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CALIBRATION OF THE CRYOGENIC DENSITOMETER - AN UPPER ATMOSPHERE DENSITY PROBE

by W. W. Youngblood

Prepared by

NORTHROP CORPORATION

Huntsville, Ala.

for George C. Marshall Space Flight Center

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • APRIL 1971





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FOREWORD

This report presents the results of the Cryogenic Densitometer Calibration performed by Northrop-Huntsville, Huntsville, Alabama, while under contract to the Aero-Astroynamics Laboratory of Marshall Space Flight Center (NAS8-20082). The task was conducted in response to the requirements of Appendix A-1, Schedule Order No. 32.

The work was performed with the technical coordination of Mr. Robert E. Smith of the Aerospace Environment Division, Aero-Astroynamics Laboratory, MSFC.

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Section I

INTRODUCTION

The Cryogenic Densitometer is an instrument designed to provide unique mass density measurements of the upper atmosphere. The particular instrument that is the subject of this report was designed to be used as part of a sounding rocket experiment in the approximate altitude range of 140 km to 180 km. Originally designed and constructed by the Celestial Research Corporation of South Pasadena, California, the instrument was calibrated by Northrop Corporation, Huntsville, Alabama (Northrop-Huntsville is presently preparing the instrument for flight qualification). Upon completion of flight qualification, the instrument is scheduled to be launched as a part of the MUMP 10 (Marshall, University of Michigan Measurement Probe) instrument payload during Winter 1970 from Wallops Island, Virginia, as part of a joint study of the University of Michigan Space Physics Research Laboratory and the Aerospace Environment Division of the Marshall Space Flight Center.

The sensing element of the Cryogenic Densitometer is a thin, half-inch diameter quartz crystal (AT cut 39° 45'). By cryogenically cooling the crystal to near liquid helium (LHe) temperature, the crystal acts as a miniature condenser (cryopump) freezing out the atmospheric constituents arriving at its surface. An oscillator circuit forces the crystal to resonate in its thickness shear mode. The measurement of the mass of the atmospheric constituents condensing on the oscillating crystal is based on the linear response of such a device according to the relationship,

$$\frac{\Delta f}{\Delta t} = C \frac{\dot{m}}{A_c} f_r^2 \quad (1-1)$$

where: $\frac{\Delta f}{\Delta t}$ = Response of the crystal per unit time, Hz/sec

C = Crystal constant

\dot{m} = Rate of mass buildup on crystal, gms/sec

A_c = Crystal area, cm²

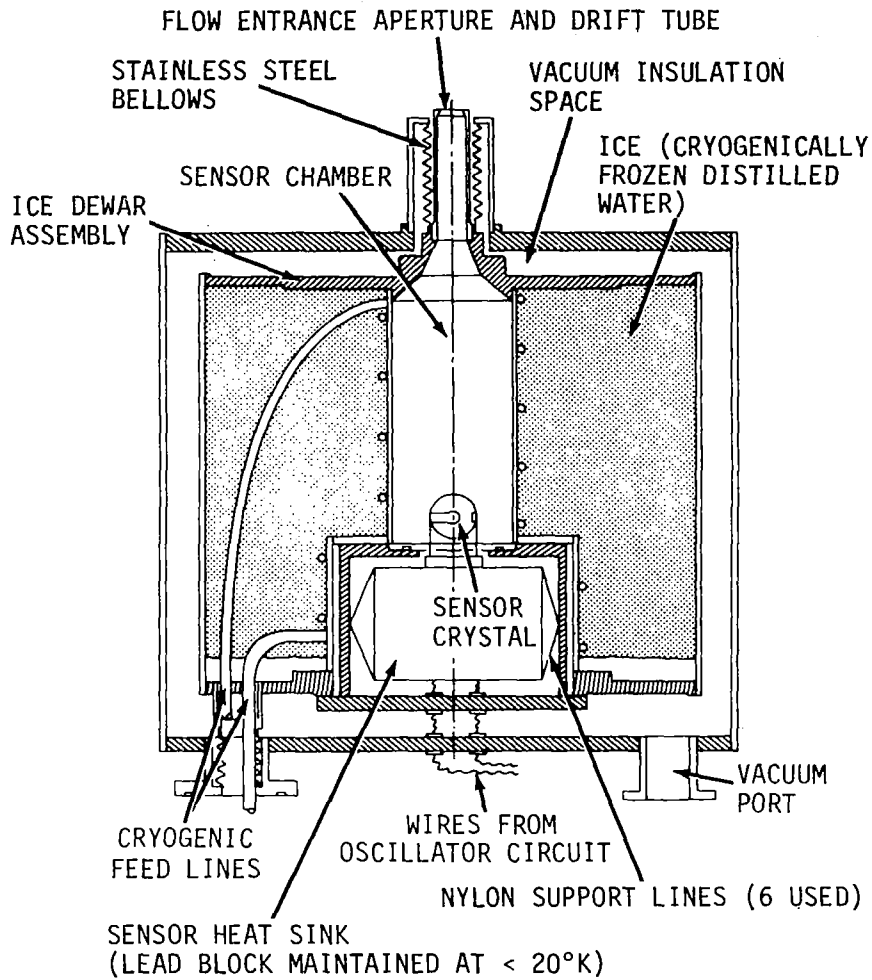
f_r = Resonant frequency, Hz.

The value of C in equation (1-1) is generally about 2.26×10^{-6} for the type of crystal used in the Cryogenic Densitometer (ref. 1). Thus, one could theoretically use equation (1-1) directly to reduce the frequency response data to determine m (after accounting for the transmission probability of the entrance aperture). However, this would not provide satisfactory confidence in the data since each crystal may have a slightly different constant, C , and difficulty would be experienced in predicting the effective crystal area, A_c . Therefore, a calibration was required to determine experimentally the crystal's response to either a known mass of condensable gases or to a known static pressure and temperature. The choice of the calibration method is discussed in Section II.

The configuration of the Cryogenic Densitometer is shown schematically in Figure 1-1. The mechanical portion of the instrument consists of three distinct regions: (1) the sensor chamber, (2) the radiation guard reservoir, and (3) the vacuum insulation space. These three regions are described briefly in the following paragraphs:

Sensor Chamber: The sensor chamber is the innermost cavity of the Cryogenic Densitometer and contains only the quartz crystal and its heat sink. The quartz crystal is mounted by stainless steel clips on a small, lead-filled stainless steel cylinder which serves as the crystal's heat sink (the crystal and its heat sink are cooled to approximately 10°K). The crystal heat sink is suspended from its enclosure by six small diameter nylon cords. The sensor chamber is completely sealed except for an orifice and drift tube which communicate the sensor chamber with the atmosphere. In the flight configuration, the orifice and drift tube are likewise sealed off with a spring-loaded cap. The interior of the sensor chamber is painted a flat black to minimize radiation tunneling.

Radiation Guard Reservoir: The radiation guard reservoir is simply an ice-filled (distilled water) dewar designed to surround the sensor chamber except for the orifice-drift tube and a small flange which supports the crystal heat sink housing. The guard reservoir contains approximately 1640 cm^3 of water which, when cooled to liquid-nitrogen temperature ($\sim 78^\circ\text{K}$), provides some four hours of time before the reservoir warms up beyond the temperature



NOTE: THIS SKETCH IS DESCRIPTIVE ONLY (FINE DETAIL NOT SHOWN)

Figure 1-1. SCHEMATIC OF CRYOGENIC DENSITOMETER

at which it ceases to be an effective radiation barrier. Water was chosen as a heat sink material for its high specific heat; however, a considerable amount of simplicity in design was lost in choosing water over metal as a heat sink material. During the cooldown procedure of the instrument, the water (ice block) is actually cooled as low as possible ($\sim 10^{\circ}\text{K}$) using LHe from a commercial dewar. Cooldown of the crystal and its heat sink is accomplished by conduction heat transfer (by introducing gaseous helium (GHe) into the sensor chamber to a pressure of about $100\mu\text{ Hg}$). Upon completion of the crystal heat sink cooldown the guard reservoir is slowly warmed to about 60°K .

Vacuum Insulation Space: The outer wall of the guard reservoir and the outer shell of the entire Cryogenic Densitometer form a cavity which is evacuated prior to any cryogenic conditioning. All surfaces of this cavity are covered with reflective material (polished aluminum foil) and several square centimeters of surface area on the guard reservoir are coated with activated charcoal to serve as a cryosorption pump. This region serves, therefore, as a vacuum insulation between the guard reservoir and the outer surface of the Cryogenic Densitometer. Its specific purpose is to maintain the guard dewar at a temperature in the 60°K to 85°K range as long as practicable.

Details of the design and construction of this Cryogenic Densitometer are given in a final report by Celestial Research Corporation (ref. 2). No further discussion of the instrument's design and construction will be given in this report except where some difficulty was experienced and/or a modification was made. Section II discusses the method of calibration used and compares it with the original calibration performed by Celestial Research Corporation. Section III describes the calibration equipment used and outlines the procedures. The results of the calibration are presented in Section IV. Comments are made in Section V relevant to the anticipated flight measurements. Section VI presents the conclusions and recommendations and Section VII lists the references.

Section II

METHOD OF CALIBRATION

This section discusses two methods of calibrating the Cryogenic Densitometer: (1) the known mass method (used by Celestial Research Corporation) and (2) the constant pressure method used by Northrop-Huntsville in the present calibration.

2.1 THE KNOWN MASS METHOD

In the known mass method, an accurately measured volume of the test gas (N_2) is allowed to be slowly admitted to the vicinity of the cryogenically cooled quartz crystal. If the pressure and temperature of the known volume are likewise accurately measured, then the amount of mass introduced to the crystal is known. Celestial Research Corporation performed this calibration on the crystal in two different configurations (ref. 3). The first configuration placed the crystal in an incremental mass calibration system (the Celestro Digivac) mounted so that the flight mounting would be duplicated as closely as possible. The results of this test appear to be quite consistent and both the condensation sticking coefficient and the crystal sensitivity were determined. The numerical values for the sticking coefficient, σ , and the crystal sensitivity, H , were determined as 0.81 and 1.65×10^8 Hz/gm, respectively. The reader is referred to reference 3 for details of this calibration.

Following the above Digivac calibration, CELESCO installed the crystal in the Densitometer package (in its basic flight configuration). The calibration again proceeded using a known mass of gas. Eight good runs were averaged giving a mean sensitivity of $H = 1.38 \times 10^8$ Hz/gm. However, there was a considerable amount of scatter in the results, from a low of 0.5 percent deviation from the mean to a high of 15.5 percent deviation from the mean. No explanation was given for the scatter in the results for this calibration. Since the calibration was performed with the Densitometer in its flight configuration, the sensitivity derived from this calibration is the one that should logically be associated with the crystal. However, it would have been desirable for there to have been more exhaustive testing with less scatter in the data.

2.2 THE CONSTANT PRESSURE METHOD

Northrop-Huntsville was asked to participate in an additional calibration of the Densitometer prior to its launch. A high and ultrahigh vacuum calibration system was immediately available at the MSFC Aerophysics Division's Experimental Physics Section (S&E-AERO-AEP). This facility, under the direction of James A. Carter, provided all the necessary supplies and instrumentation for the calibration.

The basis for this calibration is the High Vacuum Calibration System (HVCS) designed and built by the Geophysics Corporation of America, Inc. This system (Figure 2-1) is designed to permit calibration of most vacuum gauges against McLeod Gauges over a vacuum range of a few Torr to approximately 10^{-8} Torr. Since this range adequately covers the pressure environment expected to be experienced by the Densitometer in flight, it was decided to compare the crystal's response to a known pressure rather than using the known mass technique of CELESCO. The Densitometer is simply connected to a large calibration chamber on the HVCS which may be evacuated to about 10^{-9} Torr (with an adequate bakeout). This calibration chamber is also communicated to the McLeod gauges. Thus, both the Densitometer and the McLeod gauges are exposed to the same pressure simultaneously. The calibration chamber is large enough that the small amount of flow caused by the cryopumping of the Densitometer crystal is negligible when compared to the HVCS pumping system.

