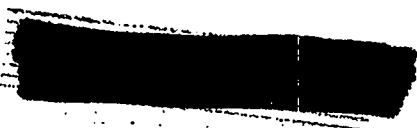C.3

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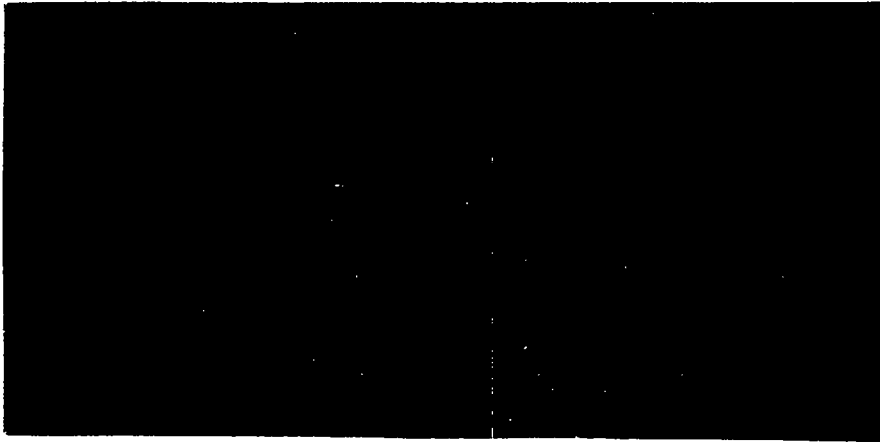
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Report written:
   June 1954

Report distributed: AUG 1 7 1954

PUBLICLY RELEASABLE
Per _M. Jain_ , FSS-16 Date: _10-13-95_
By _Marin Bailey_ CIC-14 Date: _11-13-95_

LA-1683

THE CALIBRATION OF TRITIUM
MONITORING DEVICES

Classification changed to UNCLASSIFIED
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INSTRUMENTATION

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ABSTRACT

The increased use of tritium monitoring devices has made it necessary to devise a scheme for the calibration of such instruments. Several methods for setting up known concentrations of tritium were considered. A dynamic system containing tritium as HTO vapor or gaseous HT was selected because of the ease of control of variables. The variables that have been considered are:

1. The effect of rate of air flow through the instruments.

2. The effect of humidity on instrument response.

3. The effect of ion collection ahead of the ion chamber.

4. The variation of "hold time" from the generation of tritium to its passage through the ion chamber.

5. The instrument response to several concentrations of tritium as HTO vapor and gaseous HT.

The following three types of monitoring devices have been calibrated:

1. Model 101 Sniffer of Group P-1.

2. CI-1 Vibrating Reed Electrometer of Group CMR-7.

3. Modified T1B of Group H-6.

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Experimental

The generation of a known concentration of tritium was accomplished by saturation of an air stream with water containing a known amount of tritium.[1] Gaseous tritium was generated by passage of this air stream through metallic zinc at high temperatures.

Figure 1 is a schematic drawing of the calibration equipment. The direction of air flow is indicated by the arrows. The stream of air, or nitrogen (K), was passed through a rotameter (E) at a constant back pressure reading (manometer F) and dried in a calcium chloride drying tower (G_1). The dried air was saturated with water containing tritium oxide by passing it through fritted glass bubblers (J) at a constant temperature. The saturated air next flowed through the by-pass (H), or through the zinc furnace (I) when gaseous tritium was desired, and into the mixing chamber (M).

The diluting air was supplied by a Sutor-Bilt pump (O). The diluting-air volume was controlled by a bleeder valve (N) and measured by an orifice meter (L). When dry air was used, the known volume of diluting air was dried in a calcium chloride drying tower (G_2). When high humidities were desired, this same tower could be converted to a humidifier by filling it with wet glass wool. The diluting air, dried or humidified, then passed into the mixing chamber (M) where it was mixed with the air containing tritium.

This mixed air, of known volume and tritium concentration, passed from the mixing chamber directly through the monitoring device (C), or if desired, could be held an extra length of time in a by-pass consisting of two 55-gallon drums (D). For certain information, the tritium water vapor was collected in the liquid air traps (B). The entire system was exhausted through a 4-inch duct (A) into the room hood. Figure 2 is a photograph of the equipment (without the liquid air traps).

Saturation of the air stream through the tritium water was checked by dew point measurements. The values obtained showed 98 to 99 per cent saturation for volumes up to three liters of air per minute through the water.

Collection of the tritium water vapor from the apparatus was made possible by utilizing liquid air traps. Recovery of the tritium water appeared to be a function of the time elapsed between removal of the liquid air and washing the contents of the traps into stoppered flasks. Enough evidence was gained to assure that the calculated tritium concentrations being introduced into the system were, in fact, the actual concentrations.

Assuming that the liquid air traps would collect none of the tritium existing as HT, the above procedure was used to check the conversion of tritium water vapor to the gas in the zinc furnace. Under the experimental conditions employed, it was observed that the efficiency of conversion amounted to 60 per cent.

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Model 101 Sniffer

Although the Model 101 Sniffer was designed primarily as an alarm device, it was nec-
essary to have the instrument calibrated in terms of tritium concentration for routine monitoring
procedures. The Sniffer, shown in Fig. 3, is a simple ion chamber with a collecting voltage
of 90 volts, through which air is drawn at a known rate. The ionization current of the air is
monitored by an electrometer tube circuit and read directly on the microammeter. The sensi-
tivity of the instrument ranges from 10^{-9} ampere on the 2 scale to 10^{-12} ampere on the 5 scale;
the 1 scale is the zero-set scale. The switch at the end of the ion chamber is used to select
the appropriate sensitivity scale. Figure 4 shows the Sniffer in position for calibration.

In the calibration of the Model 101 Sniffer, one of the most stubborn problems to be over-
come was the buildup of static charge on the glass mixing chamber. This phenomenon caused
the mixing chamber to act as an ion trap. The difficulty was overcome by wrapping the cham-
ber with aluminum foil and suitably grounding it.

The preliminary calibrations were confusing because the concentrations of tritium nec-
essary for instrument readings did not agree in orders of magnitude with previous theoretical
calculations or with a rough calibration performed in 1951.[2] It was then pointed out by E. A.
Bemis of Group H-6 that the "hold time" from the generation of tritium to the passage of tritium
contaminated air through the Sniffer was of utmost importance. An equilibrium concentration
of ions was not being realized in the 1.9 seconds that the tritium contacted the air before pas-
sage through the ion chamber. Wait and Parkinson[3] have experimental data to show that the
mean life of both small and large ions in the atmosphere will range from 5 to 20 minutes.
(The mean life of an ion is the average time interval between formation and recombination of
the ion.) Figure 5 shows a plot of Sniffer readings for given concentrations of tritium against
"hold time" in seconds. The "hold time" was varied by means of two 55-gallon solvent drums
with a combined volume of 0.4 cubic meter. The mixing chamber volume was 0.012 cubic
meter, and by means of valves the ionized air at a given rate of flow could be held for three
lengths of time, i.e., in the mixing chamber and in one or both 55-gallon drums.

For convenience, 85.9 seconds was the time selected as the point to make all the calibra-
tions. Figure 5 shows that this time represented more than 80 per cent of the ionization
equilibrium condition. All the calibrations with the Sniffer were performed at an air flow rate
of 0.3 cubic meter per minute. This is the normal rate of air flow for the fan now being
used on the Sniffer.

The effect of change in air flow rate through the instrument for given tritium concentrations
is shown in Fig. 6. It will be noted that the change in Sniffer reading amounts to about 20
per cent with a change in air flow rate from 0.3 cubic meter per minute to 0.15 cubic meter

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per minute. The reading drops off rapidly below an air flow rate of 0.15 cubic meter per minute. The collection of lint and dirt on the ion chamber screen is the principal cause of a diminished air flow rate and it is important that the screen be kept clean.

Figures 7 and 8 show the average calibration curves for five different Sniffers, both battery- and AC-operated, on the 4 and 5 scales. It is interesting to note that the 4 scale does not appear to be a true decade function of the 5 scale. Calibrations were not made on scales 2 and 3. Values obtained on scales 2 and 3 would be far in excess of the air tolerance figure and, as a rule, would not be used in routine monitoring procedures. For practical purposes, it might be convenient to assume that these scales are decade functions of scale 4.

All the calibrations were based upon an average relative humidity value of 30 per cent. It was of interest to increase the relative humidity to 70 to 75 per cent and perform the same calibrations. The increase in humidity was accomplished by converting the drying tower ahead of the mixing chamber into a humidifier. No significant difference in Sniffer readings was seen for a given tritium concentration at the increased humidity.

A 90-volt ion trap was inserted ahead of the Sniffer's ion chamber and calibrations were performed. The ion trap collected a large percentage (greater than 90 per cent) of the ionization produced by the tritium before reaching the Sniffer. As a consequence, the sensitivity of the Sniffer is decreased as it then responds only to the ionization which the tritium produces in its short transit time through the ion chamber. The calibration curve is given in Fig. 9.

