THE DESIGN AND CONSTRUCTION OF A MASS SPECTROMETER

By

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THE DESIGN AND CONSTRUCTION

OF A MASS SPECTROMETER

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PREFACE

This report describes the design and construction of a mass spectrometer built for the Physics Laboratory at Oklahoma Agricultural and Mechanical College, Stillwater, Oklahoma. The principal features of its design are patterned after a design presented by Dr. O. A. Nier of the University of Minnesota, in a paper published by "Review of Scientific Instruments", Vol. II, p. 212 (1940).

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GENERAL ARRANGEMENT

The general arrangement of the magnetic and electric field is shown in Fig. 1.

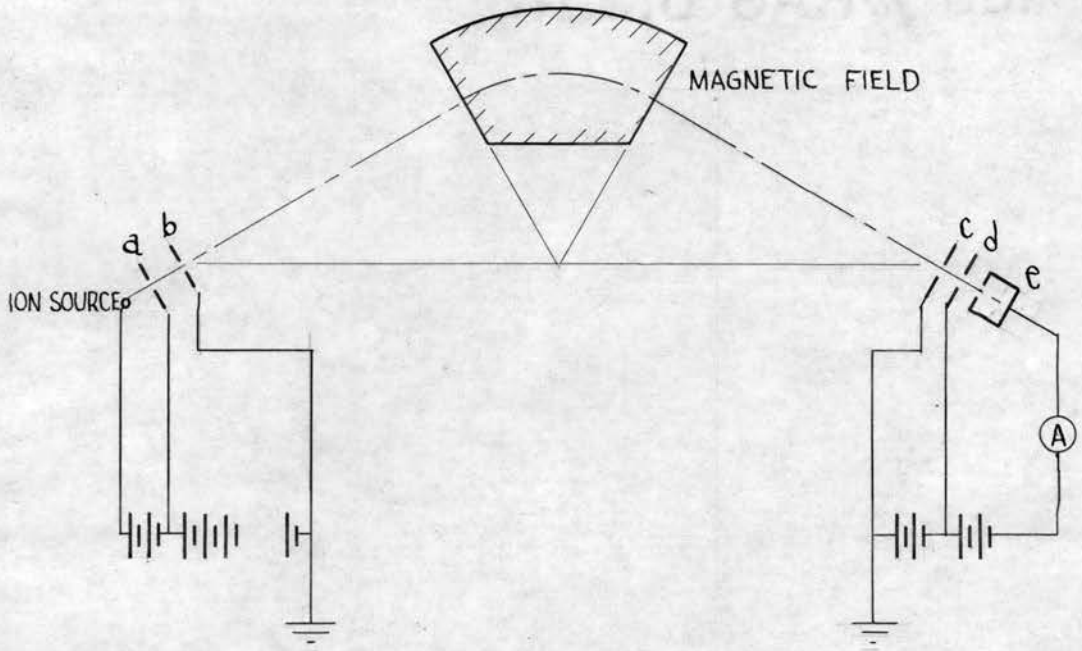

Fig. 1

The positive ions are emitted from the ion source and accelerated to the first plate (a) by a small potential. After passing through the slit (a) the ions are accelerated by a much larger potential to slit (b) where they enter a field free space. After traversing this space they encounter the magnetic field which causes the ions to curve and travel toward the slits (c) and (d) where, after leaving through slit (d), the ions are captured in the Faraday cup (e). The ion current

then is measured by the current measuring device (A).

The ion path is directed along a 3/4 inch brass tube which is bent in a horizontal plane around a radius of 12.5 cm to its center. One end of this tube is mounted in the ionization chamber which contains the ion source and accelerating slits, while the other end is mounted in the collecting chamber which contains the Faraday cup. These two chambers are supported by 1-1/2 inch brass tubes which are screwed through flanges to a table.

The spectrometer is evacuated through the tube supporting the collecting chamber and this tube is connected to the vacuum pumps and traps located beneath the table.

For purposes of adjustment, the magnet is supported on top of the table on wooden tracks.

Details of all components of the instrument are presented in the drawings and descriptions accompanying this report.

THEORY

Consider an ion with mass m, a charge e and having a velocity v directed along line AE. As it enters normal to the boundary of the magnetic field its center of curvature in the magnetic field must be along line ED. If it is required that the particle pass through the point B it then must also leave the magnetic field normal to its boundary and its center of curvature must be located at point D.

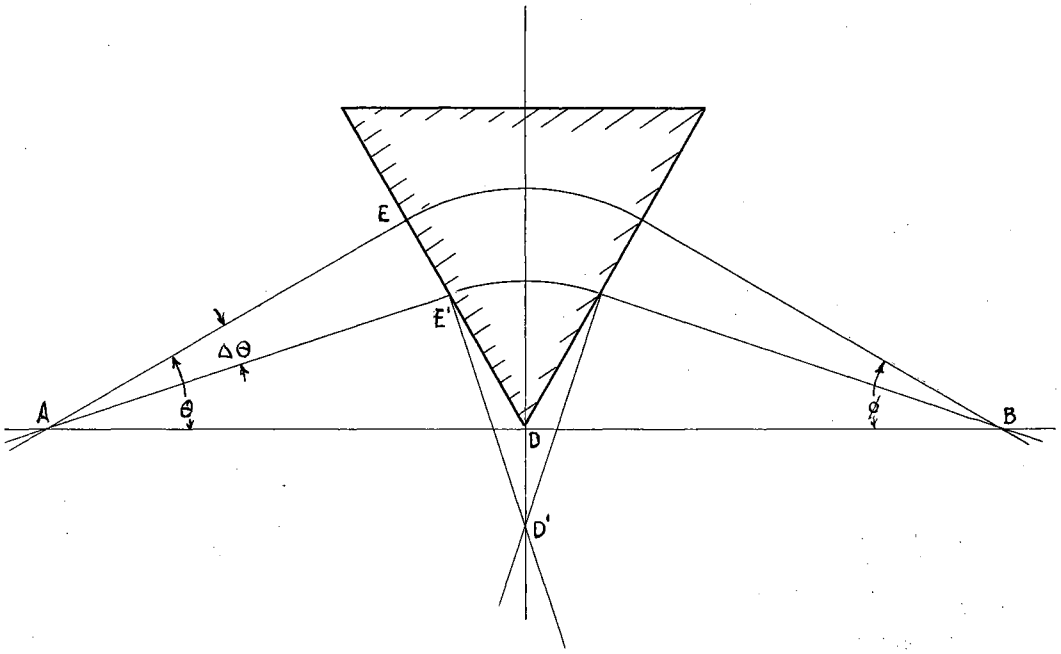

Fig. 2

In Fig. 2, AD = DB, $\angle\theta = \angle 0$ and $\angle AED = \angle DFB = 90^\circ$

The magnetic field exerts on the ion a centripetal force which can be represented by Eq. 1.

$$\frac{Hev}{c} = \frac{mv^2}{R} \qquad (1)$$

where

H = magnetic field intensity in gauss

e = ionic charge in ESU

v = velocity in cm/sec

m = mass in grams

R = radius of curvature in cm

c = 3×10^{10} cm/sec

If the ion obtains its velocity by falling through a potential

difference V, then its kinetic energy can be represented by Eq. 2.

$$\frac{1}{2} mv^2 = \frac{eV}{300} \text{ ergs} \tag{2}$$

where V is in volts.

