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RESPIRATORY GAS ANALYSIS WITH A TIME-OF-FLIGHT MASS SPECTROMETER

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AEROSPACE MEDICAL DIVISION
AIR FORCE SYSTEMS COMMAND
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FOREWORD

This study was made in the Altitude Protection Branch of the Aerospace Medical Research Laboratories. The work was done in support of Project No. 7164, "Biomedical Criteria for Aerospace Flight," and Task No. 716404, "Physiological Criteria for Altered Environments." The evaluation reported was studied during a three year period from June 1961 to June 1964.

This technical report has been reviewed and is approved.

WAYNE H. McCANDLESS
Technical Director
Biomedical Laboratory

ABSTRACT

A modified remote sampling system has been devised for the Bendix Model 17-210 time-of-flight mass spectrometer. This has been adapted for use in measuring respiratory gases breath by breath. The stability of the mass spectrometer output shows a variation of ± 5%. The sensitivity of the system for oxygen is in the range of 0.2% absolute concentration. The accuracy for oxygen is within ± 5%.

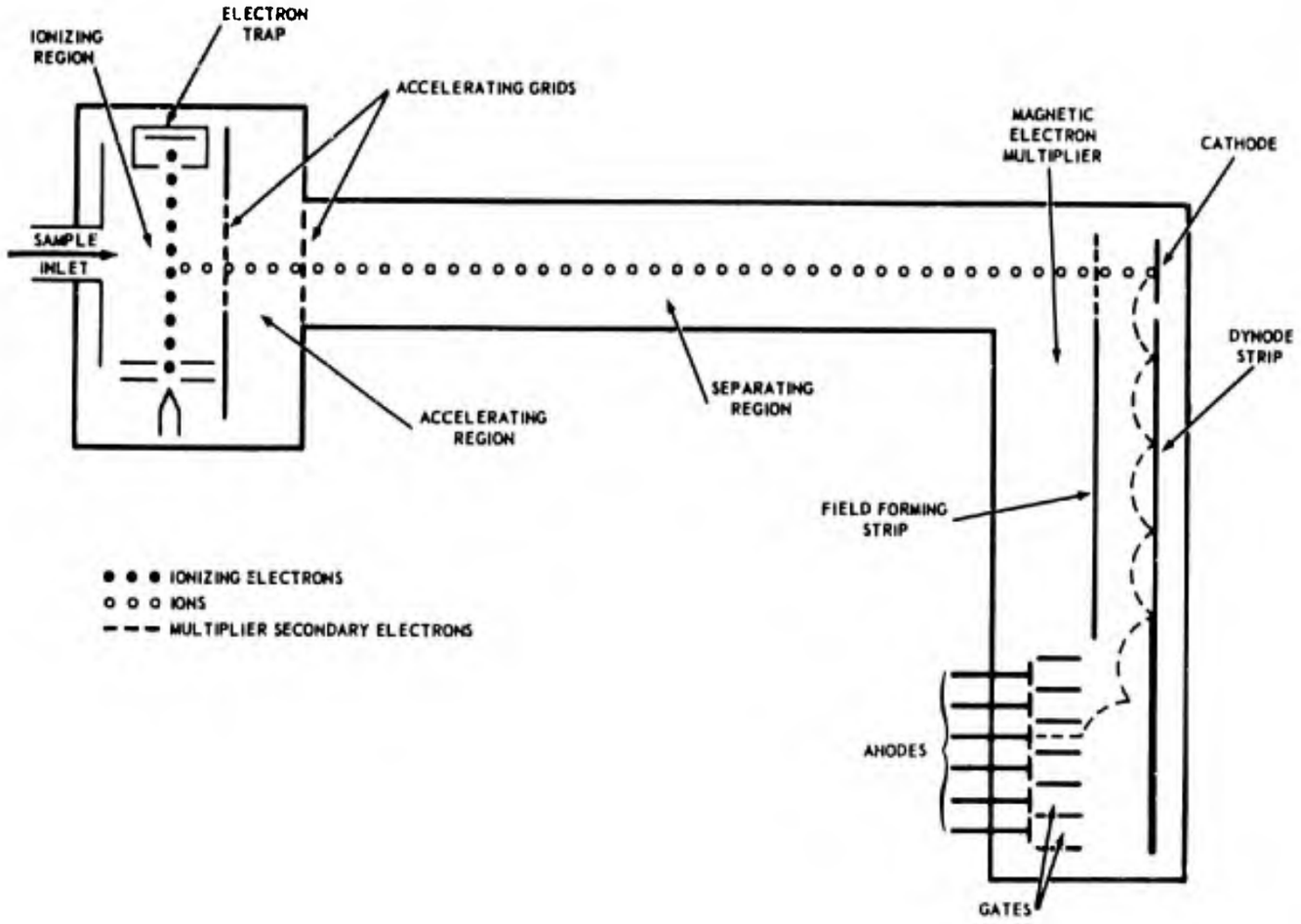

Figure 1. Schematic of Bendix Model 17-210 Time-Of-Flight Mass Spectrometer.

RESPIRATORY GAS ANALYSIS WITH A TIME-OF-FLIGHT MASS SPECTROMETER

SECTION I

INTRODUCTION

This report describes our experience using a Bendix model 17-210 time-of-flight mass spectrometer built by the Research Laboratories Division of the Bendix Corporation. As with any highly complex instrument, the reliability of the results depends on the way it is used. This study, then, was not intended to evaluate the instrument, but was performed solely to apply to measurements of respiratory gases.

SECTION II

DESCRIPTION OF MASS SPECTROMETER

