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Technology - Tritium Production

AEC Research and Development Report

IONIZATION CHAMBERS FOR MEASURING TRITIUM IN PROCESS STREAMS

by

J. D. Anthony
Engineering Assistance Section
Works Technical Department
Savannah River Plant

August 1962

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IONIZATION CHAMBERS FOR MEASURING TRITIUM IN PROCESS STREAMS

by

John D. Anthony

August 1962

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ABSTRACT

Ionization chambers of three different designs were developed for continuous measurement of tritium in the presence of hydrogen in process pipes and tanks. The chambers cover the range of 0.01% to 100% tritium concentration.

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IONIZATION CHAMBERS FOR MEASURING TRITIUM IN PROCESS STREAMS

INTRODUCTION

Ion chambers capable of continuously monitoring the tritium process were developed to replace and supplement mass spectrometers for tritium analysis of gaseous process streams. Ion chambers continuously monitoring these streams would indicate process upsets immediately and thus would simplify process control. Some work had been done on the design of an ion chamber(1) suitable for process monitoring; however, the chamber as designed could not be used for in-line monitoring of tritium in concentrations higher than a few per cent because it lacked ion traps and because its materials of construction were unsatisfactory.

SUMMARY

Three types of ion chambers were designed for monitoring tritium gas in process lines and tanks in the range of 0.01% to 100% tritium. These ion chambers have active volumes of 5.5, 20, and 930 cc and are designed to monitor tritium in hydrogen at pressures between 50 and 760 mm Hg. The chambers are made of stainless steel and have glass insulators. The ranges of the three types of chambers are as follows:

Chamber Volume, cc	Range, % T_2	Operating Voltage, v
5.5	1 - 100	200
20	0.1 - 10	600
930	0.01 - 1	600

Each type of chamber is in use, and all perform satisfactorily. The accuracy of the chambers and associated instrumentation is within ±10%. The 930-cc chamber is used for both in-line and in-tank monitoring.

The chambers were calibrated after being connected into the process lines. Initial calibration equations were developed by measuring chamber current as a function of pressure, because it was impractical to vary the tritium concentration. In developing these equations, it was assumed that current was proportional to concentration at constant pressure. Subsequent comparisons of chamber readings with mass spectrometer results showed this assumption to be correct.

DISCUSSION

Ion chambers present a simple means of monitoring tritium by measuring the ionization caused by the beta particle associated with the radioactive decay of tritium. The beta particle ionizes the gas through which it passes and the ions are collected on electrodes. The resulting current flow is proportional to the amount of tritium present, assuming all the ions formed are collected. Because the average energy of these beta particles is small (5690 ev)[2], their inability to penetrate detector walls makes it necessary to have the tritium in the active volume of the chamber.

GENERAL DESIGN

Design Basis

The design of the ionization chambers for use in the tritium process was based on the following requirements:

1. The chambers must operate between 50 and 760 mm Hg absolute pressure and measure concentrations between 0.01% and 100% tritium.
2. Each chamber must be capable of measuring the tritium concentration in process streams and tanks while the gas is continuously flowing and the tritium concentration changing.
3. The chambers must have a leak rate of less than 10^{-7} cc/sec of helium during operation at pressures below 10 microns Hg.
4. The components which come in contact with the process gas must be constructed entirely of stainless steel or glass.

The following chambers were designed to meet these requirements:

1. In-line ion chamber - 5.5-cc volume
2. In-line ion chamber - 20-cc volume
3. In-line ion chamber - 930-cc volume
4. In-tank ion chamber - 930-cc volume

Each of the in-line chambers was designed to be mounted directly in the process line and to monitor the gas flowing through the line. The in-tank chamber is inserted into the center of a process tank.

In each of these chambers the components which were to be exposed to the process gas were made of 304 stainless steel or glass with the exception of the electrical feedthrough terminals which were "Kovar" metal sealed to glass. The stainless steel is necessary because mercury is present in the system. The insulator material was glass because most other suitable insulators absorb tritium and give undesirable background readings.

Electrical Design

The basic electrical design of each type of chamber is the same, as shown in Figure 1. The beta radiation emitted from the tritium ionizes the gas between the electrodes. These ions are collected and the resulting current, which is proportional to the tritium in the chamber, is amplified and displayed on an electrometer amplifier.

At each end of the chamber is an ion trap which will collect free ions before they can drift into the chamber. These traps ensure that only the ions formed within the chamber will be collected. If ions originating outside the chamber were collected the signal would depend not only on tritium concentration but also on gas flow rate since the electrons and ions do not recombine immediately.

IN-LINE ION CHAMBER - 5.5-cc VOLUME

The 5.5-cc in-line ion chamber was designed for use at a constant pressure. The design range of the chamber was for tritium concentrations from 10% to 100% with 200 volts applied across the electrodes. However, because the background buildup of tritium on the chamber walls is less than 0.1%, the useful range is actually 1% to 100%.

A sketch of the chamber is shown in Figure 2. Figures 3 and 4 show the chamber disassembled and assembled. The over-all length is 7-3/4 inches. The active volume is 5.5 cc and the total volume between flanges is 27 cc.

Ion Trap

Since ion traps are at both ends of the chamber the gas flow through the chamber can be in either direction. The ion trap is 100% efficient with flow rates up to 20 liters per minute.

Ion Chamber Saturation Characteristics

Figure 5 is the saturation curve of the chamber with 86% tritium concentration in hydrogen at a chamber pressure of 760 mm Hg. The chamber saturates at 150 volts at this concentration and will saturate with 200 volts applied with 100% tritium.

Ion Chamber Response

The chamber current with 86% tritium in hydrogen at a pressure of 760 mm Hg is 7.3 microamperes. However, the sensitivity will not change proportionally with pressure. The range of the tritium beta particle in hydrogen at atmospheric pressure is approximately 0.5 cm, which is comparable to the electrode spacing. Because of this, a significant portion of the energy from the beta particles is lost to the walls and is not measured. As the pressure is reduced in the chamber and the range of the beta particles becomes greater, more of the energy will be lost to the walls causing the response to be nonlinear with change in pressure. Figure 6 is a response curve for the 5.5-cc chamber over a range of pressures. Because this chamber was intended for use at a fixed pressure, it was not necessary to determine the equation for the curve represented in Figure 6. However, should the usage be extended to cover a wide range of pressures, a calibration curve based on experimental data can be determined as described in the Appendix.

IN-LINE ION CHAMBER - 20-cc VOLUME

The 20-cc in-line chamber has a design range of 0.1% to 10% tritium with 600 volts applied. Figure 7 is a drawing of this chamber. A photograph of the chamber both assembled and disassembled is shown in Figure 8. The total chamber volume between the flanges is 75 cc. The sensitive volume is 20 cc and the over-all length of the chamber including flanges is 16 inches. The large flanges shown in Figure 8 were used to adapt the chamber to existing process piping.

Ion Trap

This chamber has an ion trap at only one end and the chamber must be installed so that the gas flow is in the direction shown in Figure 7.

Ion Chamber Saturation Characteristics

Figure 9 is a saturation curve of the 20-cc ion chamber. This chamber saturates at approximately 20 volts for a tritium concentration of 0.86% at a pressure of 283 mm. The chamber will saturate properly with 600 volts applied up to at least 10% tritium concentration.

Ion Chamber Response

Figure 10 is the response curve for the 20-cc ion chamber to variations in chamber pressure. This chamber was intended for use in determining tritium concentration over a wide range of pressures. Therefore, it was necessary to determine the general equation for a family of curves which is represented by the curve in Figure 10. The method used for determining an approximate equation based on experimental data is described in the Appendix. The equation for this response curve is

$$C = \frac{I}{P} \left(322e^{-0.00505P} + 131\right) \times 10^7$$

where
C = tritium concentration, %
I = chamber current, amperes
P = chamber pressure, mm Hg

The chamber current with 1% tritium in hydrogen at 760 mm Hg pressure is 0.55 microampere.

IN-LINE ION CHAMBER - 930-cc VOLUME

Figure 11 is a drawing of the 930-cc ion chamber which has a range of 0.01% to 1% with 600 volts applied. Figure 12 is a photograph of the chamber. The active volume is 930 cc and the total volume is approximately 3 liters. The high voltage electrode is supported by two glass rings which are held in place by metal stops. The signal electrode is supported by a glass tube which is held at both ends by metal cups. The signal electrode is insulated from the cups by glass spacers. A spring is used at each end of the signal electrode in the support assemblies to hold the glass insulating spacer and support tube snug yet permit differential expansion. The high voltage and signal lead enter through "Kovar" feedthrough terminals.

