GCA Technical Note

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GCA CORPORATION
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Bedford, Massachusetts

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PLASMA BEAM CALIBRATION
MASS SPECTROMETER

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Section 1

INTRODUCTION

It is necessary to calibrate a time of flight ion mass spectrometer under conditions which resemble the space environment. Apart from the problem of space simulation in the laboratory, this requires the means to determine a relationship between the plasma density of a specific ionic gas and the peak height given by the mass spectrometer. If the plasma consisted of only one kind of ions the problem could be solved by establishing as parameters the plasma density, the ion energy and the degree of neutralization, with respect to the indication of the mass spectrometer. The measurements can be made by Langmuir probes and Faraday cups. However, in addition to the possibility of uncontrolled contamination in the plasma beam, it is beyond the state-of-the-art for any known plasma source to produce only atomic or molecular ions of the gases of interest. While it is possible to change the ratio either in favor of the molecular or the atomic species to a fair extent, it will still be a mixture as regards the required accuracy. The latter will be particularly true for a calibration with atomic ions. It is at once evident that the problem can be solved when the composition of the plasma - in particular the ratio of atomic to molecular ions - can be established. With this ratio and the total density known, the partial density is readily calculated and related to the measured peak heights. It was the objective

of this part of the sub-contract to design and build a mass spectrometer capable of determining these ratios and actually to measure these ratios in the plasma beam produced by the plasma guns developed under the prime contract by PTL.

Section 2

DESCRIPTION OF THE MASS SPECTROMETER

2.1 THE ANALYZER

The mass spectrometer was designed for the exact determination of an ion or plasma beam particularly with regard to the ratios of N/N_2, O/O_2, and H/H_2. Whereas a resolution of 2 would be sufficient for this purpose, a higher resolution is desirable to gain additional information about other impurities such as C, H_2O, CO, and CO_2. These impurities are very likely to occur in a hot filament ion source.

The resolution of a 60° magnetic sector mass spectrometer as utilized here is given by:

$$A = \frac{M}{\Delta M} = \frac{1}{\frac{S' + S''}{a} \quad \frac{\Delta V}{V}} \tag{1}$$

with S' and S'' being the width of the entrance and exit slit respectively, a the radius the ions describe in the magnetic field and $\frac{\Delta V}{V}$ the relative energy spread of the ions. In our case, the radius a equals 2" and the slit widths S', $S'' = 0.05"$. As $\frac{\Delta V}{V}$ is expected to be less than 0.01 it can be neglected and the resulting resolution is:

$$A = \frac{a}{S' + S''} = \frac{2}{0.1} = 20 \tag{2}$$

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This is more than adequate for the purpose, and an even higher resolution
can be achieved by a further reduction of the slit width; this, however
is accompanied by a loss in intensity.

The analyzer housing is a modification of a commercial Veeco MS9 Mass
Spectrometer.

2.2 THE ION COLLIMATOR SYSTEM

The purpose of the particular ion collection system, as used here, was
to resemble the geometric conditions of the Bennett Tube to a close extent.
In our case, it is much more convenient to have the mass spectrometer outside
the vacuum chamber. Therefore, a special configuration was devised, which
collects the ions, accelerates them, and focusses them over a distance of
about 15" onto the entrance slit of the spectrometer. With this device
the mass spectrometer measures the composition of the plasma at a location
close to the position of the Farady cup and also close to the eventual
position of the orifice of the Bennett spectrometer.

We refer now to the attached ASSEMBLY DRAWING. The plasma enters the
collimator tube through a 70% transmission grid in diaphragm 1 which with
the outside of the ion collector is on ground potential. The configuration
of electrodes 1, 2, and 3 constitutes an immersion lens system.[1] This
accelerates the ions and produces a crossover on the axis about 2" behind
the entrance grid. The electrons from the original plasma will be reflected
back into the chamber. The position of this cross over does not depend on

the potential difference between electrodes 1, 2, and 3. Its sharpness depends
on the spatial velocity of the ions entering the grid and, of course, also
on the lens aberrations. The angular aperture at the crossover on the other
hand depends on the width of the entrance opening. This crossover which
constitutes the spot of highest current density along the beam is imaged onto
the entrance slit of the mass spectrometer by an Einzel lens[*] which is given
by the electrode configuration 3, 4, and 5. A further possibility for focussing
the ion beam is given by the gap between electrodes 5 and 6, but is not
utilized. Two crossed electric fields generated by the electrodes 9 allow
centering of the beam onto the entrance slit.

The potentials along the ion beam are as follows. Electrodes 1 and 2 are
on ground potential the same as the outside shell 10 of the ion collimator
tube. Thus, the ions from the plasma which are supposed to have a velocity
corresponding to an energy of 1 to 10 eV perpendicular to the entrance grid
will penetrate this grid. Electrode 3 is on the same negative potential as
electrodes 5 and 6. Also the spectrometer entrance slit 14 with the whole
shell of the mass spectrometer is on this same potential. Accordingly, the
ions already acquire their final energy when they fall through the potential
drop of the immersion lens system. Electrode 4, the middle electrode of the
einzel lens, is on a variable negative potential, which for focussing of
the beam into the entrance slit is about 1/10 of the potential of electrode 3
and 5. Electrode 9, providing the deflection perpendicular to the beam, on one
side is always connected to electrode 6, whereas the opposite electrode can
be made positive or negative with respect to the potential of 6.

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[*] op.cit.

2.3 THE ION COLLECTOR

The ion collector is equipped with a suppressor arrangement, electrodes
20, 21, 22, to prevent scattered ions which have lost energy from reaching
the collector cup. A number of baffle plates 15 a, b and 23, 24, reduce
the possibility of scattered ions reaching the collector. The width of
the exit slit 14 of the mass spectrometer is adjustable as is the entrance
slit.