Eliminating v between Eqs. 1 and 2 leads to Eq. 3.

$$\frac{m}{e} = \frac{H^2R^2}{V} \frac{300}{2c^2} \tag{3}$$

If the unit of mass is taken as that of the hydrogen atom (1.622×10^{-24}gm) and the unit of charge is 4.80×10^{-10}esu then

$$\frac{m'}{e'} = \frac{H^2R^2}{V} \frac{300}{2c^2} \frac{4.80 \times 10^{-10}}{1.662 \times 10^{-24}}$$

or

$$\frac{m'}{e'} = 4.82 \times 10^{-5} \frac{H^2R^2}{V} \tag{4}$$

where

m' = number of mass units

e' = number of charge units

H = magnetic field intensity in gauss

R = radius of curvature in cm

V = accelerating potential in volts

To show the direction focusing effect of this arrangement, assume an ion directed along a path AE' at a small angle $\triangle\theta$ to that of the previously considered ion. Considering that the speed of this ion is the same as that of the first, it will follow a path whose curvature is the same as the first and its center of

curvature will be along a line perpendicular to its path at the point where it enters the field or along the line E'D'. If the ion is to pass through the point B its center of curvature must be at D'. Then, if the length of the line E'D' can be shown to be equal to the radius of curvature ED caused by the magnetic field, the conditions for direction focusing are fulfilled.

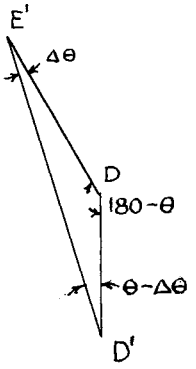

Fig. 3

Triangle E'D'D' has been re-drawn in Fig. 3 showing its angles as determined by the geometry of the figure.

$$E'D = ED - EE'$$

but

$$EE' = AE \tan \Delta\theta$$

and

$$AE = \frac{ED}{\tan \theta}$$

therefore

$$E'D = ED - \frac{ED \tan \Delta\theta}{\tan \theta}$$

or

$$E'D = \left[1 - \frac{\tan \Delta\theta}{\tan \theta}\right] \qquad (5)$$

Then by the law of sines

$$\frac{E'D}{\sin(\theta + \triangle\theta)} \quad \frac{E'D'}{\sin(180 - \theta)} \tag{6}$$

Substituting Eq. 5 into Eq. 6 and solving for E'D' leads to Eq. 7

$$E'D' = ED\left[\frac{\sin(180 - \theta)\left[1 - \frac{\tan\triangle\theta}{\tan\theta}\right]}{\sin(180 - \triangle\theta)}\right] \tag{7}$$

The coefficient of the ED term of Eq. 7 is equal to unity provided $\triangle\theta$ is small enough that

$$\tan\triangle\theta = \triangle\theta$$
$$\sin\triangle\theta = \triangle\theta$$

and

$$\cos\triangle\theta = 1$$

This means that if the angle of divergence is held to a small angle, the velocity of the two ions is constant and satisfy Eq. 4, their paths will intersect at point B of Fig. 2.

N. F. Barber[1] has presented a more general treatment of the focusing properties of this design.

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[1] N. F. Barber, Note on the Shape of an Electron Beam Bent in a Magnetic Field, Proceedings of the Leeds Philosophical Society, Vol. II, pp. 427 (33).

MAGNET DESIGN

The dimensions of the magnetic circuit are substantially the same as those presented in the Nier[2] report. The pole faces differ in design and size in order that unusuable flux may be partially eliminated, thereby increasing the flux density for a given magnetomotive force. The basis for this change in pole face size is a brief indication of the fringing effects encountered by Nier with his magnet. He states in his report that the flux can be considered uniform across the pole face and for a distance equal to the air gap from the pole boundary. At that point the flux is cut off sharply. The pole face was designed so that if this condition holds true the desired path of the ions will be at the pole face. The area of the pole face is about 10.85 square inches as compared to 17.75 square inches used by Nier. A comparison of the two pole faces is presented in Drawing 7.

The core of the magnet and the pole pieces were cast as a unit of commercial grade S.A.E. 1020 steel by the Oklahoma Steel Casting Company, Inc., at Tulsa, Oklahoma. The yoke was cut from a piece of scrap plate obtained from the same company and was accepted as the same material. Magnetic properties for the design of the magnet were taken from the International Critical Tables for a similar material. No magnetic tests were conducted to confirm these values for the material used.

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[2] O. A. Nier, "A Mass Spectrometer for Routine Isotope Abundance Measurements," Review of Scientific Instruments, Vol. II, pp. 212 (1940).

The design was based upon a maximum flux density in the iron of 15,000 gauss, and a permeability of 3,000. The reluctance of the complete magnetic circuit was calculated to be about 0.0154 CGS units of which 0.0150 units was due to the air gap. The total flux allowed by the maximum flux density of 15,000 gauss in the iron is 1.16×10^6 maxwells and to obtain this flux requires 14,100 ampere turns. With no allowance made for fringing effects, this gives a flux density between the pole faces of 8,450 gauss. Roughly accounting for fringing effects by increasing the area by the gap width results in a flux density of 5,500 gauss.

The coils were wound with No. 14 Formax covered annealed copper wire. Paper .021" thick was placed between layers. The spools were made by soldering flanges 1/8" thick and 6-1/2" outside diameter onto 4"-.065" brass tubing 8-1/4" long. As a result of a variation in winding technique, one coil contains 1,862 turns and the other contains 1,881 turns. To obtain the design magnetomotive force then requires 3.8 amperes.

THE VACUUM SYSTEM

The entire vacuum system was constructed of brass tubing and plate and the details of construction can be obtained from the accompanying drawings. All permanent joints were made with "Easy flow 45"[3], a commercial silver solder containing 45% silver and having a melting point of 785°C. Joints that could not be made with silver solder and

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[3] "Easy flow 45" can be obtained from The Metal Good Corporation, North Boston, Tulsa, Oklahoma.

joints to be disconnected were made with common soft solder.

Flanged joints provided complete accessibility to the electrodes and slits in both the ionization chamber and the collecting chamber. These joints were sealed with lead gaskets and held by eight 8-32 round head brass machine screws. A die and punch for cutting these lead gaskets has been provided.

Electrical connections to both the ionization chamber and collecting chamber were made through Kovar-Glass Seals obtained from Stupakoff Ceramic Company.[4] Soft solder was used to make all connections to these Kovar-Glass Seals because the risk of cracking the glass becomes much greater at the temperatures required for silver soldering.