Ion Trap

The ion trap at each end of the chamber is formed by the high voltage electrode and the metal cups. The gas flow can be in either direction through this chamber.

Ion Chamber Saturation Characteristics

Figure 13 is a voltage saturation curve of the chamber taken with 0.53% tritium in hydrogen at a pressure of 550 mm Hg. The chamber will saturate at 600 volts with 1% tritium at 700 mm Hg.

Ion Chamber Response

Because this chamber was designed to be operated at various pressures a characteristic response equation similar to the one used with the 20-cc ion chamber was determined. The equation is for a family of curves represented by the curve shown in Figure 14. The equation is

$$C = \frac{I}{P(2.14 - 1.877e^{-0.00668P})} \times 10^{8}$$

This equation is developed in the Appendix.

The response to 0.1% tritium concentration at 760 mm is 1.65×10^{-6} ampere. In order to render the equation more usable, a nomograph can be constructed based on the equation. Figure 15 is a typical nomograph used for determining the tritium concentration from the pressure and chamber current. To determine tritium concentration from the nomograph, a straightedge is placed between known pressure and current values and the tritium concentration is read at the point the straightedge intersects the third column.

IN-TANK ION CHAMBER - 930-cc VOLUME

The in-tank process ion chamber was designed to make possible the continuous monitoring of tritium concentration within a tank. The chamber is essentially the same design as the 930-cc in-line ion chamber except that the outer case has been removed and a support frame substituted to permit insertion of the chamber into a tank. Figure 16 is a photograph of the in-tank ion chamber. The chamber is open to the tank gas at both ends to permit equilibration of the tank and chamber gas. The signal and high voltage leads enter at "Kovar" seal feedthrough terminals at the flange and are separated by an electrostatic shield extending from the flange to the chamber. The ion chamber has the same sensitive volume as the 930-cc in-line chamber.

In addition to the ion chamber existing between the signal and high voltage electrode another ion chamber exists between the high voltage electrode and the walls of the tank. In a large tank this ion chamber can cause currents on the order of several milliamperes. Although this current is not in the measuring circuit it could cause excessive loading of the power supply. To prevent these high currents from being developed a shield made of 304 stainless steel mesh wire was designed to be placed over the chamber. Figure 17 is a photograph of this shield mounted on the chamber.

VOLTAGE BREAKDOWN WITHIN THE CHAMBER

Because the chambers are an integral part of the process they are sometimes evacuated to pressures below 10 mm Hg. At these low pressures the 600 volts applied to the chamber will discharge across the electrodes. To prevent electrical discharge, pressure switches automatically switch off the voltage in the chambers when the pressure goes below 100 mm Hg. If a pressure switch should fail and air should leak into the chamber containing hydrogen and tritium, an explosion could occur due to an electrical discharge through the mixture of hydrogen and oxygen, and the equipment could be damaged. Continuous electrical discharge might also lead to loss of tritium by oxidation. In order to determine whether electrical discharges would occur at pressures where self-sustained combustion of hydrogen and oxygen takes place and whether continuous discharge would result in loss of an appreciable amount of tritium, a series of tests was made. These tests were made to determine:

1. Where in the chamber breakdown occurs and at what voltage it occurs at various pressures.

2. If discharges of short duration would ignite a mixture of hydrogen and oxygen at pressures less than 100 mm Hg.

3. The rate of oxidation of hydrogen with continuous discharges within the chamber.

4. The lowest pressure at which hydrogen-air mixtures would support general combustion.

As a result of these tests, which are described below, it was determined that at pressures where self-sustaining oxidation of hydrogen occurs voltage discharges will not occur in the chamber and that continuous discharge will cause oxidation of almost all of the hydrogen present.

Experimental Arrangement

The experimental arrangement shown in Figure 18 was used to test the combustibility of hydrogen and air in the ion chamber. The ratio of hydrogen, oxygen, and nitrogen was near optimum combustion conditions, approximately $2H_2 + O_2 + 7N_2$.

A mercury manometer was used to measure the partial pressures of the gases as they were added and to measure the pressure in the chamber before and after electrical discharge. The accuracy of measurement was ±1 mm Hg pressure. A differential pressure cell was used to measure the pressure change and its output was recorded on a high speed Brush recorder.

The polarizing voltage was supplied to the ion chamber by a power supply with an output variable from 0 to 5000 volts.

The 930-cc in-line chamber was used throughout these tests.

Voltage Breakdown Tests

Figure 19 shows the voltage at which breakdown occurs at various pressures for the chamber in a process line exposed to tritium. The lower voltage required to cause breakdown under process conditions is due to the ionization caused by the tritium.

When a blank flange made of clear plastic was placed over one end of the chamber, the breakdown was observed to occur at the high voltage feedthrough terminal. The breakdown at the lower pressures (less than a few cm Hg) was glow discharge, and the discharge current ranged between 0.4 and 1.0 milliampere.

Gas Combustion Tests

The first of three gas combustion tests was run to determine if voltage discharges of short duration would ignite a mixture of hydrogen and oxygen within the chamber at pressures less than 100 mm Hg. Gas mixtures with a hydrogen to oxygen ratio of 2 to 1 were used throughout these tests. Table I shows the partial pressures of gases used and the voltages at which breakdown occurred. For each mixture, the voltage was increased gradually until the milliammeter in the voltage supply line indicated a voltage discharge in the chamber. When several voltage discharges would not ignite the gas, a new mixture was tried. No measurable pressure change occurred during this test.

In the second test the amount of oxidation of hydrogen during continuous discharge was determined. Mixtures with different hydrogen-to-oxygen ratios were used and the voltage discharge was allowed to continue for 40 minutes. Table II shows the data obtained from this test. To determine the amount of oxidation, the difference in pressure of the system before and after the discharge period was measured. A decrease in pressure of 4 mm Hg would result from oxidation of 8 mm Hg of hydrogen. The amount of oxidation of the hydrogen ranged from 20% to 53% in 40 minutes. This test showed that self-sustained combustion will not occur below 100 mm Hg but that oxidation will result if mixtures of hydrogen and oxygen are exposed to continuous discharge.

A third test was run to determine whether larger amounts of air added to the mixture would cause general combustion. These results are shown in Table III. In this test air was added to hydrogen in 10 mm Hg pressure increments and the voltage increased to breakdown at each step. For a hydrogen partial pressure of 20 mm, general combustion occurred at about 160 mm and the peak pressure was 340 and 310 mm on successive runs. In the second run a decrease in pressure of 12 mm was evident after a short cooling period indicating ~98% oxidation of hydrogen. The time of combustion as indicated on the recorder was ~1/2 second. Attempts were made to cause self-sustained combustion of a mixture containing 15 mm partial pressure of hydrogen with partial air pressures up to 200 mm; however, no combustion occurred.

ACKNOWLEDGEMENT

The author expresses appreciation to J. G. Lambright at the Savannah River Plant for providing assistance in obtaining data from and evaluating these ion chambers.

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Engineering Assistance Section
Works Technical Department

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APPENDIX

A method of determining the empirical equation for a family of curves representing the response of an ion chamber at different pressures was developed for the ion chambers described in this report. In an ion chamber in which the ionizing radiation to be measured is low energy beta and originates within the sensitive portion of the ion chamber some of the energy of the radiation is absorbed by the walls. As the pressure is reduced within the chamber the range of the beta becomes greater and a larger percentage of the energy is lost. Figure 20 shows the current response for a fixed tritium concentration at different pressures. From this curve, which was determined experimentally, enough data can be obtained to formulate an equation from which tritium concentration can be determined based on ion chamber pressure and current.

If there were no wall losses, the chamber current would be directly proportional to concentration and pressure.

The theoretical chamber current in amperes, assuming no losses, would be represented by the equation,

$$I_{th} = KPC \tag{1}$$

where K = a constant for a particular chamber size, shape, arrangement, and voltage

P = total pressure of the gas in the chamber, mm Hg

C = concentration of tritium in the chamber, %

To account for losses in an actual chamber, the equation becomes

$$I = I_{th} - \Delta I \tag{2}$$

where ΔI = current loss to the chamber walls

Since the rate of energy loss from very low energy beta ray spectra is approximately exponential, an equation of the following form was assumed for the current lost to the wall:

$$\Delta I = AI_{th}e^{-BP} \tag{3}$$

where A and B are constants.