Figure 10 shows a calibration curve for tritium as 100 per cent HTO and as a gaseous mixture of 60 per cent HT and 40 per cent HTO. It seemed apparent that the instrument's response was the same for tritium existing in both states.

CI-1 Vibrating Reed Electrometer Continuous Tritium Monitor

This monitor shown in Fig. 11 is described in detail in LA-1270.[4] It is unlike the Model 101 Sniffer in that it only records ionization produced by the tritium in the ion chamber. Figure 12 shows a photograph of the instrument adapted to the calibration system.

On calibrating this instrument, it was found that two hours were required for the electrometer to indicate tritium (as HTO) in a given atmosphere, with the CWS E4R3 canister filter ahead of the ion chamber. This long time might be explained by the uptake, on the filter, of atmospheric water vapor containing HTO. Apparently the atmospheric contamination is not reflected by the instrument until a water vapor equilibrium is established across the filter. Glass wool, substituted for the canister filter, reduced this equilibrium time to less than two minutes; with no filter medium ahead of the ion chamber the time required for a maximum electrometer reading is reduced to less than one minute. All these data were obtained at an air flow rate of one cubic foot per minute.

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It is obvious that if any filtration of room dusts is desired then glass wool is the medium of choice. Neither glass wool nor the canister filter will completely trap ions of cigarette smoke or welding operations.

Figure 13 shows the instrument response for several concentrations of tritium at different rates of air flow. These data confirm the statement made in LA-1270 that "the ionization current collected is independent, within wide limits, of the air flow rate." With a glass wool filter ahead of the ion chamber, the fan pulls 1 to 1.5 cubic feet of air per minute.

Good correlation was noted with the theoretical calculations of Deal[4] and the previously observed results of Anderson, Pinson, and Lotz.[5] These previous results indicated that 84 microcuries of tritium per cubic meter of air should give a full scale reading on the 100 scale. The calibration results obtained here showed a full scale reading at 85 microcuries per cubic meter of air on the same scale. Figure 14 is a calibration curve of the 100 scale.

T1B Modification

This modification of the standard T1B was designed to record tritium in the presence of other ionizing radiation. Although it had a 135-volt ion collector ahead of the ion chamber, its sensitivity to tritium was about the same as that of the Model 101. A decrease in air flow rate through the instrument caused an increase in sensitivity as shown in Fig. 15. Only the prototype of this monitor has been calibrated.

Conclusions

Variations in humidity appear to have no effect on the response of the several instruments. For any specific tritium concentration, the Model 101 Sniffer gave identical readings for tritium existing at 100 per cent HTO or a gaseous mixture of 60 per cent HT and 40 per cent HTO. Model 101 and the T1B of Group H-6 have responses that are dependent upon air flow through the chamber for a given concentration of tritium whereas the CI-1 of Group CMR-7 is independent of air flow over a range from 0 to 5 cubic feet per minute. Ion collection ahead of the Model 101 allows it to be used for tritium monitoring in the presence of other ionizing radiation.

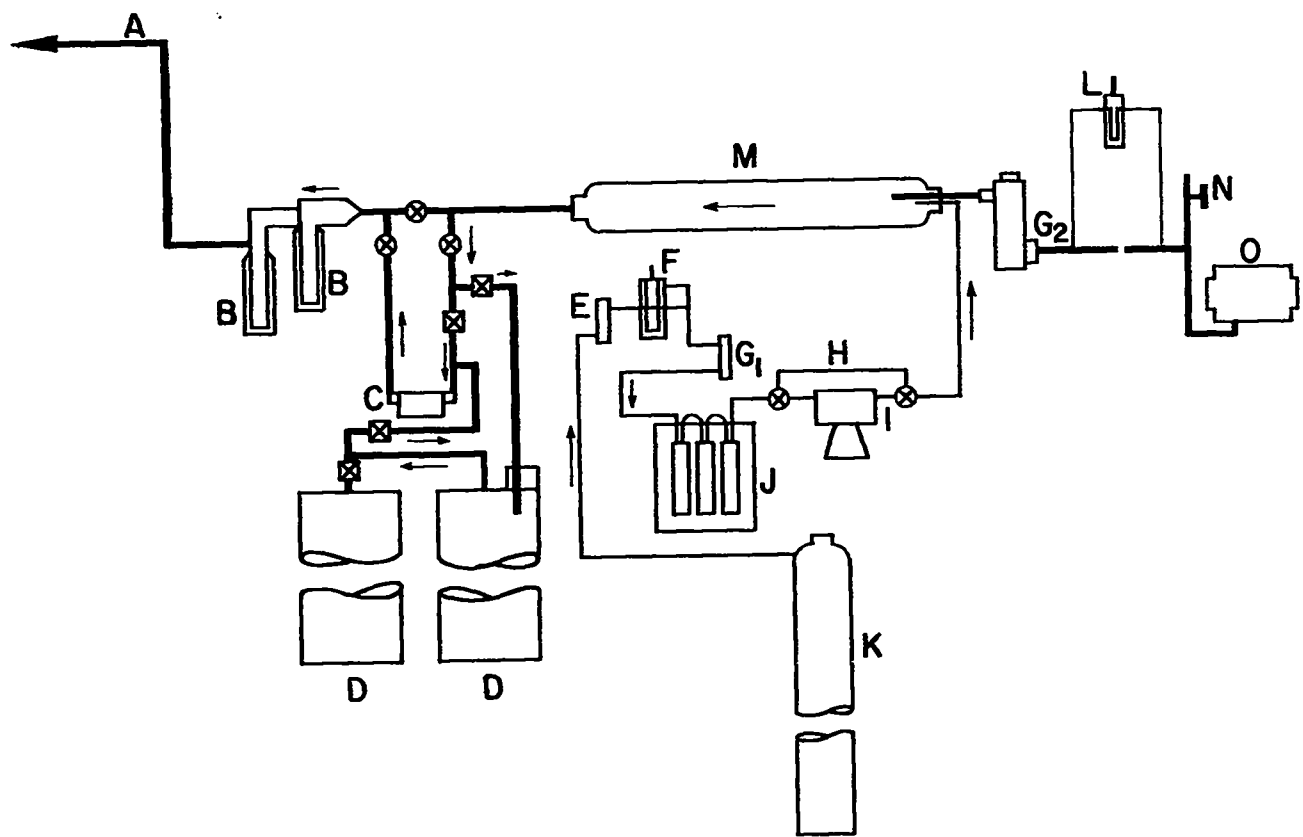The instruments calibrated showed good reproducibility from day to day as well as from one instrument to another.

Acknowledgments

Evan Campbell of Group H-5 and Felix Vigil of Group H-1 assisted us in the completion of this work.

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References

1. Tritium content of the water standards was determined by the method of McClelland and Milligan. Jean McClelland and M. F. Milligan, "Determination of Tritium in Urine and Water," Los Alamos Scientific Laboratory Report LA-1645, March 1954.