The oil diffusion pump used in the system is a VMF-10 produced by Distillation Products Company.[5] It has a capacity of 10 liters per second at 10^{-4}mm of mercury pressure and a guaranteed ultimate pressure of 10^{-6}mm of hg. The pump requires 150 cc of "Octoil" provided by the same company.[6] A 1/4" copper tube has been provided for filling the pump with oil.

A water jacket was placed around the horizontal section of the piping to condense any of the oil vapor that might escape from the

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[4] Stupakoff Ceramic & Manufacturing Company, Latrobe, Pa.

[5] Distillation Products Inc., Vacuum Equipment Division, 755 Ridge Road West, Rochester 13, New York.

[6] T. J. Morgan, "Use of Oil Diffusion Pumps in Mass Spectrometers," Review of Scientific Instruments, Vol. XVIII, pp. 926.

diffusion pump. Care must be taken to direct the flow of water through the water jacket in the proper direction. One tube extends to the top of the water jacket and is to be used as the water outlet.

Provisions were made to absorb the water vapor present by placing a phosporous pentoxide trap at the bottom of the tube connecting to the collecting chamber. The phosphorous pentoxide is contained in a heavy lipped flask and held by a brass yoke to the flanged end of the tube. The seal is made by a rubber gasket.

Swedged joints connected with soft solder provided a means of removing the diffusion pump from the system and another joint of the same type was provided immediately below the table in the tubing supporting the collecting chamber.

A brass "stop and waste" valve was placed between the diffusion pump and the fore-pump connection. This valve is a standard water valve with the threads bored out for soldered joints and the plug lapped in with fine emery. Valves of this type have been successfully used by Saxon and Richards.[7]

If it becomes necessary to install a valve between the diffusion pump and the collecting chamber, space has been provided at the end of the horizontal section of the piping through the water jacket. The plug that contains a pressure connection and seals this horizontal tube is fastened in place with soft solder for easy removal. No

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[7] D. Saxon and J. Richards, "A Vacuum Lock for Continuously Evacuated Systems," Review of Scientific Instruments, Vol. XX, pp. 745 (October 1949).

design for this valve will be presented in this report.

ION SOURCE

The ion source for gases differs in principle from that used by Nier[8] in that the magnetic field across the ion chamber is reduced to zero. Electron collision was the process used for ionizing the gases but the orientation of the electron beam was altered from the Nier design.

A very detailed analysis of the Nier design has been made by Jordan and Coggeshall[9] showing the effects of the magnetic and electric field on the paths of the electrons and positive ions. The conditions for minimum mass selectivity are presented both for an ion source completely submerged in the magnetic field and one such as described by Nier where the field is only a stray field from the analyzer magnet. The conditions imposed on the Nier type are such that the measurements made at differing magnetic field intensities can not be relied upon to better than about 5%, and that measurements made with varying electric fields can not be relied upon to better than about 1% due to space charge effects.

The electron beam is orientated so that the electrons are directed across the collimating slit as shown in Fig. 4. The electrons are accelerated from the filament across the slot of the ionization cup

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[8] A. O. Nier, Op. Cit., p. 212.

[9] E. B. Jordan and D. D. Coggeshall, "Measurement of Relative Abundance with the Mass Spectrometer," Journal of Applied Physics, Vol. XIII, pp. 539 (1942).

and are collected by the Faraday cup. An adjustable slit is used to collimate the electron beam as the electrons enter into the ionization cup. Ions formed in the ionization cup are accelerated by a small potential between the ionization cup and the first accelerating plate and are further accelerated to the second accelerating plate by a much higher potential.

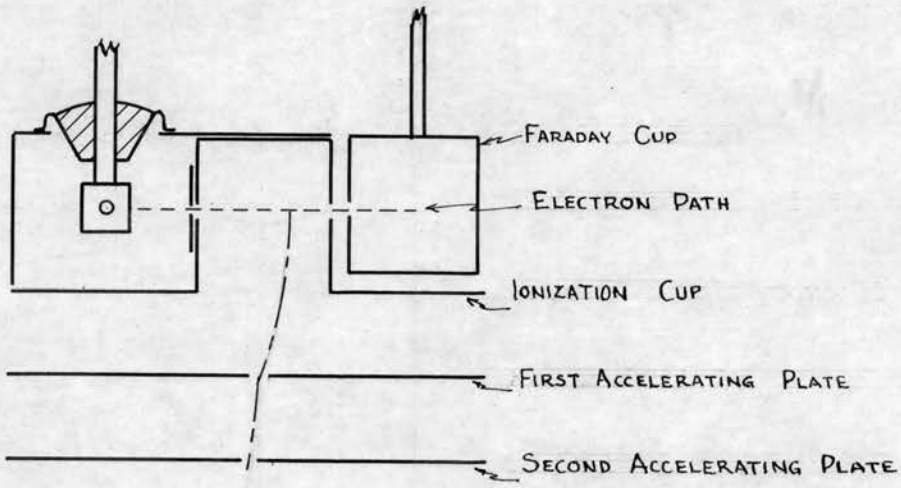

Fig. 4

The electrostatic field of the ionization cup was mapped by the electrolytic tank method and the potential lines are shown in Fig. 5. It can be noted that a very definite focusing effect exists and that this effect is such as to produce a larger ion current and possibly produce some selective effects to ions of different mass charge ratio .

Ions formed by the electron beam will fall through about 94% of the potential between the ionization cup and the first accelerating

plate regardless of the position at which the ion is formed along the electron path. The width of the slit in the first accelerating plate will determine the upper limit to this voltage. The spread of the potential through which the ions will fall will range from 94% of the potential to an upper limit determined by the slit width.

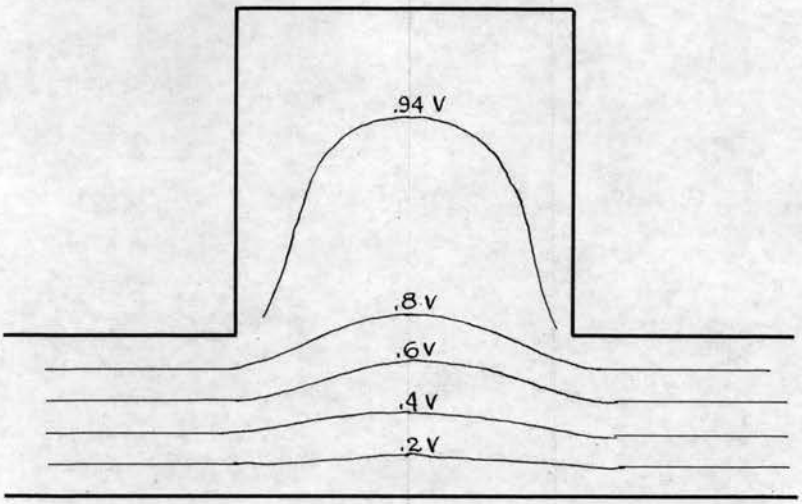

Fig. 5

In order to minimize the energy spread it is necessary to use small potentials between the ionization cup and the first accelerating plate. Utilizing small potentials allows the space charge effects in the ionization cup to decrease the energy spread still further.[10]

With the design employing these principles, a few difficulties arise which leave no doubt as to the advisability of the design. First, the focusing of the electron beam must be accomplished by elec-

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[10] C. F. Robinson, "Effects of Space Charge on the Focusing Properties of a 180° Mass Spectrometer," Review of Scientific Instruments, Vol. XX, pp. 745.

trostatic fields and second, the increase of ion current expected by the configuration will be decreased due to space charge effects. Further, there is some doubt that this method does actually provide less mass discrimination than one employing a magnetic field. A complete analytic study of the mass discrimination problem has been stated to be impossible by Jordan and Coggeshall[11], but no experimental data are available to confirm this point.