The essential features of this model 17-210 Bendix time-of-flight mass spectrometer are shown in figure 1. The gas to be analyzed is carried from its source by a remote sample system to the sample inlet. The gas enters the low pressure ionizing region through a capillary molecular leak of one to two one thousandth inch diameter. The gas is ionized by a pulsed electron beam emitted from a tungsten filament. The positive ions are ejected from the source region by a pulse applied to the accelerating grids. All ions leave the source region with the same energy and achieve velocities proportional to their masses. Groups of ions separate according to mass during passage through the drift tube. At the end of the drift tube the ion pulses impinge on the cathode emitting a proportionate number of secondary electrons. These are multiplied during their travel down the dynode strip and gated by a pulse to the gate electrode, which is timed with the arrival of the electron multiplier pulse corresponding to the desired mass peak.[1]

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[1]G. J. O'Halloran and W. R. Wyess. The Model 17 Series Bendix Time-Of-Flight Mass Spectrometer. Presented at Eleventh Annual Conference on Mass Spectrometry and Allied Topics. May 1963. San Francisco, California.

SECTION III

MODIFICATION OF SAMPLE SYSTEM

Certain modifications were necessary to adapt this unit to continuous breath by breath measurements of respiratory gases. The sample head was modified as shown in figure 2. This decreased the dead space volume by one half to permit a more rapid response time. The remote sample line consisted of a length up to 20 feet of polyethylene tubing fitted with modified hypodermic needles as shown in figure 3. To construct the sample line, a 25-gage 0.25-inch needle was filed to a blunt end. A stylet was placed in the needle to prevent blocking during its insertion into one end of the desired length of polyethylene tubing. This joint was sealed with household cement, and then adapted to the modified sample head. The connection was lubricated with a silicone gel to make it air tight. The other end of the sample line was similarly fitted with a needle that was then inserted into a mouthpiece. So long as care was taken to avoid plugging the line with silicone gel at the sample head fitting, difficulties with obstruction of the capillary molecular leak were very infrequent.