2. Private communication, Frank Dunn with Richard Watts.

3. Thomas F. Malone, editor, Compendium of Meteorology, pp. 120-127, Waverly Press, Baltimore, Maryland, 1951.

4. James B. Deal, Jr., "Continuous Tritium Monitor," Los Alamos Scientific Laboratory Report LA-1270, Feb. 15, 1951.

5. Private communication with Virginia Lotz.

A EXHAUST DUCT

B LIQUID AIR TRAPS

C TRITIUM MONITORING DEVICE

D 55 GAL. DRUMS $.4M^3$ VOLUME

E ROTAMETER

F BACK PRESSURE GAUGE

G_1-G_2 DRYING TOWERS

H BY-PASS

I ZINC FURNACE

J BUBBLERS IN CONSTANT TEMPERATURE BATH

K NITROGEN SIZE D CYLINDER

L ORIFICE METER

M MIXING CHAMBER $.01M^3$ VOLUME

N BLEEDER VALVE

O SUTOR-BILT PUMP

Fig. 1 Schematic drawing of calibration equipment.
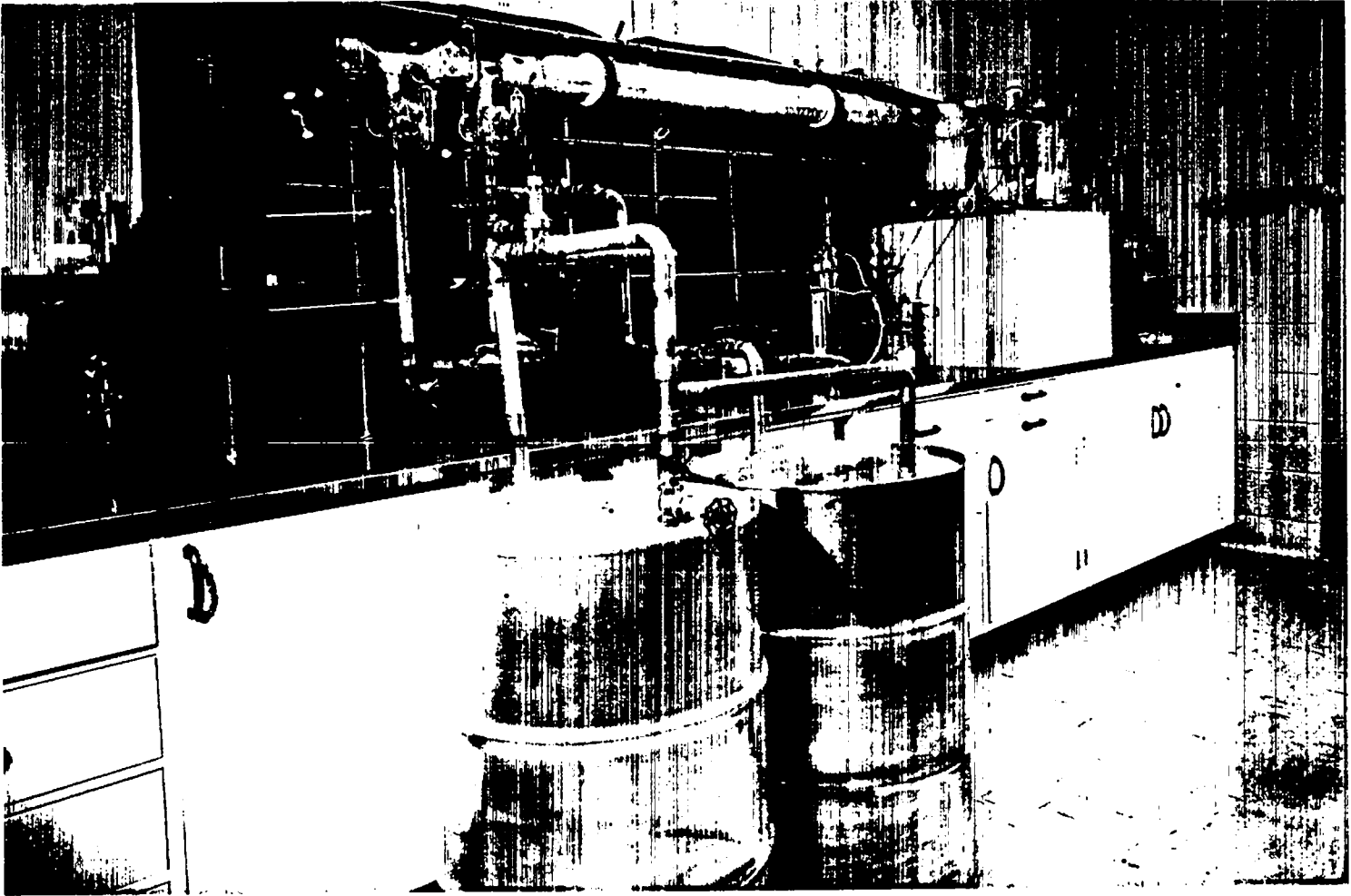
Fig. 2 Calibration equipment.
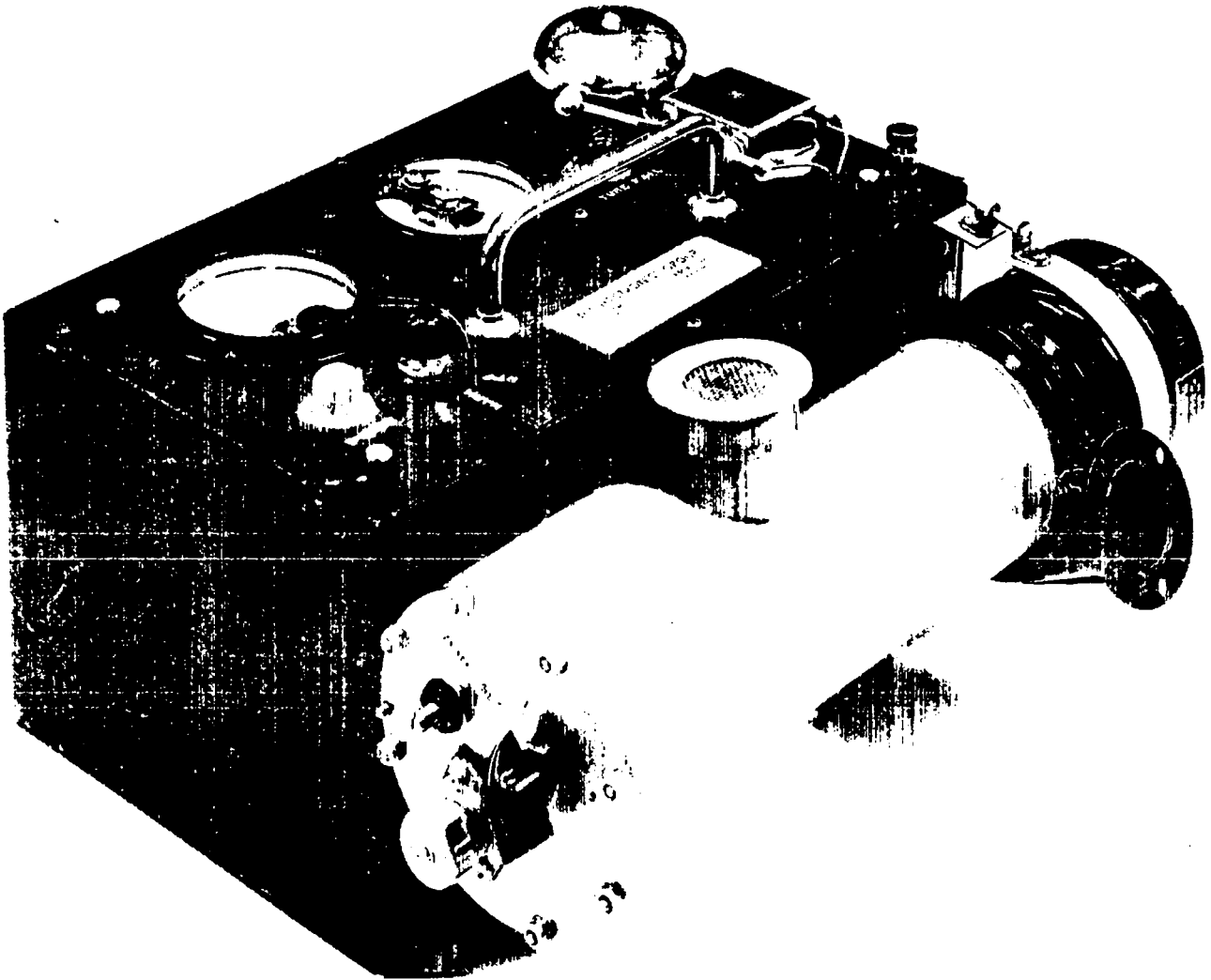
Fig. 3 Tritium monitor Model 101 Sniffer.
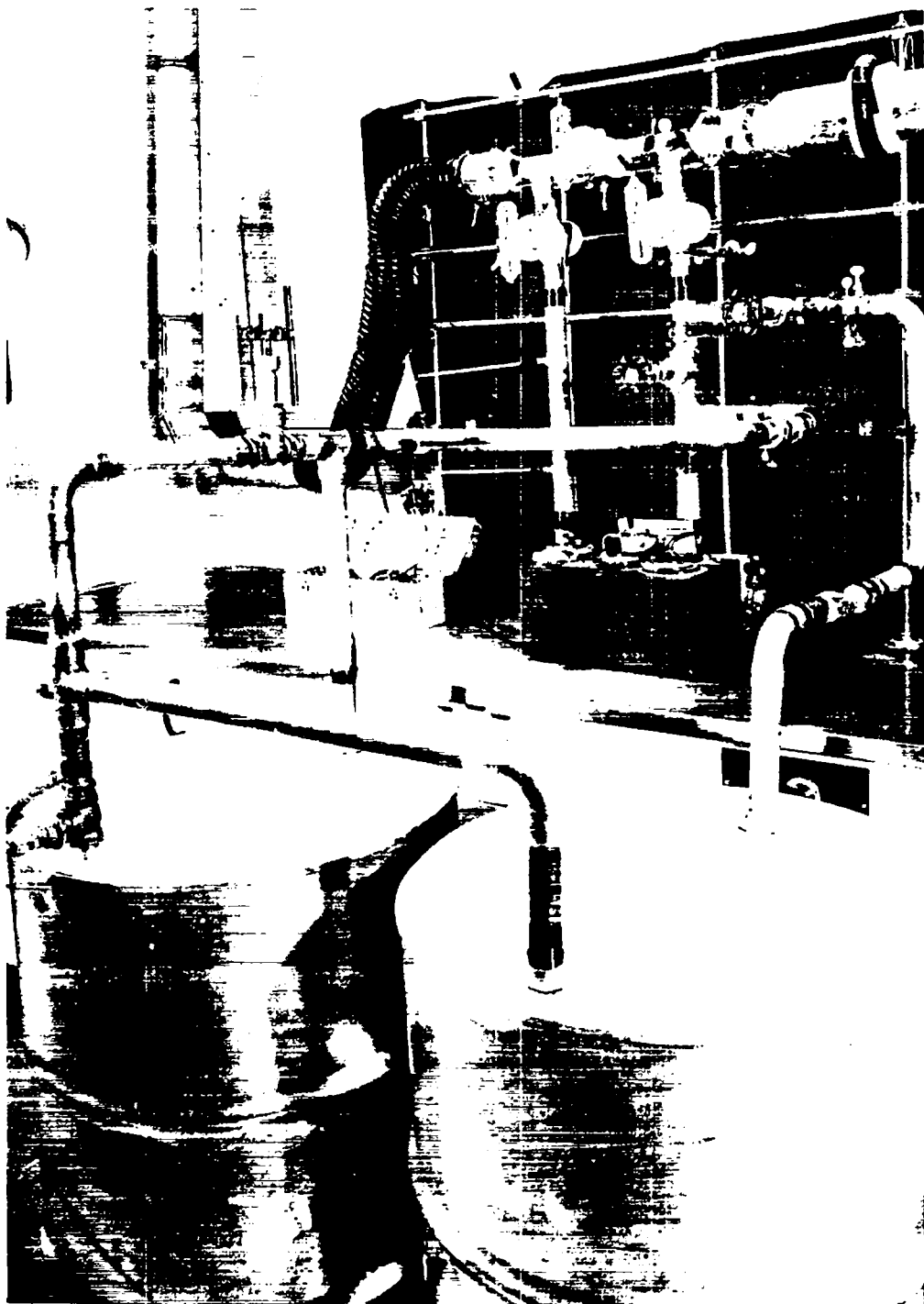
Fig. 4 Model 101 Sniffer adapted to calibration equipment.
