

RESEARCH MEMORANDUM

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SMALL VOLUMES OF A GAS

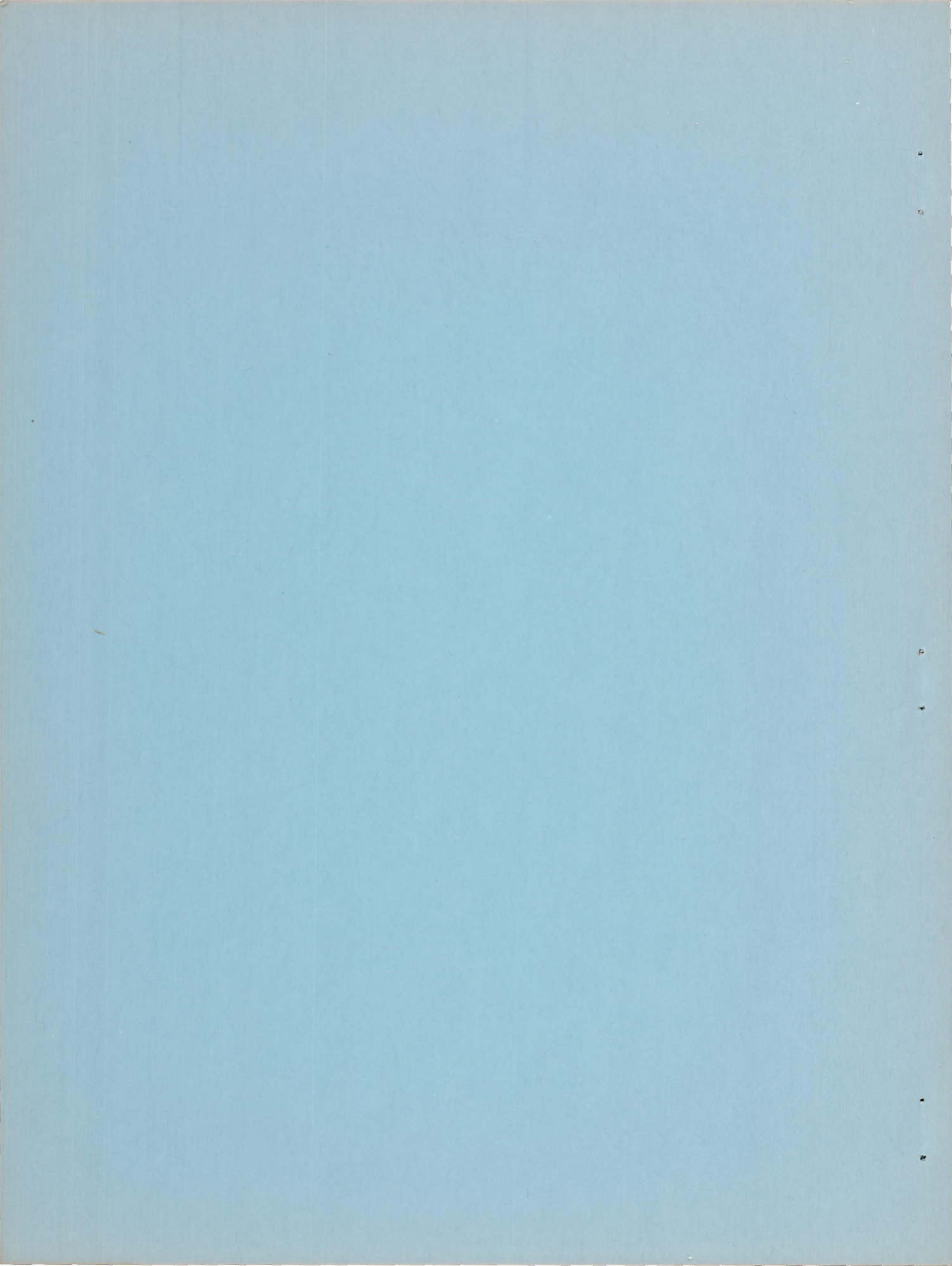
By J. C. Westmoreland

National Bureau of Standards

NATIONAL ADVISORY COMMITTEE
FOR AERONAUTICS

WASHINGTON

October 17, 1952



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SUMMARY

A flowmeter has been developed which combines a volume-time measurement of the flow, regulation of the rate, and a determination or presetting of the pressure head producing the flow. This meter is capable of accurately metering and controlling very small rates of gas flow within a closed system such as those required in a small capillary tube gas viscosimeter.

INTRODUCTION

For several years a research program on the viscosities of gases has been in progress in the Capacity, Density and Fluid Meters Section of the National Bureau of Standards. This research is based on the use of the capillary tube method. In this connection, it is customary to use the classical equation of Poiseuille (reference 1) for expressing the flow of a fluid through a capillary tube. In its simplest form this equation is

$$Q = \frac{\pi R^4}{\mu 8L} h \quad (1)$$

where

R radius of tube, cm

L length of tube, cm

h pressure drop from inlet to outlet of tube, g/cm - sec²

Q volume rate of flow at midsection of tube, cm³/sec

μ viscosity of gas, poises

The term $\pi R^4/8L$ is a constant for any one tube and was determined by the method of Fisher (references 2 and 3). Thus, in order to determine the viscosity of a gas, it is necessary to measure, with suitable equipment, the quantities h and Q .

It will be noted that in equation (1) the quantities h and Q both appear in the first power. Hence, an uncertainty in the observed value of either h or Q will produce an uncertainty in μ of the same magnitude. As it was the objective of the current research on gas viscosities to keep the uncertainties in μ as small as possible, it seemed desirable to use some form of metering unit incorporating a volumetric displacement, because such a volume could be evaluated by conventional methods and would not involve the measurement of an auxiliary quantity. As a flowmetering device which would be capable of accurately measuring extremely small gas flow rates was not available, a meter has been developed that fulfills the above requirement and that, in addition, incorporates a means of regulating the rate of flow and of measuring or preestablishing the pressure head h .

