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CALIBRATION OF A QUADRUPOLE GAS ANALYZER USING A REFERENCE GAS*

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ABSTRACT

A quadrupole gas analyzer system was calibrated for the gases H_2, He, N_2, O_2, A, and Xe. The procedure involved first calibrating the system for argon, as the reference gas. This was done using conventional techniques. A mixture of the above gases was then introduced into the system to be calibrated. The system configuration was such that the ratio of the partial pressures of each of the test gases with respect to the reference gas would be unity in the absence of sorption processes within the test envelope. Near ideal calibration characteristics of the quadupole — combining the properties of the ion injection system, mass filter, and ion collector — are given for H_2, He, N_2, and Xe. The indicated partial pressure of oxygen was, under most test conditions, 10^{-3} to 10^{-2} times that of the reference gas.

INTRODUCTION

A quadrupole gas analyzer (Varian model #974-0016) was obtained to do gas analysis studies. Understandably, however, the manufacturer was unable to make meaningful quantitative predictions about the behavior of this instrument on our system for gases other than nitrogen and argon. Part of the uncertainty stems from possible effects our vacuum environment may have on the analyzer system with time. Of equal importance is the fact that each possible test setup will yield significantly differing results for the same mass flow rate into the system, due to the varying degree to which gas sorption processes occur within various systems. In reality the problem is reduced to one of calibrating the entire system, not just the residual gas analyzer. To further complicate matters, residual gas analyzers have incorporated in their construction, features which will result in all of the problems and mysteries associated with ionization gauge calibration,[1-8] with additional problems associated with ion injection systems, mass filtering, and electron multipliers.

The objective of our tests was to determine the characteristics of a quadrupole gas analyzer[9-12] and vacuum system (quadrupole system hereafter) for various system parameters including resolution settings, filament emission, electron multiplier voltage, etc. It became impracticable to directly calibrate the quadrupole system for even one gas, with all of the system variables. We therefore decided to first calibrate a Bayard-Alpert gauge (BAG hereafter) for argon, and then use total pressure indications of the BAG to calibrate the quadrupole system. This procedure established argon as the "reference gas." A known mixture of gases (H_2, He, N_2, O_2, A, and Xe, referred to as the "mixture" hereafter) was then introduced into the calibration volume in such a manner that, in the absence of sorption processes, the partial pressure of these gases

compared with argon would be known. Indicated partial pressures of argon then became the "measuring stick" by which calibration for other gases was accomplished. Argon was selected as the reference gas due to the low pumping speeds of the BAG for this gas.[13-15]

BAYARD-ALPERT GAUGE CALIBRATION

The numerous methods of vacuum gauge calibration are fairly well represented in the literature cited. We adopted the "static" method; static in the sense that each calibration test involved a finite, measured, quantity of gas. Figure 1 is a schematic representation of the vacuum system used. Volume #1 (V_1) was accurately measured. A McLeod gauge was used to measure pressure in V_1. The variable leak, F_1, was then opened, and indicated pressure in V_2 measured as a function of time. The thin foil aperture conductance F_2,[16] was calculated.[17] The vacuum system was a double liquid nitrogen trap, oil diffusion pump system, capable of achieving pressures $<5 \times 10^{-9}$ Torr in the calibration volume (restricted by F_2). From mass flow rate equations[16]:

$$Q_{2A}(t) = F_{2A}\left[P_{2A}(t)-P_{3A}(t)\right] \cong F_{2A}P_{2A}(t) \qquad (1)$$

where

$Q_{2A}(t)$ = argon mass flow rate through F_2 as a function of time

F_{2A} = calculated molecular conductance of F_2 for argon

$P_{2A}(t)$ = argon pressure in V_2 as a function of time

$P_{3A}(t)$ = argon pressure on the pump side of aperture F_2

The approximation in Eq. (1) was valid for pressures in $V_2 \geq 10^{-7}$ Torr (~2% error at this pressure). Then:

$$\int_0^t Q_{2A}(t)dt = F_{2A}\int_0^t P_{2A}(t)dt = P_{1A}V_1, \quad \text{or} \quad F_{2A}\int_0^t k_A P_{2A_i}(t)dt = P_{1A}V_1 \qquad (2)$$

where

$P_{2A_i}(t)$ = indicated partial pressure of argon in V_2 as a function of time

$P_{1A}V_1$ = total measured quantity of argon introduced into V_2

k_A = required argon gauge calibration constant.

Numerous tests yielded a gauge calibration constant k_A of 1.38 ± 0.12 (relative to nitrogen), which appears in good agreement with the literature.[3] These tests were conducted at pressures ranging from $1 \times 10^{-7} - 5 \times 10^{-5}$ Torr, satisfactory for our purposes in that linearity is known to exist at lower pressures.[18]

PRINCIPLE OF QUADRUPOLE SYSTEM CALIBRATION

The method used is similar, in principle, to that described in glow-discharge experiments by Sullivan and Buser,[19] and in gas ionization cross section studies by Rapp and Englander-Golden.[20] Referring to Fig. 1, assume volume V_1 contains a mixture of gas X and Y. Assume pressures of gas X and Y in V_1 (P_{1X} and P_{1Y} respectively) were determined, or at least their proportion, during mixing. F_1 and F_2 represent molecular conductances. Let the total system be such that $P_3 \ll P_2 \ll P_1$; P_1, P_2 and P_3 correspond to the three pressures in the system resulting from mass flow through F_1 and F_2. Using notation similar to Eq. (1) gives:

$$Q_{1X} = F_{1X}(P_{1X} - P_{2X}) \cong F_{1X}P_{1X} = C_1(m_X)^{-1/2}P_{1X} \qquad (3)$$

where C_1 is a constant independent of the gas species, and m_X is the molecular weight of gas X. Conservation of mass flow of gas X requires that Q_{2X} (gas X mass flow rate through F_2) equal Q_{1X}. On setting up an equation similar to Eq. (3) for Q_{2X}, and on equating the two expressions it is found that $P_{2X} = \left[(C_1/C_2)P_{1X}\right]$. Developing a similar expression for gas Y and finding the ratio of the two partial pressures in V_2 gives:

$$\left[P_{2X}\right]\left[P_{2Y}\right]^{-1} = \left[(C_1/C_2)P_{1X}\right]\left[(C_1/C_2)P_{1Y}\right]^{-1} \quad \text{or} \quad P_{2X} = (P_{1X}/P_{1Y})P_{2Y} \tag{4}$$

Equation (4) indicates that the proportionality of the partial pressures of the two gases is the same in V_2 as in V_1. This familiar result is restated here since the entire calibration scheme is based on this outcome. If P_{1X} and P_{1Y} are determined accurately (i.e., with a McLeod or Bourdon tube gauge), then their ratio may be accurately expressed in Eq. (4). If the analyzer system appending V_2 is accurately calibrated for a reference gas, such as species Y in Eq. (4), one may, without loss of generality, mix any number of gases in V_1, and as long as the proportionality of a species is known with respect to the reference gas Y, Eq. (4) will have the form: $P_{2X} = r_{XY}P_{2Y}$, (r_{XY} being the ratio of the known partial pressures in V_1). Assuming that P_{2Y} is the "absolute" partial pressure of gas Y in V_2 as the result of reference gas calibration, then Eq. (4) will take the form:

$$k_X P_{2X_i} = r_{XY}P_{2Y} \tag{5}$$

where k_X is the required relative calibration constant, and P_{2X_i} the indicated partial pressure of gas X in V_2.