Combining Equations 2 and 3 gives

$$I = I_{th} - AI_{th}e^{-BP} \tag{4}$$

or

$$I = I_{th}(1 - Ae^{-BP}) \tag{5}$$

Combining Equations 1 and 5 gives

$$I = KPC(1 - Ae^{-BP}) \tag{6}$$

The theoretical and experimental curves will coincide at high pressures (above 600 mm Hg) because wall losses become very small. The constant K is the slope of the theoretical curve, and its numerical value is 2.14×10^{-8}.

Substituting the value of K in Equation 6 gives

$$I = 2.14 \times 10^{-8}\ PC(1 - Ae^{-BP}) \tag{7}$$

The constants A and B may be evaluated by simultaneously solving Equation 7 at two different pressures.

From Figure 20 for P = 50

$$0.21 \times 10^{-6} = 50 \times 0.53 \times 2.14 \times 10^{-8}\ (1 - Ae^{-50B})$$

For P = 300

$$3.0 \times 10^{-6} = 300 \times 0.53 \times 2.14 \times 10^{-8}\ (1 - Ae^{-300B})$$

From these two equations

$$A = 0.877$$

$$B = 6.68 \times 10^{-3}$$

Substituting these two values in Equation 7 gives

$$I = 2.14 \times 10^{-8}\ PC(1 - 0.877e^{-6.68P \times 10^{-3}}) \tag{8}$$

Rearranging Equation 8 gives

$$C = \frac{I}{P(2.14 - 1.877e^{-0.00668P)}} \times 10^{8} \tag{9}$$

Equation 9 is the equation for the response of the 930-cc chamber and may be used to compute the concentration of tritium in the chamber when I and P are known.

TABLE I

Breakdown Voltage Tests
930-cc In-Line Chamber

Run	Partial Pressures, mm Hg H_2	Partial Pressures, mm Hg Air	Total Pressure (H_2 + Air), mm Hg	Voltage at Breakdown, v
1	6	14	20	750
2	9	21	30	950
3	12	28	40	1100
4	15	35	50	1200
5	18	42	60	1350
6	20	50	70	1500
7	23	57	80	1600
8	26	64	90	1700
9	29	71	100	1750

TABLE II

Continuous Discharge Tests
930-cc In-Line Chamber

Run	Partial Pressures, mm Hg H_2	Partial Pressures, mm Hg Air	Total Pressure, mm Hg	Current, milliampere	Running Time, minutes	Pressure Drop, mm	Hydrogen Combined, %
1	20	50	70	0.8	40	4	40
2	20	50	70	0.6	40	2	20
3	20	50	70	0.6	40	2	20
4	20	50	70	0.7	40	3	30
5	20	70	90	0.7	40	4	40
6	0	70	70	0.7	40	0	0
7	15	80	95	0.7	40	4	53
8	25	70	95	0.8	40	3	24
9	30	66	96	0.4	25	4	37

TABLE III

Self-Sustaining Combustion Tests
930-cc In-Line Chamber

Run	Partial Pressures, mm Hg H_2	Partial Pressures, mm Hg Air	Total Pressure, mm Hg	Voltage, v	Current, milliampere	Oxidation, %	Peak Pressure, mm	Remarks
1	20	145	165	5000	0.5	Undetermined	340	A measurement of pressure decrease was not made.
2	20	140	160	5000	0.4	98	310	A 12-mm decrease in pressure after cooling.
3	15	200	215	5000	0.4	None		No general combustion evident within range tested.

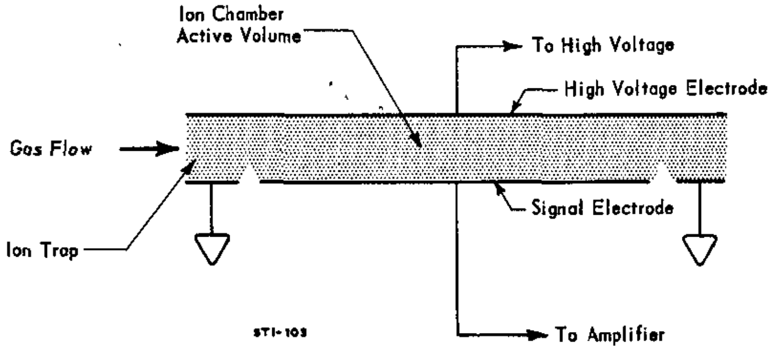

FIG. 1 ELECTRICAL DESIGN OF ION CHAMBERS

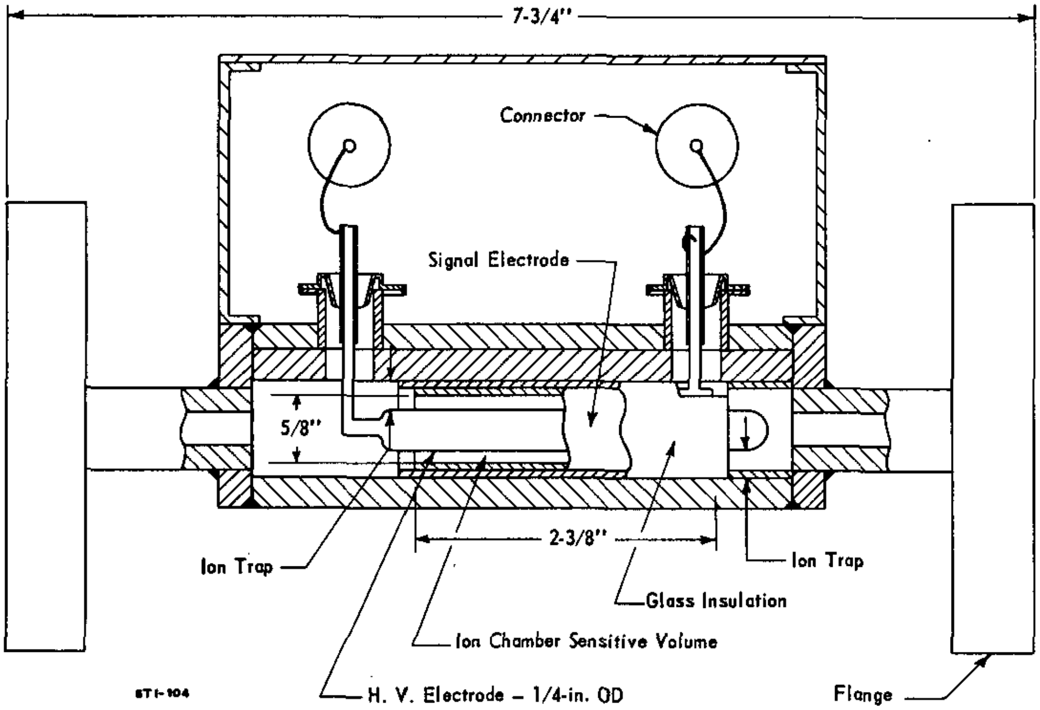

FIG. 2 DRAWING OF 5.5-cc IN-LINE ION CHAMBER

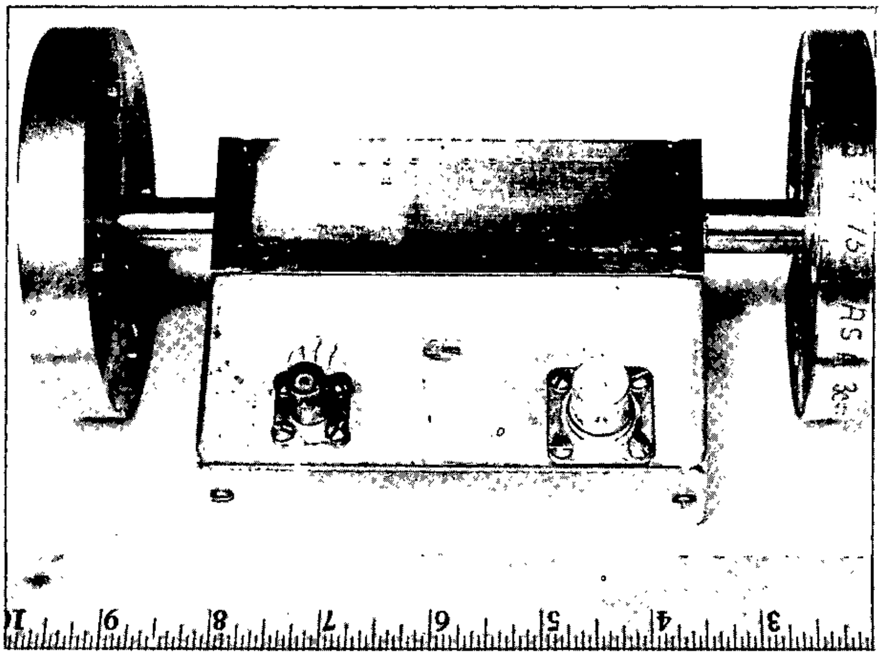

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FIG. 3 ASSEMBLED 5.5-cc IN-LINE ION CHAMBER