This work was conducted at the National Bureau of Standards under the sponsorship and with the financial assistance of the National Advisory Committee for Aeronautics.

DETAILED DESCRIPTION OF METER

The principal parts of the meter and their relative positions are shown schematically in figure 1. Figure 2 shows some of the details of the float valve and valve chamber, while figure 3 shows a completely assembled meter. A list of the component parts of the meter follows.

The volume chambers A-A' are made of glass with a necked section between the two and at the base of the lower. Platinum contact wires M, N, and O are sealed into these neck sections and the top of the upper bulb A'. These wires are bent downward to give sharp "break" signals. The primary purpose of the necked sections is to have a small cross-sectional area at the plane of the contact break.

There are no special requirements or limitations on the volume of the lower chamber A insofar as the operation of the meter is concerned. In the several models which have been made, this volume has ranged from about 25 cubic centimeters to about 100 cubic centimeters. Likewise, the volume of the upper bulb A' is not subject to close limitations. The principal purpose of this upper volume is to provide a starting period during which flow conditions may be stabilized.

The lower neck of the volume chamber A sockets into the thick top section of the float chamber B. From this socket there is a small diameter outlet into the float chamber. The lower end of this outlet passage is beveled at 60° to form a seat for the coned point of the valve.

The valve float is made enough smaller than the chamber that it will move freely within the chamber and still retain a central position. The bottom of the float is flat, but the upper surface is coned inward at 30° to 45° . There is a central hole through the float large enough to insure free passage of the mercury. The valve D is made from triangular stock with a 60° coned point. It is mounted on the coned surface of the float and coaxially with it and is cemented in this position.

The float chamber, float, and valve are made of methyl methacrylate. It has been found that a methyl-methacrylate valve and valve seat are not eroded by the flowing mercury, whereas a steel valve was. Moreover, the mercury remains clean in contact with the methyl methacrylate.

In the outlet tube from the bottom of the float chamber, there is the main control valve F. This valve may be of the simple on-off type, or it may be of a type that is adjusted and indexed for varying amounts of opening. The advantages of these two types will be discussed in later sections. From this valve the mercury is discharged into the reservoir E. This reservoir may be constructed to serve as a base for the entire unit, or it may be a separate vessel.

It is necessary that the passage from B to E, especially at the outlet end, shall be very small. In some meters the lower part of this tube has been made so small that, with a trap at the end, only one or two drops of mercury emerged from the trap per second. That the height from the mercury surface in the float chamber to the tube outlet is equal to the pressure drop across the capillary tube has been established by many pairs of measurements. However, it should be noted that this holds only when cock F is wide open. If, as in some models, cock F is replaced by a needle valve, the equality between the height of the mercury passage and the pressure drop will not obtain. It will then become necessary to use a manometer across the capillary tube.

Other features of the meter include the pressure equalization tube K between the top volume chamber A' and the top of the float chamber C and the mercury charging connection L from the bottom of the reservoir to the passage above the float valve which includes valve G. In figure 2 the pressure equalization tube K and the mercury charging connection L are shown joined in one single adapter. There are also the nipples 1 and 2 between which the capillary tube viscosimeter is connected; the gas supply connection 3, and the purging or vacuum connection 4. Additional connections, which are used sometimes but which are not shown in

figure 1, are: Pressure connections at 1 and 2 between which a differential pressure gage may be connected; an auxiliary pressure connection to the top of the reservoir E which may be branched to provide a connection for a static-pressure gage; also, an additional valve may be placed close to E in the connection to 1 and I.

One arrangement used for determining the volumes A and A' is represented in figure 4. In making this calibration it is always necessary to have the mercury surface falling when the observations are made because this is the direction in which the mercury surface will be moving when the meter is in use.

It is appropriate to mention that the materials of construction and method of assembly described above are suitable for use under gas pressure (absolute), ranging from a few millimeters of mercury to about 2 atmospheres. Modifications, which will permit the meter to be used under higher pressures, will be discussed later.

In order to use the meter, several items of auxiliary equipment will be required. One of these is an electric timing clock. However, since the current through the meter contact leads must be kept very low, not over 10 microamperes, a vacuum-tube relay unit was made through which the clock is started and stopped. Some form of pressure gage will be needed for indicating the pressure of the gas in the system. This may be a simple mercury column supplemented with a barometer. Also, a differential pressure gage (manometer) or a cathetometer may be needed to measure the pressure drop across the capillary tube.

OPERATION

With all of the mercury in the reservoir, valve I closed, and cocks F, G, and H open, a high vacuum pump is connected at 4 and the entire unit purged of all gas as completely as possible. Then, cocks F and H are closed and the gas-sample inlet valve I is opened. This must be done with some care since the rate of pressure increase in E must not be too rapid, and yet it must be at a much greater rate than gas will flow through the capillary tube. This will cause mercury to rise through the tube L into the passage above the float valve. Since F is closed, the mercury will first fill the float chamber enough to close the float valve, and thereafter will fill the volume chambers A and A'. When the mercury has reached contact O, cock G is closed. The inflow of gas is continued until the pressure within E has reached the desired value, when valve I is closed.¹

¹As mentioned above, the static-pressure gage and its connection to E are not shown in figure 1.

Before starting a measurement or run, a sufficient time interval must elapse for pressure conditions to equalize throughout the system. The following conditions then exist: Cocks F and G are closed. The mercury in the float chamber has raised the float so that valve D is pressed firmly enough against its seat that no mercury can flow from A into B. The gas pressure is the same in E, the float chamber B, and the top of A'.