It is necessary to introduce some of the Densitometer's geometry into the crystal sensitivity determination when using this known pressure calibration. This is because the instrument is "conduction limited" by the geometry of its entrance orifice and drift tube. As shown in Figure 2-2, the geometry of the calibration equipment is such that the free-molecular-flow conduction limitation from the calibration chamber to the sensor crystal is due mainly to the orifice and drift tube of the Densitometer itself. Thus, the transmission probability, K , of the entering and escaping molecules must be determined for this geometry.

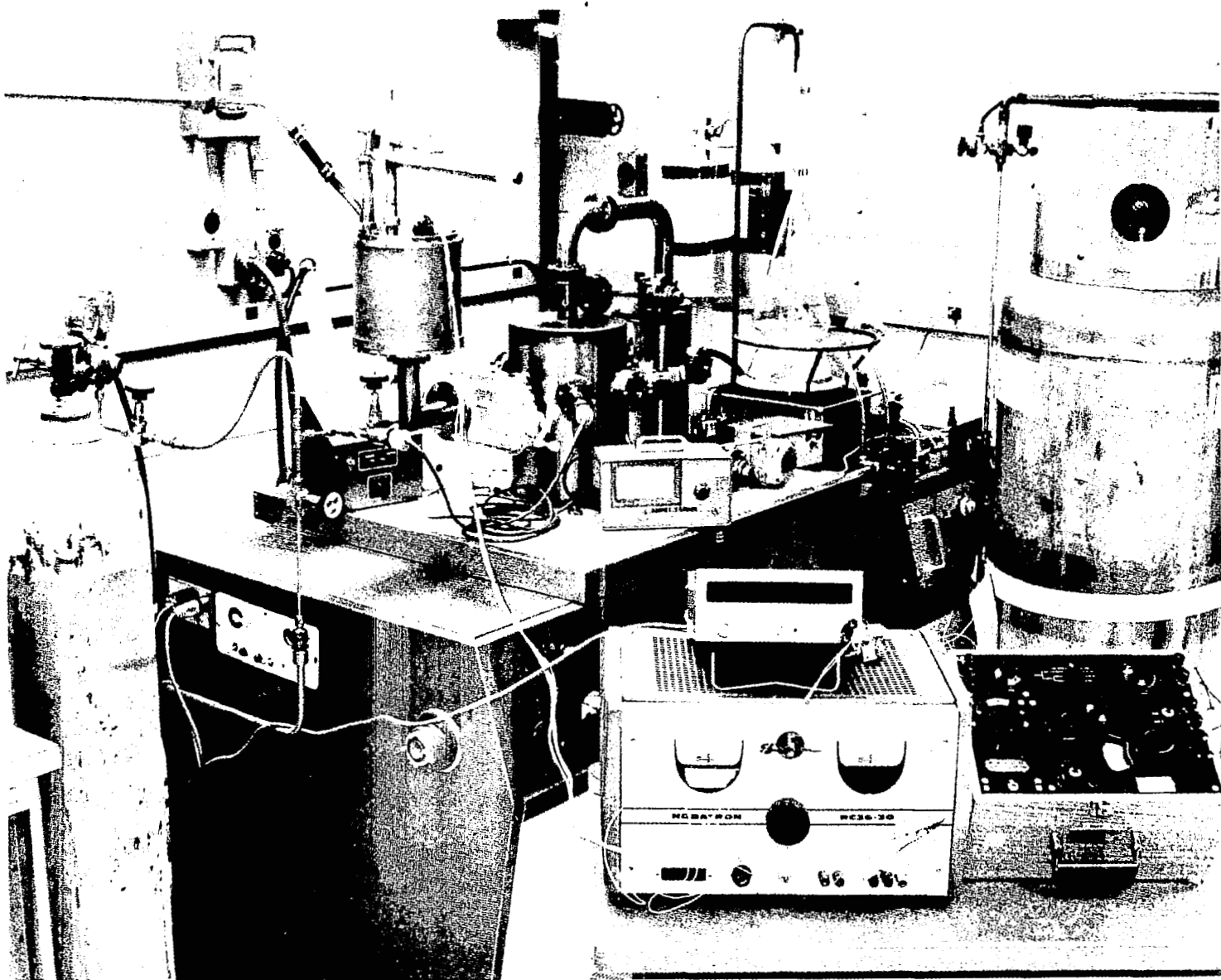


Figure 2-1. CRYOGENIC DENSITOMETER MOUNTED ON THE HIGH VACUUM CALIBRATION SYSTEM

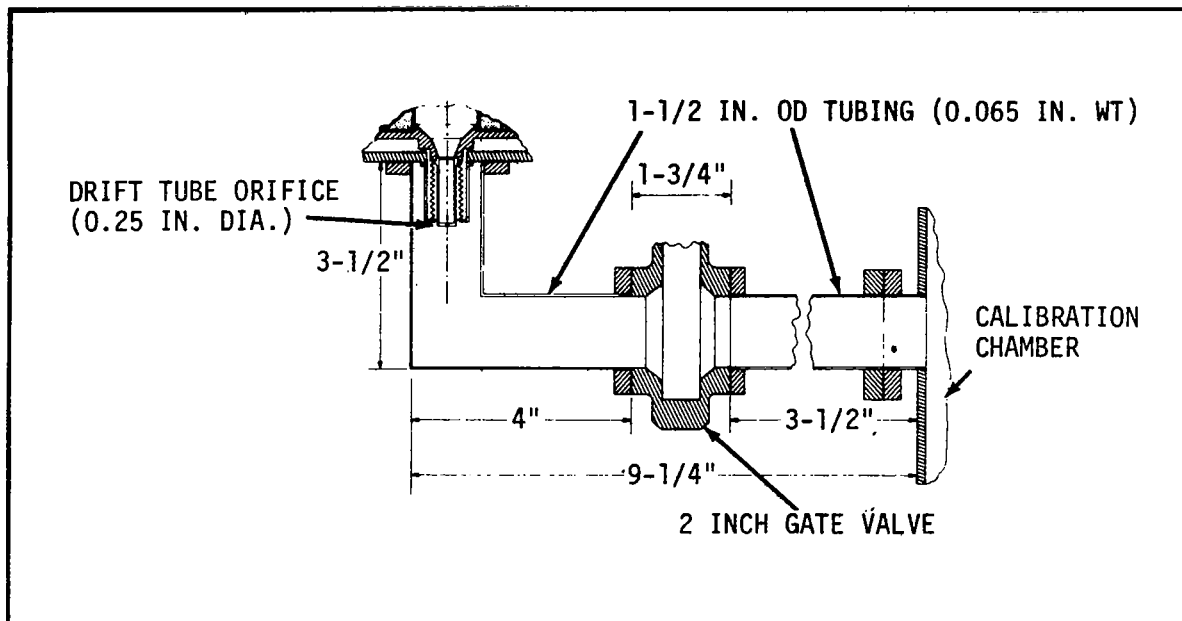


Figure 2-2. GEOMETRY OF MOLECULAR PATH BETWEEN CALIBRATION CHAMBER AND DENSITOMETER DRIFT TUBE

The output of the Densitometer instrumentation is frequency response, $\Delta f/\Delta t$, for any desired pressure. The results of the calibration and the determination of the crystal sensitivity are both given in Section IV.

Section III

CALIBRATION EQUIPMENT AND PROCEDURE

This section describes briefly the instrumentation and other hardware and supplies used in the calibration of the Cryogenic Densitometer. A very brief calibration procedure is also presented.

3.1 CALIBRATION EQUIPMENT

Figure 3-1 is a schematic layout of the various items of hardware used. The major items are as follows:

- High Vacuum Calibration System (HVCS)
- Auxiliary high vacuum system
- Gaseous helium supply
- Gaseous nitrogen supply
- Liquid nitrogen supply
- Liquid helium supply
- 28-volt dc regulated power supply
- Digital frequency counter capable of displaying 10 MHz
- Temperature measurements from room temperature to near 20°K.

Of the above items, the gaseous helium supply, the gaseous nitrogen supply, the 28-volt dc power supply, and the digital frequency counter are all common laboratory items and will not be discussed in detail here. All of the gases and cryogens used were of commercial grade and were readily available. Only the liquid helium (LHe) had not been used by this laboratory prior to this test program. Thus, a source of LHe had to be established through MSFC supply channels. In addition, a LHe transfer line had to be located and placed in operation. This required some training as none of the Experimental Physics Section personnel had ever used LHe dewars.

The major systems listed above are described in more detail in the following subsections.

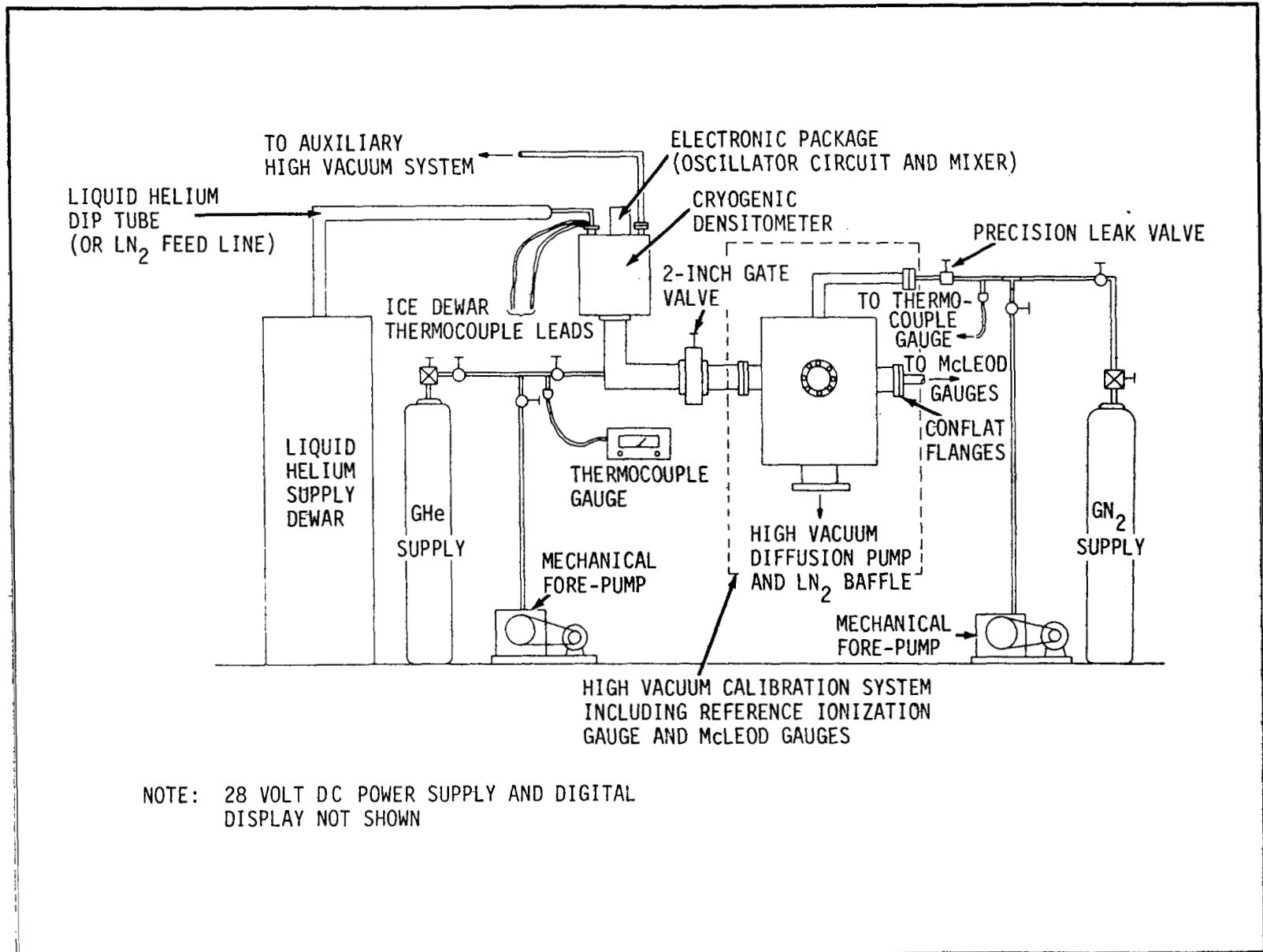


Figure 3-1. LAYOUT OF CALIBRATION TEST APPARATUS

3.1.1 High Vacuum Calibration System

The High Vacuum Calibration System (HVCS) is the key piece of equipment used in the calibration procedure. The system (shown in Figure 2-1) was built by the Geophysics Corporation of America (Mass.) to calibrate vacuum gauges over the range of 100 Torr to approximately 1×10^{-9} Torr. All calibration is made in a cylindrical stainless steel chamber (the calibration chamber) against standard McLeod gauges. Pressures down to approximately 5×10^{-7} Torr may be compared directly against the CVC type GM 110 McLeod gauge for the high vacuum range and against the CVC type GM 100 McLeod gauge for the vacuum range. Pressures below this level are compared against the CVC type GM 110 McLeod gauge through an orifice flow technique. Since the major pressure range of interest to the Densitometer is from approximately 10^{-5} Torr to 5×10^{-7} Torr, the orifice flow technique was not used.