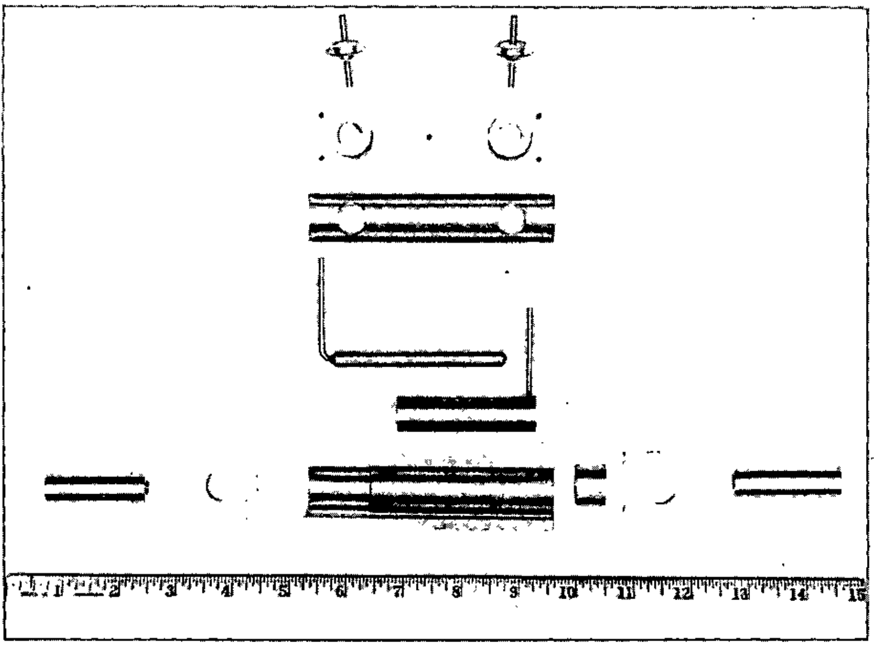

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FIG. 4 DISASSEMBLED 5.5-cc IN-LINE ION CHAMBER