2.4 THE MASS SCALE

In our case the mass scale of the spectrometer is attained by electrical
scanning. The relation between the energy of the ions and their mass is
given by the basic formula

$$a = \frac{143.6\sqrt{M.V}}{B} \qquad (3)$$

where a is the radius in cm as described by a single charged ion of the
energy eV and the atomic mass M in a magnetic field B given in gauss.
Because entrance and exit slits are in a fixed position, a is constant and
equals 5 cm. With this we can write (3) in the form

$$M = \frac{25.B^2}{2.06.10^4} \cdot \frac{1}{V} = 12.10^{-4} \cdot B^2 \cdot \frac{1}{V} \qquad (4)$$

If a mass spectrometer is scanned electrically some precautions have to be
taken in order to avoid abundance discrimination effects for different mass
numbers. For this it is imperative that the orbits of the ions within the
collimator system do not change when their acceleration energy is changed
during the scan. This is the case only if all electrode potentials within

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the collimator are changed proportionally. But this is also true only as long as the initial energy of the ions, entering through the mesh, is negligible compared with the acceleration energy. This certainly applies in our case where initial ion energies lie below 10 eV. However, any mass spectrometer which is scanned by changing the acceleration voltage has discriminatory effects which cannot be avoided. Because of theoretical reasons, the ion transmission increases with increasing acceleration voltage. Thus, higher masses seem to have higher intensity.

To eliminate this problem in the calibration of N/N_2 and O/O_2, we use two different permanent magnets. The magnets are magnetized in such a ratio that the first will transmit ionic mass M at about the same acceleration voltage at which the other one will transmit 2M. With this arrangement it is also possible to find the effective change in the transmission between M and 2M as introduced by purely electrical scanning. This is then taken into account for the measurement of H/H_2 for which only one small magnet is provided.

2.5 THE SPECTROMETER VOLTAGE SUPPLY

The circuitry is given in Schematic 1. All voltages necessary to run the spectrometer are taken from a battery powered supply, specifically designed for this spectrometer. Unusual here is the grounding of the positive side. But, in our case the ions have to be picked up at ground potential with only little kinetic energy, and for additional acceleration the spectrometer has to be on a negative potential with respect to ground.

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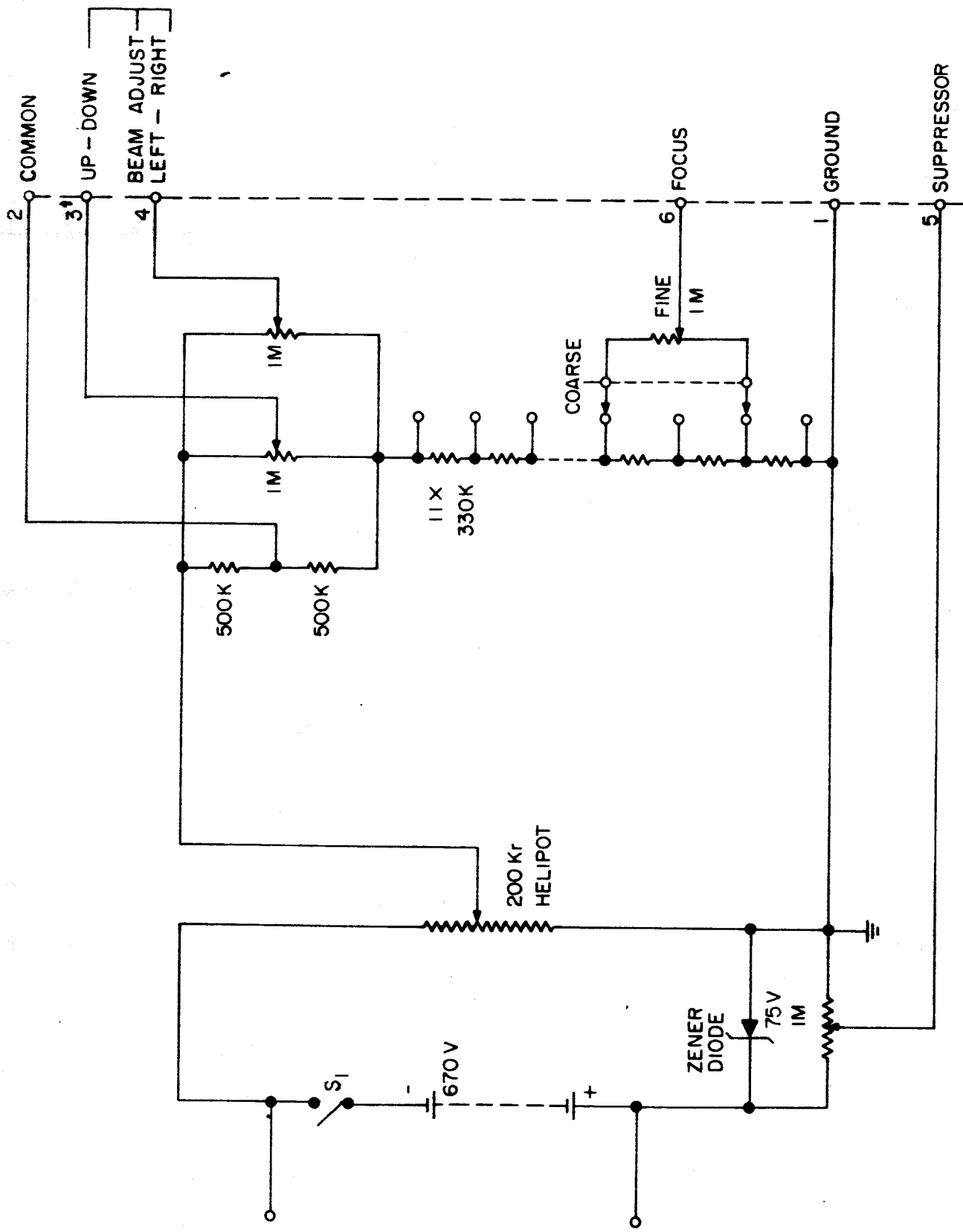
Schematic 1. Power Supply