The Densitometer was mounted onto the HVCS by means of the 1.5 inch stainless steel elbow shown in Figures 2-2 and 3-1. The elbow was fitted with rotatable Conflat flanges on both ends. Standard rotatable Conflat fittings were used except that the bolt circle of the flange which attaches to the Densitometer is 2.5 inches. Also, a 2-inch, high vacuum gate valve was fitted into the horizontal leg of the elbow. The purpose of this important valve is threefold: (1) it provides an easy means for the Densitometer to be backfilled with GHe without interfering with the vacuum in the HVCS, (2) it allows the Densitometer to be removed from the HVCS without the necessity for bringing the system to atmosphere, and (3) it provides an easy means for isolating the Densitometer from the HVCS during transition from one test pressure level to another.

3.1.2 Auxiliary High Vacuum System

An auxiliary high vacuum system had to be used to maintain the pressure in the vacuum insulation space below about 1×10^{-4} Torr. Ideally, the pressures in the vacuum insulation space should be held well below 1 μ Hg to prevent excessive cryopumping during cooldown with LHe. If this were not possible, the release of some of the LHe cryopumped gases might cause the vacuum insulation space pressure to rise unnecessarily high. This higher than desired pressure would contribute to increased heat transfer between the radiation guard reservoir and the warm outer wall of the instrument, thus limiting the hold time.

The auxiliary high vacuum system used in this calibration was a VEECO unit capable of maintaining a pressure of about 1×10^{-6} Torr without the use of an LN_2 cold trap. A 0.75 inch stainless steel tube was used to connect the vacuum insulation space to the VEECO system (Figure 3-1). The pressure in the Densitometer vacuum insulation space was monitored by an ionization gauge mounted on the bell jar of the VEECO system. Thus, the pressure in the vacuum insulation space had to be inferred from the VEECO bell jar pressure.

3.1.3 Temperature Measurements

The original designers of the Cryogenic Densitometer had hoped to monitor the temperature of both the ice dewar (the radiation guard reservoir) and the lead block (the crystal's heat sink). Unfortunately, it became impossible to measure the lead block temperature directly because of the excessive heat short created by the addition of any thermocouple wires.

Accurate measurement of the ice dewar temperature was easily accomplished by inserting copper-constantan thermocouples into the dewar's unfrozen water. Two such thermocouples were used, one being placed near the bottom of the dewar and the other being placed near the center region of the dewar (from top to bottom). These two thermocouples were used in an effort to cryogenically cool the dewar uniformly, as experience had shown that serious and damaging stresses resulted in the dewar walls from non-uniform cooling. Temperatures throughout the range from room temperature to approximately 25°K were readily monitored using a potentiometer. The emf readings were converted to temperature by use of a set of thermocouple tables published by the National Bureau of Standards (ref. 4). Temperatures below 25°K were monitored only as a reference, since accurate, quantitative temperature measurements below 25°K are not possible with simple, uncalibrated thermocouples. However, even though direct measurements of the lead block heat sink temperature were not possible, no drawbacks were observed. It was found that the ice dewar's thermocouple measurements were relatively consistent in indicating the lowest temperatures reached by the dewar. In addition, it was observed that the crystal itself was an excellent indicator of temperature. This will be explained and discussed in more detail in the next section.

3.2 CALIBRATION PROCEDURE

A detailed outline of the actual calibration procedure (with pertinent comments) is given in Appendix A. A very brief description of the calibration is given in this section.

The Densitometer is mounted on the High Vacuum Calibration System (HVCS) as shown in Figures 2-2, 3-1, and 3-2. This places the entrance orifice down and the electronic package up. Both the vacuum insulation space and the sensor chamber (through the orifice and drift tube) are highly evacuated. It is suggested that the sensor chamber, in particular, be evacuated under a hard vacuum for a period of three or more days.

At any time during the above vacuum conditioning, the ice dewar (radiation guard reservoir) may be filled with distilled water (approximately 1640 ml). It is important that no more than 1640 ml of water be used or problems may be experienced with stresses caused by the ice as it freezes and expands.

Cryogenic cooldown of the ice dewar can begin at the conclusion of the vacuum conditioning. At this time the regulated 28-volt dc power supply and frequency counter may be connected. Cooling begins, naturally, with the liquid nitrogen (LN_2) supply and proceeds slowly until the ice is frozen and until the LN_2 temperature ($\sim 78^\circ\text{K}$) is attained. From this point, the cooldown proceeds with liquid helium (LHe) and is continued until the ice dewar and the lead heat sink have been thoroughly chilled at the lowest temperature attainable ($\sim 10^\circ\text{K}$). During the cryogenic conditioning, the sensor chamber is backfilled with gaseous helium to a pressure of approximately 200 μHg (as indicated by a conventional thermocouple vacuum gauge). The backfilling gas serves as the major mode of heat transfer to chill the lead block heat sink. Heat removed from the lead block is transferred directly to the ice dewar and continues until the unit is thoroughly chilled. Chilling continues until the lead block heat sink and the sensor crystal are in thermal equilibrium with the ice dewar.

Upon completion of the cryogenic conditioning, the backfill gas (GHe) is pumped out rapidly and immediately afterwards the flow of LHe to the ice dewar is ceased. At this time the crystal frequency should be very stable and the

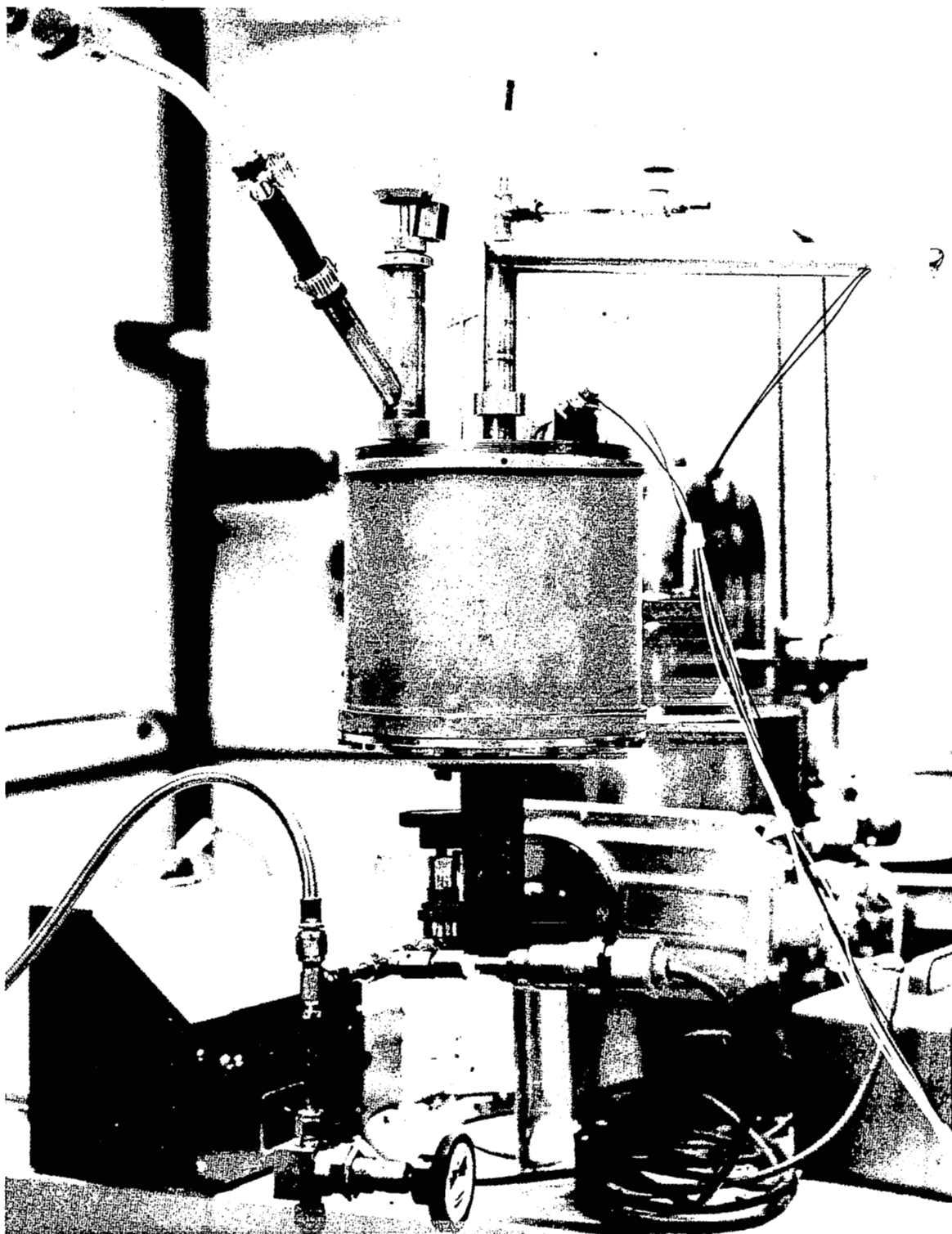


Figure 3-2. MOUNTING OF DENSITOMETER ON HIGH VACUUM CALIBRATION SYSTEM

vacuum in the HVCS should have recovered to a pressure as low as 1×10^{-7} Torr or lower. The temperature of the ice dewar is, of course, still close to the minimum temperature attained by the cooldown. Thus, the ice dewar has to be slowly warmed until its temperature is approximately 60-70°K. If its temperature is maintained any lower than this, it is possible that some of the calibration gas may condense on the walls of the sensor chamber. If the ice dewar temperature is allowed to increase to a value significantly above 80°K, unwanted warming of the crystal and its lead block heat sink would result.

Calibration can begin immediately after the above steps, but must be completed in three to four hours. This is simply because the Densitometer is designed to maintain its heat sink temperatures for no longer than four hours at best. Thus, both the conditions of calibration and launch must be completed within these constraints.

Calibration is the simplest part of the total process and is far simpler than the cryogenic conditioning. The Densitometer is isolated from the HVCS by the 2-inch gate valve while the HVCS is being brought to some constant calibration pressure. The calibration gas used is nitrogen (commercial grade) although air may be used with little error. The pressure in the HVCS may be satisfactorily monitored by an ionization gauge during this portion of the process. Ideally, the initial calibration pressures should start with lower values first, and then work up to the higher pressures over the range of 5×10^{-7} Torr to 1×10^{-3} Torr (1 μ Hg). After the HVCS is brought to some value, the pressure is allowed to stabilize for two or three minutes. After the pressure is stabilized, the 2-inch gate valve is opened and the sensor chamber and crystal of the Densitometer are exposed to the HVSC calibration chamber. The HVCS McLeod gauge has been exposed to the calibration chamber throughout this process and maintains equilibrium with the calibration gas.

No data is taken immediately after the Densitometer and the HVCS calibration chamber are communicated since some time is required for the sensor chamber to reach equilibrium. There is, of course, a small mass flow between the two chambers since the sensor crystal of the Densitometer is "cryopumping" (condensing or freezing) most of the molecules of the calibration gas that

arrive at its surface. This mass flow is, however, negligible when compared to the mass flow into the HVCS diffusion pumping system. Thus, the Densitometer and the McLeod gauge are essentially exposed to the same static pressure.