To place the meter in operation, cock F is opened. The set of conditions which will be established when a steady rate of flow is reached are: The outflow of mercury from B will lower the float and valve admitting mercury from A into B. If this inflow from A exceeds the outflow, the float and valve will be raised, choking down the rate of inflow. Because of the pressure equalizing tube K the gas pressure in the upper part of the valve chamber B will be the same as that above the mercury in A-A'. The head h producing the flow is equal to the vertical height from the surface of the mercury in the float chamber to the outflow point of the tube below F. This may be determined by the use of a cathetometer or by a differential manometer connected between the capillary tube terminals 1 and 2.

Again, it has been found that there is no "hunting" action of the float valve in maintaining the rate of outflow from A equal to that from B. However, the flow through the float valve must be in the laminar region.

Since the gas pressure in the float chamber is the same as that above the mercury in A-A', the pressure or force of the mercury upon the projected area of the point of the valve will vary with the height of the mercury in A-A'. The opposing force exerted by the valve is derived from the buoyant action of the mercury upon the float. As this force must be greater when A-A' is full than when nearly empty, it follows that the depth of the mercury in B will be greater at the start than at the end of a run. This change in depth in B is also attributed to the fact that the force that causes the flow of mercury from A to B through the restriction at the valve seat decreases as a run progresses. To sustain a constant flow of mercury through the valve port with a progressively lower driving force the restriction must widen. Since mercury flow out of chamber B into chamber E tends to exceed that from A into B, the mercury depth in B decreases sufficiently to produce this widening of the valve passage. Calculations have shown that this change in depth is very small, about 0.0014 centimeter. In the model on which this value applies, this would be equivalent to a decrease in h of less than 0.01 percent.

COMMENTS ON THE USE AND CONSTRUCTION MODIFICATIONS

In the description of the meter operation, valve F was treated as being of a full-open or full-closed type. For convenience this has been called "unrestricted" operation. For this type of operation, the rate of flow of the mercury, at equilibrium, will be determined by the quantity of gas that the pressure differential h can force through the capillary restriction J at the test conditions. If the capillary J were blocked, then no gas could enter the metering chamber and there would be no mercury flow into the chamber E. A hydrostatic balance would then exist where the gas pressure P_E in the chamber E less the pressure P_B in the measuring chamber A-A' and the float chamber B would be equal to the value of the mercury column h . Mercury flow rates may then be obtained from zero to some critical value where pressure losses in the discharge section are not negligible. The critical rate is governed by the shape and size of the discharge section.

This point is illustrated by the curves in figure 5 where comparative performance data are shown for a bent-neck and a straight-neck meter by plotting values of $P_E - P_B$ against the mercury discharge in cubic centimeters per second. The bent- or "goose-neck" mercury outlet tube is shown in figures 3 and 6 extending upward from the valves marked F.

In place of an on-off valve, a regulating valve may be used for F. In this case, the outflow tube should be of uniform size throughout. Also, the vertical distance represented by h in figure 1 would not be a measure of the pressure drop across the capillary tube. This pressure drop would now have to be measured with a manometer. Operation of the meter with this type of a valve has been called "restricted" operation.

It was mentioned that, with the type of construction described, the maximum pressure at which the meter can be used with safety is about 2 atmospheres, absolute. One method for overcoming this limitation is to place the meter within a suitable pressure chamber. Such a pressure chamber, shown in figure 6, has been used under pressures up to 5000 pounds per square inch.

Since the viscosity of a gas changes rapidly with a change of temperature, one of these meters may be used for making temperature determinations, especially at elevated levels where thermoelectric means have proven unreliable. For such use, the capillary tube would be coiled into a compact sensing unit. The unit would be charged once for all with a suitable gas. The time interval will be a measure of the average relative temperature, or actual temperature after a calibration has been made, over the time interval.

The meter has been modified so as to indicate the rate of flow of a gas sample. This modification, which is shown schematically in figure 7, is made by replacing the chamber unit A-A' with a uniformly bored tube. The contact points are replaced by a fine platinum resistance wire mounted along the axis of the tube. The ends of this wire are connected to the primary winding of an iron-core transformer and supplied from a source of direct current. As the mercury surface falls, the resistance in the circuit increases with a corresponding drop in the primary current and degree of magnetization of the primary winding. This induces an electromotive force in the secondary winding, the strength of which will depend upon the rate of change in the primary. This secondary-winding voltage may be obtained with a suitable type of voltmeter and, by a calibration, the volume rate of mercury flow and of the gas also may be read directly from the voltmeter.

Obviously, this arrangement can be combined with the temperature sensing element mentioned above. In this case, the voltmeter could be calibrated to indicate temperature, since the rate would vary proportionally with the temperature of the gas.

Another possible use of the meter would be in connection with determinations of the specific gravity of a gas by the effusion method. For this purpose, the capillary tube would be replaced by a thin metal diaphragm in which there is a small orifice of 0.008- to 0.012-inch diameter.

PERFORMANCE

For the purpose of discussion, a discharge coefficient C for this meter is defined as being the ratio of the measured pressure difference $P_E - P_B$ to the mercury column h , that is,

$$C = \frac{P_E - P_B}{h}$$

The following table shows a series of runs with which this meter was employed in a gas viscosimeter. The coefficient C is shown as defined above; the delivery time t for the particular volume indicated and the Reynolds number of the gas in the gas inlet connection are also given.

Run	Discharge time (sec)	Discharge coefficient, C	Reynolds number
Series 1 Volume of metering chamber A, 52.39 cm ³			
1	172.22	0.9867	10.68
2	172.20	.9867	10.68
3	172.29	.9867	10.68
4	172.36	.9867	10.68
5	172.49	.9867	10.68
6	172.58	.9867	10.68
Series 2 Volume of metering chamber A, 52.39 cm ³			
1	277.70	1.000	6.39
2	277.23	1.000	6.39
3	277.45	1.000	6.39
4	277.69	1.000	6.39
5	277.40	1.000	6.39
6	277.80	1.000	6.39

The difference in delivery rates has been attributed to changes in temperature that were not indicated as they affected the viscosity of the gas in the capillary tube which is the variable that was being measured.