MOLECULAR CONDUCTANCE F_1

The above derivation is invalid if F_1 is not molecular (i.e., P_2 varies linearly with P_1, and mass flow according to the inverse square root of the molecular weight of the gas species). Pressure P_2 may be maintained at pressures ranging from 10^{-10}-10^{-4} Torr, while P_1/P_2 could be 10^5-10^{12}. A commercial, all-metal, variable leak (Granville-Phillips series 203) was tested for molecularity under conditions of such large pressure ratios. Morrison had previously established linearity under such conditions.[21] Morrison's findings and the probable molecularity of the leak were substantiated in the following tests. Figure 2 represents the system used in these tests. Total pressure in V_1 (indicated with Bourdon tube gauge) was varied while pressure in V_2 was measured with a BAG. In separate tests with He, N_2 and A, the variable leak was closed during the interim roughing of V_1. Typical results are given in Fig. 3. Argon and helium data are normalized to nitrogen for convenience, assuming molecularity. Molecularity, at best, could only be demonstrated to within a factor of two if the variable leak setting were changed prior to roughing of V_1.

In subsequent tests with He and A, the variable leak was not moved during interim roughing of V_1. Typical results of these tests are shown in Fig. 4. Taking the ratio of slopes for A and He data in Fig. 4, assuming molecularity of F_1, and using published[3] average BAG relative sensitivities, agreement is found to within 3% of what is theoretically predicted.

RESULTS OF CALIBRATION OF QUADRUPOLE SYSTEM

Our experiments were divided into three test sequences. The first involved calibrating the quadrupole system for argon with a calibrated BAG; the second, conducting calibration experiments with a mixture of N_2 and A (equal partial

pressures in V_1, Fig. 1); the third, conducting calibration experiments with the gas "mixture." All gases in the "mixture" were mixed in equal proportion to the reference gas in V_1 (at 140 Torr), with the exception of Xe (at 3.72 Torr). This gives a value of unity to r_{XY} in Eq. (5), except for Xe. These proportions will change with time, however.

On concluding argon calibration, the A-N_2 mixture was introduced into the system. Typical results of these tests are shown in Fig. 5. CO desorption as a consequence of introducing A into V_2 stabilized at ~2-3% of the indicated A pressure. CO was the major background gas during A tests; the other two major peaks, H_2 and CO_2, being an order of magnitude lower. Figure 5 is therefore fairly indicative of the relative sensitivity of the quadrupole system for N_2 compared to A. Sequential data points (left to right) for A and N_2 are taken with the same resolution setting, the last data point for A being omitted. An indication of relative changes in sensitivity for various resolution settings is observed by noting changes in the slope of imaginary lines connecting sequential data points for A and N_2.

MIXED GAS EXPERIMENTS

On initially introducing the "mixed" gas into the test volume, the partial pressure of O_2 (electron multiplier current) was observed to be ~0.2% of that of A. Eventually this increased to 15.2% of the argon partial pressure. This was to be expected, as O_2 is known to be pumped very effectively (also H_2, N_2, and A, but to lesser extents) by several mechanisms in hot filament ionization gauges (thoria-coated iridium filament in our case).[13-15] At a state of equilibrium, and with an argon partial pressure of 3.8×10^{-6} Torr, experiments were conducted to determine relative sensitivity compared with A, as a function of quadrupole resolution settings. Figure 6 summarizes these findings.

normalized to A at the lowest resolution setting shown. Findings are similar to those of Harra,[11] who normalized data to CO_2 (determined to be similar to A in our tests), except that only m/e 14 and 16 appear to be significantly depressed below the argon line in this case. All of Harra's data (including m/e 1, 2, 12, 18, and 28) fell significantly below the CO_2 line for certain resolution settings. It is speculated that the loss in relative transmission of m/e 14 and 16 in our data is probably due to scattering effects on dissociation of molecular nitrogen and oxygen. The behavior of m/e 20 substantiates this.

Figure 7 shows all data taken in the particular test sequence given in Fig. 6 (now including CO_2), except that data are shown normalized to m/e 20 approximately mid-point in the data surface. This figure best illustrates the sensitivity-resolution compromise characteristic of quadrupole mass filter systems.

Figure 8 is a typical "mixed" gas mass spectrum observed with a resolution setting of 6 (see Fig. 7). The Xe spectrum superimposed in Fig. 8 was obtained by switching to the high mass range of the instrument, and therefore should not be compared directly with low mass range findings in terms of sensitivity and resolution. This spectrum indicates that water vapor (18) and CO_2 are formed when introducing the "mixed" gas. For the same quadrupole system setting (with BAG off, as in most of these tests), typically, the proportion in electron multiplier current H_2O: CO_2: A in the A-N_2 experiments was 1:5:2000, while in the "mixed" gas experiments it was approximately 1:2.5:16. From A-N_2 calibration experiments, the increase in CO on introducing the "mixture" was probably negligible, compared to increases in CO_2 and H_2O.

Table 1 summarizes "sensitivity" of the quadrupole system for the various gases at an acceptable resolution setting, and assuming negligible sorption processes within the calibration volume. This assumption is, of course,

absurd for active gases such as O_2 and may also lead to large errors in calibration for N_2 and A. Such problems were encountered early in our experiments, and are illustrated in Fig. 9. This figure shows the effect produced by first turning off the system BAG (operating at 2 mA emission), and then reducing the quadrupole filament emission. A state of equilibrium was acheived for each successive reading (progressing from right to left in the figure). Assuming that increases in indicated partial pressures were primarily due to reduced gauge pumping rather than gauge desorption processes,[26] gauge pumping speeds for A and N_2 were calculated to be 0.068 and 0.47 ℓ/sec respectively. Pumping speeds of the quadrupole at 1.0 mA emission for A and N_2 were calculated to be 0.082 and 0.097 ℓ/sec, respectively. In these tests F_2 conductance (see Fig. 1) was ~ 0.25 ℓ/sec (N_2). This was increased to ~ 0.5 ℓ/sec in all subsequent tests, and quadrupole filament emission was maintained at ≤ 0.5 mA.

CONCLUSION

A simple method was discussed for calibrating a quadrupole gas analyzer. Perhaps through use of gas mixtures, including an inert reference gas, gas-system interactions such as occur in Bayard-Alpert gauges and gas analyzers maybe more thoroughly understood. Our experiments were by no means refined, but did demonstrate the feasibility of the technique of calibration through use of a reference gas.