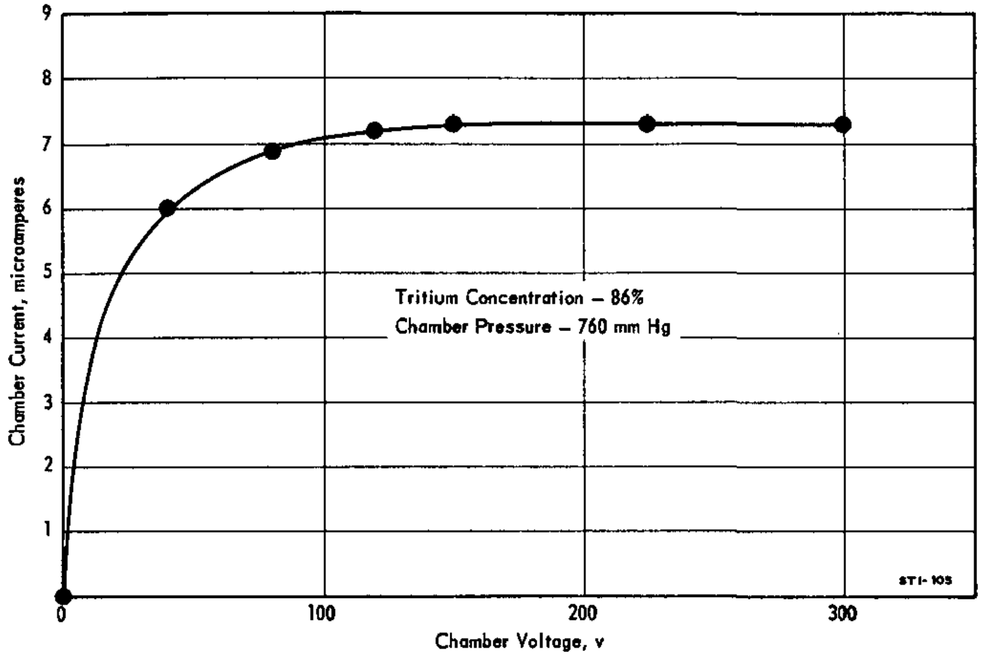

FIG. 5 SATURATION CURVE OF THE 5.5-cc IN-LINE ION CHAMBER

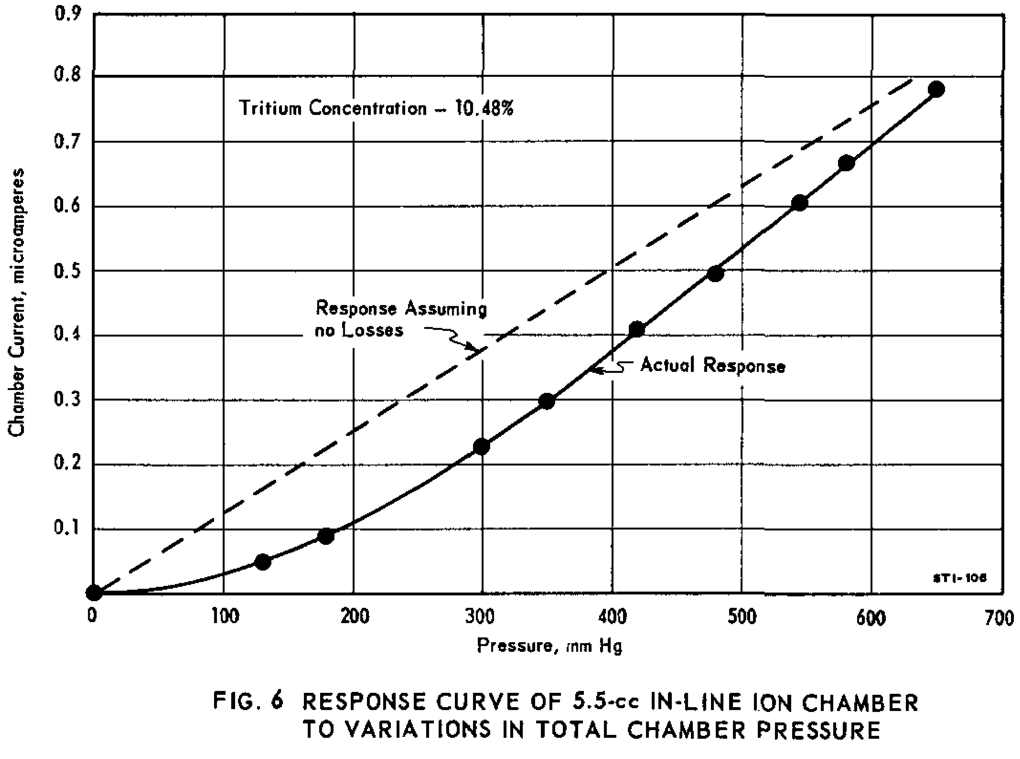

FIG. 6 RESPONSE CURVE OF 5.5-cc IN-LINE ION CHAMBER TO VARIATIONS IN TOTAL CHAMBER PRESSURE

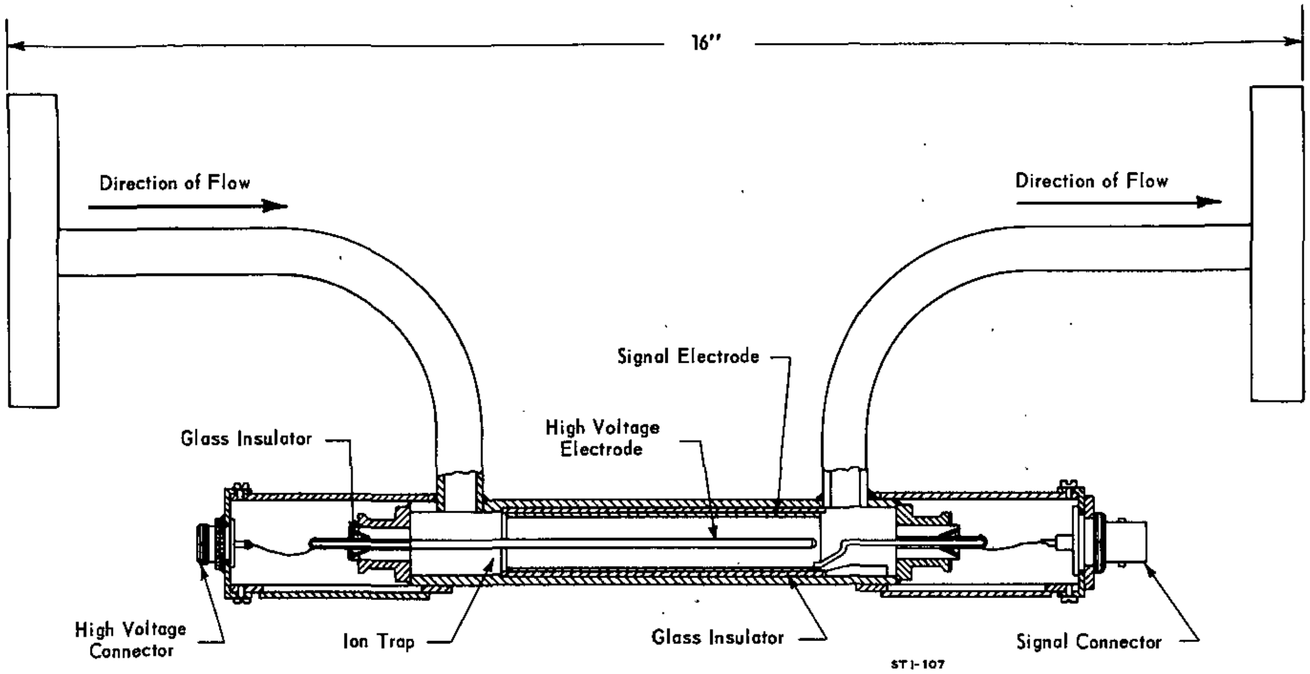

FIG. 7 DRAWING OF THE 20-cc IN-LINE ION CHAMBER

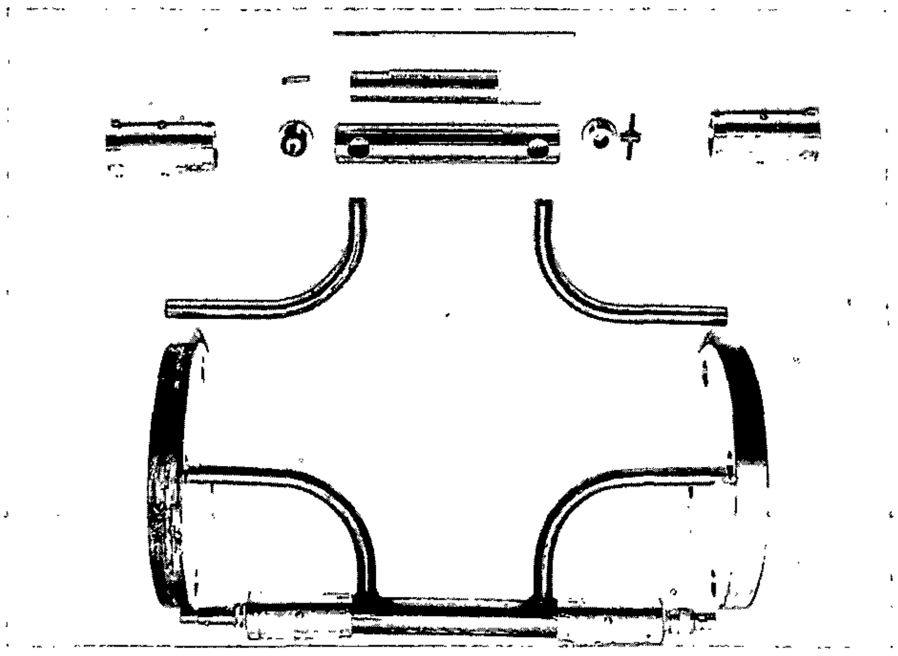

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FIG. 8 ASSEMBLED AND DISASSEMBLED 20-cc IN-LINE ION CHAMBER