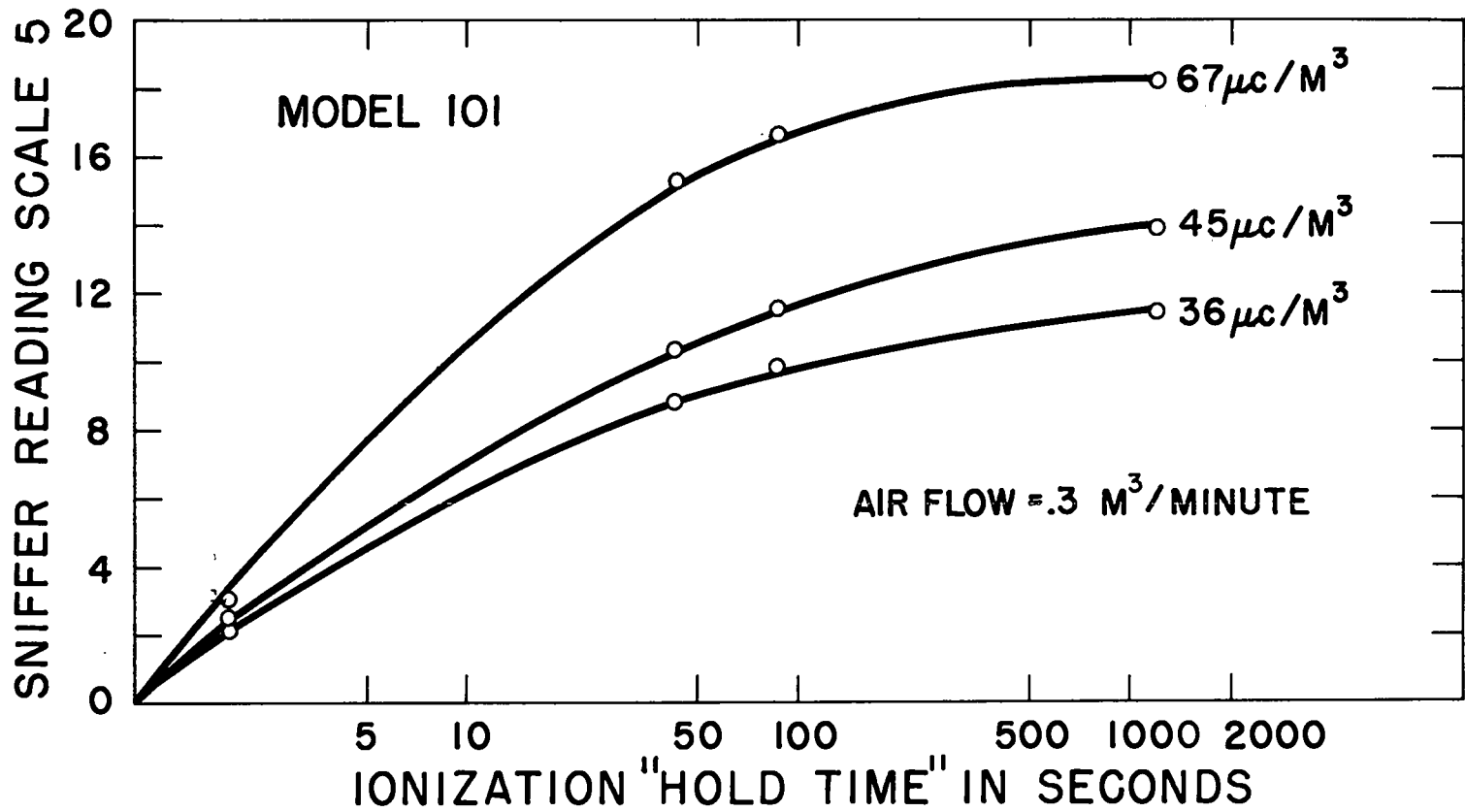
Fig. 5 "Hold time" of tritium contaminated air from generation of the tritium to its passage
through the Sniffer ion chamber plotted against Sniffer readings for tritium concentrations.
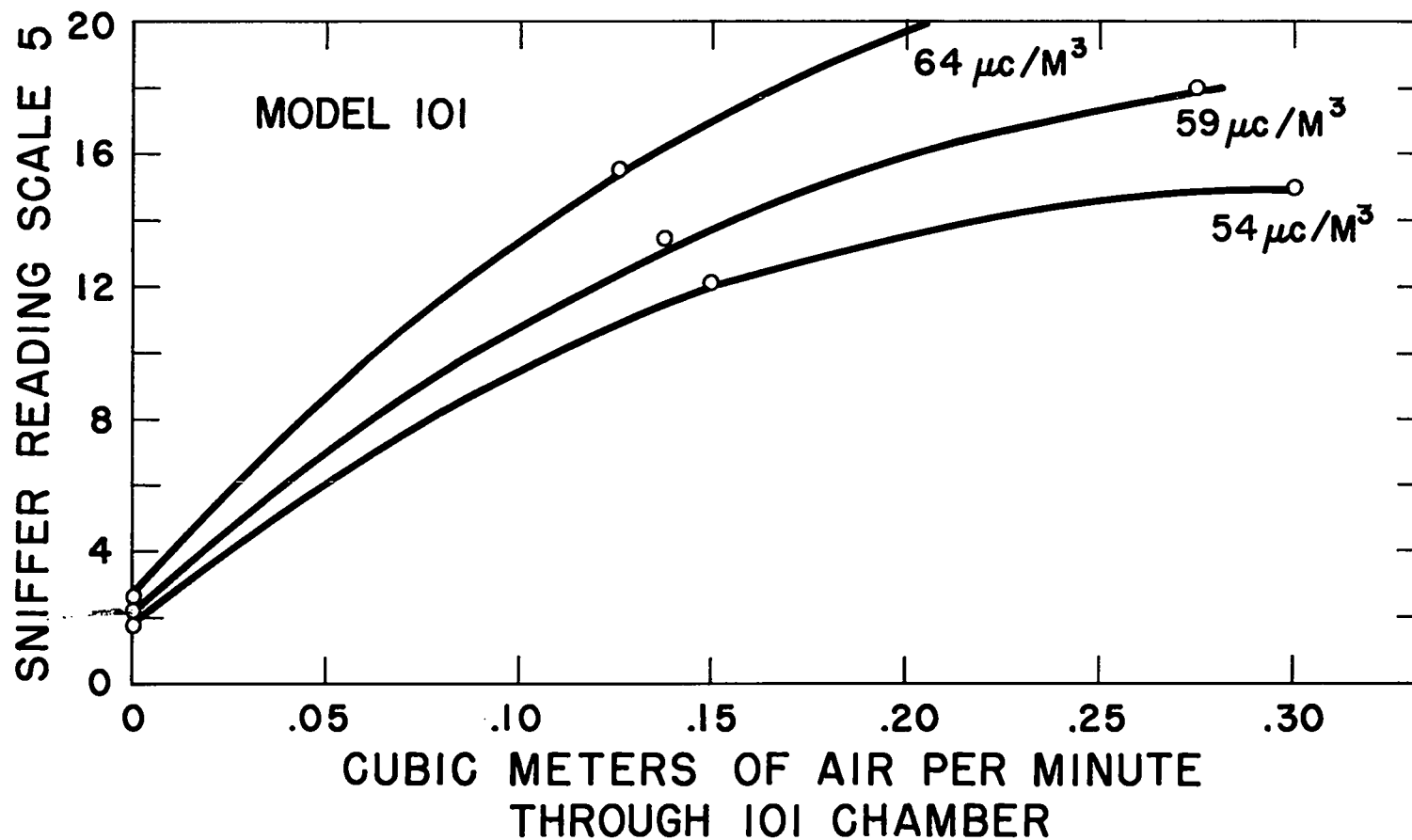
Fig. 6 The effect of change in air flow rate upon Sniffer readings for several tritium concentrations.
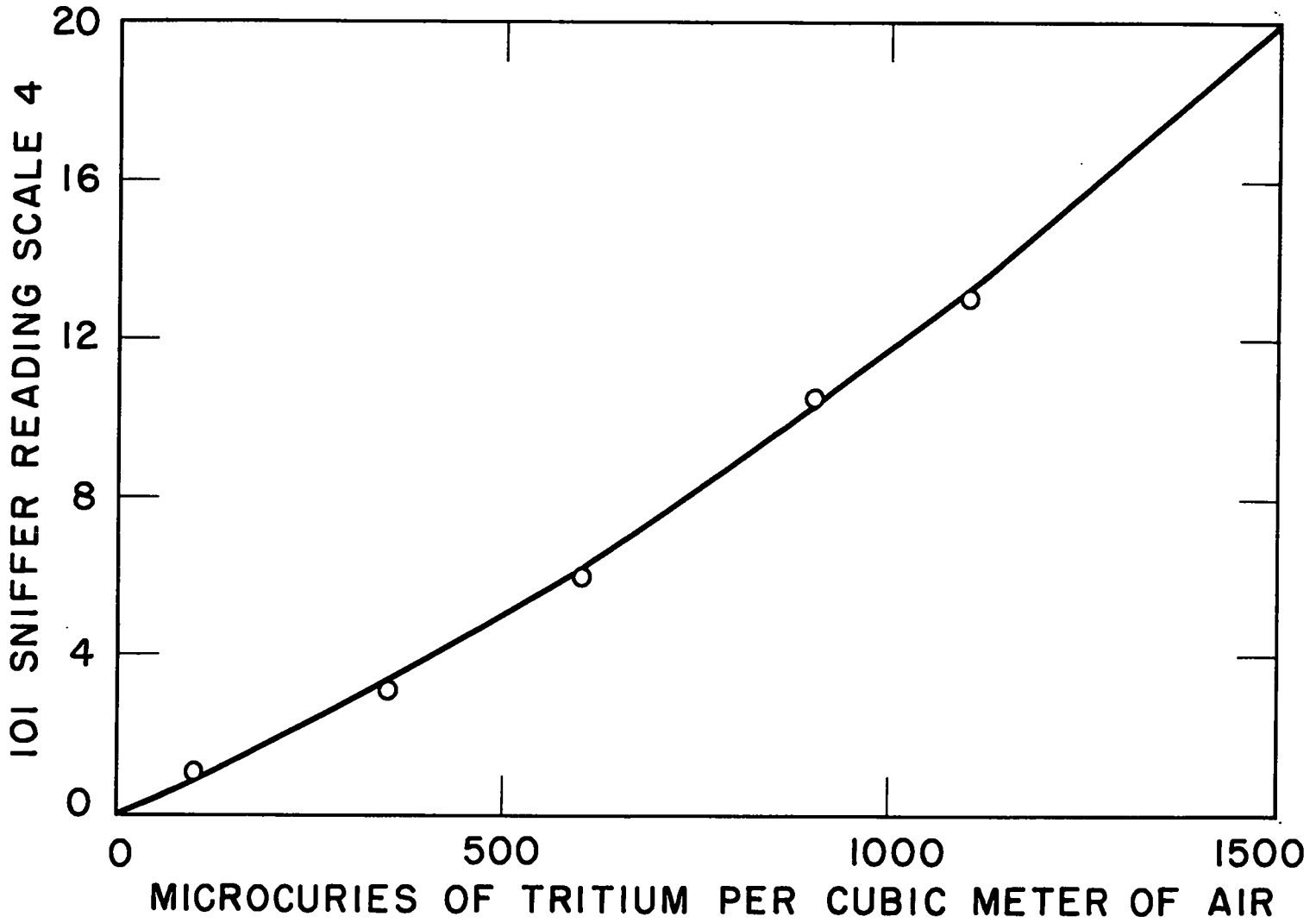
Fig. 7 Sniffer calibration curve on the 4 scale.
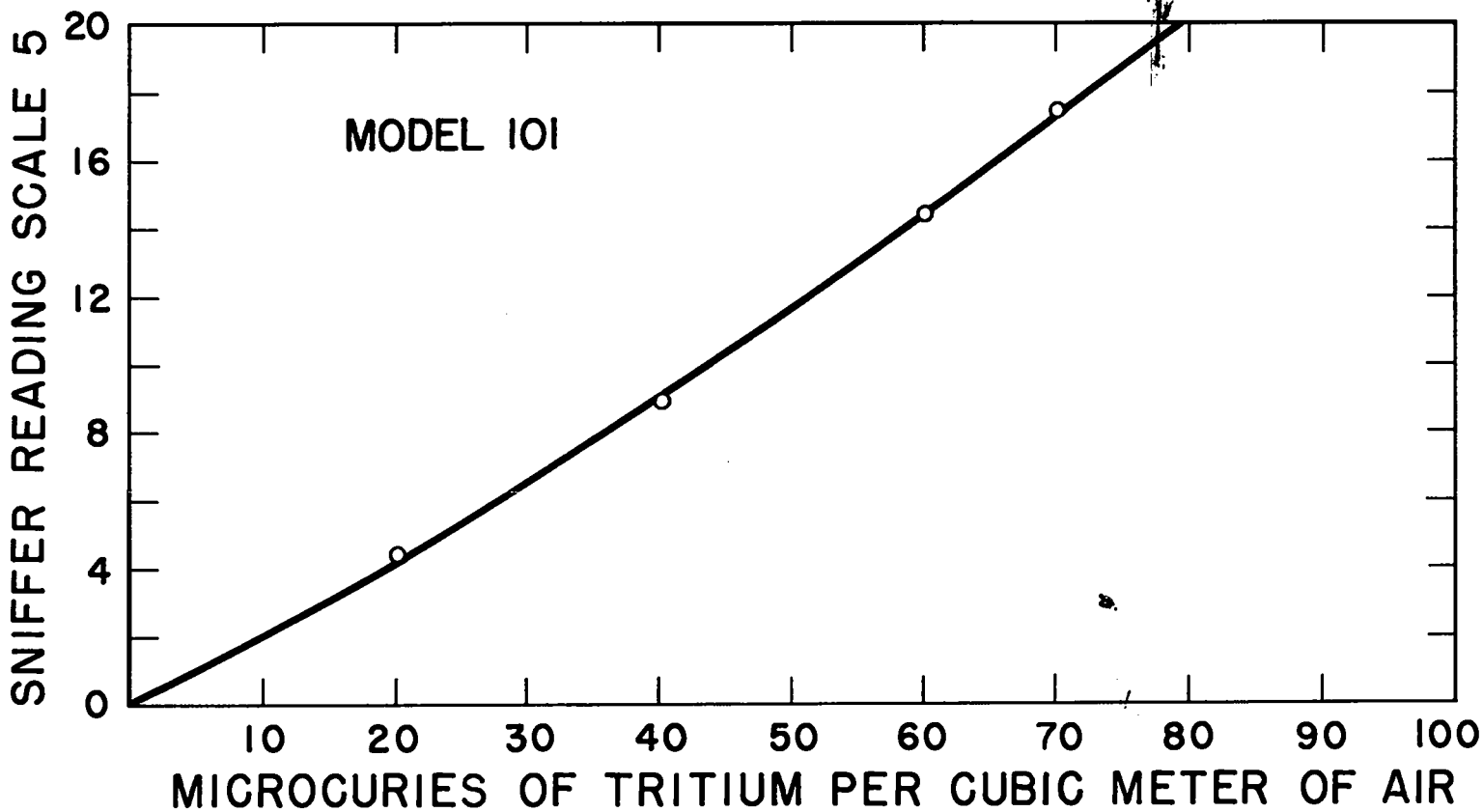
MODEL 101