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TABLE 1

OBSERVED QUADRUPOLE SYSTEM "SENSITIVITY" ASSUMING ABSENCE OF GAS SORPTION PROCESSES WITHIN CALIBRATION VOLUME AND WITH ARGON PARTIAL PRESSURE OF 3.8×10^{-6} TORR

m/e	Low Mass Range (0-50 m/e) (Quadrupole Emission 250 μA)		High Mass Range (2-250 m/e) (Quadrupole Emission 85 μA)		Electron Multiplier Gain $\times 10^3$
	"Sensitivity" - Torr^{-1} I_+/I_-P	Resolution M/ΔM	"Sensitivity" - Torr^{-1} I_+/I_-P	Resolution M/ΔM	
2	52.2 (0.21)*	18	23.2 (0.33)	4.6	5.19
4	44.8 (0.18)	25	20.1 (0.29)	7.9	5.11
28	250.0 (1.0)	85	69.1 (1.0)	65.0	3.64
32	30.0 (0.12)	93	10.0 (0.14)	74.0	3.72
40	353.0 (1.41)	106	66.3 (0.96)	70.0	2.28
129	---	---	2.29 (0.033)	430.0	1.27

*Values in parenthesis "sensitivity" relative to nitrogen.

LIST OF FIGURES

1. Schematic representation of apparatus used in quadrupole gas analyzer system calibration experiments.
2. Schematic representation of apparatus used in testing vacuum conductance of variable leak F_1.
3. Results of tests for linearity of variable leak for argon, helium and nitrogen.
4. Bayard-Alpert gauge indicated pressure in volume number 2 as a function of Bourdon tube gauge pressure in volume number 1, for helium and argon with variable leak fixed.
5. Quadrupole system sensitivity for argon and nitrogen as a function of resolution.
6. Quadrupole system relative transmission of gas species as a function of quadrupole resolution.
7. Quadrupole system relative sensitivity as a function of resolution. All values normalized to doubly ionized argon near center of data surface.
8. Typical quadrupole mass spectrum observed when introducing equal proportions of hydrogen, helium, nitrogen, oxygen, and argon, with trace of xenon (0.53% of gas mixture).
9. Sorption of argon and nitrogen by Bayard-Alpert gauge and quadrupole system attached to calibration test volume.

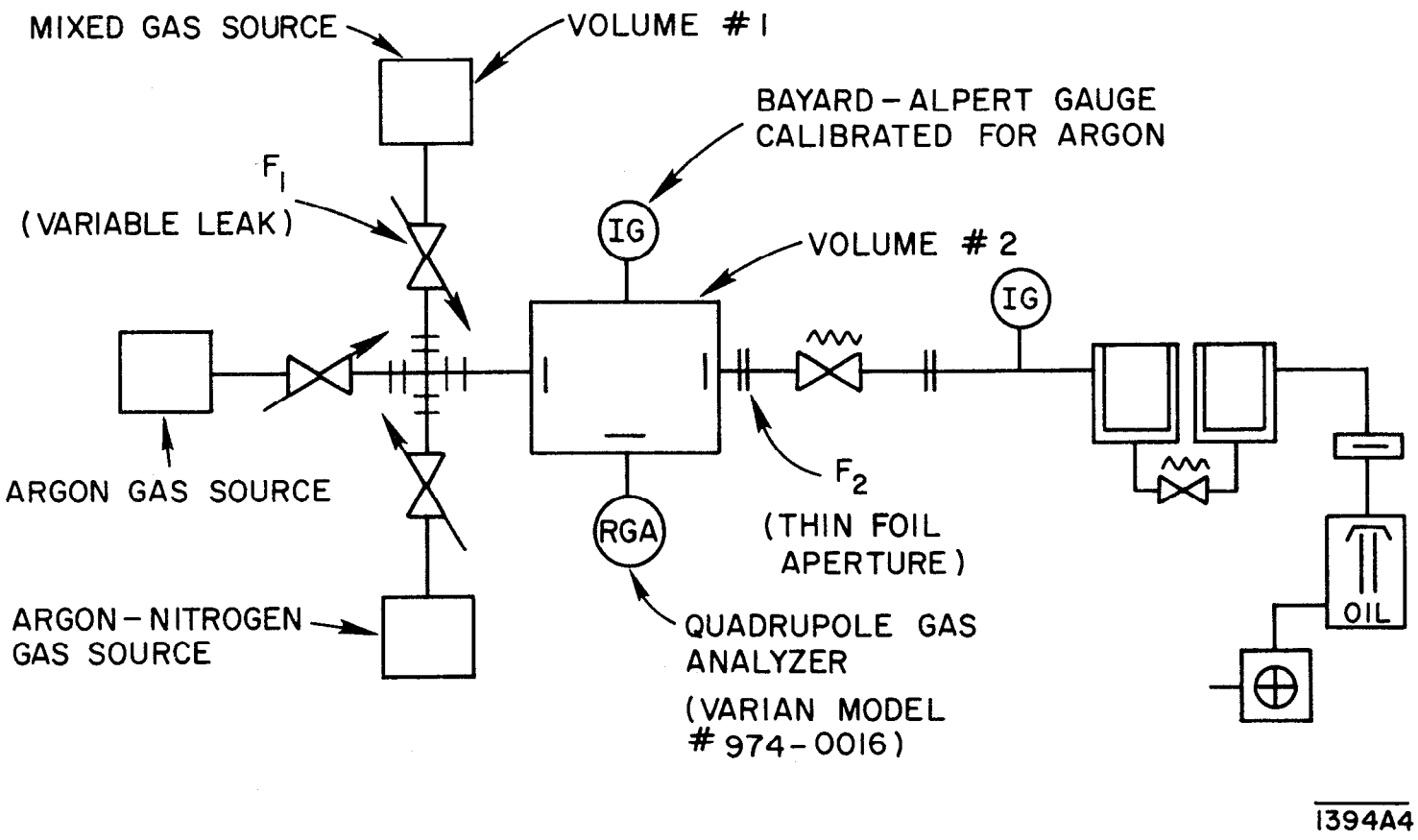
MIXED GAS SOURCE
VOLUME #1
BAYARD-ALPERT GAUGE
CALIBRATED FOR ARGON
F1
(VARIABLE LEAK)
IG
VOLUME #2
IG
ARGON GAS SOURCE
F2
(THIN FOIL
APERTURE)
RGA
ARGON-NITROGEN
GAS SOURCE
QUADRUPOLE GAS
ANALYZER
(VARIAN MODEL
#974-0016)
OIL
1394A4