The spectrometer potential which corresponds to the acceleration energy and thus determines the mass is scanned by a 10-turn helipot. All other potentials like FOCUS and BEAM ADJUST are changing proportionally with the scanning voltage as required in paragraph D above. Only the suppressor voltage is kept constant during the scan because this adjustment depends only on the initial energy of the ions.

It is possible to replace the batteries by an adequate high voltage power supply which must have a floating output.

2.6 THE ELECTROMETER

It has already been mentioned that the mass spectrometer is on a negative potential equal to the acceleration voltage. Theoretically it is possible to keep the electrometer and with it also the ion collector cup on ground potential. By virtue of their initial energy the ions still would be able to reach the collector. But under these conditions the electrometer will be extremely sensitive to changes of the scanning voltage. This annoying drawback, however, can be avoided by keeping the electrometer at the same potential as the spectrometer. Without interference from the a.c. line this can only be done with a battery powered meter. A Keithley Model 600 Electrometer has worked very satisfactorily under this condition.

Section 3

THE EXPERIMENTAL PROCEDURE

The complete mass spectrometer assembly as shown in photographs 1 and 2
is mounted onto the 5¼" porthole in the large 12" porthole flange of the
vacuum chamber. The spectrometer may be mounted in any of the eight
possible positions but the most convenient is with the magnet riding
upright on it.

The electric connections to the voltage supply are made according to
Schematic 2. To connect the electrometer to the ion collector it is
necessary to use a well-shielded, well-insulated, low noise cable. The
electrometer must be put on some good insulating supports. After the
pressure in the test chamber has reached about 1×10^{-5} and the plasma gun
has been started and adjusted, the electrometer is switched on and set to
read 1×10^{-12} A, FULL SCALE ($10^{-11} \times 0.1$), in position FAST. According
to the mass range of interest the proper magnet is selected.

MAGNET I, B = 3100 GAUSS

From Equation 4 in Section II,D we get:

$$M = \frac{11000}{V_I}$$

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Photographs 1 and 2. Mass spectrometer assembly.

For this magnet we list the voltages for masses which are likely to occur in our experiments

MASS SCALE

M	V_I	SPECIES	M	V_I	SPECIES
14	785 Volts	N	28	395 Volts	N_2, CO
16	690 Volts	O	32	345 Volts	O_2
17	650 Volts	OH	44	250 Volts	CO_2
18	610 Volts	H_2O	56	195 Volts	Fe

MAGNET II, B = 2100 GAUSS

$$M = \frac{5350}{V_{II}}$$

MASS SCALE

M	V_{II}	SPECIES	M	V_{II}	SPECIES
12	445 Volts	C	18	300 Volts	H_2O
14	380 Volts	N	28	190 Volts	N_2, CO
16	335 Volts	O	32	165 Volts	O_2
17	315 Volts	OH	44	120 Volts	CO_2

MAGNET III, B = 650 GAUSS

$$M = \frac{510}{V_{III}}$$

MASS SCALE

M	V_{III}	SPECIES	M	V_{III}	SPECIES
1	510 Volts	H	12	42 Volts	C
2	255 Volts	H_2	14	36 Volts	N
3	170 Volts	H_3	16	32 Volts	O
4	130 Volts	He			

It is very important to note that these voltages were derived under the assumption of zero initial ion energy. Any initial energy simply adds to the acceleration energy. Thus, if for instance, the N_2 peak centers at 365V instead of 395V this must be due to an initial energy of the N_2^+ of 395 eV - 365 eV = 30 eV. If the mass M and the acceleration voltage necessary for transmission throughout the analyzer is known, the mass spectrometer can be used as an energy spectrometer for the determination of the initial ion energy.

The spectrometer voltages are switched on with S_1' at the voltage supply. Then the mass range is slowly scanned with the helipot. As soon as a peak shows up, the intensity is optimized by using the BEAM ADJUST and FOCUS controls.

CAUTION - AS LONG AS THE SPECTROMETER IS SWITCHED ON, THE SPECTROMETER HOUSING AND THE ELECTROMETER ARE ON HIGH VOLTAGE AND SHOULD NOT BE TOUCHED.

If the setting of the electrometer has to be changed the spectrometer voltage should be switched off.

For the measurement of the acceleration voltage a Simpson meter between spectrometer shell potential and ground can be used.

Section 4

THE MEASUREMENTS

4.1 MEASUREMENTS WITH THE R.F. PLASMA SOURCE

The first measurements performed with the mass spectrometer were on a plasma beam produced by the R. F. Plasma Source. Test gas was Nitrogen.

One strong peak of 2.2 x 10^{-9}A appeared at 215 V_I with Magnet I.

A second peak of 2.5 x 10^{-10}A was easily detected at 200V_{II} with Magnet II.

