will improve conditions in this regard.

3. In all regards the apparatus behaved well and appears to have the capability of giving results of the quality desired.

CONTINUING PROGRAM

Within the next few months it will be our objective to complete measurements enabling the description of transition flows in porous media. Interpretation of these results will require the independent characterization of the medium in terms of pore size and configuration. Such characterization will be accomplished by a combination of optical and displacement methods and by a knowledge of the size and configuration of particles (beads, rods, etc.) used for the preparation of each sample. Materials of various descriptions will be formed into specimen slabs, with sintering being viewed as the most promising technique at this time. Thus, one may summarize by stating that the next phase of our activity will consist of four essential parts: 1) the preparation of suitable samples, 2) the physical characterization of these samples, 3) the measurement of flow characteristics, and 4) the interpretation of results.