Two or three minutes after the Densitometer is exposed to the calibration chamber, data may be recorded. Data were taken visually from the frequency counter every 10 seconds for at least a 2-minute run. At least three runs were taken at every calibration pressure. Also, at least two McLeod gauge readings were taken during the three runs, generally near the beginning and near the end of the runs. If the two McLeod readings agreed closely, then the pressure probably did remain relatively stable throughout a run. If, however, the two McLeod readings did not closely agree, then the pressure either drifted during the test runs or the McLeod readings were faulty. A new set of test runs were made whenever there was doubt about the validity of the measured pressure.

After a satisfactory set of test runs were made at any particular pressure level, the 2-inch gate valve would again be closed and a new pressure level would be established. The 2-inch gate valve was closed to minimize the amount of mass condensed on the crystal at times other than during an actual calibration. This process was repeated for as many calibrations as possible within the constraints of the "hold-time" of the instrument. Also, since the crystal loaded rapidly with mass at the higher pressures (above 1×10^{-4} Torr), the amount of calibration time was severely limited. Calibration was generally restricted to frequency shifts from about 77 kHz to 5 kHz. This is because experience has shown that the response of the crystal becomes somewhat non-linear for frequency shifts greater than one percent of the resonant frequency. However, a limited number of calibrations performed on the Densitometer after the instrument passed through the zero beat frequency showed no significant nonlinearities for frequency shifts as high as two percent of resonant frequency.

Section IV

RESULTS

This section presents the data produced by the Cryogenic Densitometer during its calibration. As discussed in Section II, the purpose of the calibration was to determine the sensitivity, H , in Hz/gm of the Densitometer's sensor crystal. The value will be a critical part of the flight data reduction.

4.1 STATIC CALIBRATION DATA

The raw calibration data were recorded manually, generally at 5 or 10 second intervals, over total run times of 1 to 2 minutes. A typical sample of raw data recorded in this manner is given in Table 4-1. Along with the frequency response data, the time, ice dewar temperature and average McLeod gauge pressures were recorded. The data were plotted on rectangular coordinate paper with the frequency, Δf , plotted versus time, Δt . A sample of this is shown in Figure 4-1 for the data of Table 4-1. As discussed in Section III, if the pressure in the HVCS calibration chamber remains relatively constant during a given run, then the plot of the data should produce a straight line. This is the case of the data for Run 1B given in Figure 4-1. However, Run 1A in Figure 4-1 shows a definite nonlinearity indicative of a drifting calibration chamber pressure. This was verified by additional McLeod gauge checks which showed that the pressure in the calibration chamber was relatively constant by the time Run 1B was started.

An additional consideration must be given to this frequency response data. It was observed that there was a small but measurable drift in the beat frequency even with the 2-inch gate valve closed. This shift in the beat frequency is assumed to be a result of the minor gas loads (outgassing and virtual leaks) from within the sensor chamber itself. If this is the case, then it is reasonable to assume that these loads would become negligible after a sufficiently long hold time. Thus, in each calibration run made, the drift beat frequency is accounted for in the calibration data. The drift beat frequency measured preceding Runs 1A and 1B is shown in Figure 4-1. The slope of this

Table 4-1. SAMPLE OF CALIBRATION TEST DATA (TEST 10-2-69, RUNS 1A & 1B)

TIME (sec)	DRIFT TEST ① BEAT FREQUENCY Δf (Hz)	CALIBRATION TEST BEAT FREQUENCY, Δf (Hz)		AVERAGE CALIBRATION PRESSURE, P_c ③ (Torr)
		RUN 1A ②	RUN 1B	
0	69.651	69.457	69.346	2.0×10^{-6}
5	69.648	69.445	69.340	2.0×10^{-6}
10	69.645	69.437	69.335	2.0×10^{-6}
15	69.643	69.429	69.330	2.0×10^{-6}
20	69.642	69.423	69.324	2.0×10^{-6}
25	69.642	69.415	69.319	2.0×10^{-6}
30	69.642	69.409	69.313	2.0×10^{-6}
35	69.641	69.403	69.308	2.0×10^{-6}
40	69.639	69.397	69.303	2.0×10^{-6}
45	69.637	69.392	69.297	2.0×10^{-6}
50	69.635	69.387	69.290	2.0×10^{-6}
55	69.635	① Drift test was run with the 2-inch gate valve closed to observe background response. ② Calibration pressure was observed to drift during run 1A. Thus, run 1A was discarded as a calibration point. ③ P_c , the calibration pressure, was monitored in the calibration chamber by means of the high vacuum McLeod gauge. NOTE: The average dewar temperature was 58°K.		
60	69.632			
65	69.630			
70	69.629			
75	69.629			
80	69.626			

NOTES: FOR RUN 1B,

$$\Delta f/\Delta t = 1.10 \text{ Hz/sec} - \text{DRIFT}$$

$$= 1.10 \text{ Hz/sec} - 0.28 \text{ Hz/sec}$$

$$\Delta f/\Delta t = 0.82 \text{ Hz/sec}$$

$$P_c = 2.0 \times 10^{-6} \text{ torr}$$

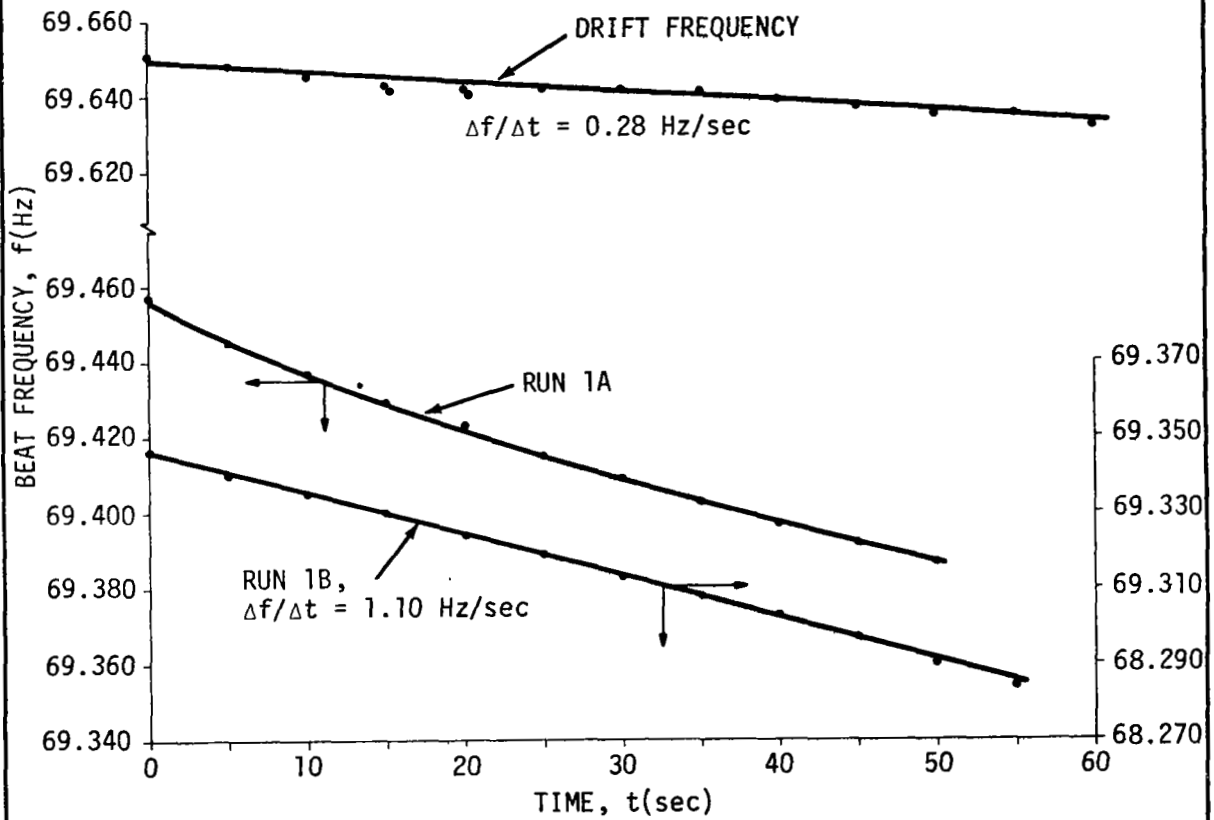


Figure 4-1. PLOT OF CALIBRATION DATA FOR TEST 1 (RUNS 1A & 1B)

drift frequency line is subtracted from the slope of the Run 1B slope, resulting in the response of the Densitometer, $\Delta f/\Delta t$ (in Hz/sec), for the calibration pressure.

Generally, two or three such runs were made for each calibration pressure. Approximately 19 good runs were made as given in Table 4-2 and plotted in the very important format of Figure 4-2. The data of Figure 4-2 defines the pressure response of the instrument. In purely free-molecular-flow, the rate of mass flow through an opening is directly proportional to the upstream pressure, i.e.,

$$\dot{m} \propto P \quad (4-1)$$

for constant temperature. From equation (1-1), the frequency response of a quartz crystal oscillating in its thickness shear mode is

$$\Delta f/\Delta t \propto \dot{m} f_r^2 \quad (4-2)$$

Therefore, it follows that

$$\Delta f/\Delta t \propto P \quad (4-3)$$

or

$$\Delta f/\Delta t = C P \quad (4-4)$$

assuming all other quantities (temperature, geometry, etc.) are held constant.

It was gratifying to find that the calibration data did fall very close to a straight line. The equation of this straight line was determined by fitting the best line through the data that also satisfies equation (4-3). When this was done, it was found that the value of C in equation (4-4) is 3.65×10^5 (within the limits of experimental testing) for an ambient temperature of about 300°K. That is,

$$\Delta f/\Delta t \approx 3.65 \times 10^5 P \quad (4-5)$$

where P is measured in Torr and $\Delta f/\Delta t$ is in Hz/sec.

Equation (4-5) constitutes the complete static calibration of the Densitometer at room temperature. This information may now be used to determine

Table 4-2. CALIBRATION RESULTS FOR ALL TESTS

TEST AND RUN NO.	AVERAGE DEWAR TEMP. (°K)	AVERAGE CALIBRATION PRESSURE, P _c (TORR)	INSTRUMENT RESPONSE Δf/Δt (Hz/sec)
1. RUN 1B	58	2.0 × 10 ⁻⁶	0.82
RUN 2B	61	2.0 × 10 ⁻⁵	7.81
RUN 2C	61	2.0 × 10 ⁻⁵	7.55
RUN 3A	64	3.5 × 10 ⁻⁵	12.30
RUN 3C	64	3.5 × 10 ⁻⁵	12.68
2. RUN 1A	55	2.4 × 10 ⁻⁴	78.8
RUN 1B	55	2.4 × 10 ⁻⁴	77.8
RUN 1C	56	2.4 × 10 ⁻⁴	81.3
3. RUN 1C	58	1.0 × 10 ⁻⁵	4.03
RUN 2B	59	6.1 × 10 ⁻⁵	22.0
RUN 2C	59	6.1 × 10 ⁻⁵	20.9
RUN 2D	59	6.1 × 10 ⁻⁵	21.2
RUN 3A	61	6.2 × 10 ⁻⁴	211.0
RUN 3B	61	6.2 × 10 ⁻⁴	213.0
4. RUN 1A	61	1.65 × 10 ⁻⁴	55.0
RUN 1B	61	1.65 × 10 ⁻⁴	57.5
RUN 2	61	7.0 × 10 ⁻⁴	214.0
5. RUN 1A	60	5.95 × 10 ⁻⁴	214.8
RUN 1B	60	5.95 × 10 ⁻⁴	209.8

- (1) The drift beat frequency has been accounted for in all of the listed responses.
- (2) Each bracketed group of data was taken at a single pressure. The instrument responses were averaged and plotted in Figure 4-2.