There is no doubt that the 2.2 x 10^{-9}A peak is due to N_2^+ and the 2.5 x 10^{-10}A peak corresponds to N^+. Comparing the voltages for these peaks with the mass scales in the preceeding chapter one can instantly see that the ions must have had considerable initial energy. N_2^+ should have appeared at 395 V_I and N^+ at 380 V_{II}. Thus the energy of the plasma ions was 180 eV. This came as a surprise because the energy was thought to be 10 eV. A check with the Faraday cup confirmed the higher energy. It was not possible to reduce the energy to 10 eV at this time. The same measurements were made with oxygen and hydrogen and yielded closely the same results within the uncertainties of the system. Generally the atomic species constituted between 10% and 15% of the beam. This seemed unexpectedly low for a R. F. discharge and it was suspected that a structural element of the plasma gun was the cause of a high recombination rate. When it was dismantled to correct this, an error in the connection of the acceleration and the deceleration grid was found to have been the cause of the descrepancy in the initial ion energy.

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The measurements were then repeated and the amount of atomic ions with all three gases increased very considerably and was between 50% and 70%. This percentage, however, was very critically dependent on a number of adjustments of the plasma source and it was still not possible to establish reproducible conditions.

Also, it was not possible to set the energy of the ions within the right range of 1 to 10 eV; even more serious was a very large energy spread of about 50 eV, which was obvious from the widening of the mass peaks. It was then decided to put the R.F. source aside for later investigations and to test the filament plasma gun.

4.2 MEASUREMENTS ON THE FILAMENT PLASMA GUN

The first test gas was Nitrogen. After some experimenting in order to set the ion energy to about 10 eV, the following result was obtained:

MAGNET II

VOLTAGE	INTENSITY	INTERPRETATION
430V$_{II}$	2×10^{-13} A	C^+
360	6.6×10^{-12}	N^+
315	5×10^{-13}	O^+
295	1×10^{-12}	OH^+
280	1.4×10^{-12}	H_2O^+
180	5×10^{-12}	N_2^+

MAGNET I

VOLTAGE	INTENSITY	INTERPRETATION
395V$_I$	1.3×10^{-11}A	N_2
250	2×10^{-13}	CO_2

Here the ratio of atomic to molecular ions was unexpectedly high with $6.6 \times 10^{-12}/1.3 \times 10^{-11}$ which gives about 30% atomic ions. It was suspected that secondary electrons from the acceleration mesh are accelerated backwards into the plasma. As the mesh was on a potential of about 160V negative to the plasma, high energy electrons can contribute to the strong dissociation observed. Indeed, reduction of the mesh voltage from -160V to -50V gave:

$$N^+ = 8 \times 10^{-12}A \text{ and } N_2^+ = 4.5 \times 10^{-11}A,$$

and further reduction to 15V yielded:

$$N^+ = 9.2 \times 10^{-12} \text{ and } N_2^+ = 2 \times 10^{-10}$$

The precentage of N^+ thus has changed from 30% down to 5%.

Remarkable is the fact that the absolute intensity of N^+ changed only little while the intensity of N_2^+ increased by a factor of 15. The intensity of the contaminants under the latter conditions was $O^+ = 1.4 \cdot 10^{-12}A$, $OH^+ = 2.5 \times 10^{-12}A$, $H_2O = 5 \times 10^{-12}A$, $CO = 7 \times 10^{-13}A$. This constitutes an only three-fold increase of the contaminants, which are the usual ones for a hot filament ion source.

These results have proved to be fairly reproducible. Thus, it is felt that the reporting of all the measurements actually made is of no pertinent value.

It was also tried to perform similar measurements with oxygen and hydrogen. But the tungsten filaments did not work too well in the oxygen atmosphere and there were difficulties in getting a stable discharge with hydrogen within the time available for the tests.

Evidently, the original purpose of this part of the sub-contract, namely, the final calibration of the plasma beam composition with regard to the concentration of atomic and molecular species of hydrogen, nitrogen and oxygen, could not be completed because both plasma sources are still in a state of development. Even in the case of the filament plasma gun working with nitrogen some further investigations of all parameters involved are indicated.

However, it should be stressed that the information gained so far is extremely valuable for further development, and it is also obvious that the mass spectrometer is an indispensible tool for the continuation of this work. Anyone who is acquainted with the difficulties of the generation of intense plasma beams of low energy will appreciate the achievements of the prime contractor. The tests with the mass spectrometer have shown some insufficiencies but also indicated the direction of improvements. The high recombination rate for instance, which was observed in the R. F. gun can certainly be reduced by lining some parts with pyrex glass. The difficulties in obtaining the desired ion energy were already reduced by some modifications in the circuitry of the power supply for the plasma gun. The hydrogen discharge can certainly be sustained better with Bariumoxide-coated filament cathodes because of the higher emission and lifetime. Rhenium

filaments, on the other hand, have proved to work very satisfactorily under an oxygen atmosphere.