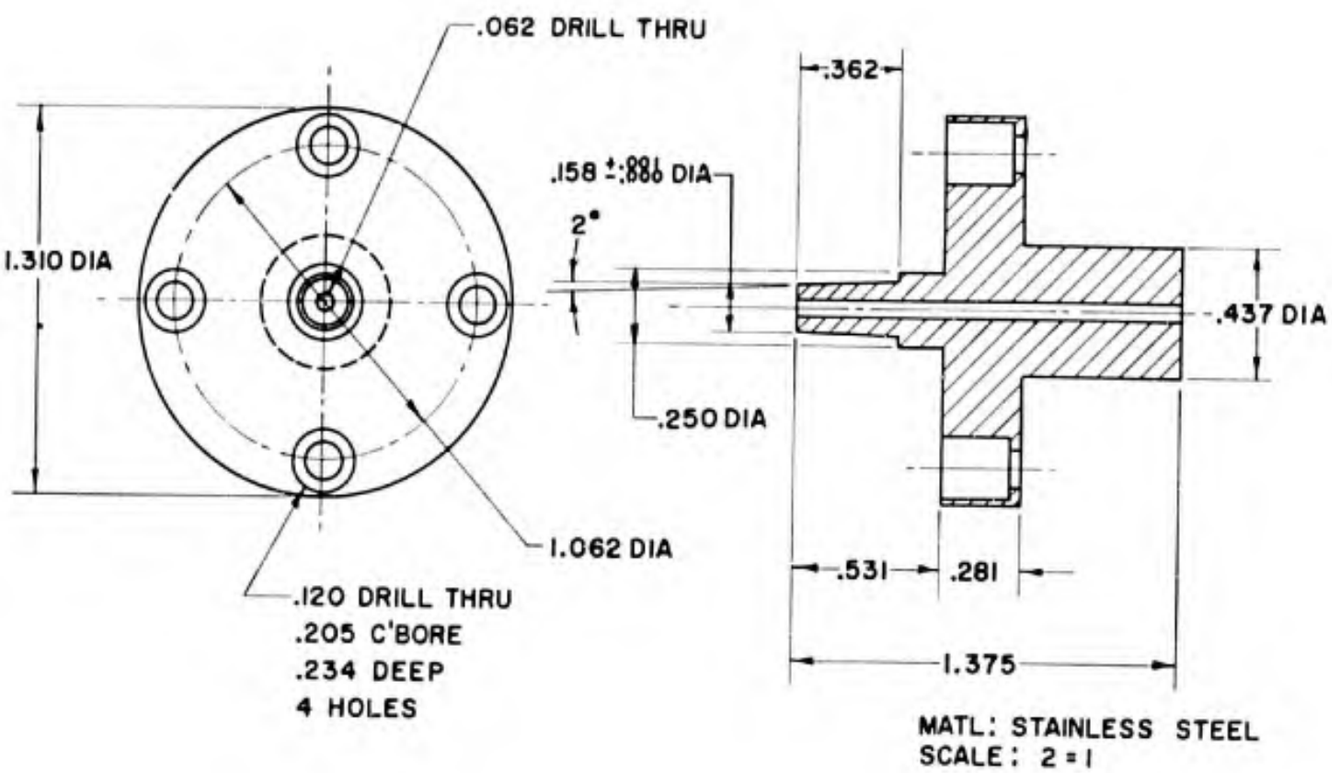

Figure 2. Modified Sample System Head.