The mechanical design of the ion source allows adjustment of the slit widths and orientation of the slits relative to each other. Complete access to all electrodes is had through the end plates of the chamber. All electrical connections were made through Kovar-Glass Seals soft soldered to the end plates of the chamber. The electrodes were made from nickel sheets.

THE COLLECTING CHAMBER

The collecting chamber is identical with the ion chamber in external appearance. The nickel collecting slit is adjustable in width and ions entering through the slit are collected in a nickel Faraday cup after passing through a grid which is held positive with respect to the Faraday cup. This grid is to eliminate any secondary emission caused by ions bombarding the Faraday cup.

The grid and cup were mounted on the cover plate of the chamber through Kovar-Glass Seals as shown in Drawing 3.

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[11] Jordan and Coggeshall, Op. Cit., p. 545.

THE GAS ADMITTANCE VALVE

Details of the gas admittance valve are given in Drawing 5. The valve operates on the principle of the differential screw. A screw of 27 threads per inch operates in a threaded piston. A standard 4/0 steel taper pin is pressed into the threaded piston and fits into a brass seat. One turn of the differential screw moves the piston .0013 inches and changes the cross sectional area of the opening about .0004 square inches.

A metallic bellows is used to eliminate all packing. A coarse adjusting screw with a lock is provided which moves the steel taper pin .037 inches per turn.

It should be noted that this type valve or any other type which uses a gas flow and throttling process produces some selectivity by allowing lighter gas molecules to diffuse more easily than the heavier molecules.

AUXILIARY EQUIPMENT

The following auxiliary equipment will be required for the successful operation of this instrument:

1. Voltage source for ion gun
2. Current source for magnetic analyzer
3. Current measurement device for collector
4. Gas handling equipment
5. Pressure gauges

None of this equipment was constructed, but some consideration was

given to the design of these components. A list of references to previous designs of these components follows.

Voltage Source for Ion Gun

The accelerating voltage must be stable to one part in several thousand.[12] A battery could be used for this purpose or an electronic device such as described by Nier[13] can be employed.

Current Source for Magnetic Analyzer

The current source for the magnet must be at least two times as stable as the accelerating voltage since the magnetic field strength appears as a squared term in Eq. 4. A storage battery can supply this current or it can be regulated by an electronic device. The magnet was designed to be operated at relatively high currents which would be difficult to control with ordinary vacuum tubes. Nier[14] has presented a design for a current regulator for 110 ma max., but this design could not be used for this particular magnet without modification. An earlier paper by Nier[15] presents a design for a current regulator which was built for a magnet of similar design. This device adjusts the accelerating voltage to compensate for magnetic field fluctuations.

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[12] Hipple, Grove, Hickam, "Electronic Problems Involved in the Practical Application of the Mass Spectrometer," Review of Scientific Instruments, Vol. XVI, pp. 69 (April 1945).

[13] O. A. Nier, "Mass Spectrometers for Isotope Analysis," Review of Scientific Instruments, Vol. XVIII, pp. 398.

[14] Ibid, Vol. XVIII, p. 398.

[15] O. A. Nier, Review of Scientific Instrument, Vol. VI, pp. 254(35).

Current Measurement Device for Collector

A major consideration for the current measuring device other than being sensitive to current of the order of 10^{-15} to 10^{-16} amp. is its time of response. To make relative abundance measurements it is important to have the response time sufficiently short to assure that the ion current does not change during the readings.

Very extensive literature exists on the design of simple electrometer circuits employing the FP-54 electrometer tube.[16 - 17] Other less expensive tubes can be employed in a variety of circuits but with less sensitivity.[18]

Recording mechanisms of various designs can be employed to simplify the measurements but the requirements for such a device demand very careful design. Two such designs, one by Nier[19] and another by Hipple, et al[20] have proven satisfactory. Companies who produce mass spectrometers commercially have developed very satisfactory devices.[21 - 22]

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16 D. B. Penick, Review of Scientific Instruments, Vol. VI, pp. 115.

17 L. A. DuBridge, "The Amplification of Small Direct Currents," Physical Review, Vol. XXXVII, pp. 392 (31).

18 V. J. Caldecourt, "An Electrometer Amplifier," Review of Scientific Instruments, Vol. XX, pp. 748 (October 1949).

19 O. A. Nier, Op. Cit., p. 254.

20 Hipple, Grove, Hickman, Op. Cit., p. 69.

21 Consolidated Engineering Corp., 300 N. Sierra Madre Villa, Pasadena 8, California.

22 Westinghouse.

It should be pointed out that the response time of the recording device can become of minor importance provided some type of automatic ion current regulator can be devised. Such devices have been employed by Winn and Nier.[23] Two different principles are employed; one controlling the filament current, the other controlling space charge.

Gas Handling Equipment

The requirement that the gas sample be introduced into the ionization chamber at a pressure of the order of 10^{-4} mm of Hg. with minimum separation of particles of different mass is the only important consideration. The nature of the gas and its source will control to a great extent the particular equipment employed.

Pressure Gauges

Standard pressure gauges can be installed at two points provided. One gauge outlet is located at the ionization chamber. Another gauge outlet is located in the pumping manifold.

DESIRABLE MODIFICATIONS

It would be very desirable to completely redesign the ion source to eliminate many of the undesirable features. The filament mounting is very poor and should be corrected to provide tension and to allow expansion of the filament when heated. A better focusing method should be provided for the electron beam.