Summing up, it is anticipated that the prime contractor will on continuation of the contract, with the help of the mass spectrometer, reach a state of development where a meaningful final calibration of the composition of the plasma beam can be performed.
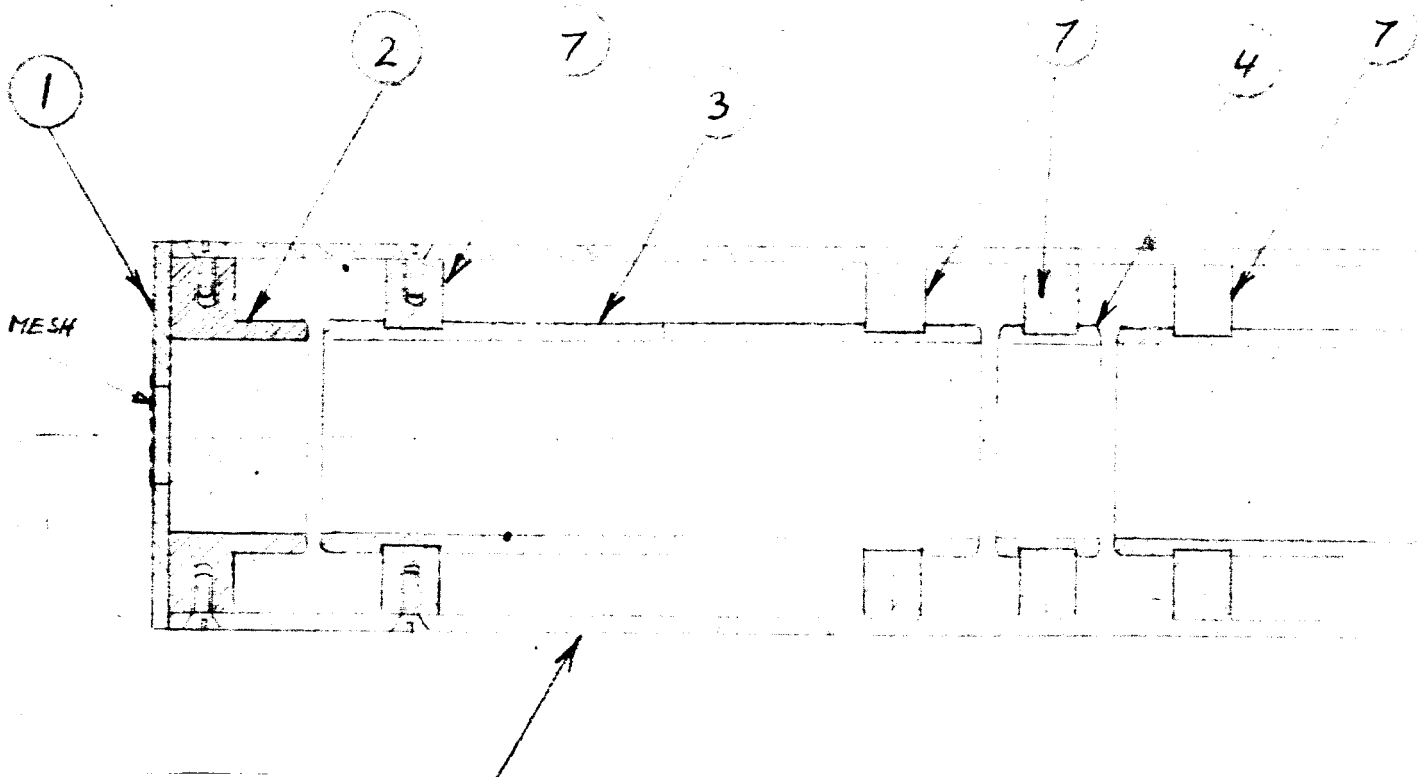
REFERENCES

1. M. v. Ardenne, Tabellen zur angewandten Physik, V.E.B. Deutscher Verlag der Wissenschaften, 1st Ed., 1956, pp. 34 and 47.