Fig. 1

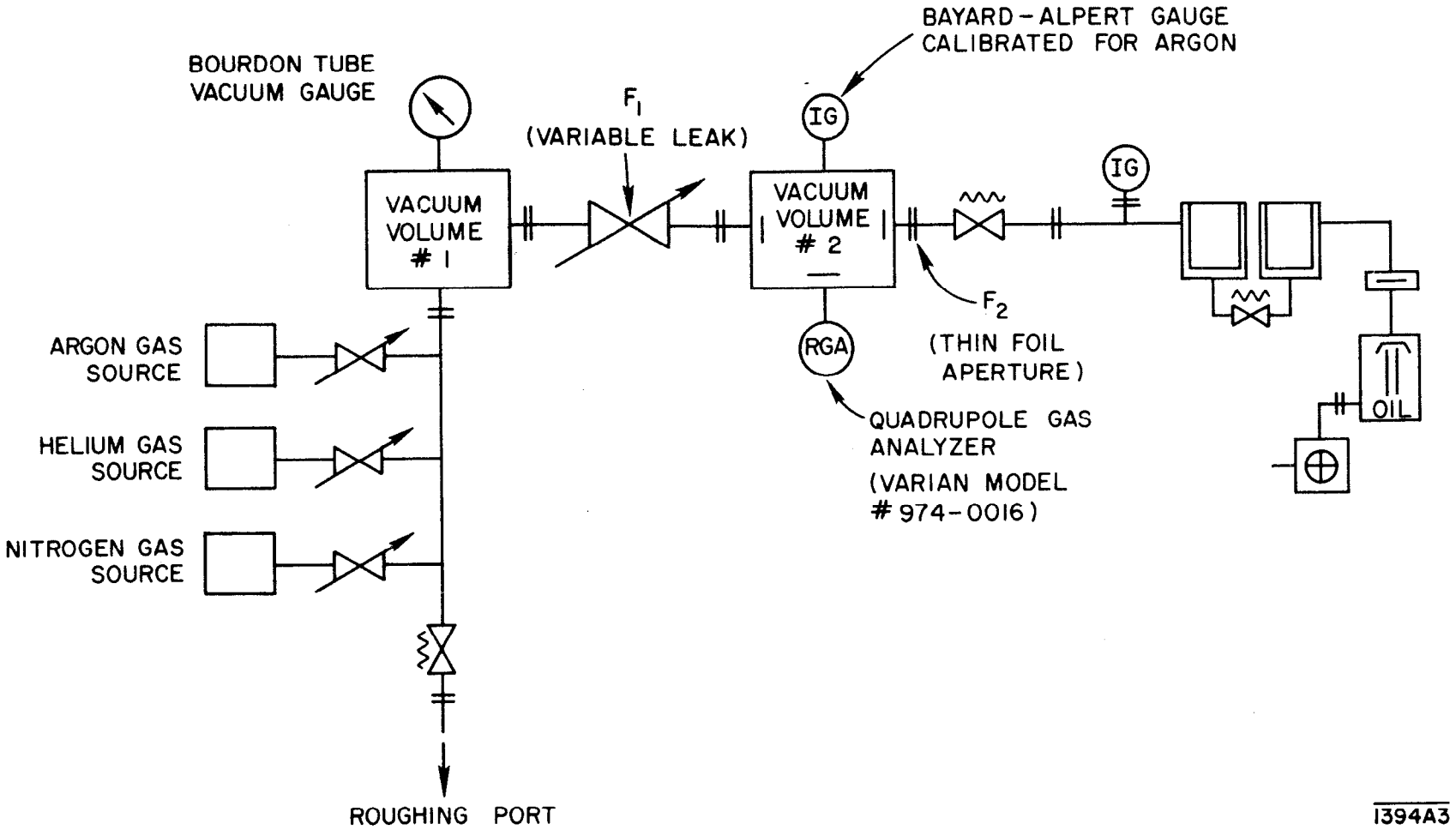
BAYARD-ALPERT GAUGE
CALIBRATED FOR ARGON
BOURDON TUBE
VACUUM GAUGE
F1
(VARIABLE LEAK)
IG
IG
VACUUM
VOLUME
1
VACUUM
VOLUME
2
F2
(THIN FOIL
APERTURE)
RGA
QUADRUPOLE GAS
ANALYZER
(VARIAN MODEL
974-0016)
OIL
ARGON GAS
SOURCE
HELIUM GAS
SOURCE
NITROGEN GAS
SOURCE
ROUGHING PORT
1394A3

Fig. 2

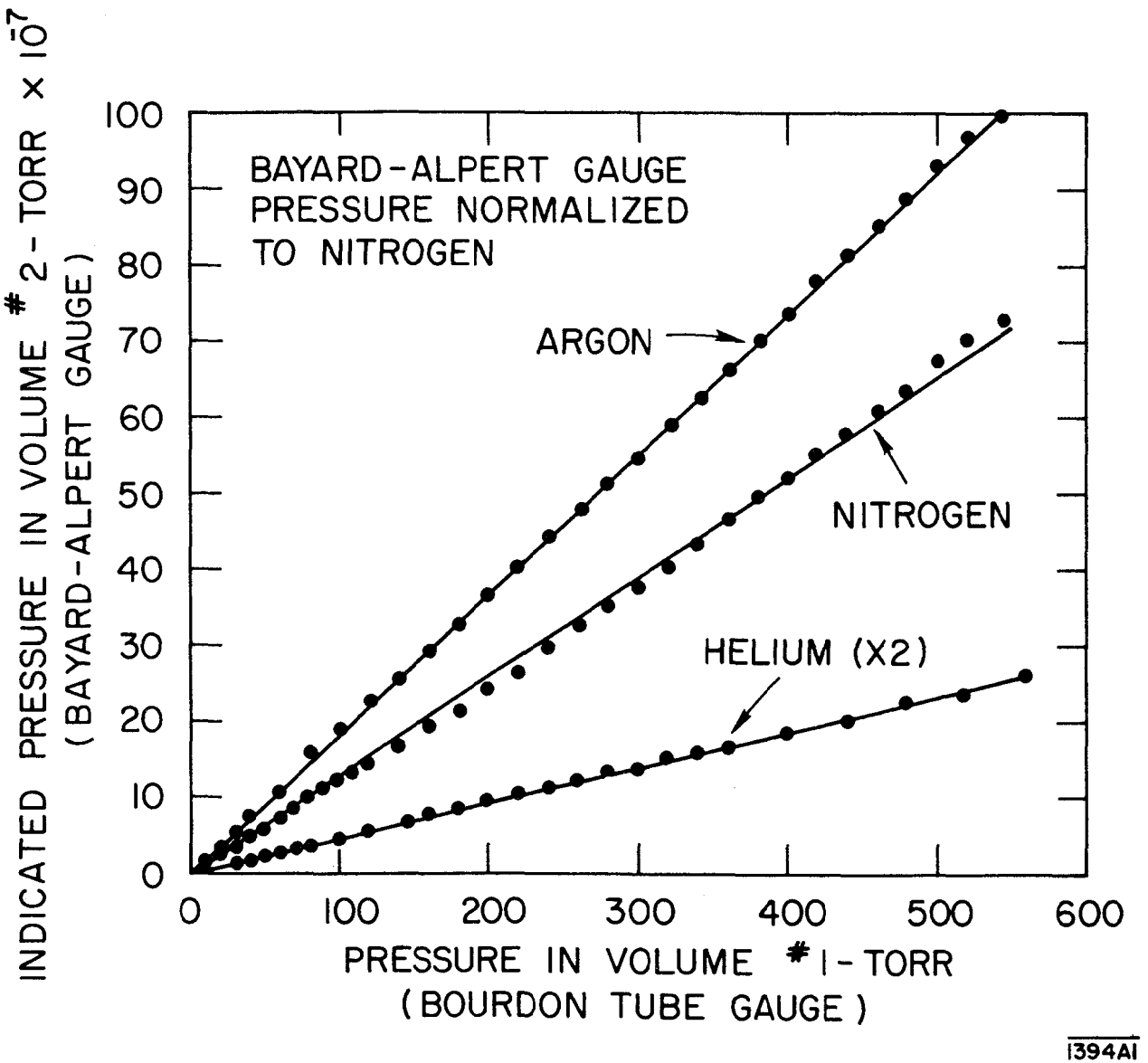
INDICATED PRESSURE IN VOLUME #2 - TORR x 10⁻⁷
(BAYARD-ALPERT GAUGE)
BAYARD-ALPERT GAUGE
PRESSURE NORMALIZED
TO NITROGEN
ARGON
NITROGEN
HELIUM (X2)
0
10
20
30
40
50
60
70
80
90
100
0
100
200
300
400
500
600
PRESSURE IN VOLUME #1 - TORR
(BOURDON TUBE GAUGE)
1394A1