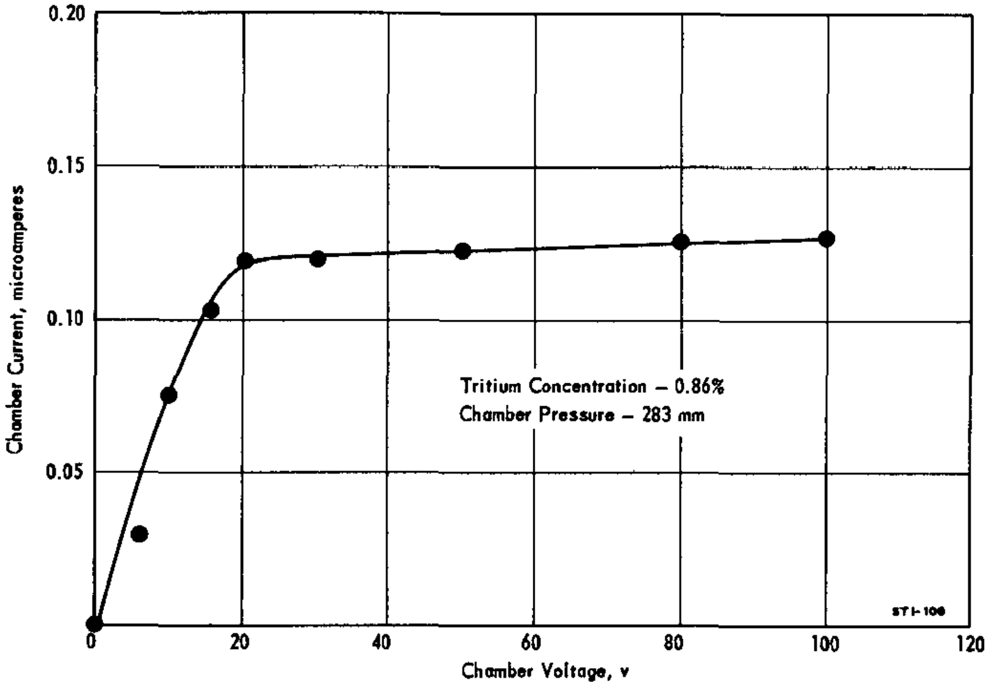

FIG. 9 SATURATION CURVE OF THE 20-cc IN-LINE ION CHAMBER

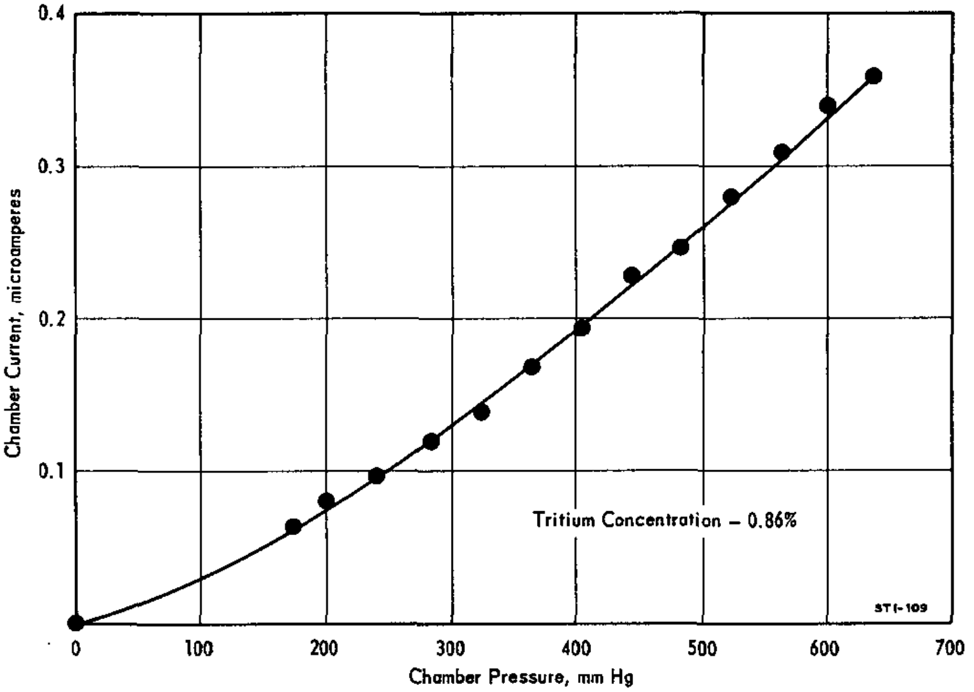

FIG. 10 RESPONSE CURVE OF THE 20-cc IN-LINE ION CHAMBER
TO VARIATIONS IN TOTAL CHAMBER PRESSURE

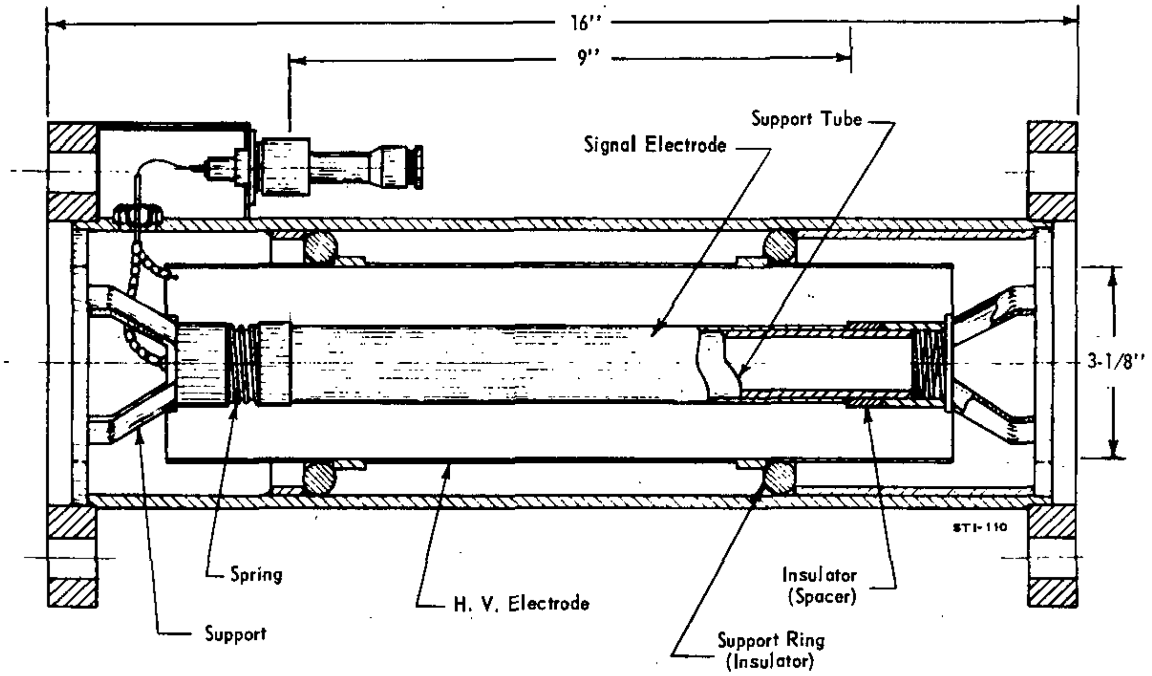

FIG. 11 DRAWING OF THE 930-cc IN-LINE ION CHAMBER

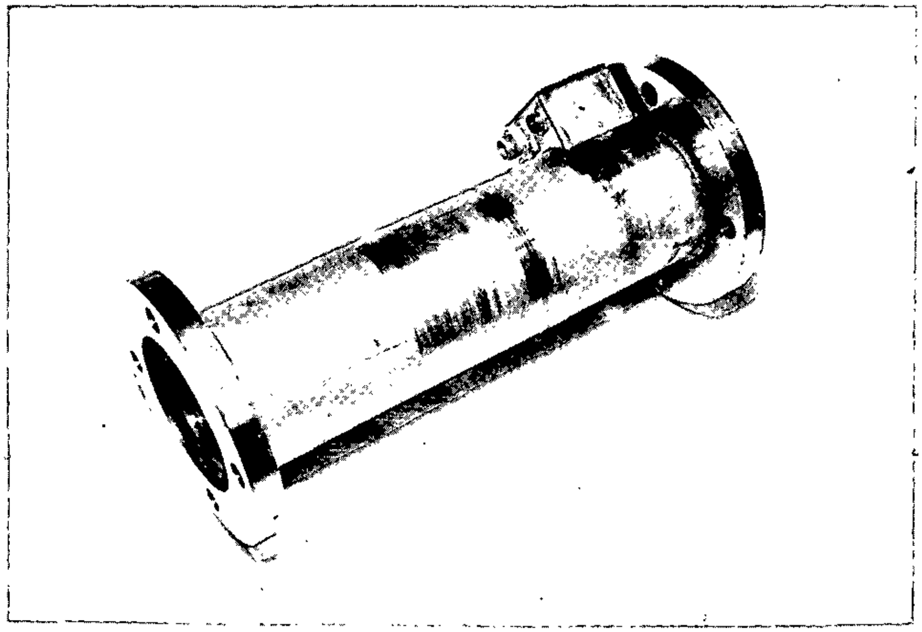

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FIG. 12 ASSEMBLED 930-cc IN-LINE ION CHAMBER