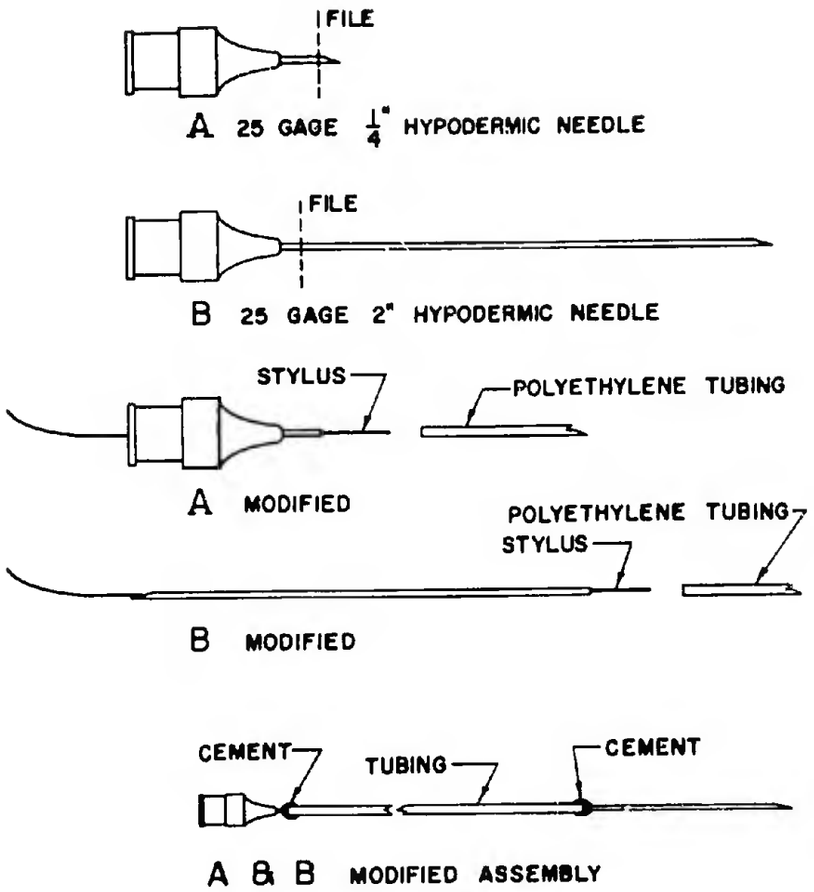

Figure 3. Remote Sample Line Construction.

For most purposes a sample line length of 5-10 feet with a 0.015-inch ID polyethylene tubing maintained at a pressure between 0.8 and 1.0 mm Hg gave the desired result. The output voltage for any given gas in this system is, of course, linearly related to the sample system pressure. See figure 4. This data was obtained by sampling from a closed tube connected to the reduction valve on a tank of pure nitrogen. By altering the reduction valve aperture, the pressure in the sample line could be regulated and maintained while voltage output on mass 28 was recorded.

The recording system used was a D-C amplifier and a model 5-124 Consolidated Electronics Corporation oscillograph. A 90% response time for the entire system of 1 second was obtained with a mass spectrometer time constant setting of 0.5 second. This gave a stable recording and was an appropriate response time for breath-by-breath measurements at a frequency of 10 breaths per minute. The 90% response time could be shortened to 0.3 second by adjustment of the time constant setting. The 90% response time was determined by measuring the time for 90% of a total response after abruptly removing the sample line from a pure oxygen source to room air.

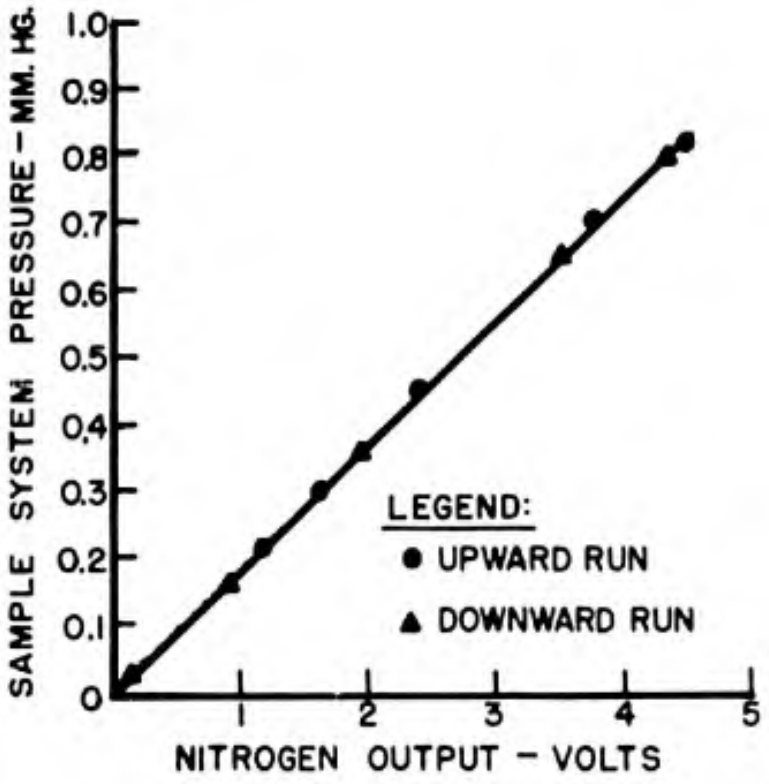

Figure 4.
Relation of Nitrogen Output
to Sample System Pressure.