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ASSEMBLY

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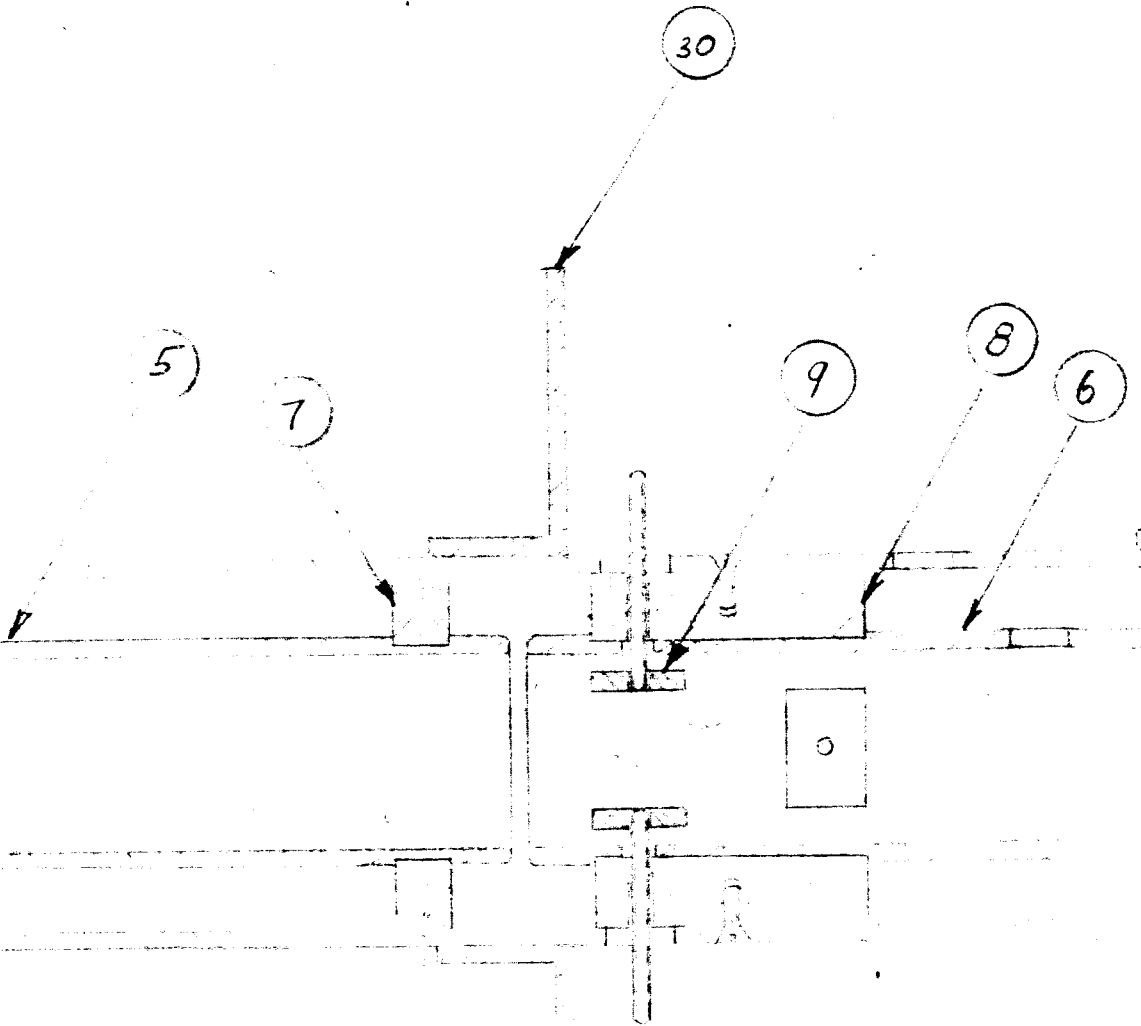
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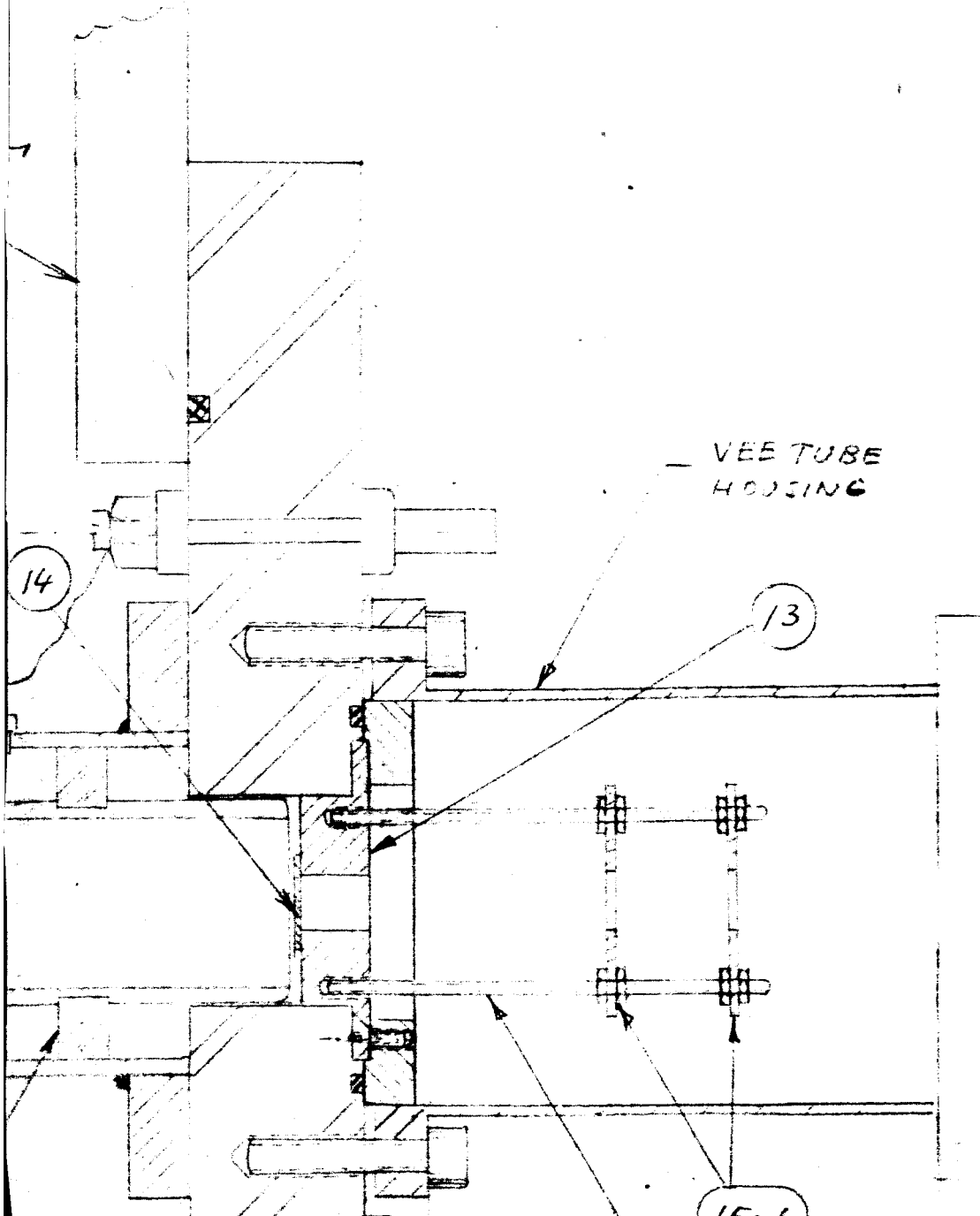