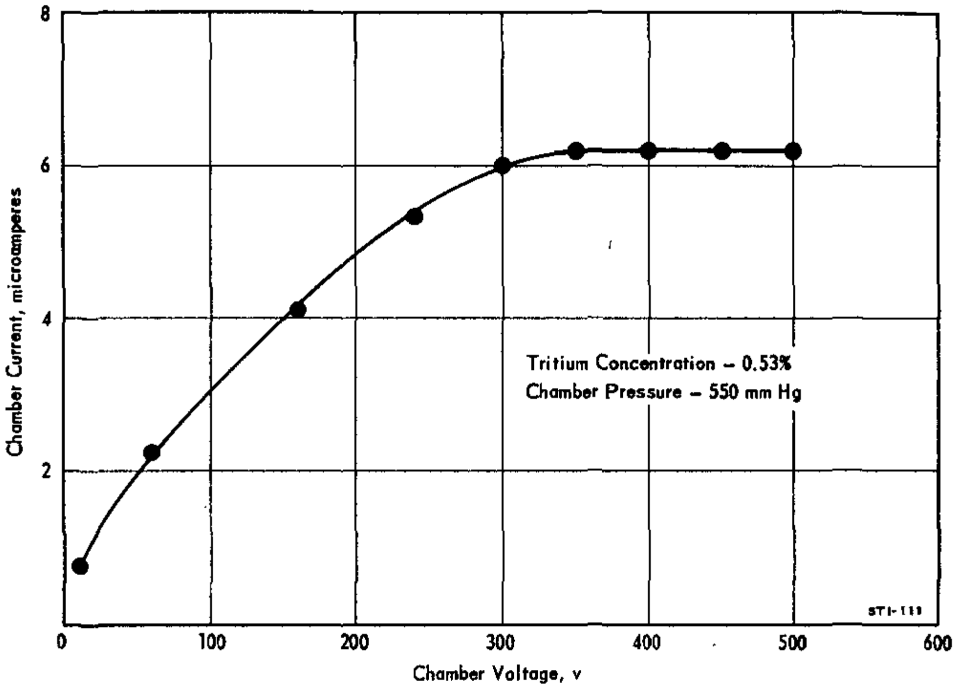

FIG. 13 SATURATION CURVE OF THE 930-cc IN-LINE ION CHAMBER

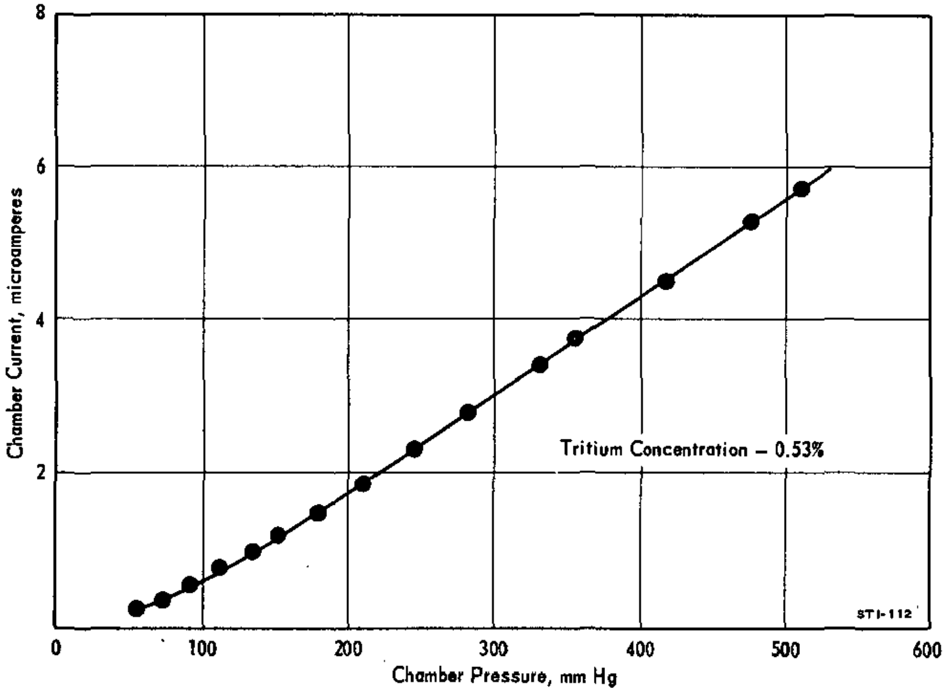

FIG. 14 RESPONSE CURVE OF THE 930-cc IN-LINE ION CHAMBER TO VARIATIONS IN TOTAL CHAMBER PRESSURE

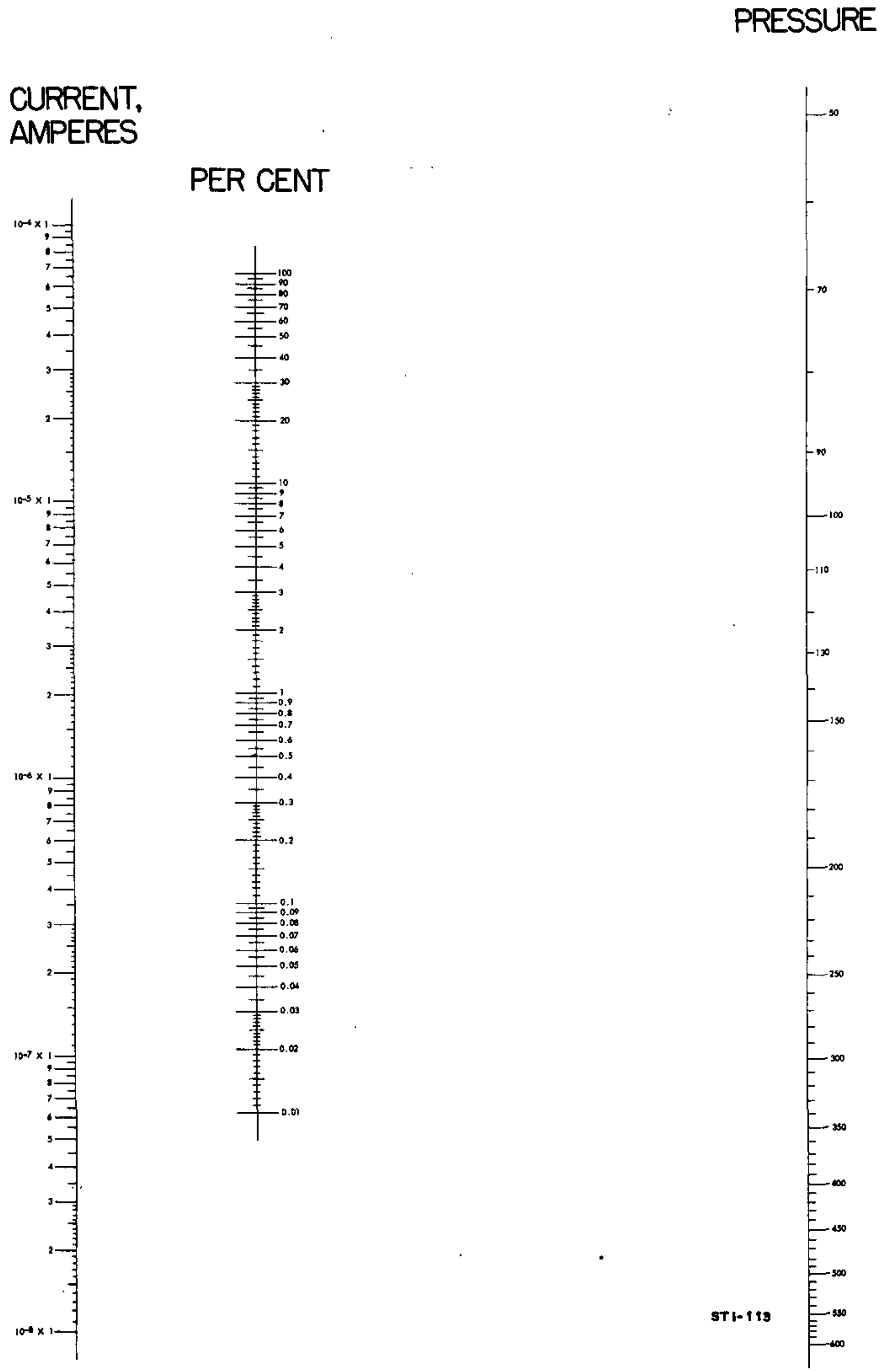

FIG. 15 TYPICAL NOMOGRAPH FOR USE IN DETERMINING TRITIUM CONCENTRATION WHEN CHAMBER PRESSURE AND CURRENT ARE KNOWN

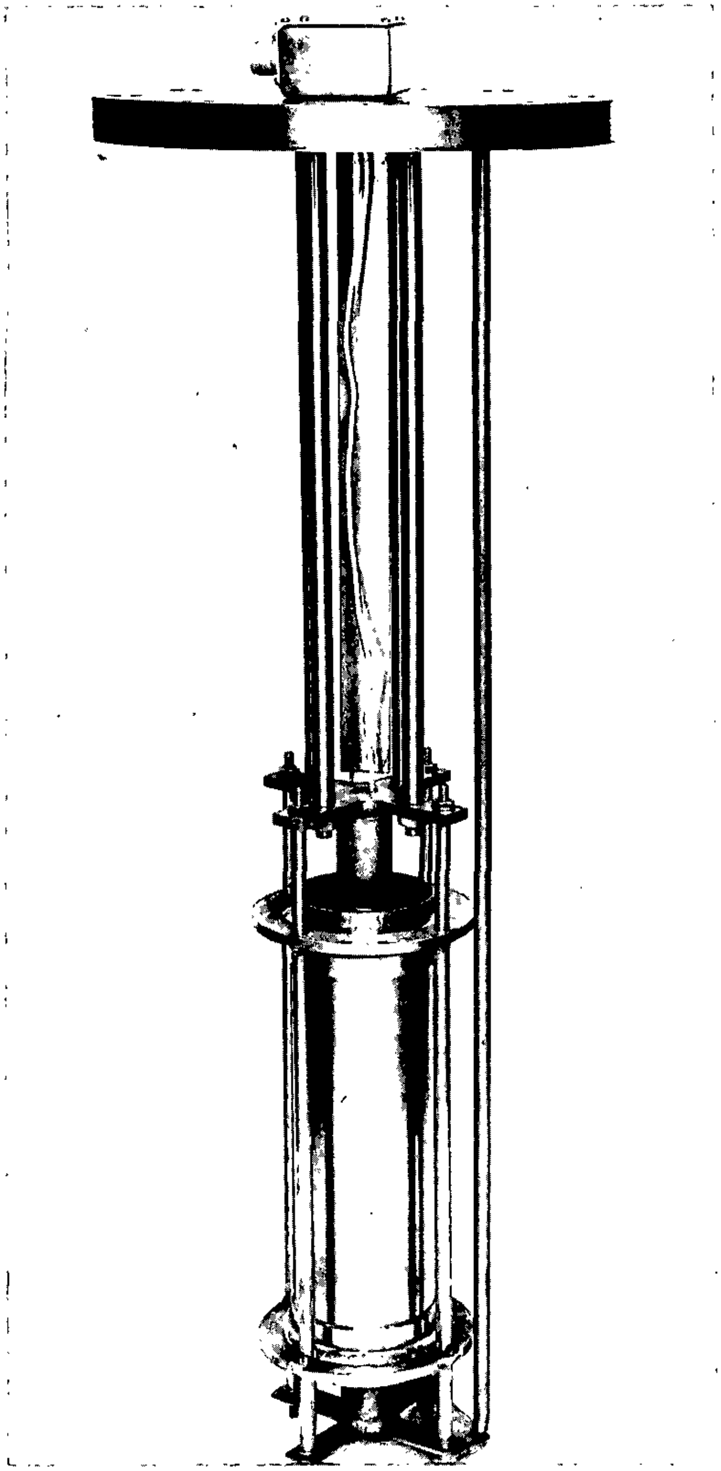

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FIG. 16 THE IN-TANK ION CHAMBER