SECTION IV

OVERALL STABILITY OF SYSTEM

The overall system stability was determined by continuous sampling from a 100-liter Tissot spirometer containing pure nitrogen. The voltage output on mass 28 was measured periodically and found to vary by about ± 5%. A representative run is shown in figure 5. The gate was checked prior to each determination so that gate drift should not be a factor in this instability. These results illustrate the need for frequent calibration in the use of this machine.

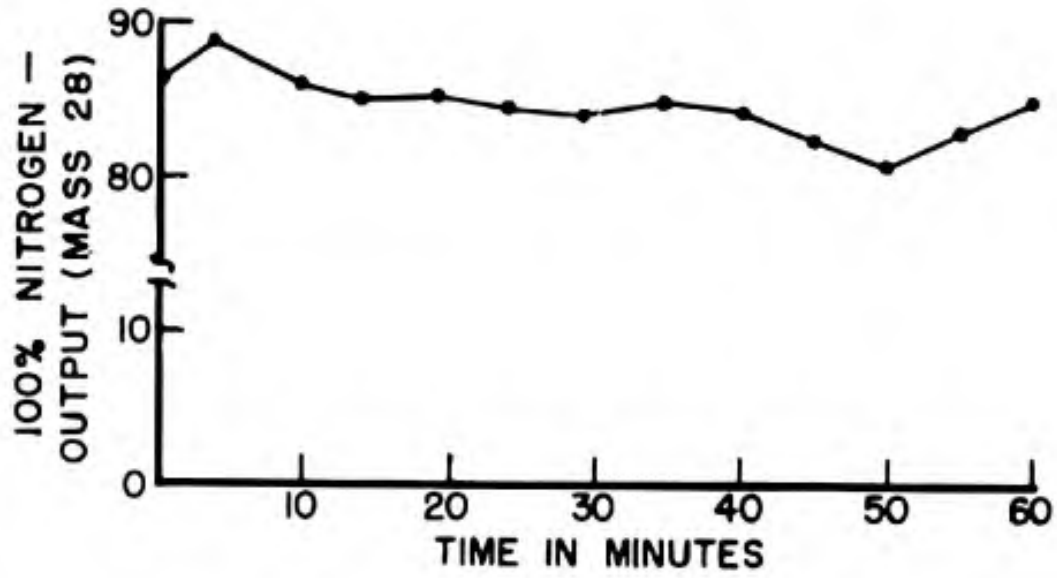

Figure 5. Periodic Determinations of a Single Sample of Pure Nitrogen.

SECTION V

ACCURACY OF OXYGEN MEASUREMENT

The accuracy of gas measurement was tested by comparing the mass spectrometer analysis of various oxygen in nitrogen mixtures to that of the micro-Scholander gas analyzer. The mixtures were prepared in a 100-liter Tissot spirometer and mixed by fan for at least 20 minutes. A sample was collected in a greased syringe, then the mass spectrometer analysis was made at the collecting site, and finally a second syringe sample was obtained. The two syringe samples were analyzed in duplicate on the micro-Scholander. The standard deviation for these analyses was $\pm$ 0.1%.

The mass spectrometer was calibrated with gaseous aviator's breathing oxygen (99.5%) and gaseous water pumped nitrogen (99.5%). Only mass 32 was measured because previous analysis indicated that the splitting of oxygen into masses 32 and 16 was in constant 5:1 ratio over the range of 1-100% concentrations. See figure 6. To avoid any possible error due to gate drift, the spectrum was scanned at a scan rate of 7, time constant setting of 0.02 second and recorded on the oscillograph. The output on mass 32 was then measured on the paper deflection. Background levels of mass 32 were determined at the multiplier voltage and any range settings used during the measurement and subtracted from the measured values of mass 32. The concentrations were then calculated from the measured outputs from the relationship,

$$\frac{99.5}{K_1 - B} = \frac{X}{K_2 - B}$$

where

X is the unknown concentration

B is the background output of mass 32 under operating conditions

K_1 is the output on aviator's breathing oxygen

K_2 is the output of the unknown gas.

The results of this comparison are shown in figure 7 and indicate an accuracy within about $\pm$ 5% over the range of concentrations measured.

Below concentrations of oxygen of 0.2% the mass spectrometer analysis usually indicated zero concentration. The sensitivity of the mass spectrometer for oxygen, then, appears to be in this range of concentration.