ACCURACY OF METER

In considering the parameters associated with the operation of the flowmeter, the following compilation of accuracies gives the total accuracy at which the meter has been observed to perform:

Accuracy, percent, of:

Volume chamber A	0.10
Time measurement	0.05
Differential head	0.20

Absolute accuracy, percent	0.35
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DISCUSSION

The order of magnitude of the gas flows that are being metered is the same order as those which occur in convection currents set up by small temperature gradients within a gas system. In some regulating-meter systems, this phenomenon would prove a distinct disadvantage and would present a serious source for control drift. The fact that the present meter operates at only one pressure differential by design results in continuous response to any pressure fluctuations. The movement of gas into the meter itself is the controlling factor for flow regulation, a fact which makes the device self-regulating.

The dynamics and structural effects of very small head meters such as nozzles and orifices do not seem to have been thoroughly investigated; however, consideration of the theoretical basis on which these meters operate leads to the conclusion that their calibration would be a function of the gas employed and its state at the time of calibration. This would necessitate a complete family of calibration curves for each size of meter used. It may be added also that the flowmeter here described has been utilized in studying the performance of some of these small-head meters. The results of these studies indicate that the small-head meters do not have the desired degree of repeatability.

In metering extremely small rates of gas there is very little kinetic energy available in the gas stream for use in driving a meter of the positive displacement type such as a wet test meter. The driving forces and the balancing forces have to be necessarily small; this results in a delicately balanced force system that is subject to considerable hunting action.

When the flowmeter regulator herein described is employed in the unrestricted flow manner, it provides a constant driving force (motor construction) within a gas system that is capable of inducing a gas flow through the system that is susceptible to this driving force. By this means, no energy from the gas is required for its motivation, nor does there have to be an energy transformation within the gas stream to indicate the magnitude of flow. A total flow measurement is deduced from a volume-time measurement. This principle of operation has made it possible to meter accurately volumetric flow rates of gas up to 500 atmospheres pressure.

National Bureau of Standards

Washington 25, D. C., April 17, 1952

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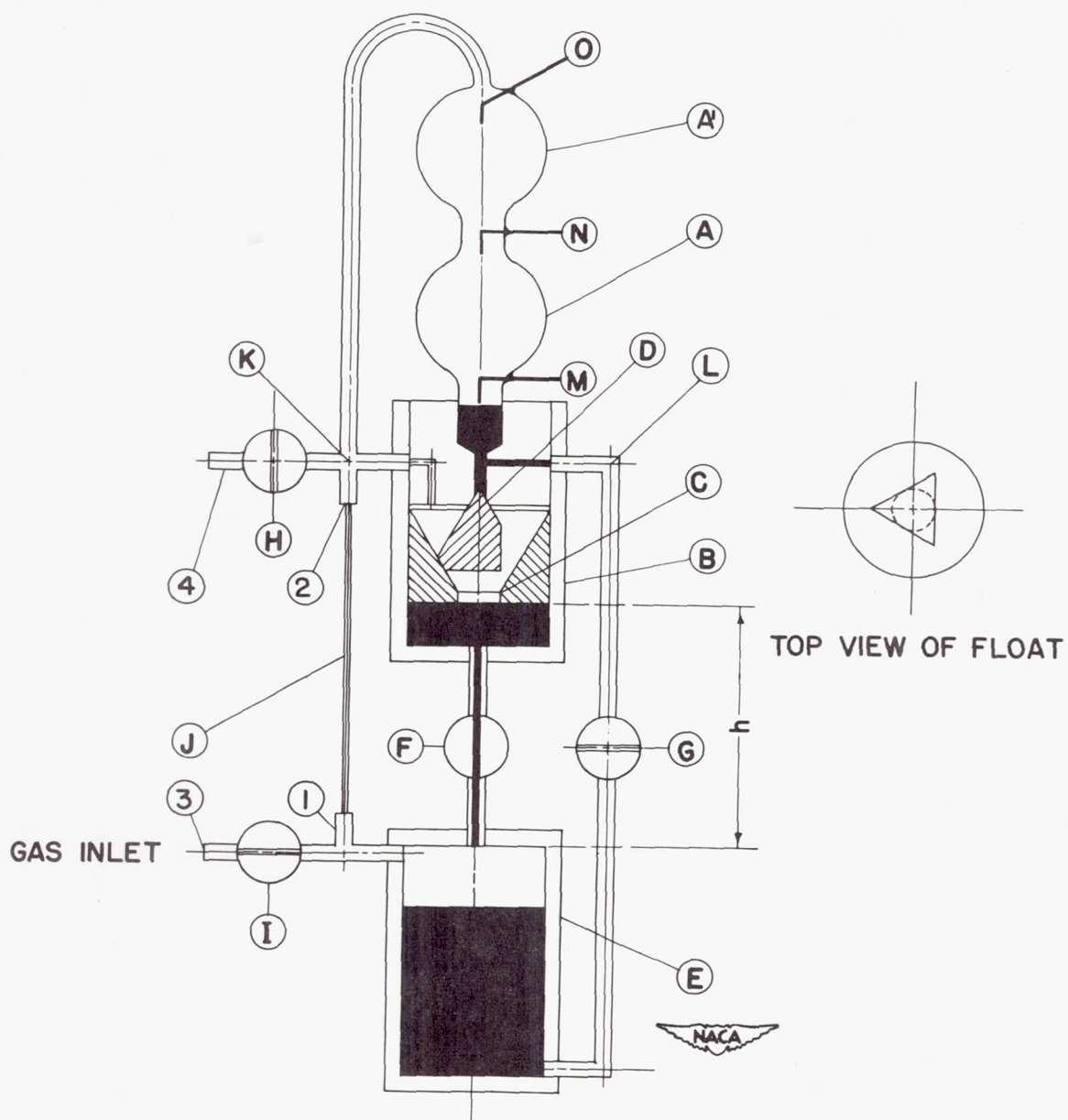


Figure 1.- Schematic diagram of the meter regulator.