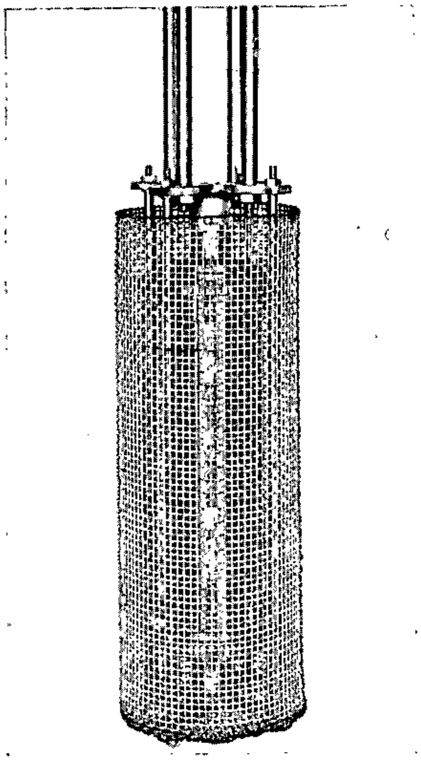

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FIG. 17 THE IN-TANK ION CHAMBER WITH SHIELD

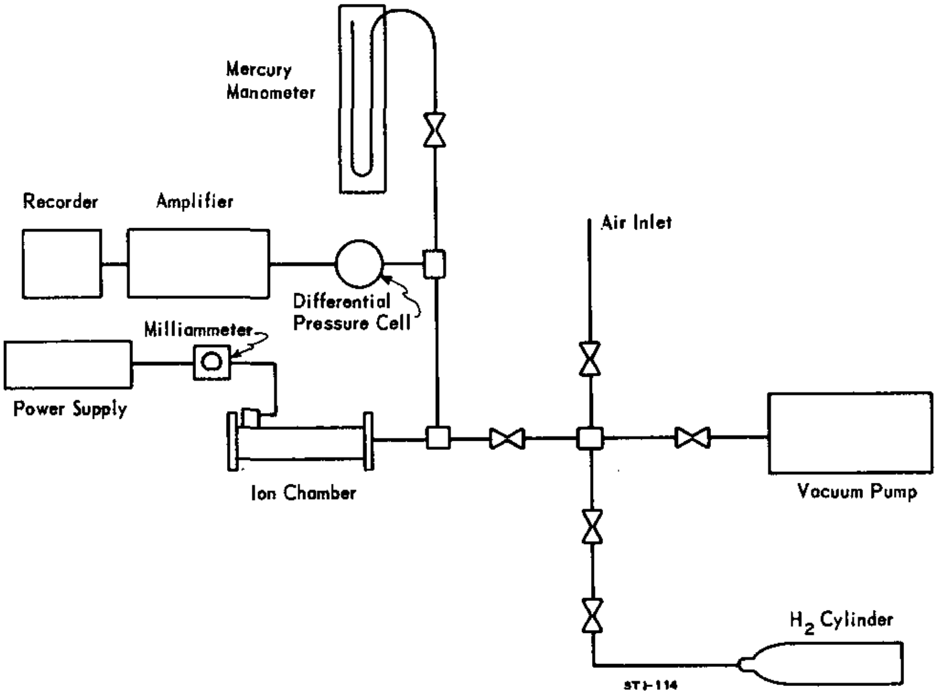

FIG. 18 TEST SETUP FOR MEASURING VOLTAGE BREAKDOWN IN THE ION CHAMBER

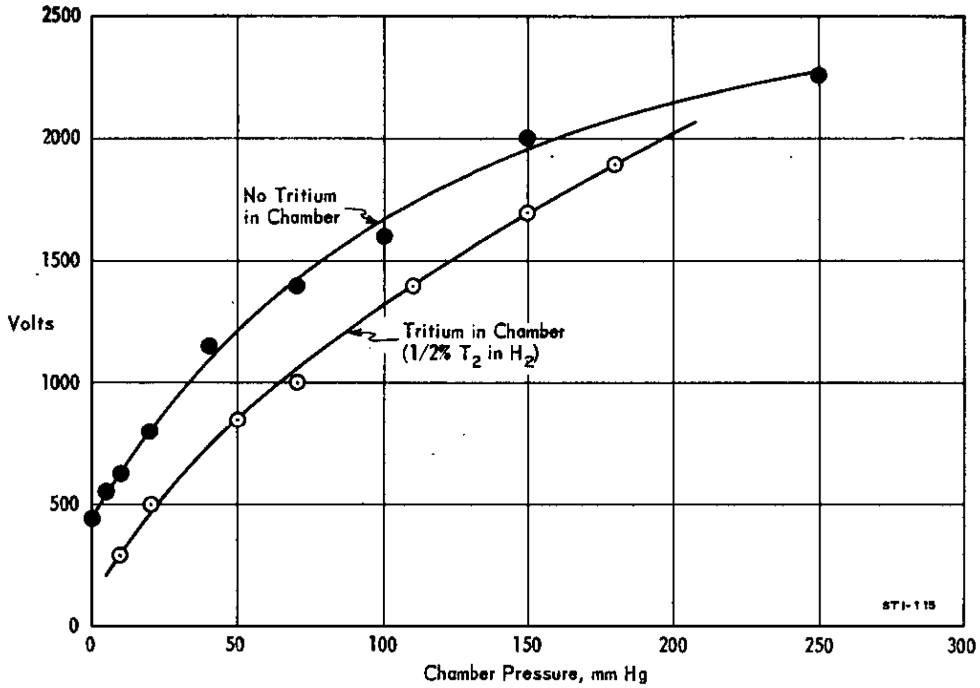

FIG. 19 VOLTAGE BREAKDOWN IN THE 930-cc ION CHAMBER AT DIFFERENT PRESSURES

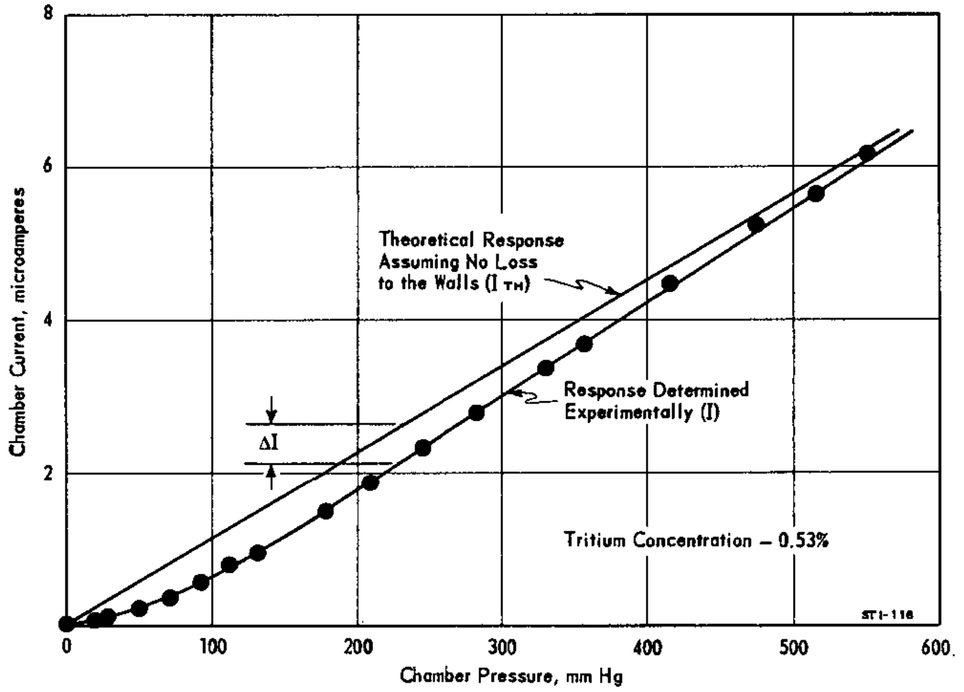

FIG. 20 RESPONSE CURVE OF 930-cc ION CHAMBER FOR USE IN DETERMINING APPROXIMATE EQUATION