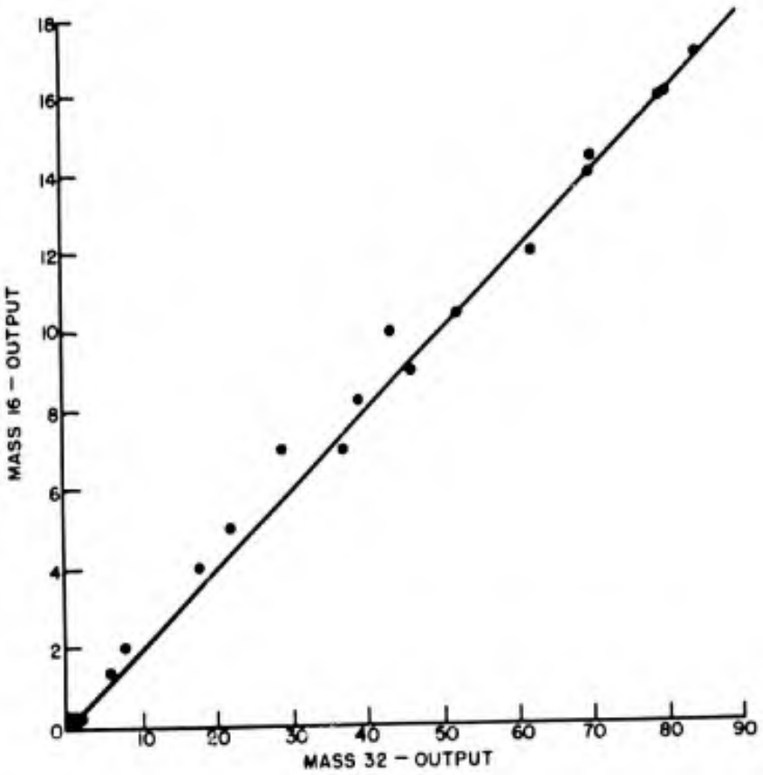

Figure 6. Ratio of Outputs on Masses 32 and 16 for Oxygen Mixtures ranging from 1-100% Concentration.

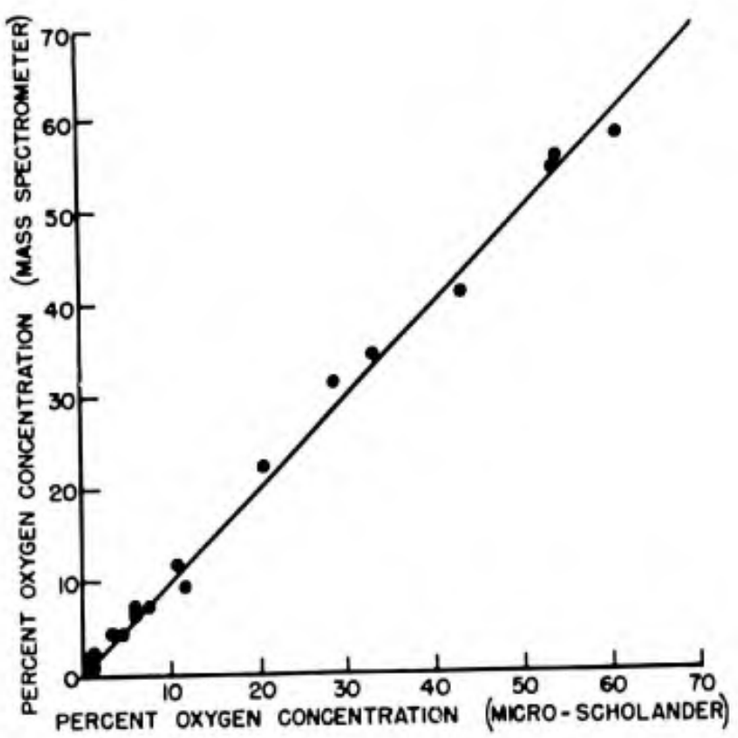

Figure 7. Comparison of Measurements of Oxygen Concentration in Various Mixtures by the Mass Spectrometer and Micro-Scholander Techniques.

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