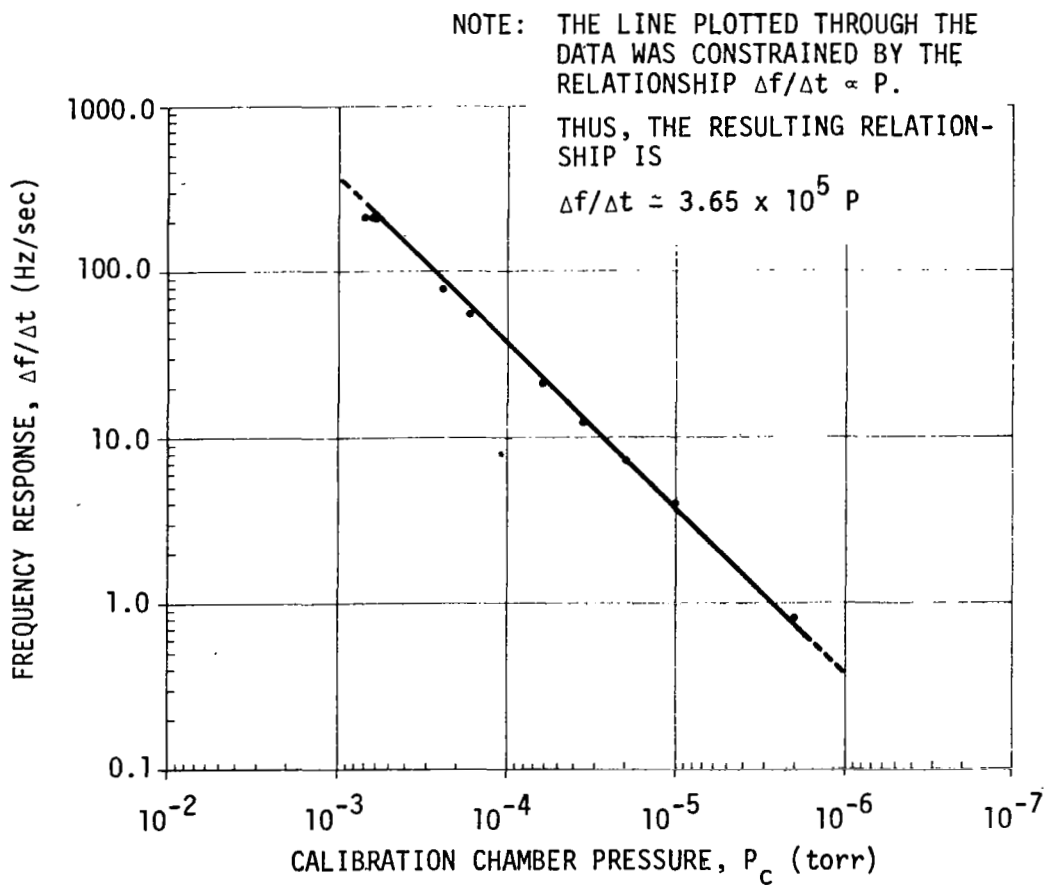


Figure 4-2. CRYOGENIC DENSITOMETER RESPONSE VERSUS CALIBRATION PRESSURE

the sensitivity of the instrument if the proper considerations are given to its geometry. Absolute accuracy of the instrument was determined to be approximately ± 5 percent.

4.2 DETERMINATION OF DENSITOMETER SENSITIVITY, H

The ambient mass reaching the instrument sensor chamber must be related to that which is accommodated (captured) by the instrument and that which is lost by migration back out of the instrument.

Following the development of reference 2 (p. 7),

$$\dot{m}_e = \dot{m}_{coll} + \dot{m}_{ec} + \dot{m}_{ea} \quad (4-6)$$

where: \dot{m}_e = total molecular mass entering the sensor chamber per unit time, gm/sec
 \dot{m}_{coll} = total mass collected on the sensor crystal per unit time, gm/sec
 \dot{m}_{ec} = total mass lost back out of the orifice during the diffuse condensation process, per unit time, gm/sec
 \dot{m}_{ea} = total mass lost back out of the orifice per unit time due to one or more collisions of a molecule being necessary before the molecular energy is below the critical value for condensation.

Equation (4-6) may be rewritten as

$$\dot{m}_e = \dot{m}_{coll} \left[1 + \frac{\dot{m}_{ec}}{\dot{m}_{coll}} + \frac{\dot{m}_{ea}}{\dot{m}_{coll}} \right] \quad (4-7)$$

where the nondimensionalized terms may be formulated as follows:

$$\frac{\dot{m}_{ec}}{\dot{m}_{coll}} = \frac{A_d K_{diff}}{A_c \sigma} \quad (4-8)$$

and

$$\frac{\dot{m}_{ea}}{\dot{m}_{coll}} = \left(\frac{v A_c \sigma}{A_w} \right) \left(\frac{A_d K_{diff}}{A_c \sigma} \right) = \frac{v A_d K_{diff}}{A_w} \quad (4-9)$$

where: A_d = drift tube cross sectional area, 0.733 cm^2
 K_{diff} = transmission probability for molecules leaving collector diffusely, 0.1701
 A_c = effective crystal area, 3.95 cm^2
 σ = sticking coefficient of molecules on crystal, 0.81
 ν = number of collisions required to reduce molecular energy below critical value for condensation
 A_w = collector wall area, $\sim 97.1 \text{ cm}^2$

The value for K_{diff} was approximated from the dimensions in Figure 4-3 and simple transmission probability theory (ref. 6).

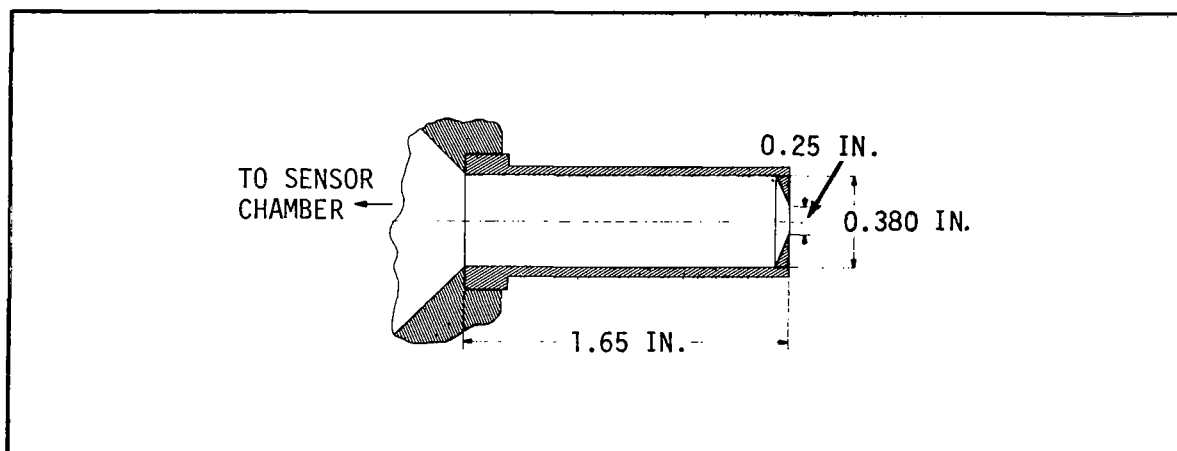


Figure 4-3. DRIFT TUBE DIMENSIONS

As explained in subsection 2.1, the sticking coefficient, σ , was determined experimentally by CELESCO (ref. 2) as ≈ 0.81 . Thus, the numerical value of equation (4-8) is

$$\frac{\dot{m}_{ec}}{\dot{m}_{coll}} \approx 0.0389 \quad (4-10)$$

Since the velocity of the rocket launched instrument package is not high (i.e., it is not hyperthermal), it is expected that the energy of the incoming molecules will be low enough that no more than one collision with the cool inner wall of the sensor chamber will be required before being condensed on the crystal. Thus, the value of ν in equation (4-9) may be taken as zero, or

no more than unity at the most. Assuming v to be unity as a very conservative estimate, the numerical value of equation (4-9) becomes

$$\frac{\dot{m}_{ea}}{\dot{m}_{coll}} \approx 0.00128 \quad (4-11)$$

or a loss contribution of only about 0.128 percent. Thus, equation (4-7) becomes

$$\dot{m}_e = \dot{m}_{coll} 1.04018 \quad (4-12)$$

Now, the response of an oscillating quartz crystal is directly proportional to the mass deposited on its surface and to the square of its resonant frequency. Since, for a given crystal, its resonant frequency and effective surface area are constant, the following expression may be written

$$\Delta f / \Delta t = H \dot{m}_{coll} \quad (4-13)$$

where H is the crystal constant (or sensitivity) in Hz/gm. In order to evaluate H , a relationship must be written between the instrument's frequency response and the kinetic theory equation for molecules entering the sensor chamber. This relationship is derived in Appendix B as

$$\Delta f / \Delta t = \frac{H \rho U A_o}{(1.04018) 2 \sqrt{\pi}} f (S_o \cos \alpha) K (S_o, \alpha) \quad (4-14)$$

If the values of ρ and U are substituted from kinetic theory,

$$\rho = \frac{P m}{k T}$$

and

$$U = \left(\frac{2 k T}{m} \right)^{1/2},$$

then equation (4-14) becomes

$$\Delta f / \Delta t = \frac{H}{1.04018} \left(\frac{m}{2 k \pi} \right)^{1/2} \frac{P}{\sqrt{T}} A_o f (S_o \cos \alpha) K (S_o, \alpha) \quad (4-15)$$

From Figure 4-4, $K(S_o, \alpha)$ is about 0.393 for S_o and $\alpha = 0$. Also, assuming the molecular species under consideration to be nitrogen and the orifice area, A_o , to be 0.317 cm^2 , equation (4-15) becomes

$$\Delta f / \Delta t = \frac{H}{1.04018} (0.038445) \frac{P}{\sqrt{T}} \quad (4-16)$$

where the proper conversion factor has been introduced for the pressure in Torr and temperature in °K.

By combining equations (4-5) and (4-16), obtain

$$3.635 \times 10^5 P = \frac{H}{1.04018} (0.038445) \frac{P}{\sqrt{T}} \quad (4-17)$$

or

$$H = 1.703 \times 10^8 \text{ Hz/gm} \quad (4-18)$$

for $T = 300^\circ\text{K}$. Thus, the sensitivity of the Densitometer, H , is 1.703×10^8 Hz/gm. The relationship between the Densitometer's frequency response and the flux of molecules entering its drift tube orifice is

$$\Delta f / \Delta t = \frac{1.703 \times 10^8}{1.0402} \frac{\rho U A_o}{2 \sqrt{\pi}} f(S_o \cos \alpha) K(S_o, \alpha) \quad (4-19)$$

The species subscripts (a) have been dropped in equation (4-19) where it is understood that ρ is the total ambient density and U and S_o ($\equiv V/U$) are weighted for the expected species concentrations.