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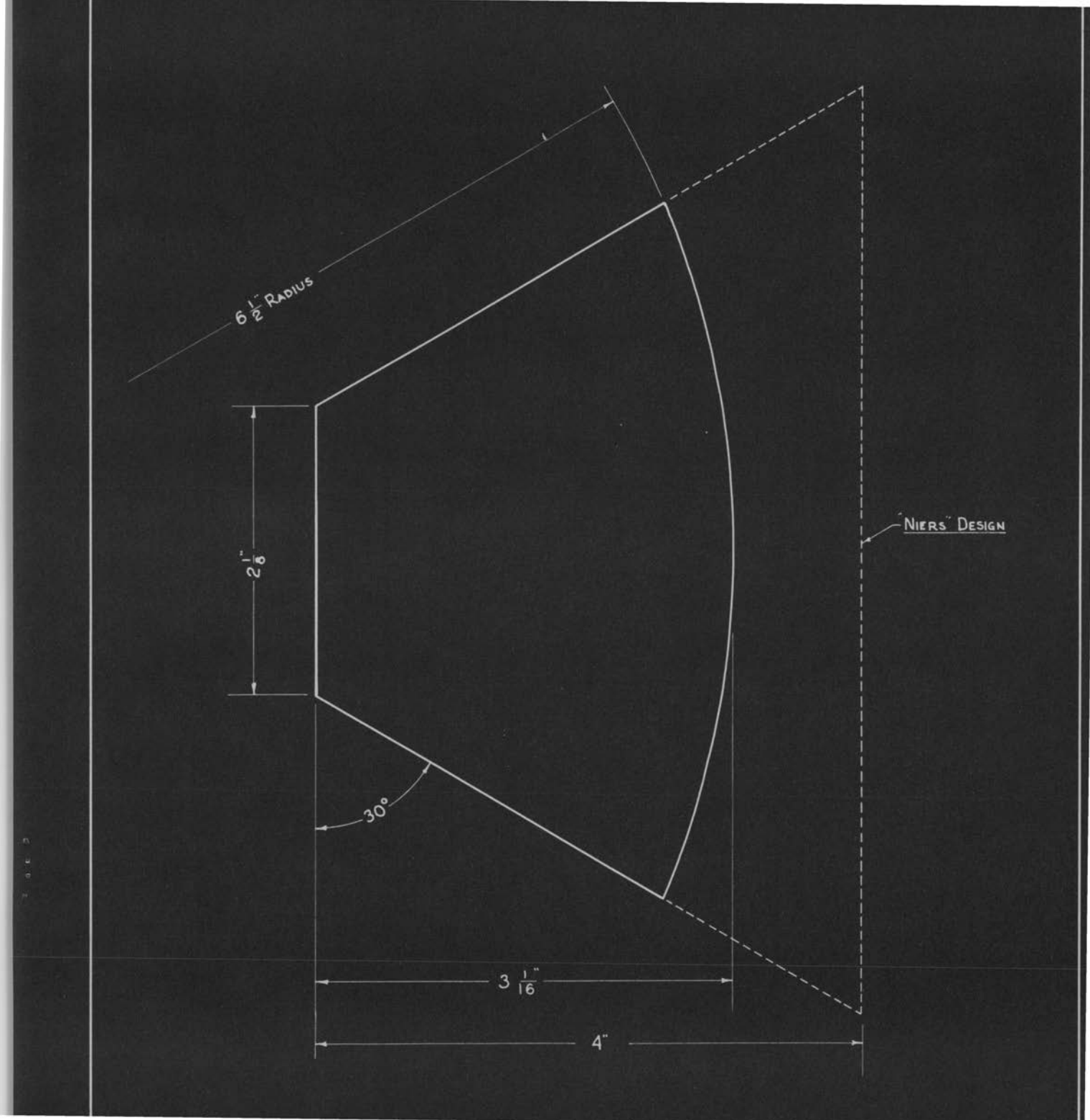
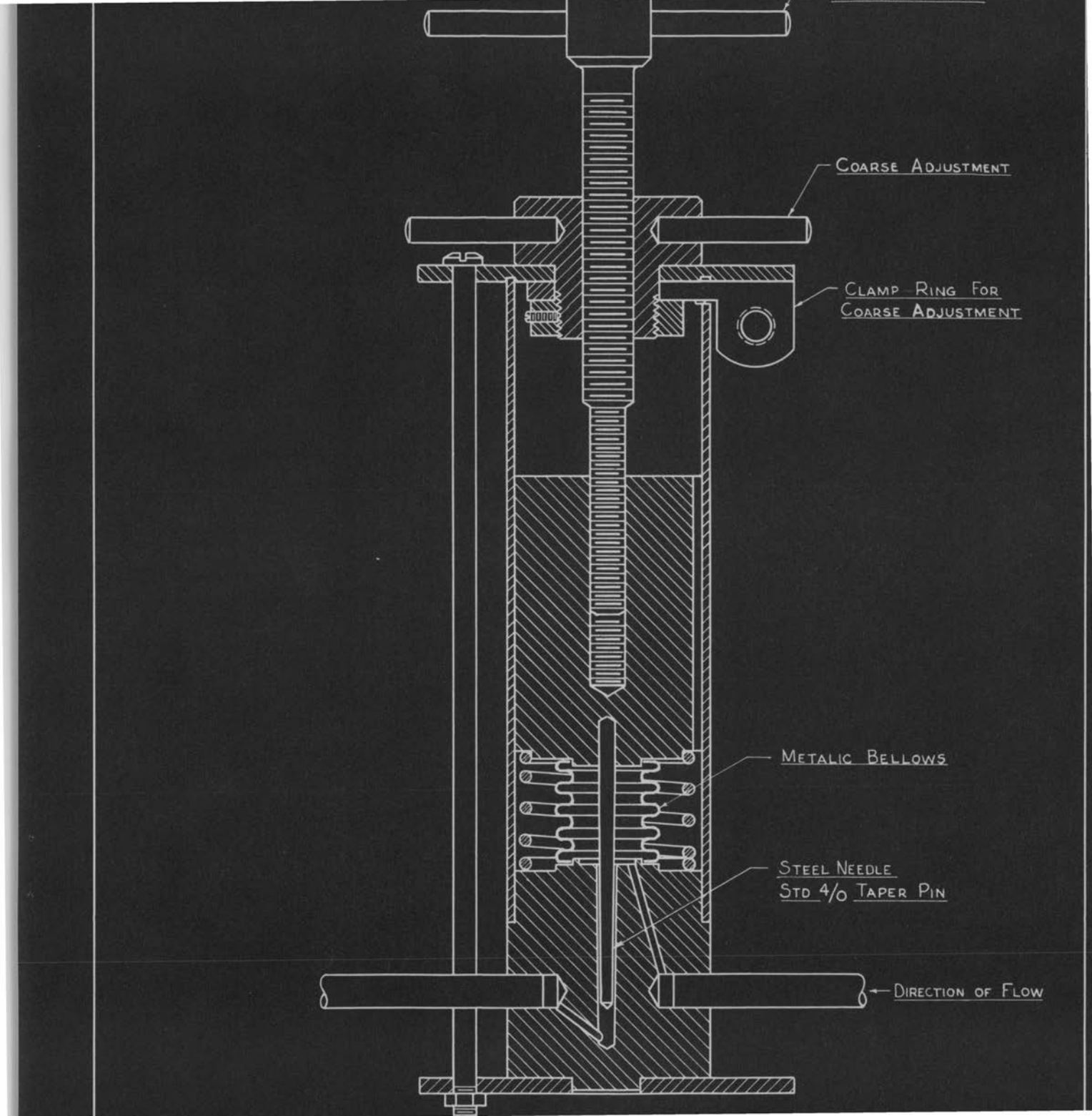
