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MASS SPECTROMETER ANALYSIS OF NEON ISOTOPES

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ABSTRACT

A 6-in.-radius, 60° sector, dual-collector mass spectrometer was modified for precise ratio measurements of neon isotopes. A bellows was attached to the manifold to regulate the sample pressure behind the leak and thus avoid leak adjustments between samples being compared. An expanded-scale meter was added to the dc amplifier to facilitate precise setting of the ion beam intensity by regulating the manifold pressure with the bellows. With ratios of isotopic ratios of 1.04, the standard deviation of a single determination was ± 0.0001.

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MASS SPECTROMETER ANALYSIS OF NEON ISOTOPES

INTRODUCTION

A mass spectrometer method was needed for measuring small isotopic differences in neon sample pairs having wide variations in sample pressures, even between two samples being compared to each other. Since the samples are admitted alternately to the mass spectrometer ion source, either through individual mixed-flow leaks for each sample or through one common leak, errors from leak-fractionation are possible. With a common adjustable leak, a wider opening is required for lower sample pressure, to maintain a fixed ion current at the collector. Previous studies in this laboratory have shown that fractionation errors can be introduced with an adjustable single-leak system. It has also been demonstrated that separate leaks for each sample may fractionate the isotopes to different degrees[1]. Capillary tubing has been used in front of the leak to create sample flow toward the leak with sufficient velocity to overcome back diffusion. However, an increase in capillary length, plus a reduction in cross section, increases the response time[2,3]. This is especially objectionable with ratio-type measurements and was avoided by using a fixed leak and adjustable bellows to equalize the pressures of the gases behind the leak.

SUMMARY

Improvements were made in the mass spectrometer manifold so that leak fractionation problems were minimized. An expandable bellows was placed on the high-pressure side of the leak to adjust the sample pressure, and a sensitive, expanded-scale meter was added to detect small changes in ion beam intensity while adjusting the bellows. The standard deviation of a single measurement was ± 0.0001 with a ratio of isotopic ratios of 1.04.

APPARATUS

A 6-in., 60° sector type mass spectrometer[4] was modified by enlarging the collector end of the analyzer tube and redesigning the collector to accommodate the $^{20}Ne^+$ and $^{22}Ne^+$ ions. The $^{20}Ne^+$ and $^{22}Ne^+$ ions have a dispersion of 0.571 in. in a 6-in.-radius mass spectrometer. The $^{22}Ne^+$ ions were focused to pass through the collector slit in a low-current plate, while the $^{20}Ne^+$ and $^{21}Ne^+$ ions were collected on the slit plate to facilitate measuring the mole ratio $\frac{^{22}Ne^+}{1-{}^{22}Ne^+}$.

The manifold, as shown in figure 1, was constructed using low-volume solenoid valves, an adjustable bellows, and a diaphragm leak[5] with a capillary 5 in. long and 0.006 in. ID. Valves were provided for trapping and pipetting 1.8 cc of the sample into the larger volume of the manifold to bring the sample pressure within the adjustable range of the bellows.