Fig. 3

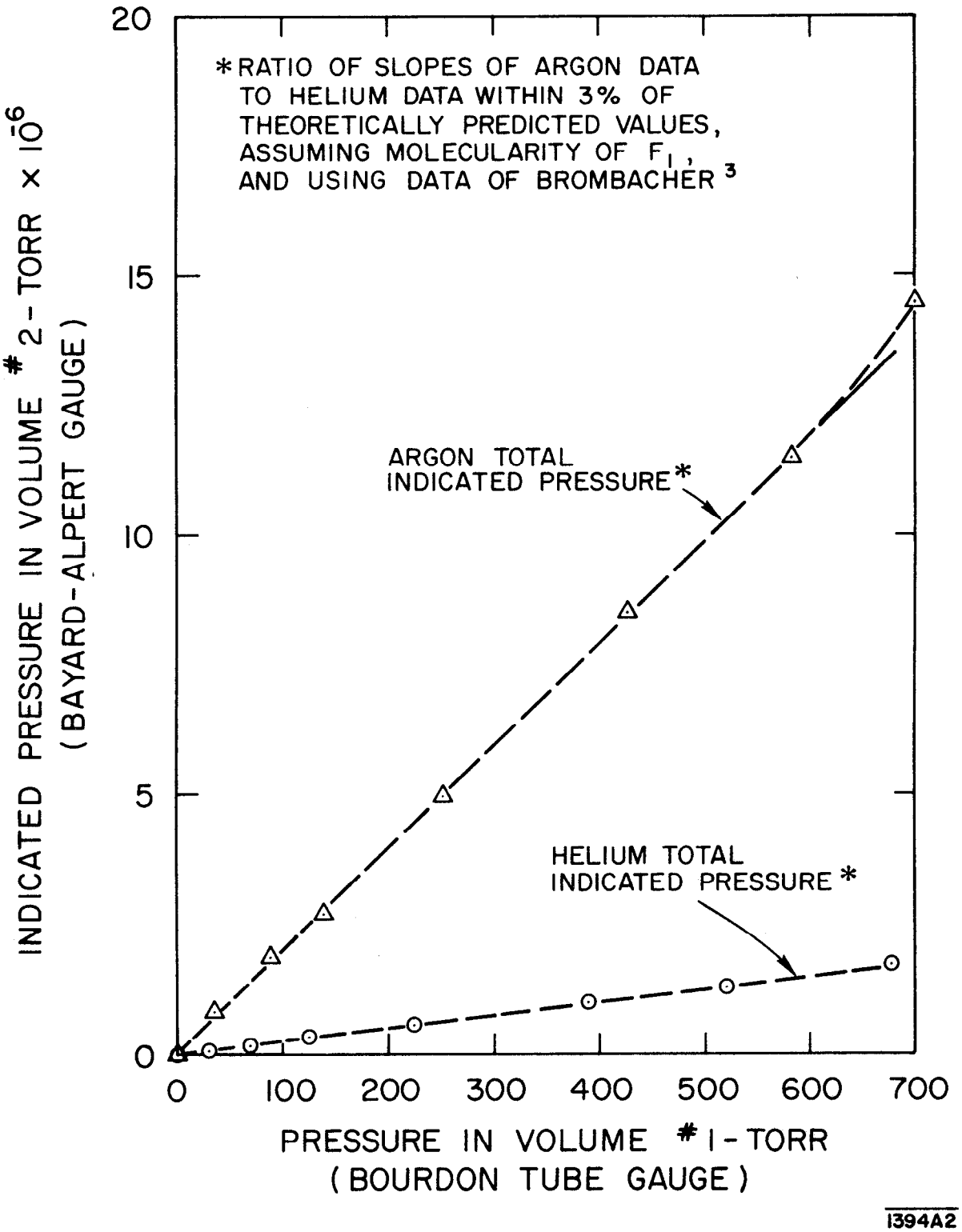
20
15
10
5
0
0
100
200
300
400
500
600
700
*RATIO OF SLOPES OF ARGON DATA TO HELIUM DATA WITHIN 3% OF THEORETICALLY PREDICTED VALUES, ASSUMING MOLECULARITY OF F_1, AND USING DATA OF BROMBACHER [3]
ARGON TOTAL INDICATED PRESSURE*
HELIUM TOTAL INDICATED PRESSURE *
INDICATED PRESSURE IN VOLUME #2 - TORR x 10^{-6} (BAYARD-ALPERT GAUGE)
PRESSURE IN VOLUME #1 - TORR (BOURDON TUBE GAUGE)
1394A2