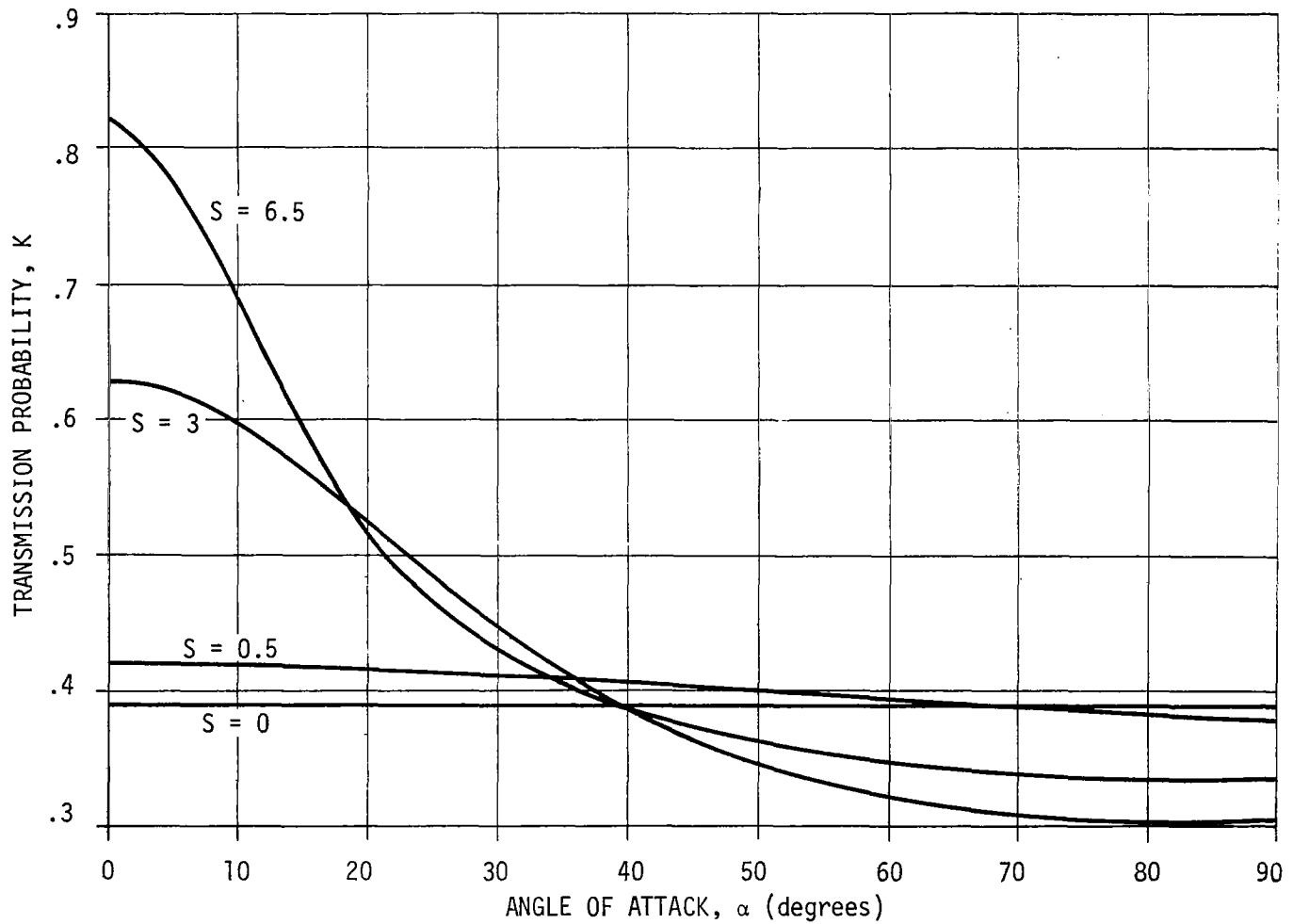


Figure 4-4. TRANSMISSION PROBABILITY FACTOR VERSUS ANGLE OF ATTACK FOR VARIOUS SPEED RATIOS

Section V

COMMENTS ON FLIGHT MEASUREMENTS

The Cryogenic Densitometer is designed to operate in the altitude range of 140 to 180 km. Under the present plans, the instrument is scheduled to be launched from Wallops Island, Virginia, aboard a Nike-Tomahawk launch vehicle.

The sensor chamber of the instrument will be exposed to the atmosphere at about 140 km by means of a spring-loaded cap and the instrument package will be tumbled at a rate close to 1 cps. The only data that will be telemetered from the Cryogenic Densitometer experiment will be the beat frequency (the sensor crystal response) and a thermistor temperature of the middle deck on the electronic package. The beat frequency is expected to be at a level of about 65 to 75 kHz when opened to the atmosphere and should shift to approximately 40 kHz by the time the instrument package reaches 180 km. The instrument is also expected to respond to the atmosphere on the downleg of the flight and should have shifted to approximately 5 to 10 kHz by the time it returns to the 140 km altitude level. These estimates of the anticipated total beat frequency shift were made by a simple computer analysis which used a standard atmosphere as the input (ref. 6).

The rate of change of the frequency shift will be constantly varying due to the passage of the instrument through the atmosphere and due to the intentional tumbling motion. Thus, a representative portion of the flight data may be expected to appear as shown in Figure 5-1.

The reduction of the flight data will be performed initially as follows:

- Step 1. The slope of the frequency response data, $\Delta f/\Delta t$, will be obtained from the plots similar to those of Figure 5-1 for various values of flight time, t . Independent measurements will have been made of the instrument package's attitude and trajectory from which the velocity V , altitude h , and angle-of-attack may be derived. A further independent measurement is expected to allow determination of the ambient temperature T_a of the neutral constituents of the atmosphere.
- Step 2. Sufficient information is now available to permit calculation of the ambient density, ρ , from equation (4-19)

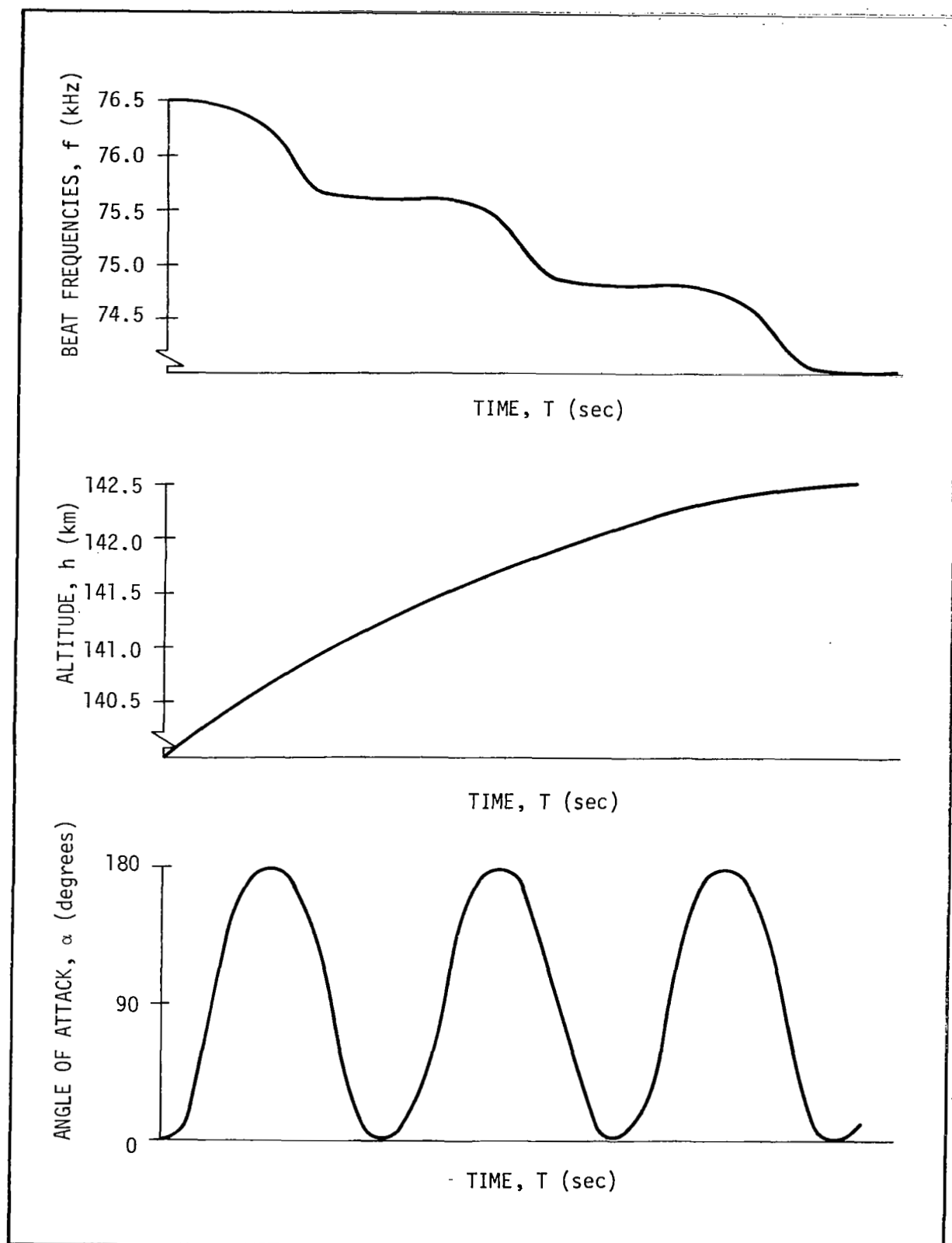


Figure 5-1. REPRESENTATION OF ANTICIPATED DATA FROM FLIGHT OF CRYOGENIC DENSITOMETER

$$\Delta f / \Delta t = \frac{1.703 \times 10^8 \rho U A_o}{2 (1.0402) \sqrt{\pi}} f(S_o \cos \alpha) K(S_o, \alpha) \quad (4-19)$$

All of the quantities in equation (4-19) have been identified before. The constants and flight data may be listed as follows:

Flight Data: h = altitude, km

T = ambient temperature of local atmosphere, °K

V = velocity of instrument package with respect to the ambient environment, cm/sec

α = angle-of-attack between the normal to the entrance orifice and the velocity vector.

Constants: A_o = Entrance orifice area, 0.317 cm²

$\dot{m}_e / \dot{m}_{coll} = 1.0402$, (equation 4-12)

H = Crystal sensitivity (equation 4-18), 1.703 x 10⁸ Hz/gm

m = Mean mass of ambient molecule, gm

k = Boltzmann's constant, 1.3803 x 10⁻¹⁶ erg/deg

The value of U is calculated from

$$U = (2 kT/m)^{1/2}$$

and S_o is defined as

$$S_o \equiv \frac{V}{U}$$

Now, the conventional method of writing the product $f(S_o \cos \alpha) K(S_o, \alpha)$ in equation (4-19) may be replaced by a numerically calculated value, $f'(S_o, \alpha)_{correct}$. This value is determined from $\eta(S_o, \alpha) f(S_o)$ where $\eta(S_o, \alpha)$ is a correction to simple transmission coefficient theory obtained from curves similar to Figure 4-4 and $f(S_o)$ is simply

$$f(S_o) = e^{-S_o^2} + \sqrt{\pi} S_o (1 + \text{erf } S_o)$$

Thus, equation (4-19) becomes

$$\Delta f / \Delta t = \frac{1.703 \times 10^8 \rho U A_o}{2 (1.0402) \sqrt{\pi}} f' (S_o, \alpha) \text{ correct.} \quad (5-1)$$

Equation (5-1) may now be solved by substituting instantaneous values of $\Delta f / \Delta t$ as a function of time in flight and plotting the results versus altitude.

Section VI

CONCLUSIONS AND RECOMMENDATIONS

The laboratory calibration of the Cryogenic Densitometer was completed successfully after a series of mechanical and electronic problems were solved. The following points summarize the results of the calibration.

- The Cryogenic Densitometer is quite accurate and repeatable over the static pressure range of approximately 1×10^{-6} Torr to 1×10^{-3} Torr.
- The sensitivity of the unit for dry nitrogen gas was determined experimentally to be 1.703×10^8 Hz/gm.
- The experimentally determined relationship between the unit's frequency response and calibration pressure was found to be approximately $\Delta f/\Delta t = 3.65 \times 10^5$ P.
- The beat frequency of the unit was extremely stable during calibration.
- A "dead-band" exists in the frequency response from approximately +5 kHz to -5 kHz. This is inherent in the unit's electronics and prohibits measurements from being taken in this frequency range.
- Absolute accuracy of the unit was determined to be approximately +5 percent.

The series of mechanical failures experienced during calibration casts serious doubt on the ability of this prototype cryogenic Densitometer to perform reliably as a thermospheric probe. It is highly recommended that the unit be redesigned to increase its mechanical integrity and to permit more accessible entry for adjustments and repairs. The following specific recommendations should be considered:

- Eliminate the efficient but troublesome water heat sink in favor of a metallic heat sink for the sensor chamber walls.
- Eliminate all soft solder joints in favor of heliarc welded joints in any region experiencing extremely low cryogenic temperatures.
- Provide an optimum number of high vacuum flanges to permit easier access to the interior of the unit.
- Redesign the sensor crystal mount to isolate it from the extremely high shock loadings induced by the sensor crystal heat sink during launch.
- Provide a reliable vacuum valve to the vacuum insulation space.
- Investigate the use of materials other than nylon (e.g., rayon) for use as the sensor crystal heat sink support cords.

Section VII

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Appendix A

DETAILED CALIBRATION PROCEDURE

This appendix was written to outline the total calibration procedure of the Cryogenic Densitometer including preliminary vacuum and cryogenic conditioning. The procedure is given step-by-step to enable one to avoid many pitfalls which were learned through experience.