Fig. 8 Sniffer calibration curve on the 5 scale.
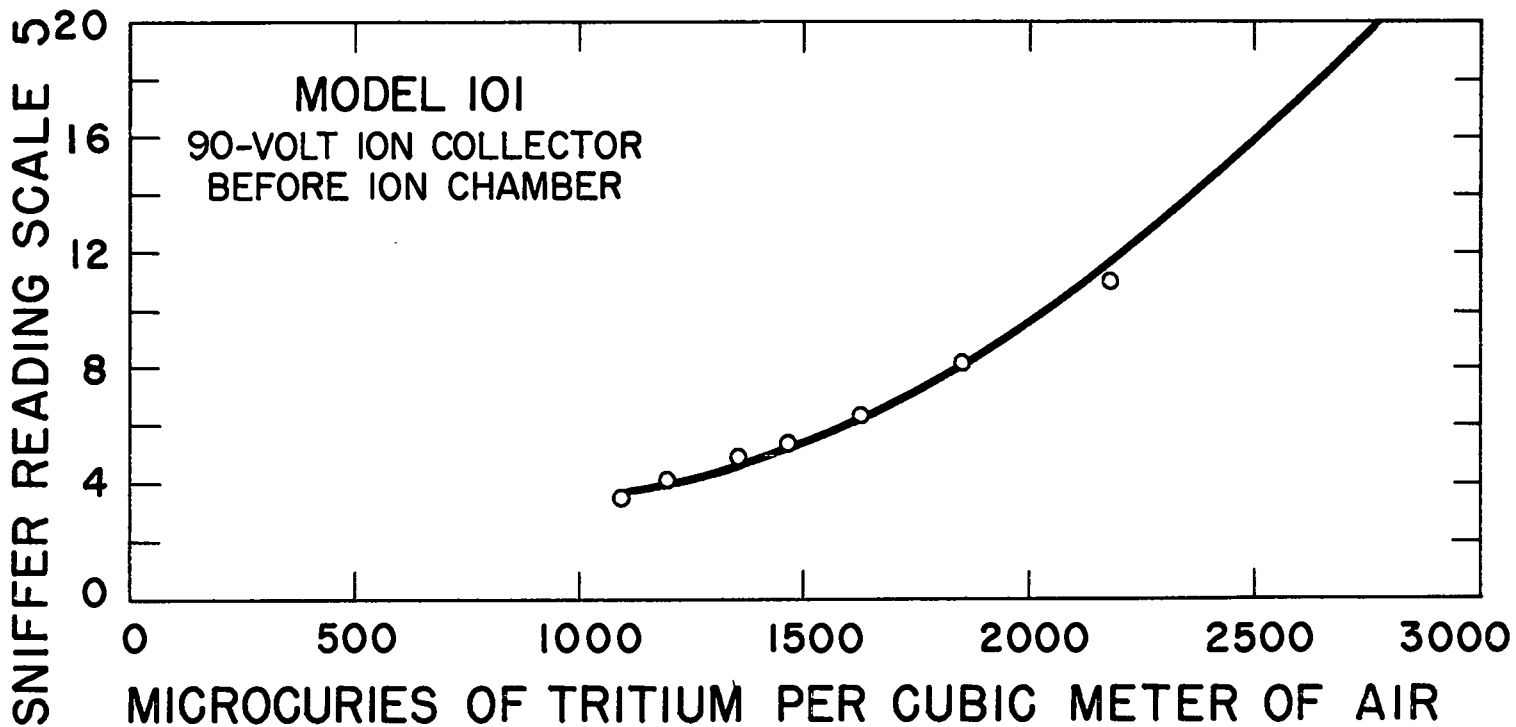
Fig. 9 Scale 5 calibration curve of Model 101 Sniffer with 90-volt ion trap ahead of the Sniffer ion chamber.
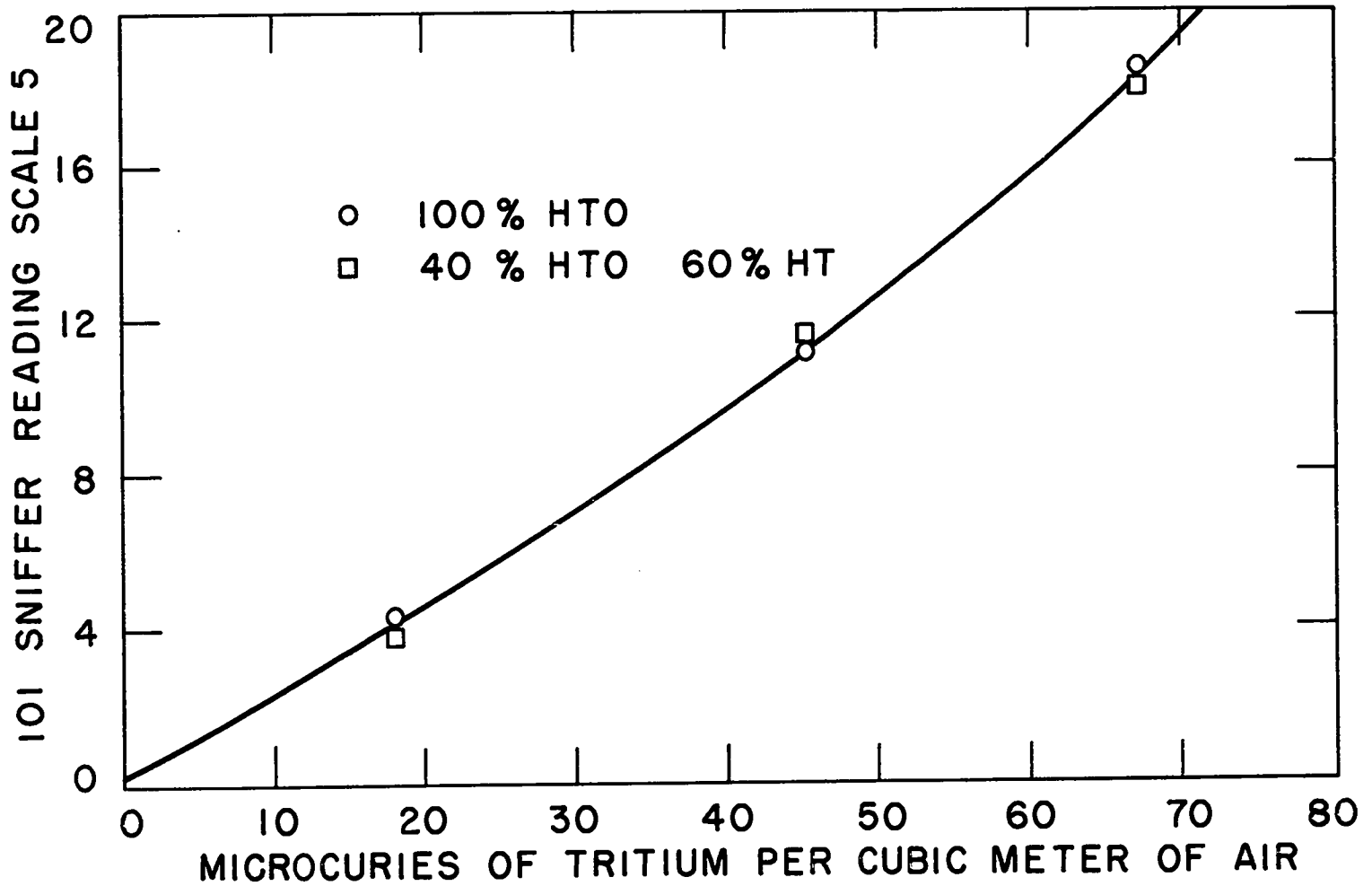
Fig. 10 Scale 5 Sniffer calibration curve for tritium as 100 per cent HTO and as a gaseous mixture of 60 per cent HT and 40 per cent HTO.
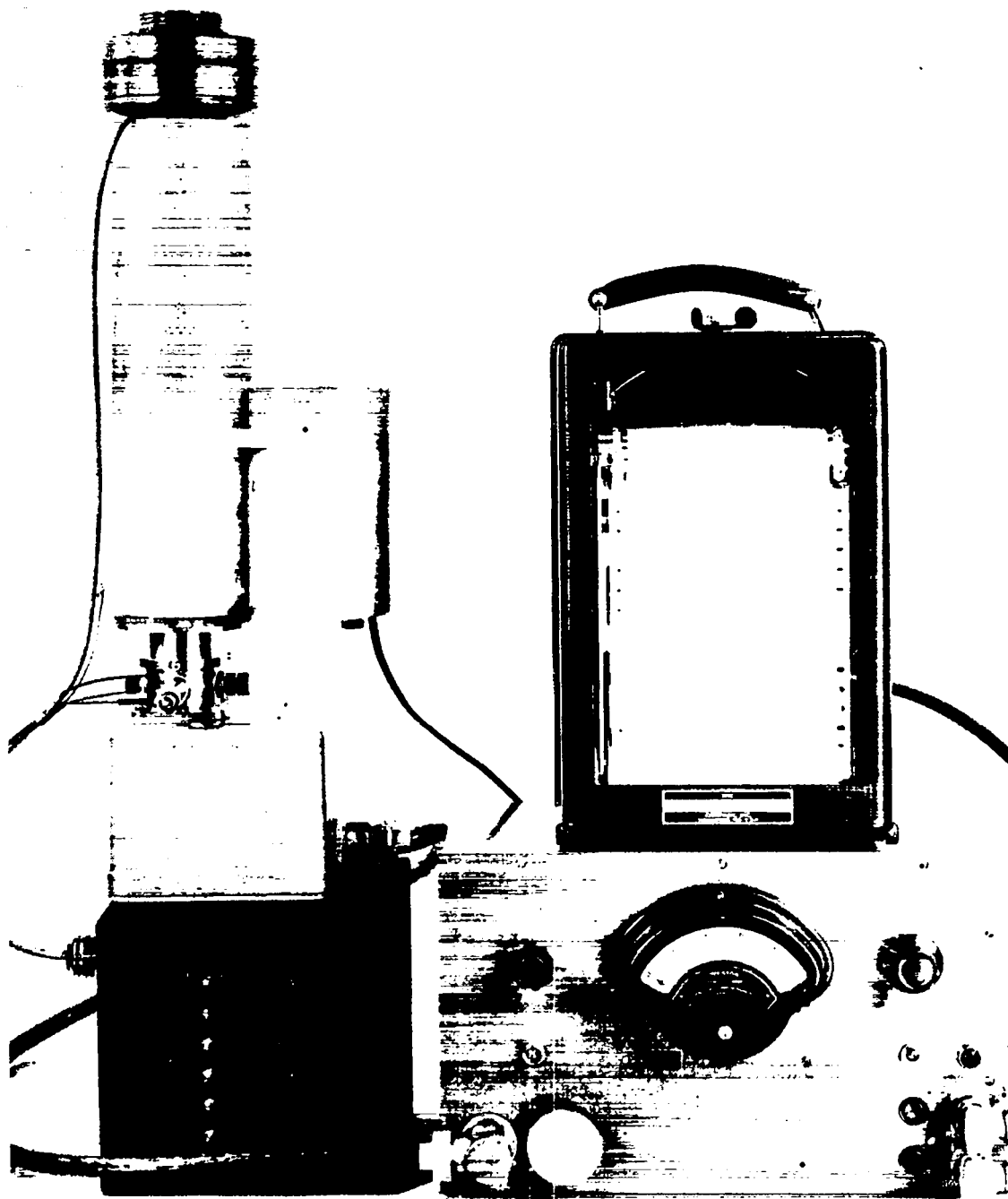
Fig. 11 Continuous tritium monitor CI-1.
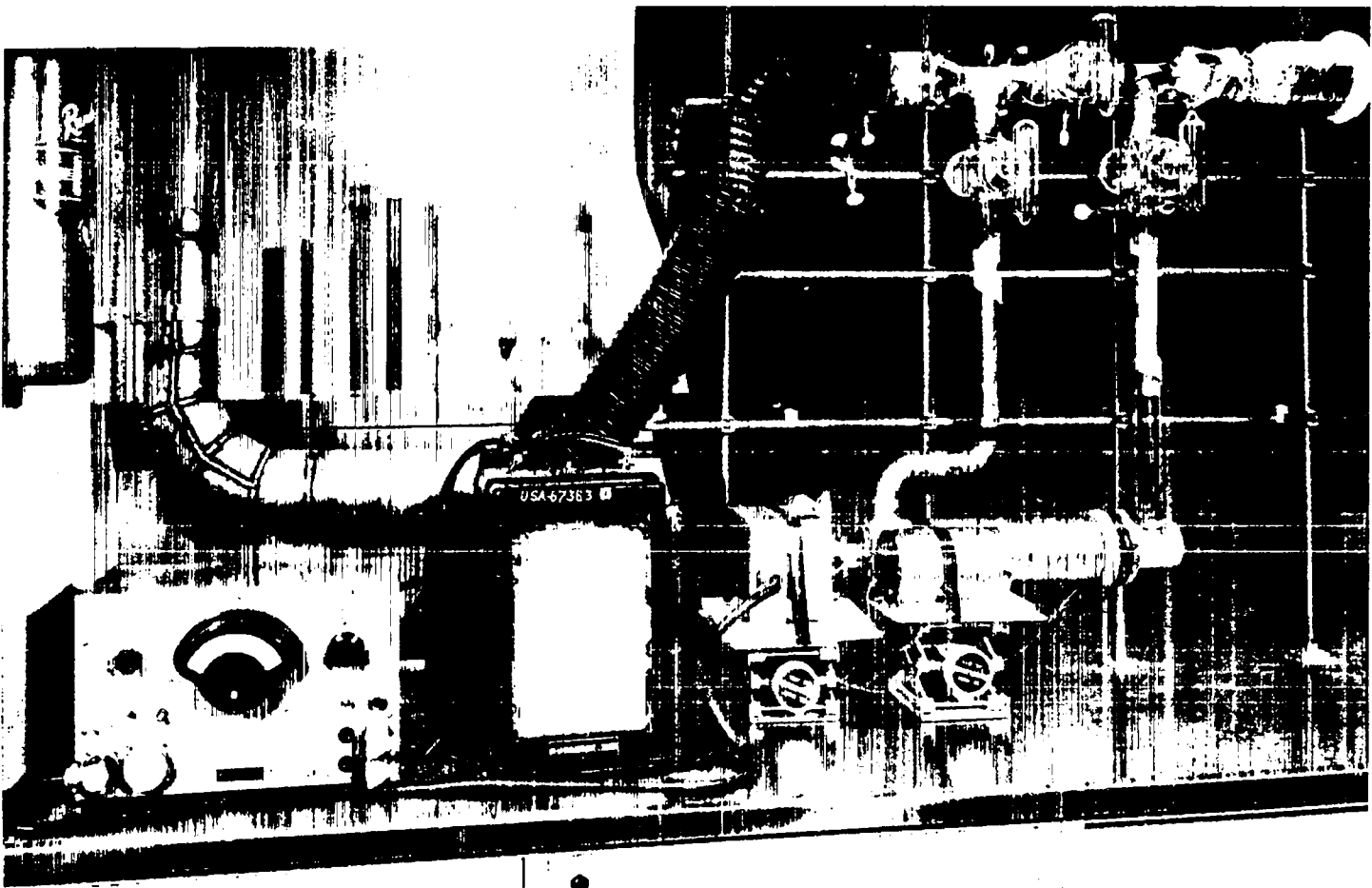
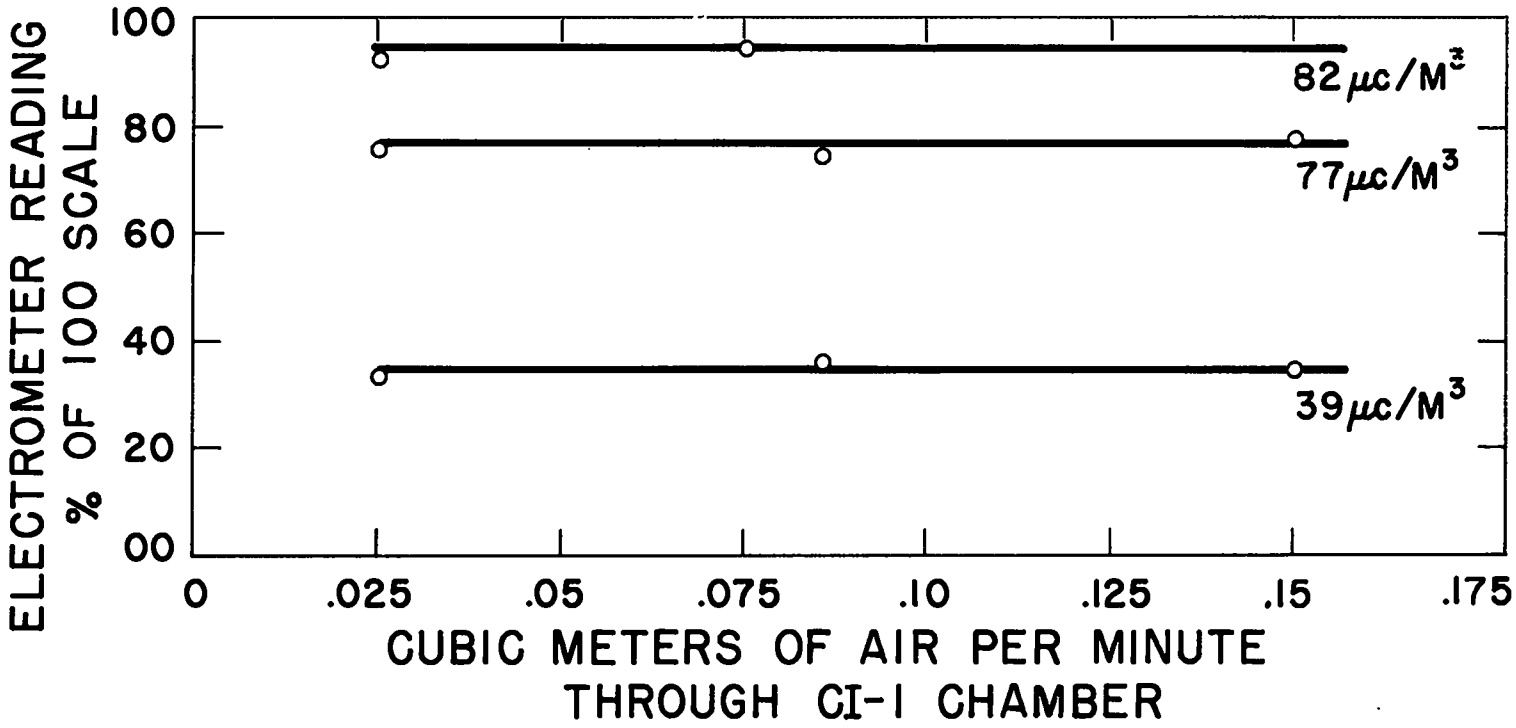Fig. 12 Continuous tritium monitor CI-1 adapted to calibration system.