Fig. 4

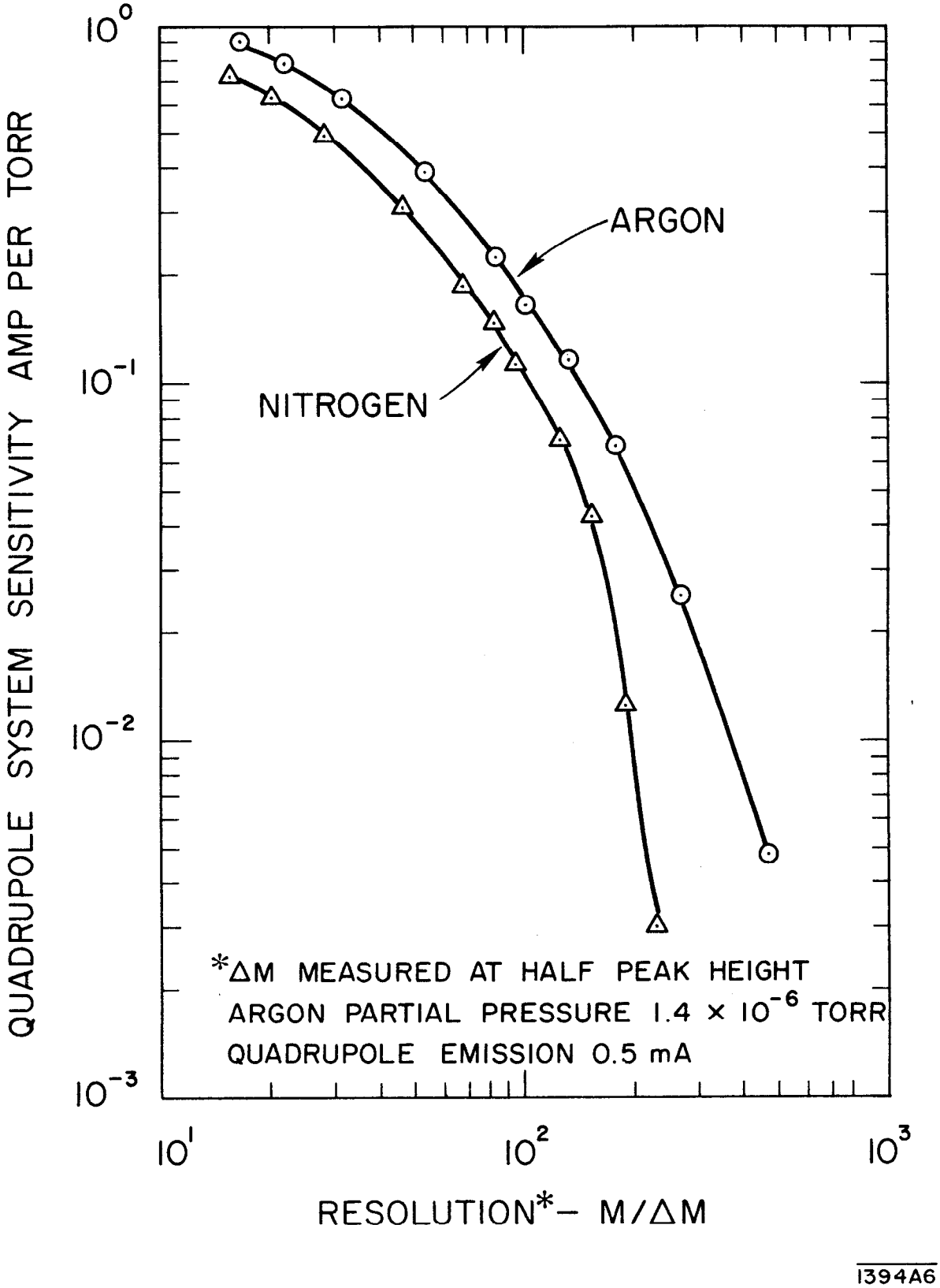
QUADRUPOLE SYSTEM SENSITIVITY AMP PER TORR
10^0
10^{-1}
10^{-2}
10^{-3}
ARGON
NITROGEN
*ΔM MEASURED AT HALF PEAK HEIGHT
ARGON PARTIAL PRESSURE 1.4 × 10^{-6} TORR
QUADRUPOLE EMISSION 0.5 mA
10^1
10^2
10^3
RESOLUTION*– M/ΔM
1394A6

Fig. 5

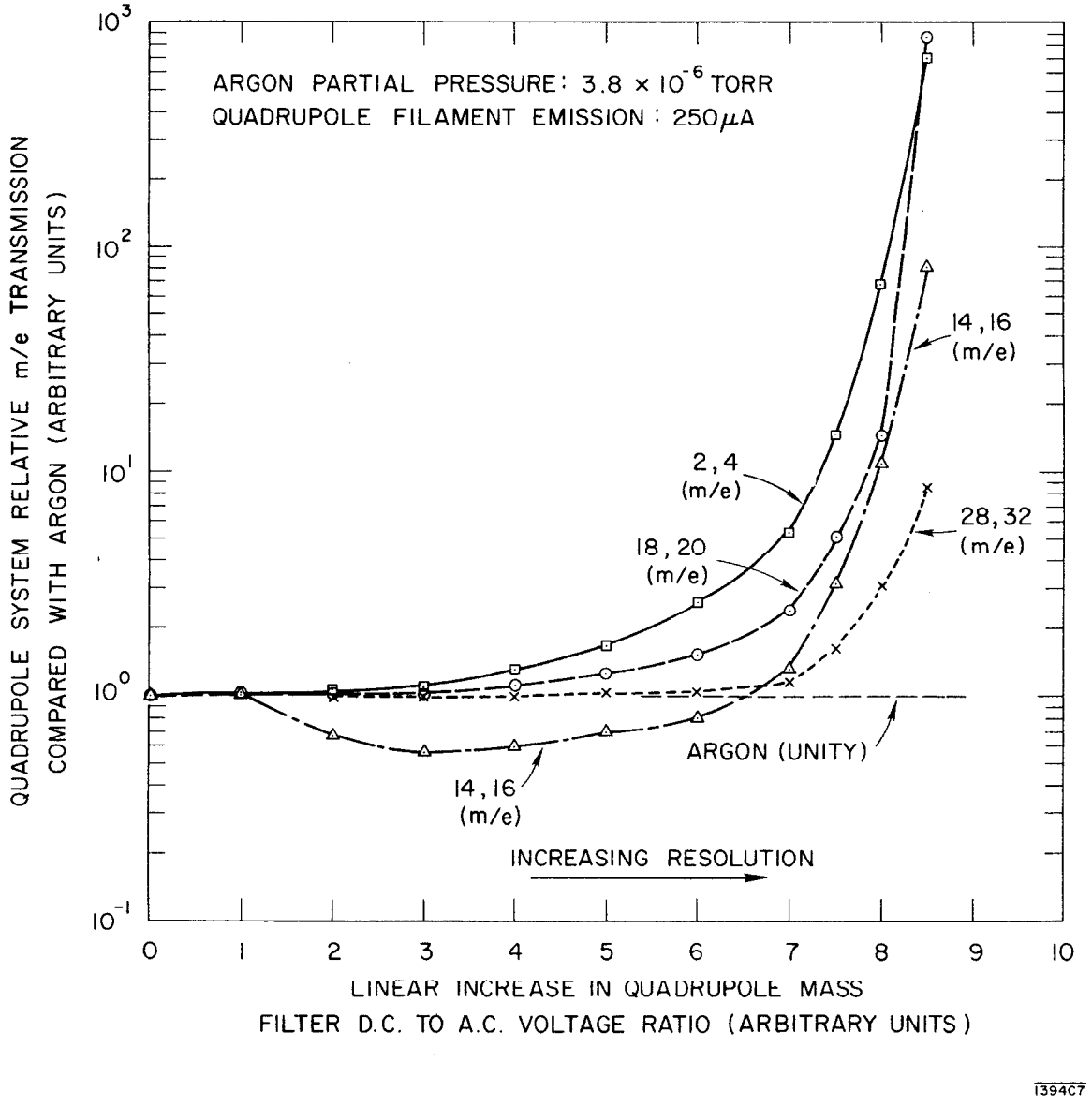
ARGON PARTIAL PRESSURE: 3.8 x 10⁻⁶ TORR
QUADRUPOLE FILAMENT EMISSION : 250μA
QUADRUPOLE SYSTEM RELATIVE m/e TRANSMISSION
COMPARED WITH ARGON (ARBITRARY UNITS)
10³
10²
10¹
10⁰
10⁻¹
2,4 (m/e)
14,16 (m/e)
18,20 (m/e)
28,32 (m/e)
ARGON (UNITY)
14,16 (m/e)
INCREASING RESOLUTION
0 1 2 3 4 5 6 7 8 9 10
LINEAR INCREASE IN QUADRUPOLE MASS
FILTER D.C. TO A.C. VOLTAGE RATIO (ARBITRARY UNITS)
139407