1. VACUUM CONDITIONING

- 1.1 The Densitometer and its auxiliary equipment should be setup as shown in Figure 3-1, except that the liquid-helium dewar will not be connected initially.
- 1.2 Rough pump the sensor chamber and the vacuum insulation space with the mechanical vacuum pumps of the High Vacuum Calibration System (HVCS) and the Auxiliary High Vacuum System (AHVS) respectively. During this time, the 2-inch gate valve between the Densitometer and the HVCS calibration chamber is open.
- 1.3 If no leaks are apparent, pump both the sensor chamber and the vacuum insulation space with their respective diffusion pumps. Ideally, the vacuum in the vacuum insulation space should be $\leq 1 \times 10^{-4}$ Torr as read by an ionization gauge located as close to the Densitometer as reasonable. The vacuum in the sensor chamber and the HVCS calibration chamber should be a hard vacuum, preferably into the 10^{-8} Torr range.
- 1.4 Continue the vacuum cleanup for approximately 72 hours prior to initiating the cryogenic conditioning. Inspect the system periodically for minute vacuum leaks.
- 1.5 During the vacuum conditioning process, a power supply and digital frequency counter may be connected to the Densitometer's electronic package. The connections are shown on the electronic circuit diagram (Figure A-1). The power supply should be a well-regulated 28-volt dc supply capable of furnishing up to 1.5 amps continuously. The frequency counter should be capable of displaying up to 99.999 kHz.

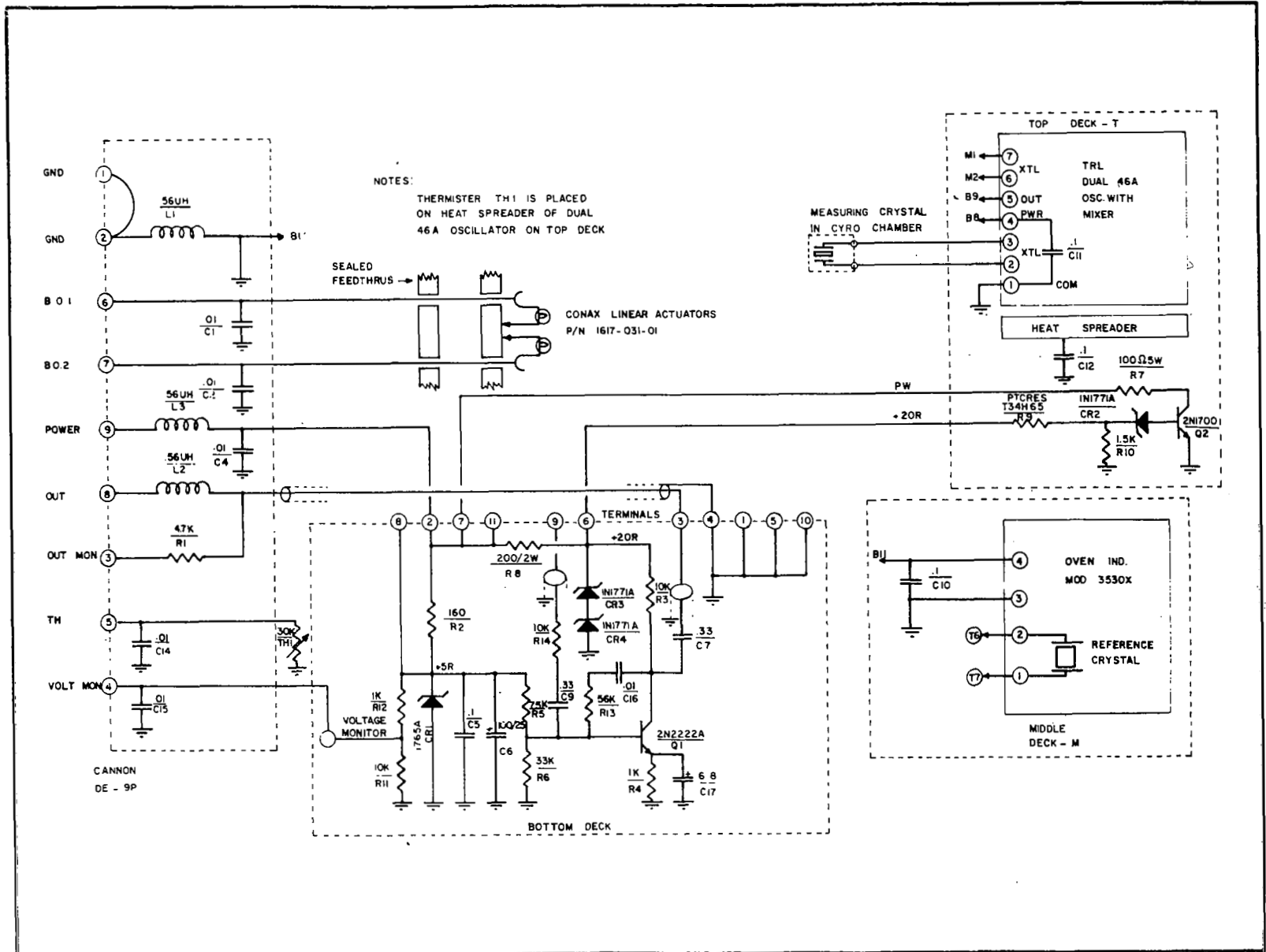


Figure A-1. REVISED ELECTRONIC CIRCUIT DIAGRAM FOR CRYOGENIC DENSITOMETER

2. CRYOGENIC COOLING (LN₂)

The following steps relate the cryogenic conditioning of the ice dewar with liquid nitrogen (LN₂). The LN₂ is used initially to conserve on the cost of liquid helium (LHe).

2.1 Fill the guard reservoir with 1640 ml of distilled water. It is important to avoid overfilling of the ice dewar because of the mechanical stresses resulting from the expansion of the frozen water. The distilled water may be easily poured into the small opening in the top of the ice dewar by using a 100 ml graduated flask.

2.2 Insert two copper/constantan thermocouples, T₁ and T₂, into the ice dewar, one near the bottom of the dewar and the other near the mid-section of the dewar. An LN₂ reference junction should be used for the greatest accuracy, but in the calibration reported here an ice bath reference junction was used satisfactorily.

NOTE: A special manifold was built to allow the cryogenic exhaust gases to be directed away from the upper surface of the Densitometer. This manifold (Figures 2-1 and 3-2) passes the liquid cryogen from the supply dewar through the center of the exhaust gas portion of the manifold and into the ice dewar. This manifold was found to be extremely useful in preventing unwanted freezing and resultant leaks in teflon seals on top of the Densitometer.

2.3 The LN₂ dewar may be connected to the Densitometer's cryogenic feed lines by means of an 1/8-inch ID Teflon tube. Typically, an adapter will have to be made for the LN₂ dewar.

2.4 Cooldown may now begin by opening the LN₂ supply valve. The initial freezing of the ice block should be performed with great care to avoid the large mechanical stresses resulting from asymmetrical freezing. Observation of the two thermocouples, T₁ and T₂, will prevent too rapid cooling. After the water in the ice dewar has been carefully frozen, the cooling rate may be increased some. If T₁ and T₂ stay

within about 5°K of each other, then the cooling rate may be assumed satisfactory.

2.5 As the temperature of the ice dewar approaches that of LN₂, the sensor chamber should be back-filled with gaseous helium (GHe) to a pressure of between 100 μHg and 200 μHg as indicated by a thermocouple pressure gauge. This backfilling process can be easily accomplished by a GHe supply such as is shown in Figures 2-1 and 3-1. The system of valves in the GHe supply system allows the system to be adequately purged and evacuated prior to the actual backfilling process. The 2-inch gate valve between the Densitometer and the HVCS calibration chamber is closed. Then the GHe supply bottle is opened to the GHe supply system with the fore pump operating. This allows the pressure in the supply line to be established at about 200 μHg (as indicated by a thermocouple gauge located in the line) prior to admitting the GHe to the Densitometer sensor chamber. The small valve between the supply line and the sensor chamber is then opened and the pressure is adjusted until the supply line again equalizes at between 100 μHg and 200 μHg. The valve to the sensor chamber is again closed isolating the charge of GHe in the sensor chamber.

2.6 Cooling continues until the ice dewar reaches LN₂ temperature (~ 78°K). After this temperature is reached, the LN₂ flow is reduced until the ice dewar temperature just maintains ~ 78°K. This condition should be held for at least a half hour to ensure thorough chilling of the entire ice dewar. The exact amount of LN₂ used in one cooldown cycle has not been precisely measured, but is approximately 20 to 25 liters.

3. CRYOGENIC COOLING (LHe)

Approximately 25 to 30 liters of liquid helium is required for the cool-down from ~ 78°K. A Linde 100-liter dewar was used in the tests reported here. Depending on the actual quantity of LHe initially in the supply dewar, three or four cooldowns can be accomplished with one "full" 100-liter supply dewar. A vacuum insulated transfer line is required to top the supply dewar.

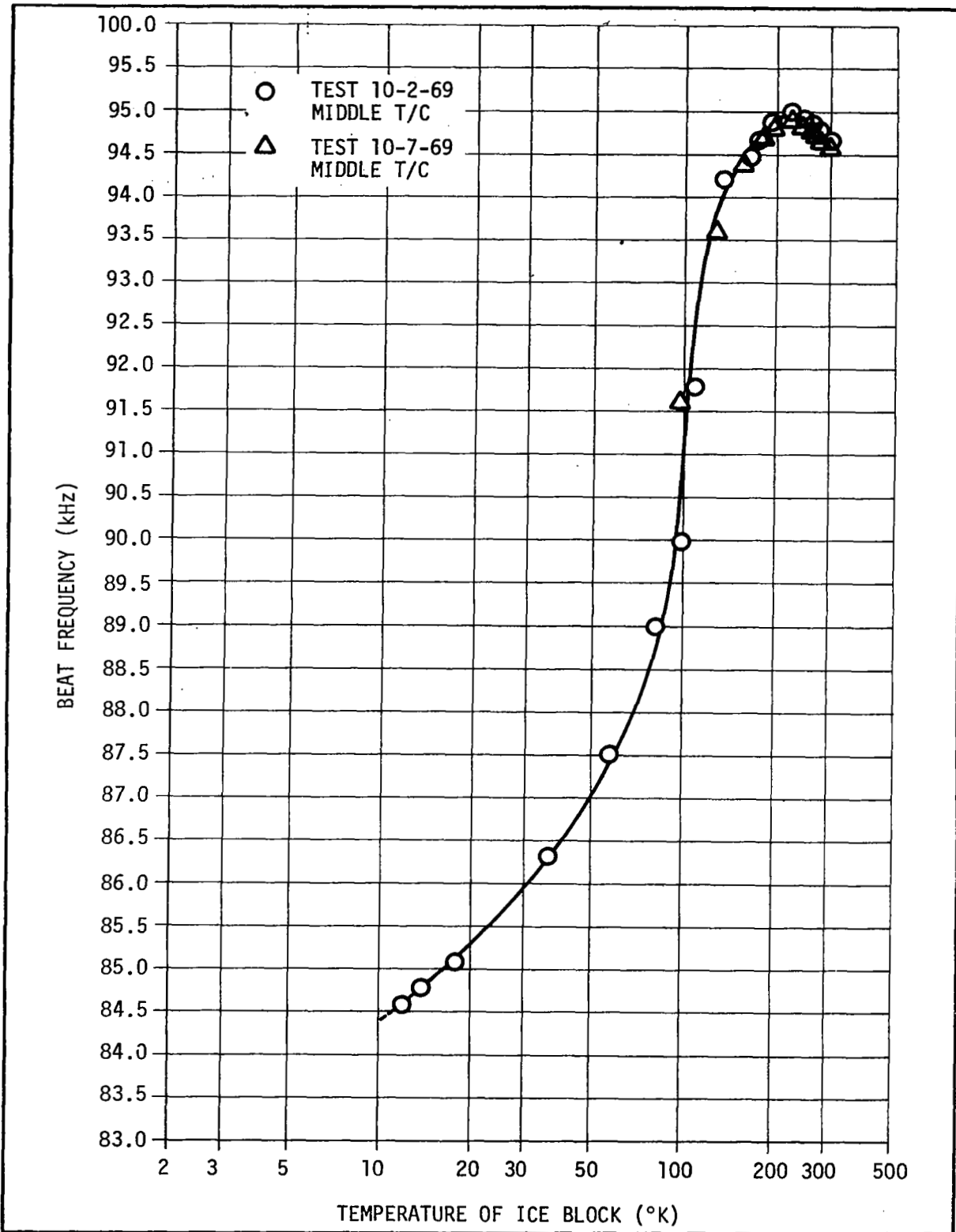


Figure A-2. TYPICAL CHANGE IN SENSOR BEAT FREQUENCY AS A FUNCTION OF TEMPERATURE

Generally, an adapter has to be made for the discharge end of the transfer line to allow an 1/8th inch Teflon coupling to tie the transfer line to the cryogenic feed line of the Densitometer. The manifold described in Step 2.2 has an additional feature that is extremely useful when continuing the cooldown with LHe. There is a tee immediately above the manifold with a small valve exhausting to the atmosphere. Since there may be no valve in the transfer line, the addition of this small valve allows some of the LHe to be initially diverted from the Densitometer, resulting in some control in the flowrate. There is an additional feature of this tee and valve which will be discussed later in this procedure.