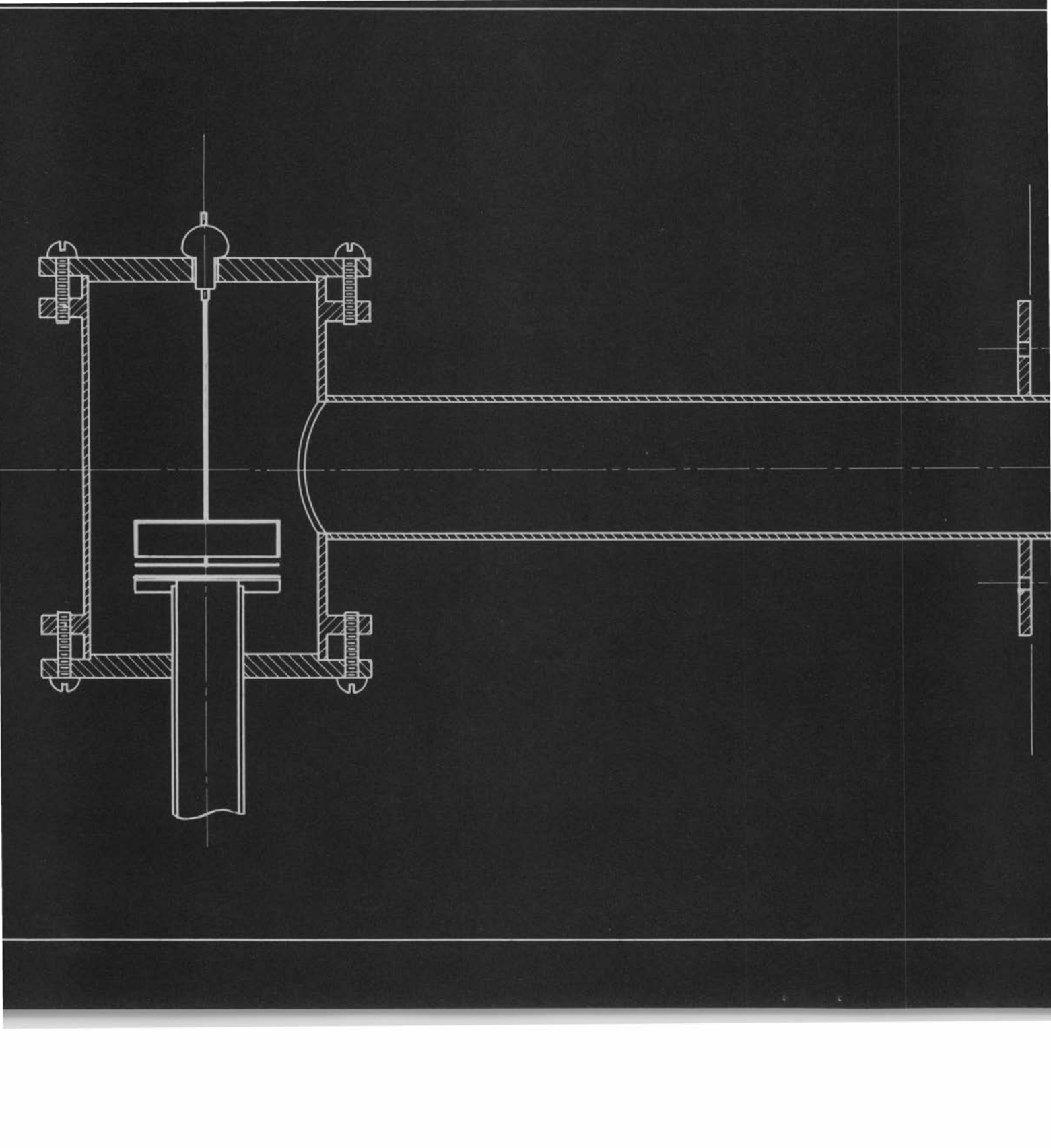
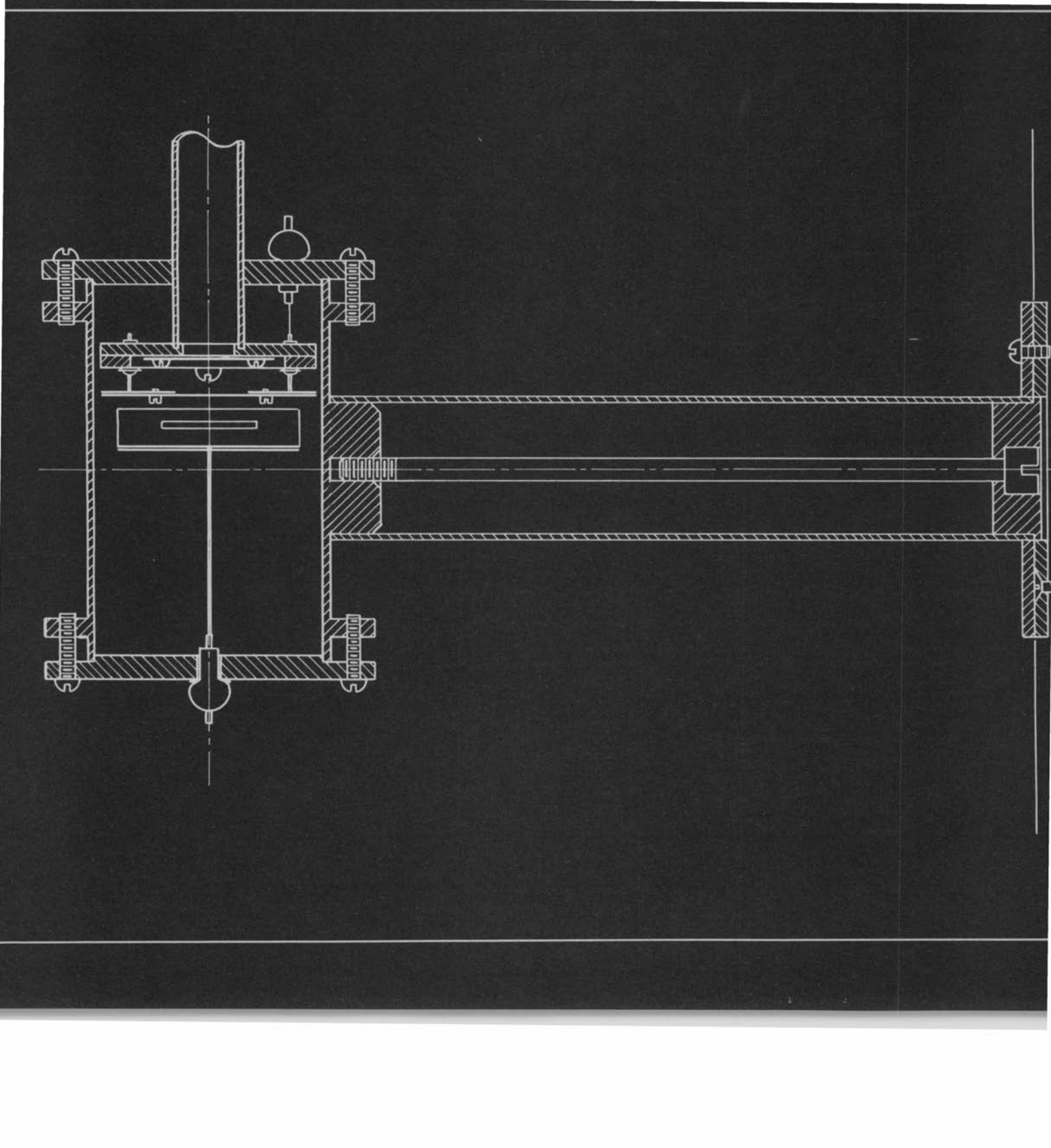
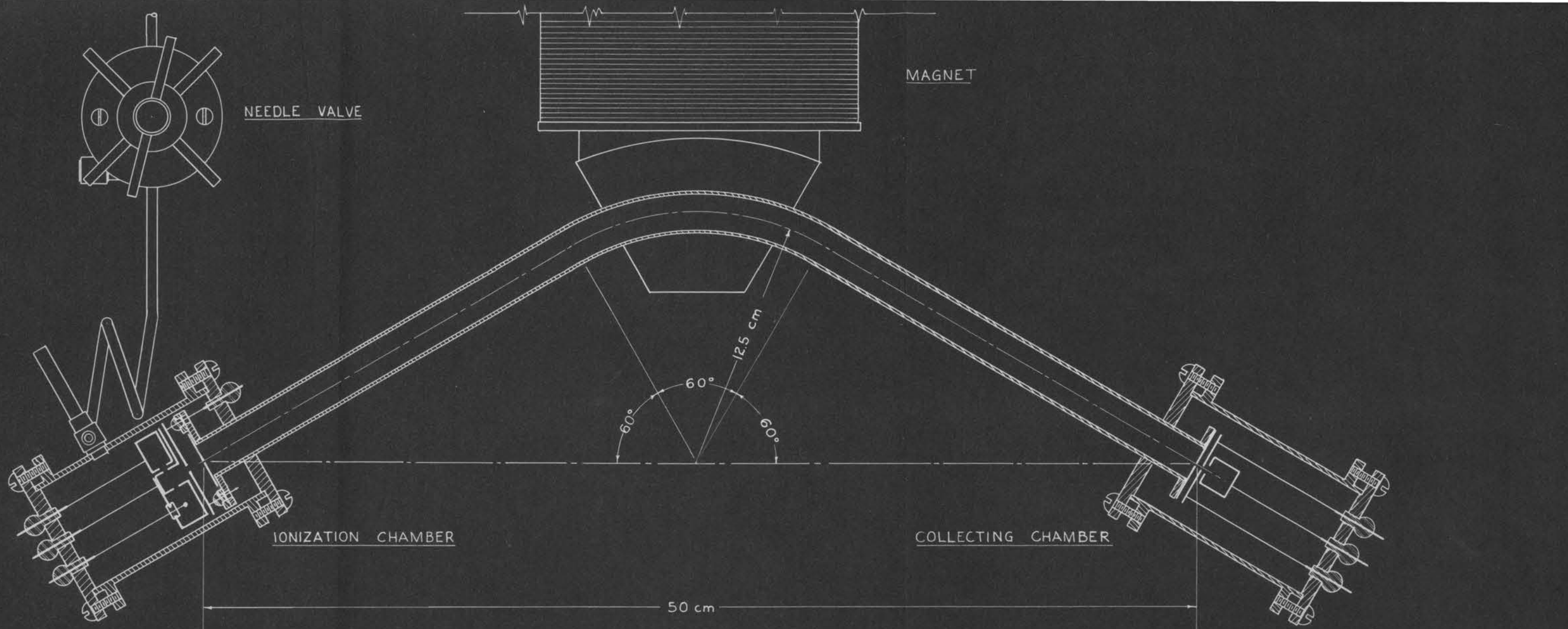
[23] E. B. Winn and O. A. Nier, "Simplified Emission Regulator for Mass Spectrometer," Review of Scientific Instruments, Vol. XX, pp. 773.