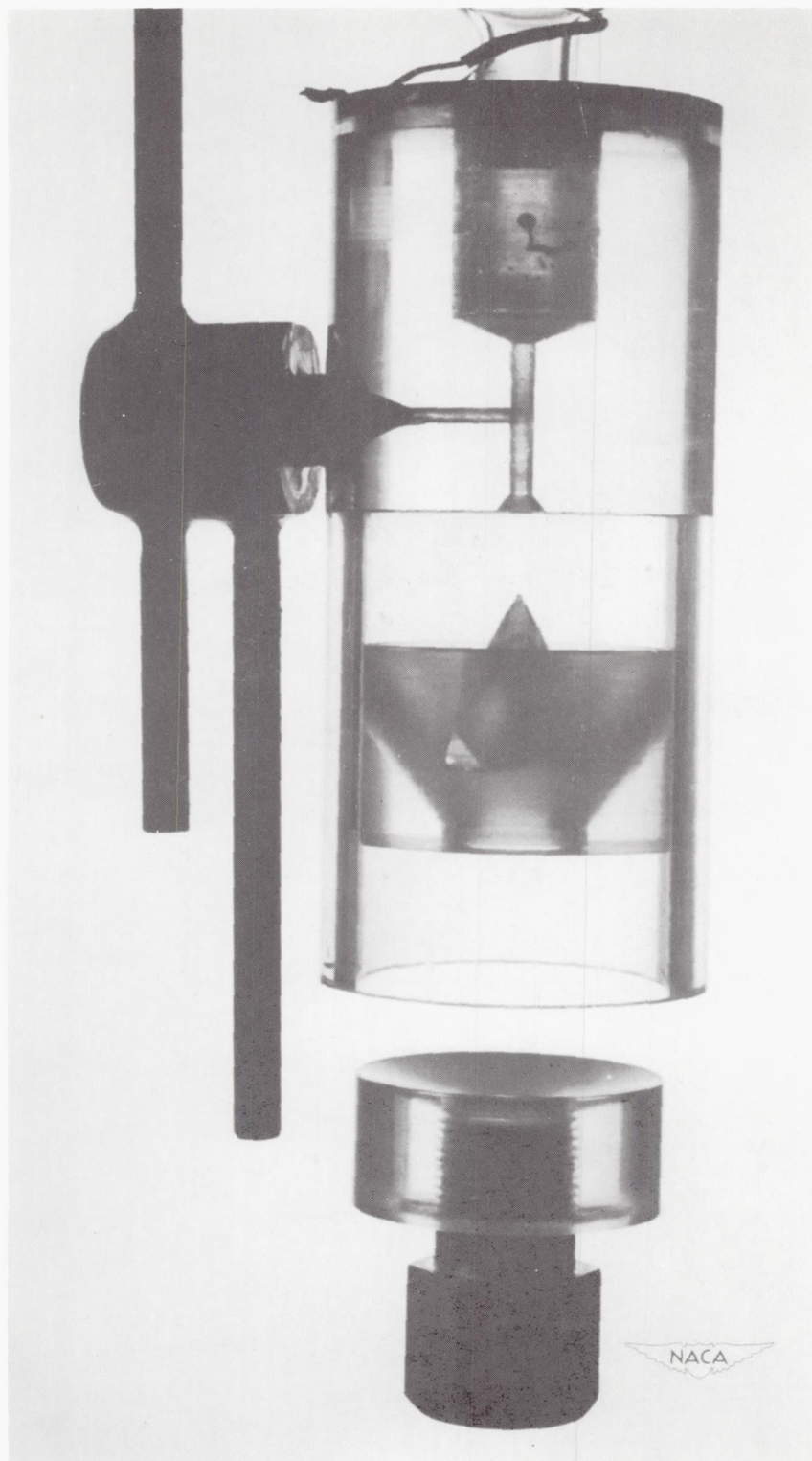


Figure 2.- Float chamber showing float, valve, and valve seat.