- 3.1 Insert the LHe transfer line in the LHe supply dewar and attach the discharge end to the manifold with a tight fitting Teflon coupling. This has to be accomplished with some finesse since, ideally, the cryogenic fluid introduced to the Densitometer cryogenic feed lines should closely match in temperature the ice dewar. In practice, this situation cannot be met exactly. However, by letting the LHe transfer line purge itself partially before attaching its discharge end to the manifold, the temperatures can be more closely met.
- 3.2 The cooling rate of the LHe cooldown may be adjusted to some extent through by-passing a portion of the LHe flow through the manifold tee. The lowest temperature attainable with the LHe should be accomplished in about 45 minutes. Although the copper/constantan thermocouples are not considered accurate below about 25°K, they can give an indication of the lowest temperature attainable in the ice dewar. Experience has shown that this indicated temperature is approximately 9 to 10°K.
- 3.3 The flow of LHe must continue until the sensor crystal and lead block heat sink are thoroughly chilled and are in thermal equilibrium with the ice dewar. This normally requires approximately 45 to 55 minutes after the ice dewar attains its lowest temperature. The pressure of the GHe backfill gas in the sensor chamber should be periodically checked to make certain that is maintained at approximately 100 to 200 μ Hg as discussed in Step 2.5.

- 3.4 As the LHe cooldown progresses, the pressure in the 100-liter supply dewar will slowly drop off resulting in a flow rate that may be too low to maintain the cooldown rate. This can be overcome by pressurizing the LHe dewar with a supply line of GHe. The normal operating pressure of the Linde 100-liter LHe dewar is 18 psia and should not be exceeded. Pressurization of these dewars should be performed only by personnel intimately familiar with such procedures.
- 3.5 In the absence of any direct measurement of the sensor crystal temperature, thermal equilibrium of the sensor crystal heat sink at the lowest attainable temperature of the ice dewar may be determined by the following observations: (1) As the cooldown progresses, the beat frequency will follow a trend such as that shown in Figure A-2. The sensor crystal will show less and less of a temperature dependence below about 25°K. Thus, when the beat frequency stabilizes, the sensor crystal is essentially in thermal equilibrium with the ice dewar. (2) Since the mass of the lead block is many times that of the sensor crystal, the lead block may not be thoroughly chilled even though the sensor crystal has appeared to stabilize. This can be checked easily through rapid removal of the GHe backfill gas from the sensor chamber by opening the 2-inch gate valve to the HVCS calibration chamber. If the beat frequency rises very slowly some 50 to 150 Hz and then stabilizes, the heat sink may be assumed to be adequately chilled. However, if the beat frequency continues to shift significantly over 150 Hz, the cooling is not complete and must be continued. This is accomplished by reclosing the 2-inch gate valve and again slowly backfilling the sensor chamber with GHe. All during this time the flowrate of LHe is continued constant.
- 3.6 After the sensor crystal and the lead block heat sink have been thoroughly chilled according to the criteria in Step 3.5, the GHe backfill gas in the sensor chamber may be rapidly removed by opening the 2-inch gate valve. The LHe flow may be terminated as soon as the sensor chamber is adequately evacuated. No more than about two minutes are required for this pumpdown as the GHe will diffuse out quickly. If

it is known that the LHe dewar is nearly empty, the flow of LHe may be continued until the dewar is empty and the warmer GHe pressurization gas is admitted to the Densitometer cryogenic feed lines. The ice dewar must be slowly warmed until its temperature is approximately 55 to 65°K. If the LHe dewar is not nearly empty, the transfer line must be carefully removed and replaced with a line of GHe. This has to be accomplished carefully or the LHe cooled Densitometer cryogenic feed lines will cryopump room air and moisture and can very easily "freeze-up" completely. This can be easily avoided by attaching the GHe supply line to one side of the manifold tee before removing the LHe transfer line. By initiating a small flow of GHe and then removing the transfer line and capping its connection point on the manifold tee, no room air is permitted into the cryogenic feed lines.

- 3.7 The ionization gauge pressure in the calibration chamber should be observed during the slow warm-up of the ice dewar. Small pressure bursts of residual amounts of N_2 , O_2 , and H_2O will be noticed as warm-up progresses. Obviously, some of these gases will deposit on the sensor crystal and will cause a significant shift in the beat frequency. This is of no concern if the shift does not exceed over 5 to 10 kHz. Warm-up of the ice-dewar should take approximately 30 minutes.

The above sequence of steps completes a typical cooldown process. In general, the amounts of elapsed time involved is approximately as follows:

LN_2 cooldown - 2.5 to 3.0 hours

LHe cooldown - 1.5 hours

Ice Dewar Warmup - 0.5 hours

4. CALIBRATION

Calibration or testing of the instrument must be completed within 3 to 4 hours from completion of the foregoing cooldown process. This assumes that no significant leaks exist in either the sensor chamber or the vacuum insulation space. A brief outline of the calibration procedure is given in subsection 3.2.

- 4.1 The HVCS McLeod gauge (CVC GM110) is assumed to be clean and ready for operation at the conclusion of the cryogenic conditioning process. There is ample time to prepare the McLeod gauge during the cryogenic cooldown of the Densitometer.
- 4.2 The 2-inch gate valve is closed isolating the Densitometer sensor chamber from the HVCS calibration chamber. If there are no leaks in the 2-inch gate valve or in the elbow on which the Densitometer is mounted, then there should be only a very minor drift in the beat frequency (see Figure 4-1). This drift reflects minor leaks and out-gassing of the system up to the 2-inch gate valve.
- 4.3 The drift beat frequency should be monitored and recorded prior to each calibration run while the 2-inch gate valve is closed and the sensor chamber is isolated from the HVCS calibration chamber. Monitoring of the drift beat frequency may be easily accomplished by recording the displayed beat frequency every five or ten seconds. The digital counter used in the present calibration was capable of displaying up to 99.999 kHz allowing sufficiently accurate readings of the drift beat frequency.
- 4.4 While the 2-inch gate valve is closed, commercial grade nitrogen gas (GN_2) is carefully admitted to the HVCS calibration chamber. This can be accomplished very easily by means of an arrangement similar to that shown in Figure 3-1. Control of the GN_2 flowrate is accomplished by means of a precision leak valve. The pressure in the HVCS calibration chamber is monitored by means of an ionization gauge during this process. When the pressure in the HVCS calibration chamber has risen to approximately 5×10^{-7} Torr, the GN_2 is regulated to maintain that pressure. This is assuming that the base pressure of the calibration chamber was well below 1×10^{-7} Torr.
- 4.5 After the HVCS calibration chamber pressure has stabilized, the 2-inch gate valve is opened exposing the Densitometer sensor chamber to the calibration chamber. About two or three minutes are required for the pressure to again stabilize.

- 4.6 The frequency response data may now be recorded for the pressure in the HVCS calibration chamber. The data may be monitored in the same manner as for the drift beat frequency, i.e., by recording the displayed beat frequency every five or ten seconds as desired. In the present tests it was found that recording the beat frequency every ten seconds for a run time of one minute provided sufficiently accurate calibration data. At least two, and preferably three, runs should be made at a given calibration pressure.
- 4.7 The 2-inch gate valve is again closed, isolating the Densitometer sensor chamber. A new HVCS calibration chamber pressure is adjusted (Step 4.4) and the drift beat frequency is again monitored (Step 4.3). Steps 4.5 and 4.6 are then repeated for the new pressure. Ideally, the system should permit calibrations at the following pressures:

- (1) 5×10^{-7} Torr
- (2) 1×10^{-6} Torr
- (3) 5×10^{-6} Torr
- (4) 1×10^{-5} Torr
- (5) 5×10^{-5} Torr
- (6) 1×10^{-4} Torr
- (7) 5×10^{-4} Torr
- (8) 1×10^{-3} Torr

As mentioned earlier, calibration must be completed in 3 to 4 hours immediately following cryogenic conditioning of the unit. As the Densitometer's sensor crystal and heat sink begins to warm up, the beat frequency will no longer respond linearly to pressure. Ultimately the gases condensed on the sensor crystal will begin to evaporate, leaving the crystal and resulting in a shift in the beat frequency opposite to that during calibration.

NOTE: No calibration should be performed when the beat frequency is within ± 5.0 kHz of zero. This is due to an electronic "locking-up" of the oscillator circuit near ± 5 kHz. As the frequency passes on through zero the beat frequency will again appear near -5 kHz.

Appendix B

DERIVATION OF RELATIONSHIP BETWEEN DENSITOMETER FREQUENCY RESPONSE AND FREE-MOLECULAR-FLUX OF MOLECULES ARRIVING AT ENTRANCE ORIFICE

The number of molecules per unit time arriving at the entrance aperture of the Densitometer in free-molecular flow may be written as (ref. 7, p. 403).

$$N_a = \frac{n_a U_a A_o}{2\sqrt{\pi}} f(S_o \cos\alpha) \quad (B-1)$$

where

n_a = ambient number density of constituent a, molecules/cm³

$U_a = (2k T_a/m_a)^{1/2}$, most probable thermal speed of an ambient particle
(assuming a Maxwellian distribution of velocities), cm/sec

k = Boltzmann's constant, 1.380×10^{-16} erg/°K

T_a = Ambient temperature of free-stream molecules, °K

m_a = mass of ambient molecule, gm

A_o = Entrance aperture cross-sectional area, cm²

$$F(S_o \cos\alpha) = \exp(-S_o^2 \cos^2\alpha) + \sqrt{\pi} S_o \cos\alpha [1 + \operatorname{erf}(S_o \cos\alpha)]$$

S_o = Molecular speed ratio, V/U_a

V = Instrument package velocity, cm/sec

α = Angle between a normal to the sensor chamber entrance orifice and the instrument package velocity vector.

The number of molecules arriving per unit time at the sensor chamber after passing through the entrance aperture and the instrument drift tube may be obtained by modifying equation (B-1) with the free-molecular-flow transmission probability, $K(S_o, \alpha)$. Equation (B-1) becomes

$$N_{as} = \frac{n_a U_a A_o}{2\sqrt{\pi}} f(S_o \cos\alpha) K(S_o, \alpha) \quad (B-2)$$

Multiply both sides of equation (B-2) by m_a to obtain

$$\dot{m}_e = \frac{\rho_a U_a A_o}{2\sqrt{\pi}} f(S_o \cos\alpha) K(S_o, \alpha) \quad (B-3)$$

where \dot{m}_e is the total mass of the ambient molecules arriving at the entrance aperture per unit time and ρ_a is the mass density in gm/cm^3 of the ambient molecular flux.

A relationship between the molecules entering the sensor chamber and those escaping without being captured by the sensor crystal was derived in subsection 4.2 as

$$\dot{m}_e = \dot{m}_{\text{coll}} 1.04018 \quad (4-12)$$

where \dot{m}_{coll} is the total mass collected on the sensor crystal per unit time in gm/sec. Further, the relationship between the instrument's frequency response, $\Delta f/\Delta t$, and \dot{m}_{coll} was written as

$$\Delta f/\Delta t = H \dot{m}_{\text{coll}} \quad (4-13)$$

where H is the crystal sensitivity in Hz/gm.

Equations (B-3), (4-12), and (4-13) may be combined to result in the required relationship

$$\Delta f/\Delta t = \frac{H\rho_a U_a A_o}{(1.04018) 2\sqrt{\pi}} f(S_o \cos\alpha) K(S_o, \alpha) \quad (B-4)$$