Fig. 6

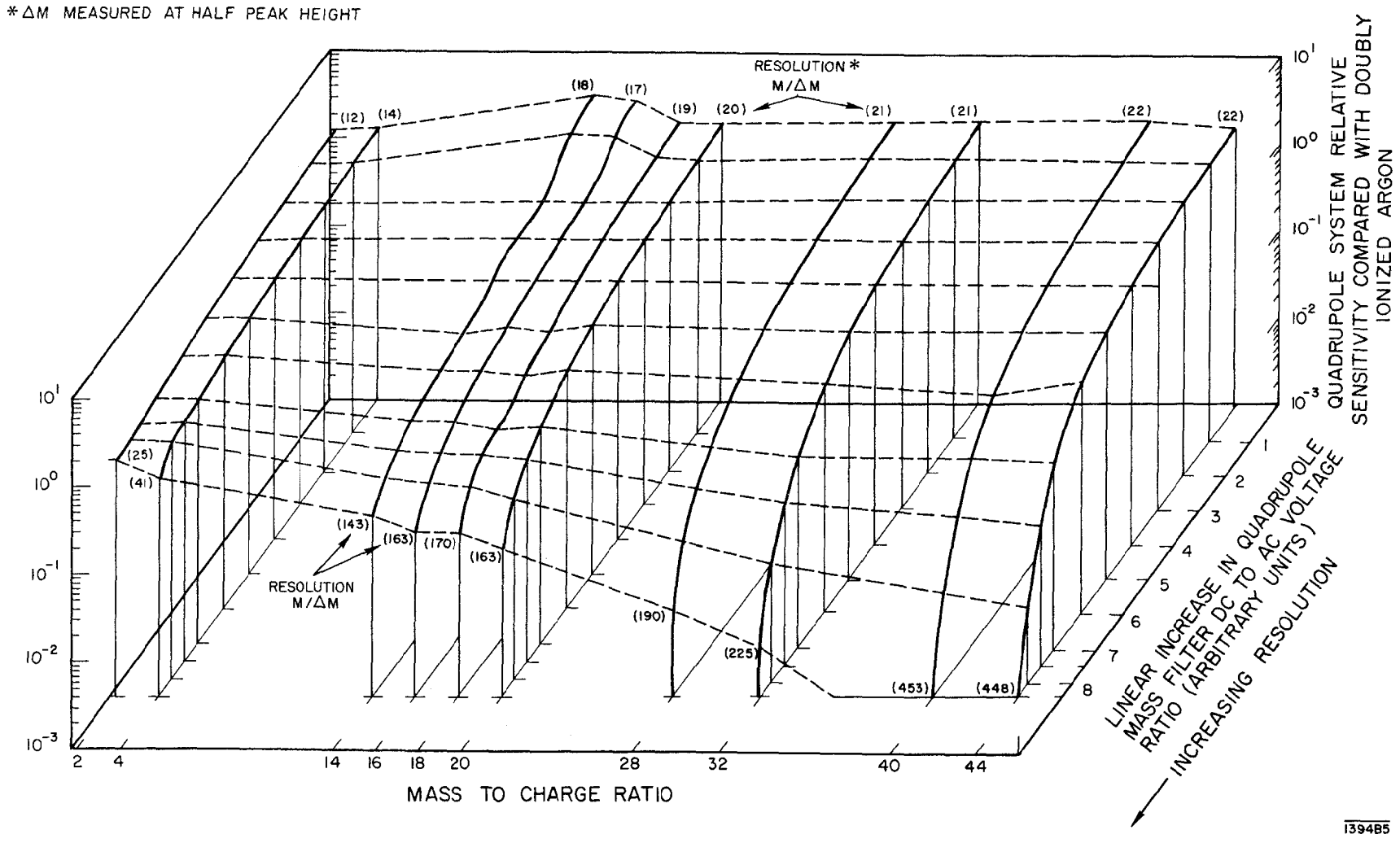
* ΔM MEASURED AT HALF PEAK HEIGHT
RESOLUTION *
M/ΔM
(12) (14) (18) (17) (19) (20) (21) (21) (22) (22)
(25) (41) (143) (163) (170) (163) (190) (225) (453) (448)
RESOLUTION
M/ΔM
10^{1} 10^{0} 10^{-1} 10^{-2} 10^{-3}
2 4 14 16 18 20 28 32 40 44
MASS TO CHARGE RATIO
1 2 3 4 5 6 7 8
LINEAR INCREASE IN QUADRUPOLE MASS FILTER DC TO AC VOLTAGE RATIO (ARBITRARY UNITS)
INCREASING RESOLUTION
QUADRUPOLE SYSTEM RELATIVE SENSITIVITY COMPARED WITH DOUBLY IONIZED ARGON
1394B5