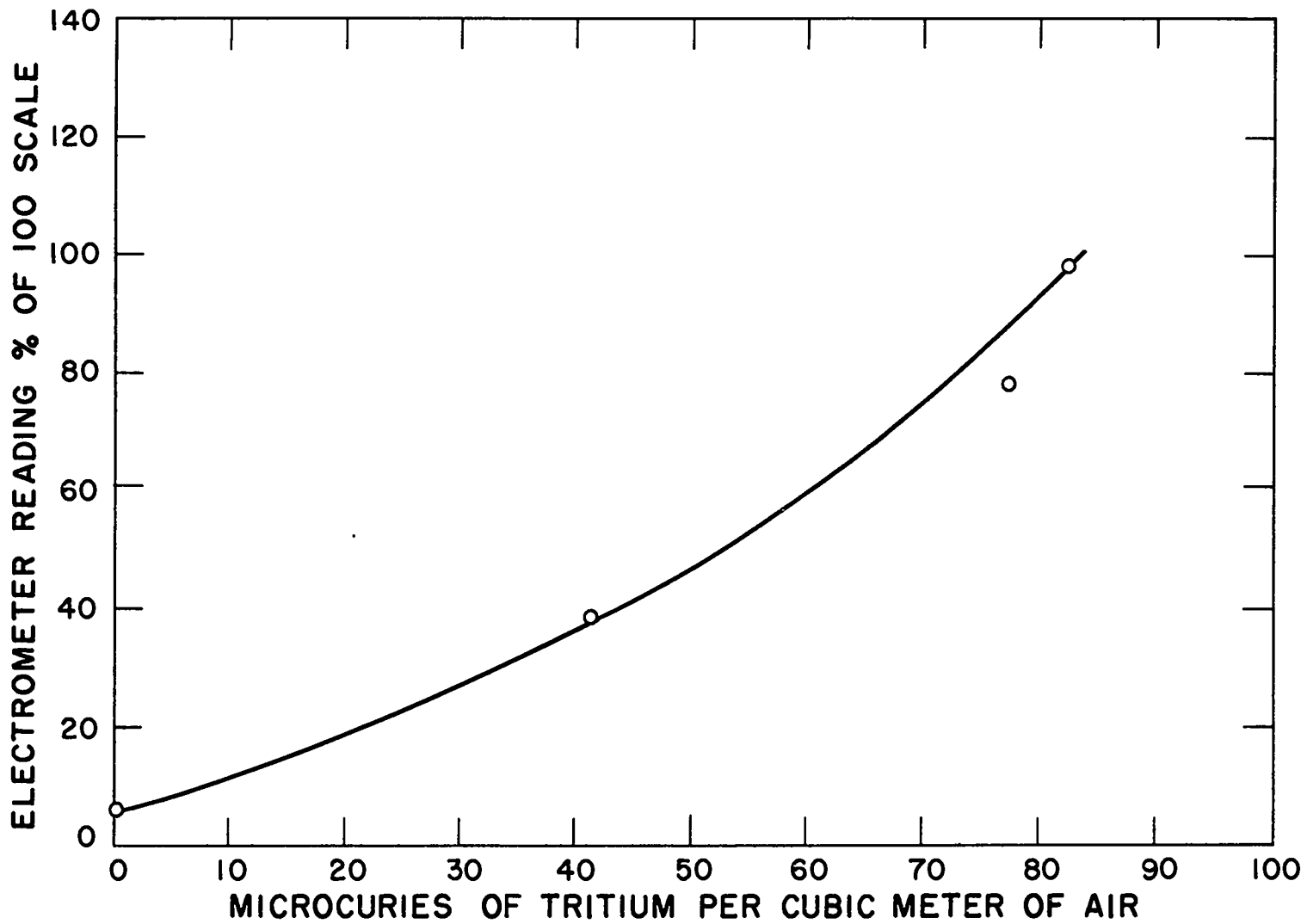Fig. 13 CI-1 response for several tritium concentrations at different air flow rates.