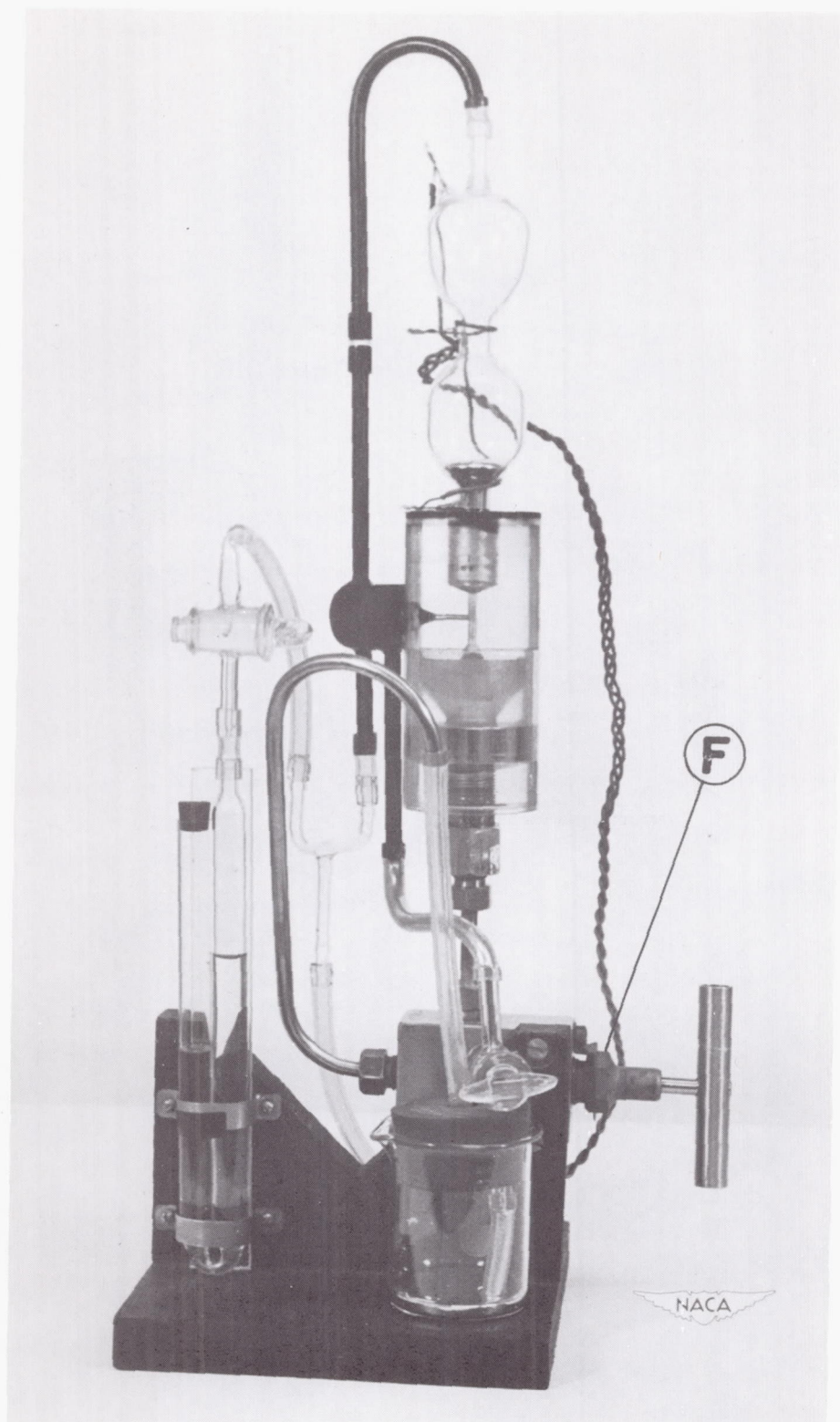


Figure 3.- One of the meters in operation.

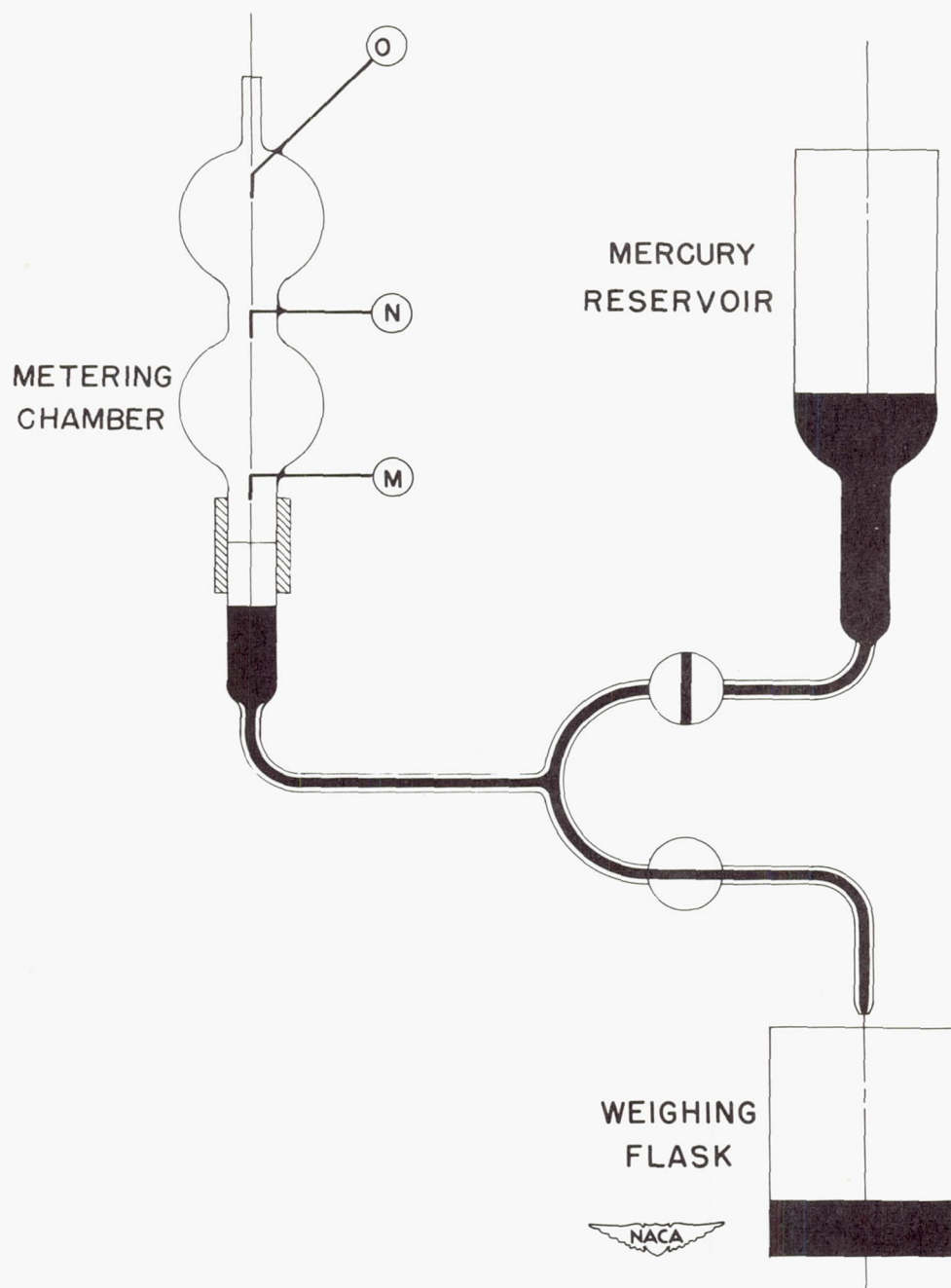


Figure 4.- One method of calibrating volume chambers.