6 1/2" RADIUS
2 1/8"
30°
3 1/16"
4"
"NIERS" DESIGN


COARSE ADJUSTMENT
CLAMP RING FOR
COARSE ADJUSTMENT
METALIC BELLOWS
STEEL NEEDLE
STD 4/0 TAPER PIN
DIRECTION OF FLOW



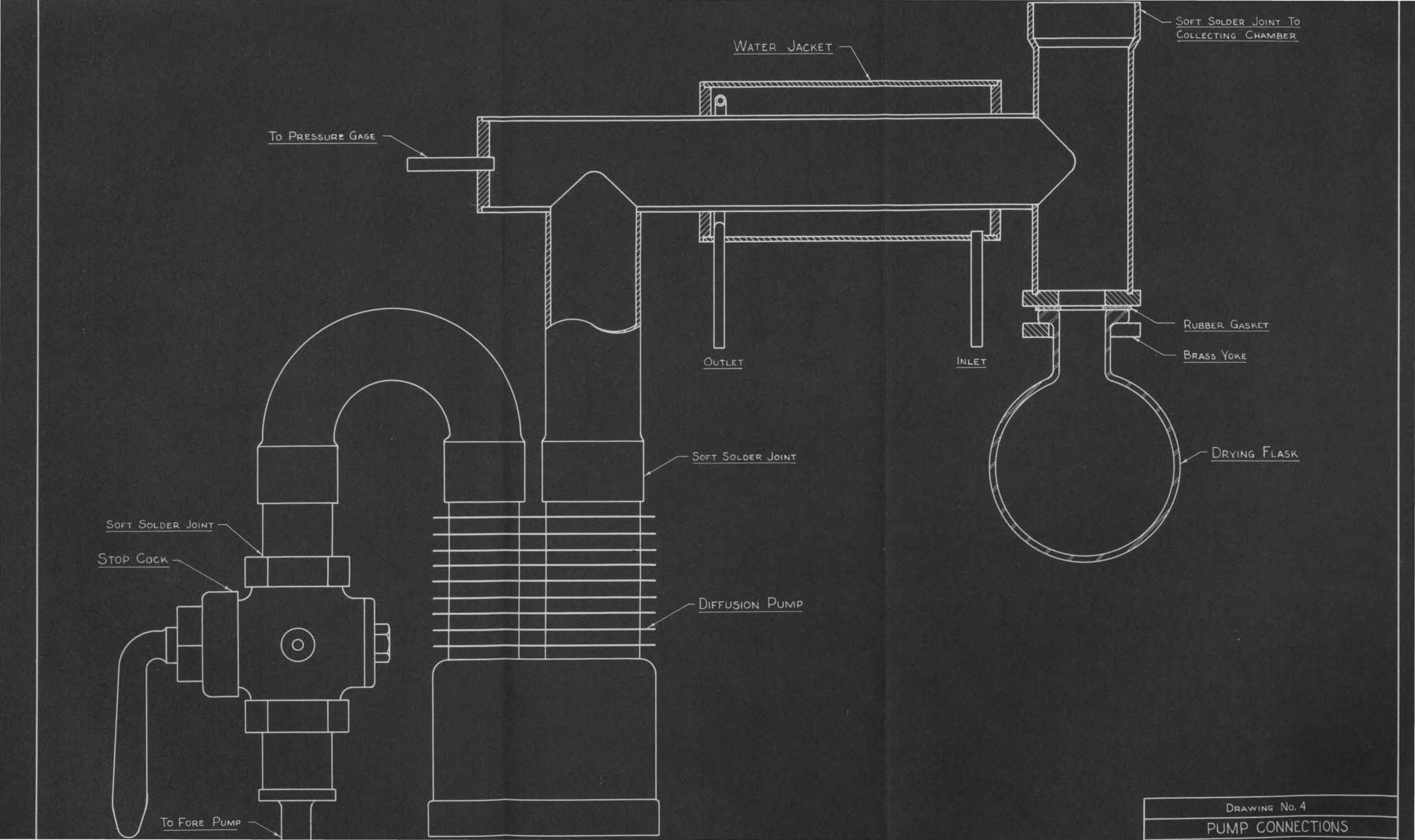




DRAWING No. 1

ASSEMBLY

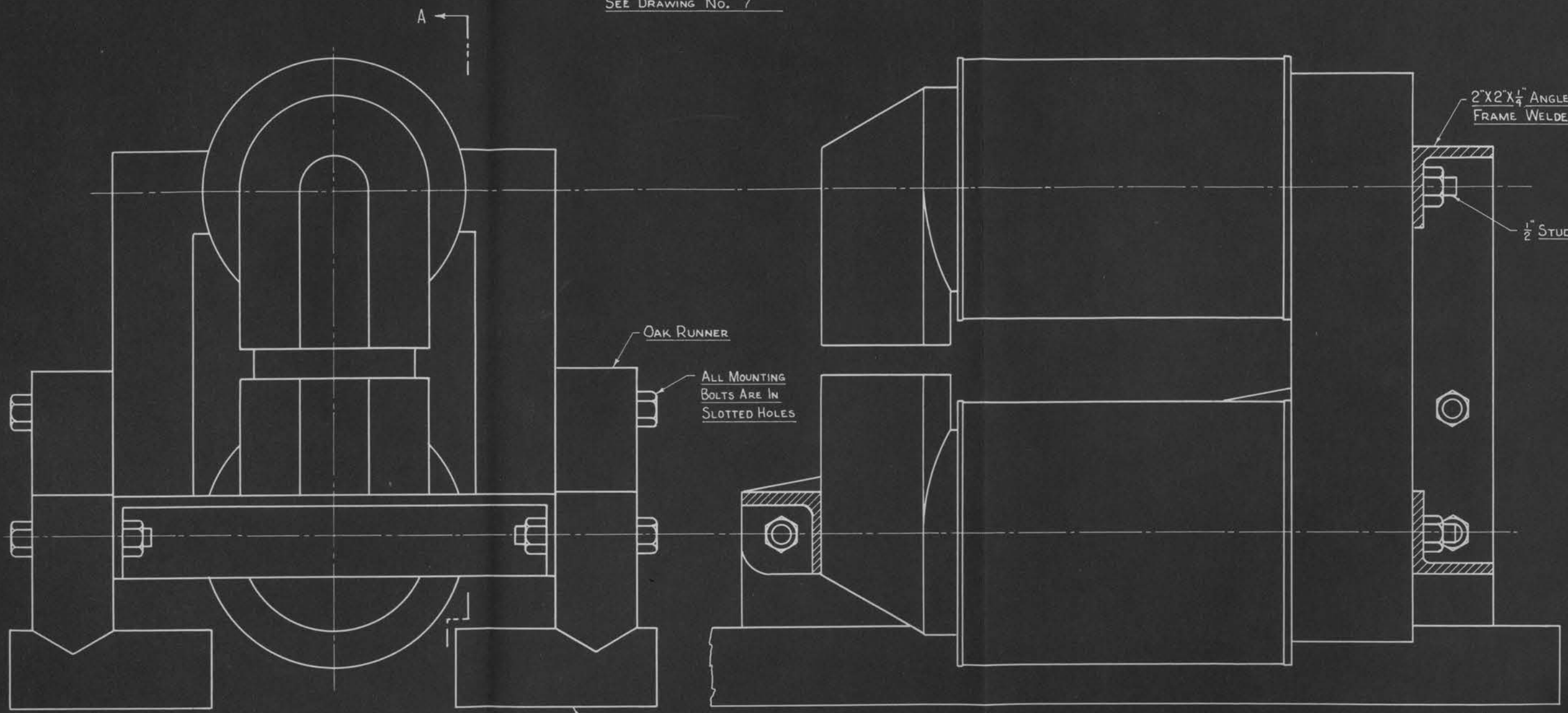
SCALE 1"=5 CM



Drawing No. 4

PUMP CONNECTIONS

For Pole Face Demensions
See Drawing No. 7



Section A-A

Drawing No 6

MAGNET

The vacuum system should include a liquid air trap if the instrument is to be used for absolute measurements of isotope abundance. This can be installed in place of the phosphorous pentoxide trap.

The capacity of the vacuum pumps may be questionable, especially when the instrument is used for gas analysis. Distillation products can supply diffusion pumps of the same series with a capacity up to 20 liters/sec at 10^{-4} mm. of Hg. in air-cooled models and up to 100 liters/sec at 10^{-4} mm. of Hg. in water-cooled models. The VMF series also includes the VMF-80 and the VMF-260 which are three stage water-cooled pumps with 80 and 260 liters/sec at 3×10^{-5} mm. of Hg., respectively. One of these pumps may have to be used to obtain a suitable pressure when gas is being admitted to the ionization chamber.

The lapped surfaces of brass stop and waste valve between the diffusion pump and fore pump were not as smooth as was thought necessary. If leaks occur in the valve it can probably be remedied by further lapping with a finer emery and rouge. If the valve still does not operate satisfactorily or if the surfaces can not be brought to a fine finish it may be possible to eliminate the trouble by replacing the brass plug by a hardened and ground steel plug. Since metal valves of this type are not standard technique, the valve was soldered into the system with soft solder for easy removal.

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Robinson, C. F. "Effects of Space Charge on the Focusing Properties of a 180° Mass Spectrometer." Review of Scientific Instruments, Vol. XX.

Saxon, D., and Richards, J. "A Vacuum Lock for Continuously Evacuated Systems." Review of Scientific Instruments, Vol. XX, (1949).

Winn, E. B., and Nier, O. A. "Simplified Emission Regulator for Mass Spectrometer." Review of Scientific Instruments, Vol. XX.

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