Fig. 14 Scale 100 calibration curve for CI-1 continuous tritium monitor.
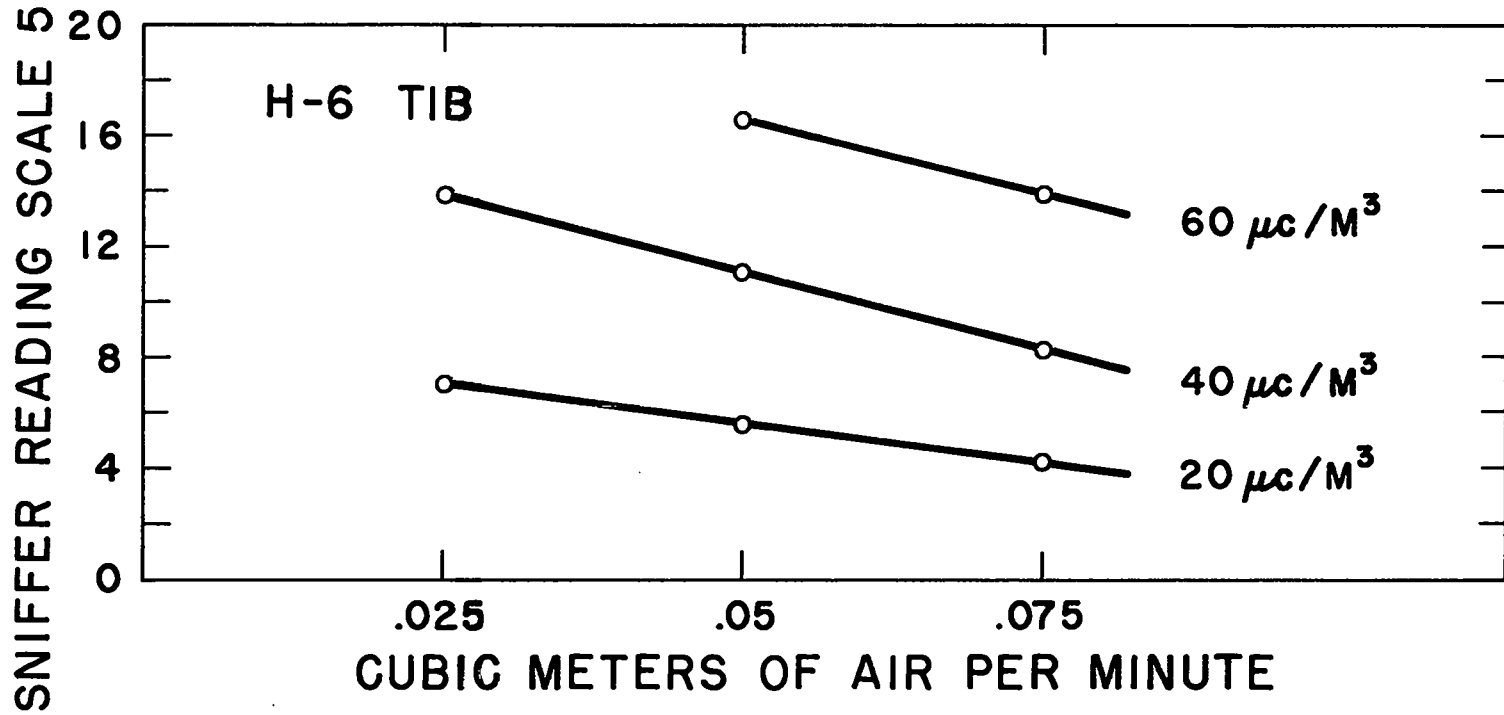
Fig. 15 T1B readings on scale 5 plotted against air flow rate at several tritium concentrations.