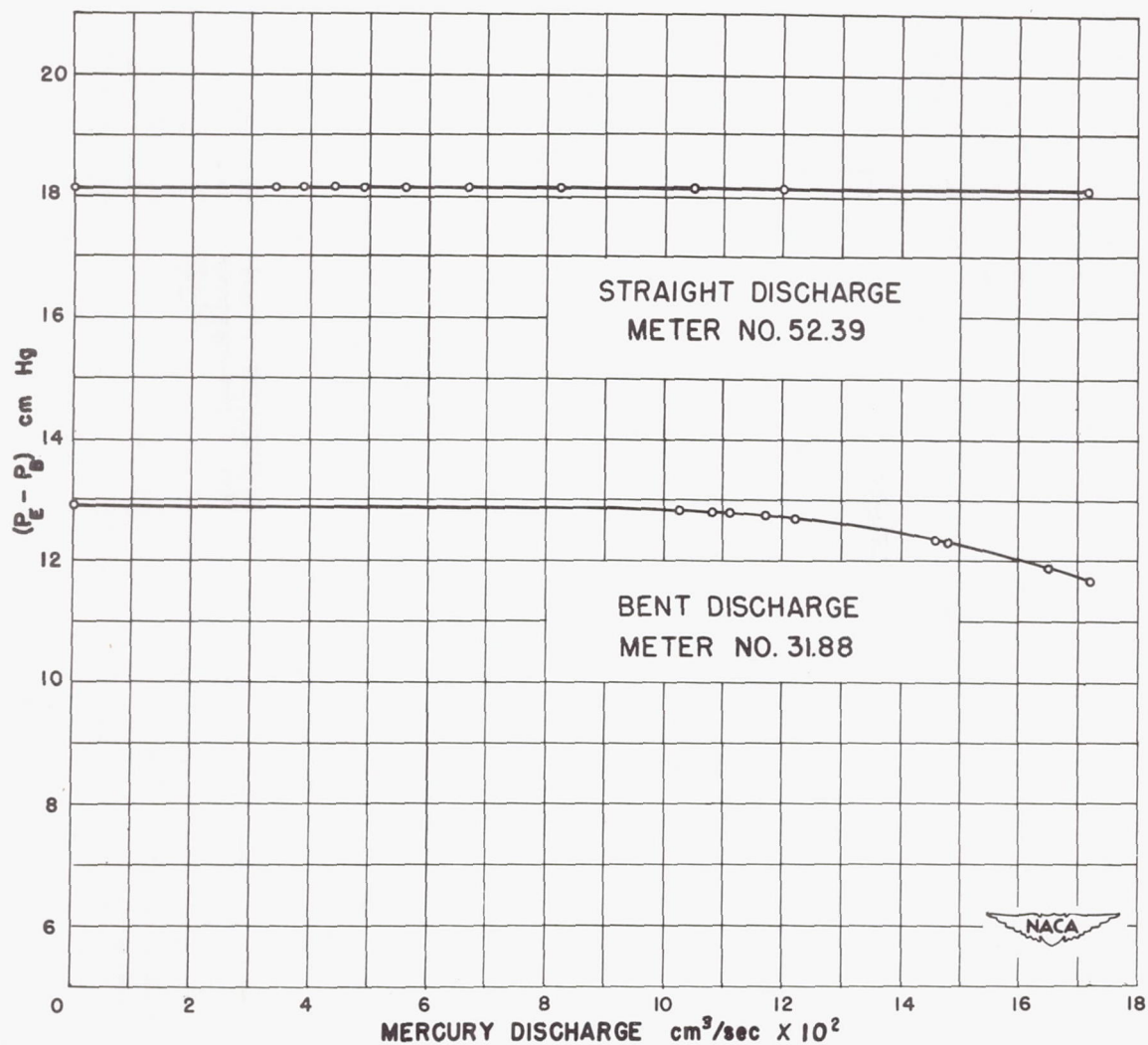


Figure 5.- Performance curves of two types of meter discharge connections.