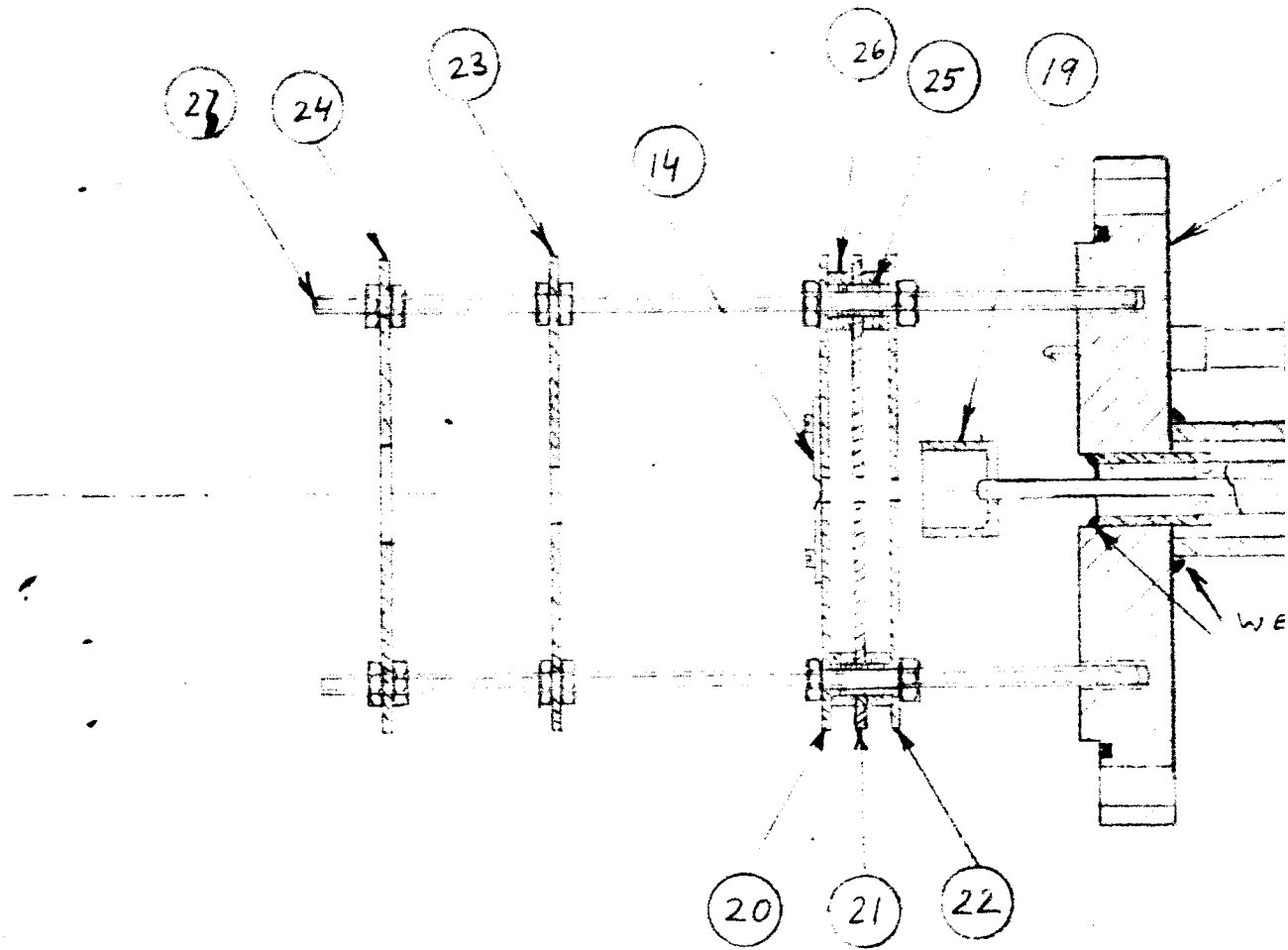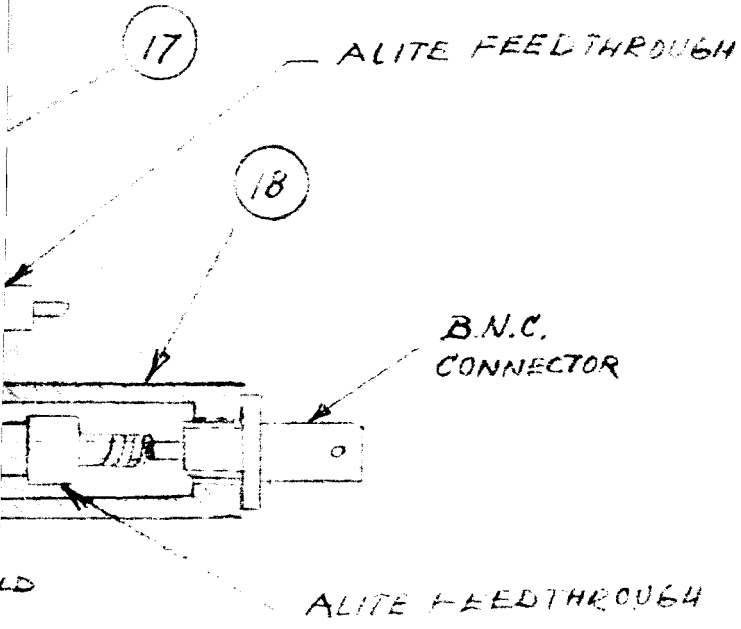
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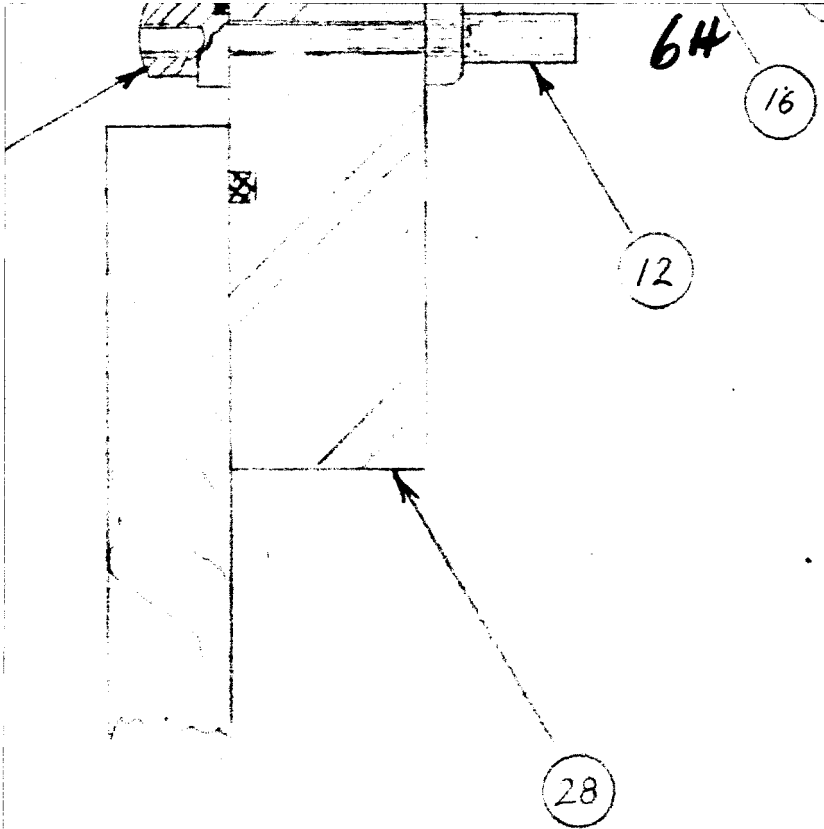
NO.	DESCRIPTION	MATL	MATL SPEC	REQD