Fig. 7

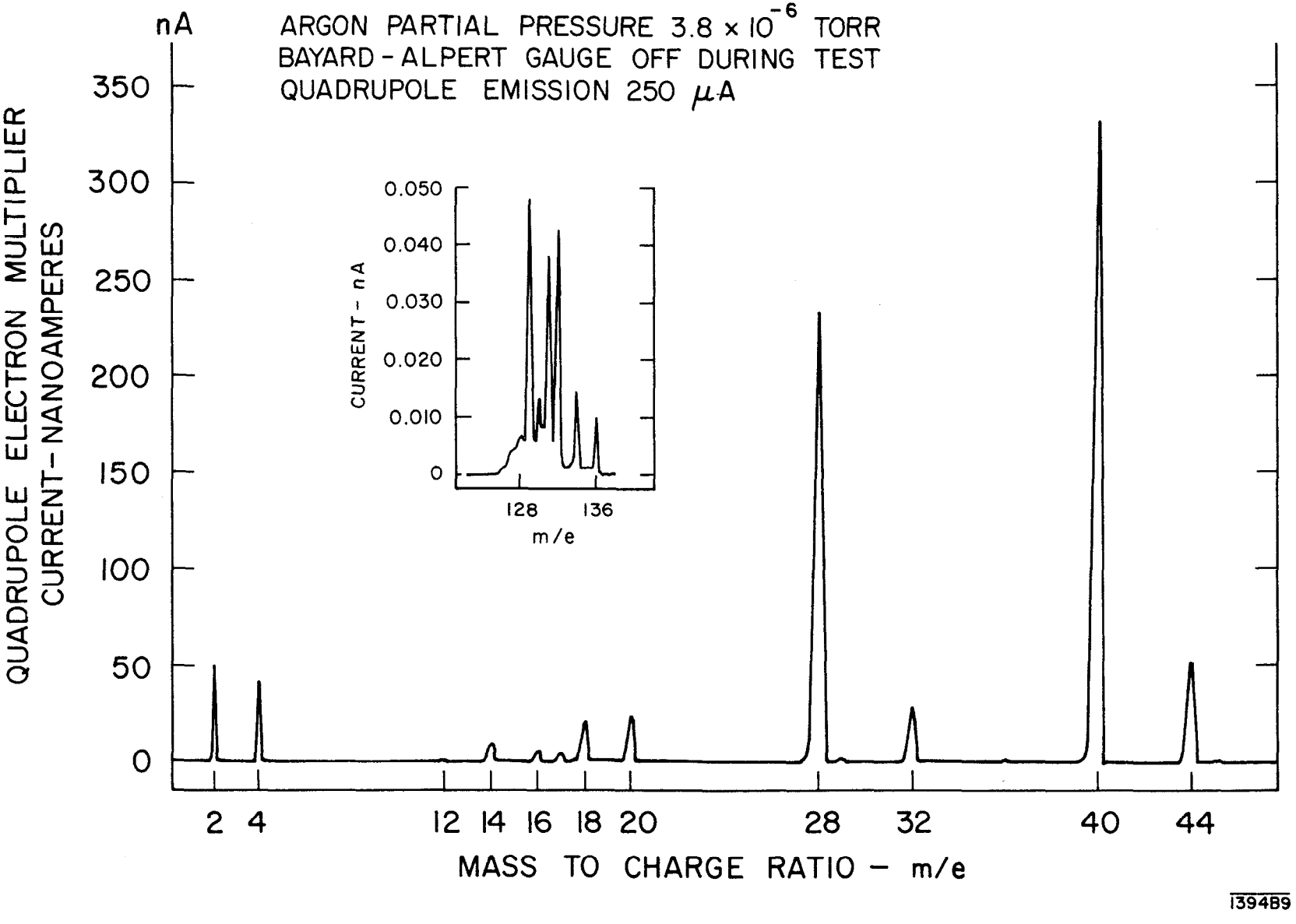
nA
ARGON PARTIAL PRESSURE 3.8 x 10^-6 TORR
BAYARD-ALPERT GAUGE OFF DURING TEST
QUADRUPOLE EMISSION 250 μA
QUADRUPOLE ELECTRON MULTIPLIER
CURRENT-NANOAMPERES
350
300
250
200
150
100
50
0
CURRENT - nA
0.050
0.040
0.030
0.020
0.010
0
128
136
m/e
2 4 12 14 16 18 20 28 32 40 44
MASS TO CHARGE RATIO – m/e
139489

Fig. 8

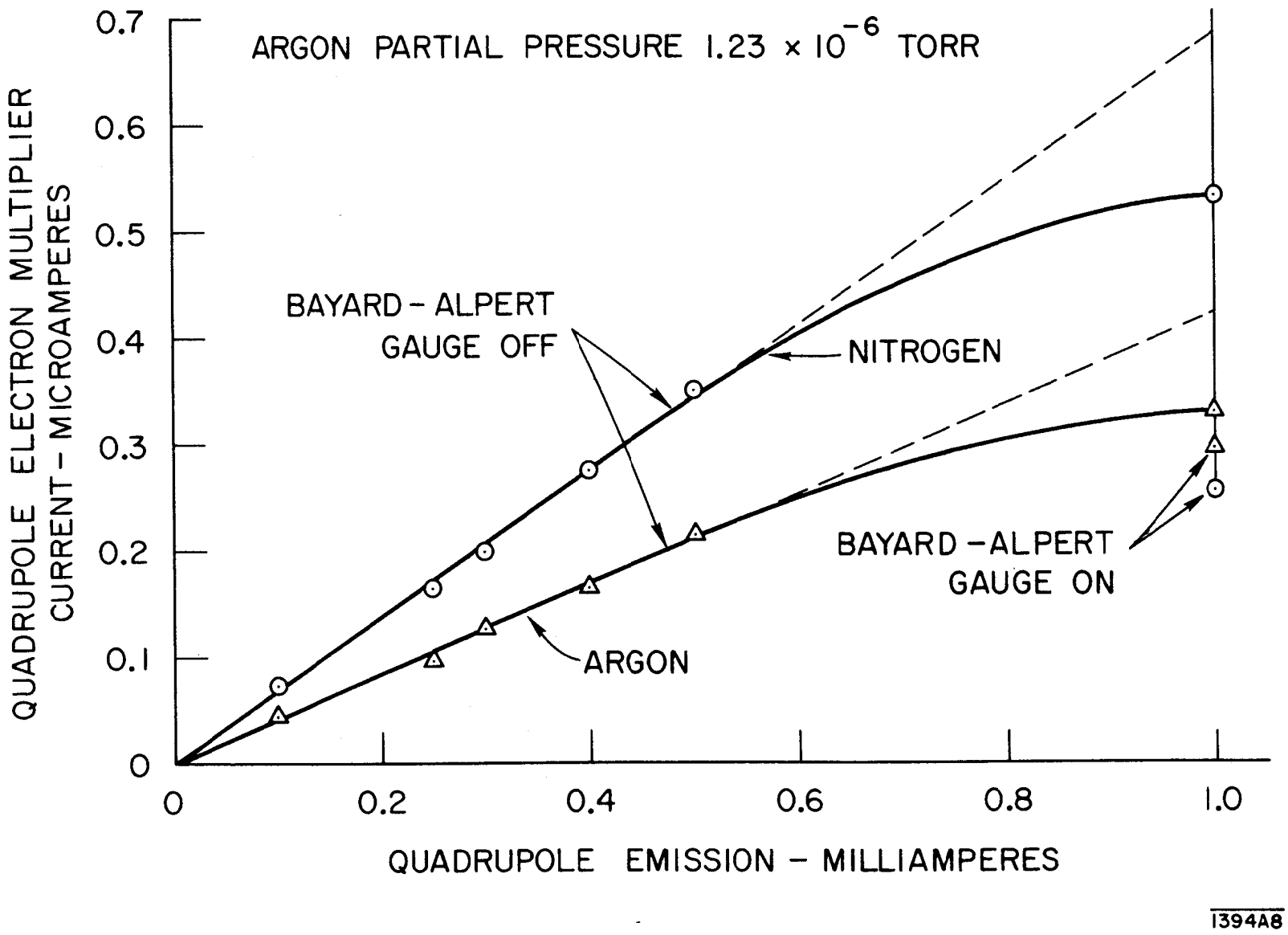
ARGON PARTIAL PRESSURE 1.23 x 10⁻⁶ TORR
QUADRUPOLE ELECTRON MULTIPLIER CURRENT - MICROAMPERES
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
BAYARD - ALPERT GAUGE OFF
NITROGEN
BAYARD - ALPERT GAUGE ON
ARGON
0
0.2
0.4
0.6
0.8
1.0
QUADRUPOLE EMISSION - MILLIAMPERES
1394A8

Fig. 9