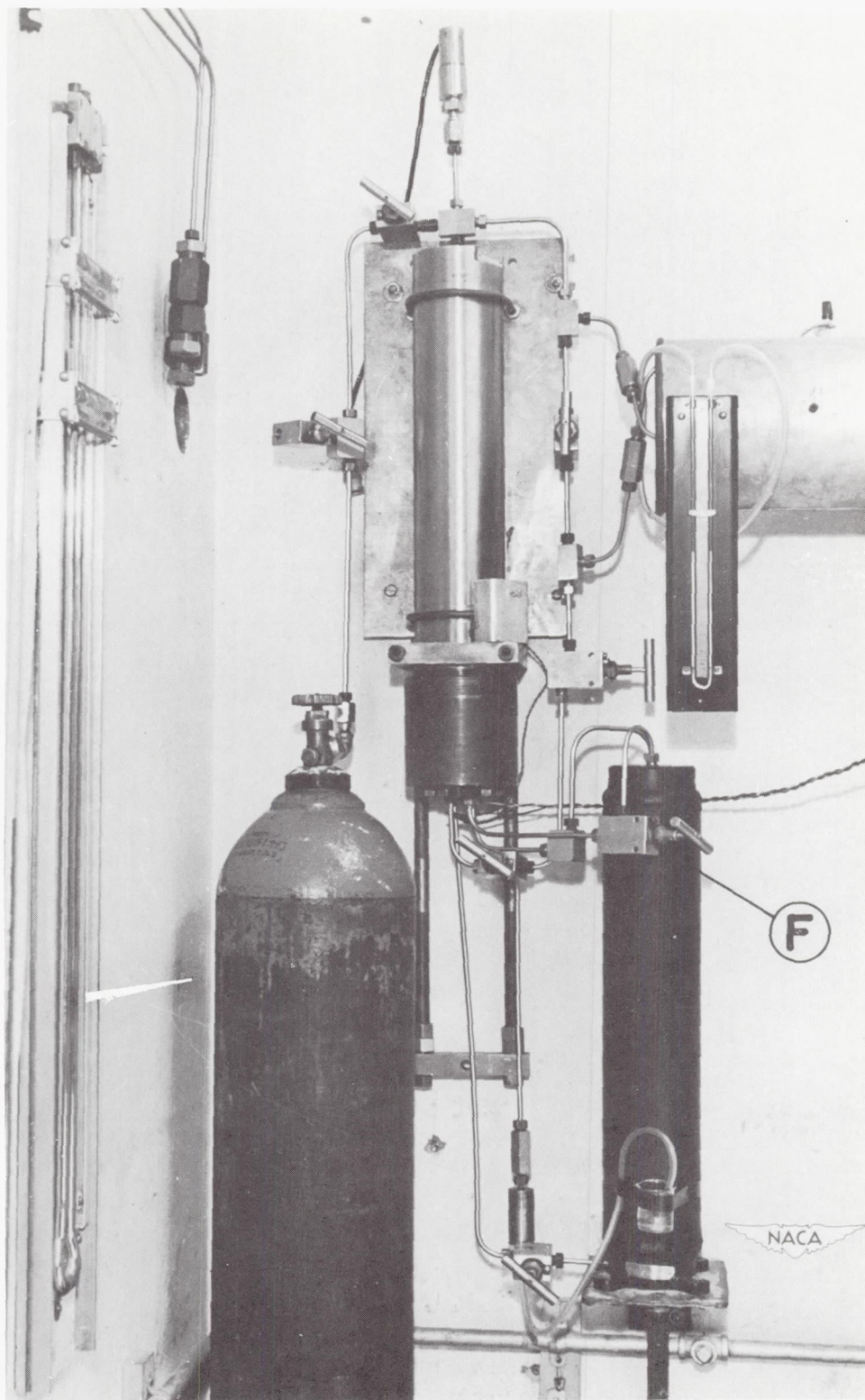


Figure 6.- Enclosure in which a meter is installed for use at high pressures.

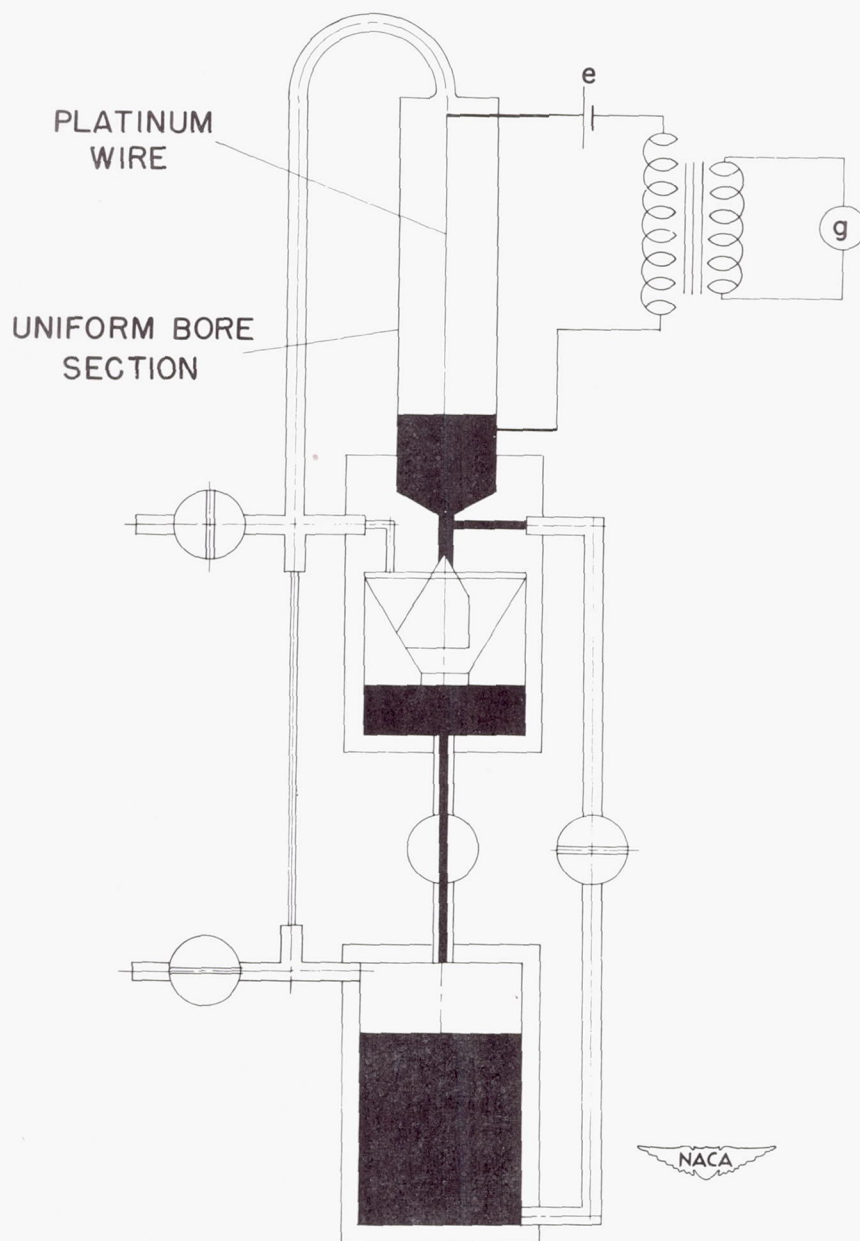


Figure 7.- Straight-tube modification.