LIST OF MATERIAL

RPORATION
CA

3-12-65

CALIBRATION

MASS SPECTROMETER

**GEOPHYSICS
CORPORATION
OF AMERICA**

DRAWING NUMBER

2 - 016 - 01

SCALE

LOC | SIZE **C** | SH. *1* OF *12* | REV

CLASSIFICATION:

COLLECTOR
SUPPRESSOR

SPECTROMETER

S

G
C
F_I

U
R
F_{II}

ION COLLECTOR

GRID

CONNECTIONS

POWER SUPPLY TO SPECTROMETER

1   G
2   C, S, F_I
3   U
4   R
5   SUPPRESSOR
6   F_{II}
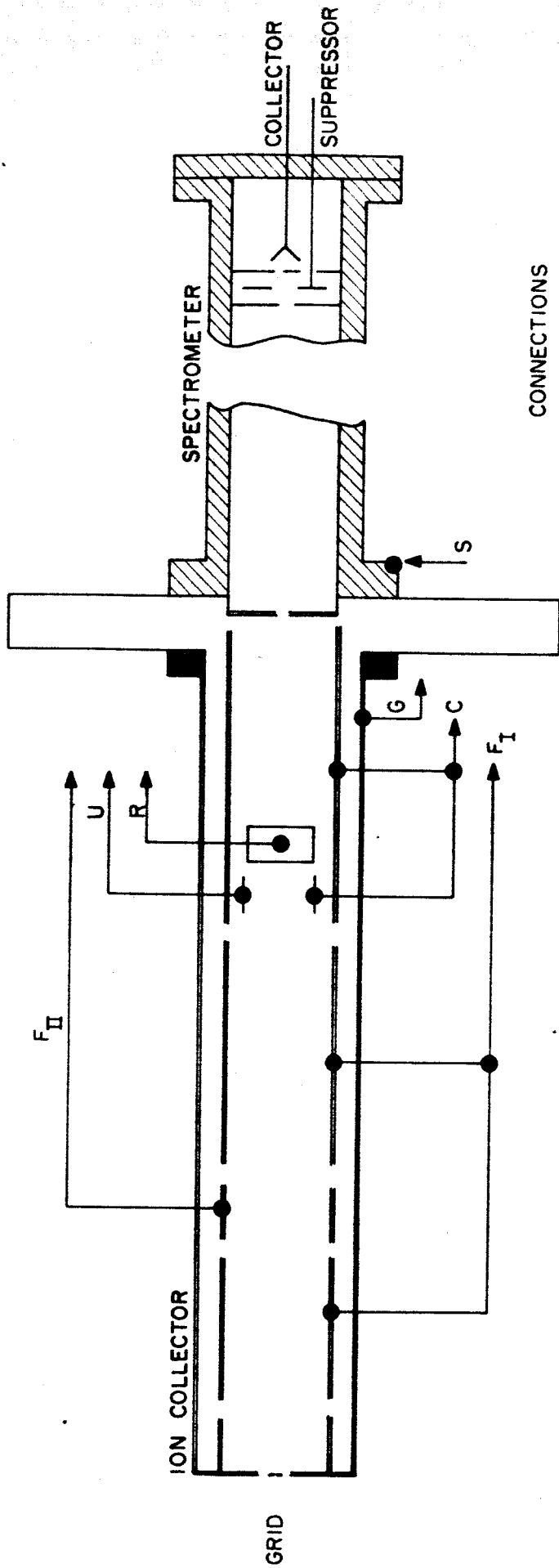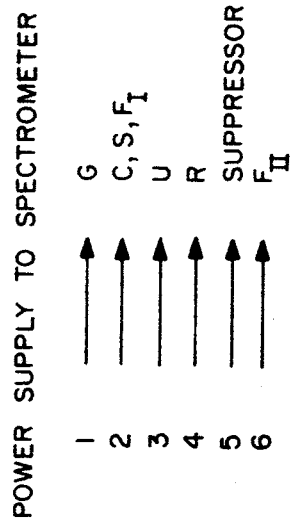
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Schematic 3. Electrical Connection