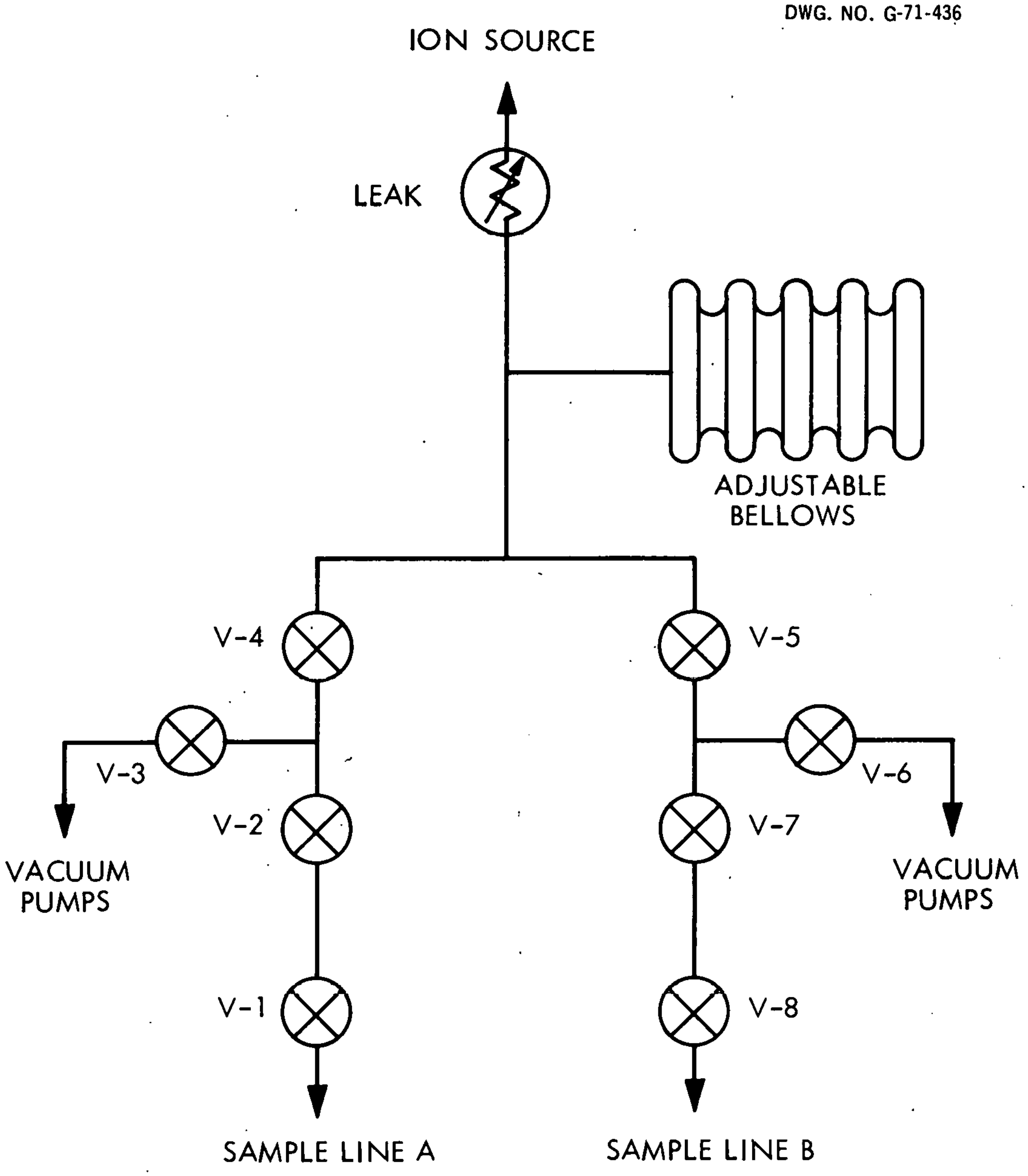

Figure 1

MANIFOLD

The bellows was sized to provide a volume change of 100% in the manifold. The leak was adjusted for the desired ion current and left at this setting. By adjusting the bellows, the ion signal level could be maintained without leak adjustment for sample pressures ranging from 50 to 100 torr.

Since the ion intensity was the only indication used to adjust the manifold pressure, an expanded-scale meter was provided to assure accurate setting of the intensity.

The measuring system was a ratio recorder with solid-state amplifiers[6].

PROCEDURE

The samples to be compared were attached at points A and B, and the trapped air removed through valves 3 and 6. After the introduction of either sample to the manifold, the bellows was adjusted to obtain the proper ion intensity. Measurements were made by alternating members of a pair so that three introductions of one sample bracketed two introductions of the other. If the pressures in the sample tubes differed by more than 100%, the gas with the higher pressure was trapped behind valves 1 or 2 in line A, or valves 7 or 8 in line B, and expanded into the remaining portion of the manifold. The bellows was then used for the final adjustment of pressure.

A pair of standards with a well-established ratio of isotopic ratios was used periodically to check the operating condition of the mass spectrometer. If the ratio did not check the accepted value, no analyses were made until the trouble was located and corrected.

SAMPLE IMPURITY EFFECT

Tests were made to determine the biasing effect of nitrogen on the ratio of isotopic ratios, since air would be the most likely sample contaminant. Mixtures of neon and nitrogen were compared to pure neon of identical isotopic composition, noting the bias of the measured ratios of mole ratios from unity. Figure 2 shows the results of these tests. The beam scattering effect on the ratio of ratios was in the same direction as observed previously[7] in uranium hexafluoride analyses. Nitrogen impurities less than 0.5% caused an error in the ratio of ratios no greater than 0.0001, and frequent mass scans on an analytical mass spectrometer served to detect and eliminate samples with higher nitrogen content.

PRECISION CHECK

A pair of standards was analyzed once a day, interspersed with routine samples. The results are tabulated in appendix A. The mean of fifty-four determinations was 1.0433 with a standard deviation of ± 0.0001 for a single determination.

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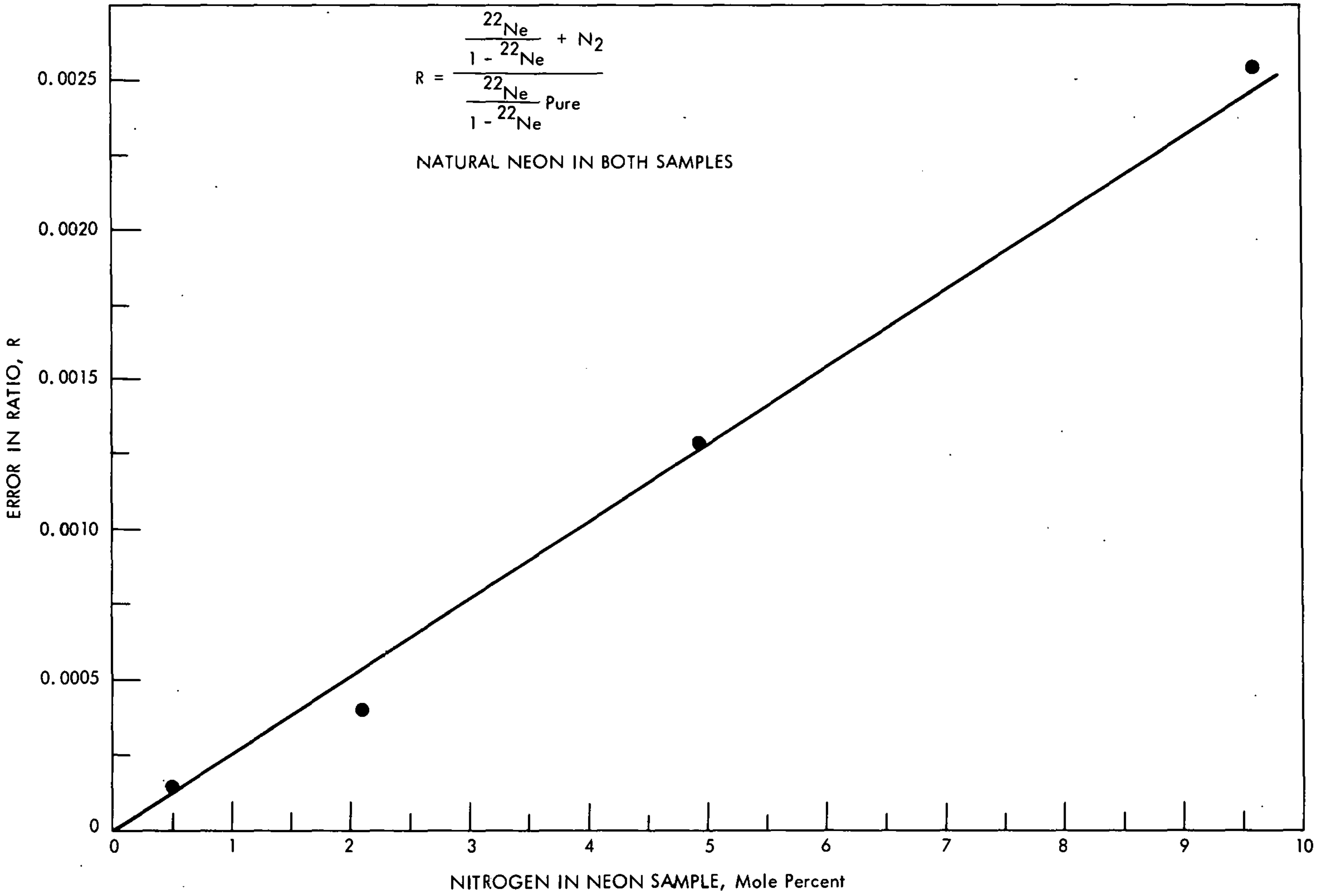

Figure 2

CONTAMINANT BIAS

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APPENDIX

NEON CONTROL ANALYSIS

APPENDIX

NEON CONTROL ANALYSIS

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