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THE NASA/MARSHALL SPACE FLIGHT CENTER  
DROP TUBE USER'S MANUAL

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Space Science Laboratory  
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16. Abstract  <b>A comprehensive description of the structural and instrumentation hardware and the experimental capabilities of the 105-meter Marshall Space Flight Center Drop Tube Facility is given. This document is to serve as an guide to the investigator who wishes to perform materials processing experiments in the Drop Tube. Particular attention is given to the Tube's hardware to which an investigator must interface to perform experiments. This hardware consists of the permanent structural hardware (with such items as vacuum flanges), and the experimental hardware (with the furnaces and the sample insertion devices). Two furnaces, an electron-beam and an electromagnetic levitator, are currently used to melt metallic samples in a process environment that can range from 10<sup>-6</sup> Torr to 1 atmosphere. Details of these furnaces, the processing environment gases/vacuum, the electrical power, and data acquisition capabilities are specified to allow an investigator to design his/her experiment to maximize successful results and to reduce experimental "setup" time on the Tube. Various devices used to catch samples while inflicting minimum damage and to enhance turnaround time between experiments are described. Enough information is provided to allow an investigator who wishes to build his/her own furnace or sample catch devices to easily interface it to the Tube. The experimental instrumentation and data acquisition systems used to perform pre-drop and in-flight measurements of the melting and solidification process are also detailed. Typical experimental results are presented as an indicator of the type of data that is provided by the Drop Tube Facility. A summary bibliography of past Drop Tube experiments is provided, and an appendix explaining the noncontact temperature determination of free-falling drops is provided. This document is to be revised occasionally as improvements to the Facility are made and as the summary bibliography grows.</b>					
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1. INTRODUCTION

During the past decade increasing interest has been shown by the scientific community in experiments to study the effects of a low-gravity environment on the solidification of materials. When materials are processed on Earth, several types of phenomena can affect the solidification process such as buoyancy, sedimentation, reactions with the container, hydrostatic head, and thermally-driven convection. When processed in low gravity these effects can be reduced to a very low level of contribution to the solidification process.

As more studies are performed, the scientific community is increasing its understanding of materials processes such that they are able to develop unique materials which can have different properties according to the processing environment. For example, higher transition temperature superconductors or lower creep rate nickel superalloys have been prepared in low gravity and/or containerless furnaces.

There are a number of ways to perform materials processing experiments in low gravity. The ultimate environment is attainable as a shuttle experiment, which is very costly and requires a very long procedure (usually in terms of years) to fly an experiment. Other alternatives exist which can be less costly and a lot quicker. There is a KC-135 aircraft which provides low-g times of up to 20 seconds. Operational procedures of these low-g facilities do not lend themselves to running a large number of samples cost effectively, particularly when all the necessary experimental parameters are not known.

NASA also has available ground-based facilities which provide low-g or containerless processing capabilities located at Marshall Space Flight Center (MSFC) in Huntsville, Alabama. These facilities are the Drop Tube and Drop Tower. They provide a low-g time of approximately 4.5 seconds. Drop Tower experiments are performed by free-floating a materials processing package inside a much larger aerodynamically-shaped capsule which drops 105 meters during the processing time. These experiments are typically materials processing or fluid dynamic experiments that are contained in crucibles or fluid cells.

The Drop Tube, however, serves as a unique facility for performing materials processing research in a low-gravity and containerless environment. Since the facility operates by melting the material of interest and then allowing the molten drop to fall to the bottom of the tube, solidification and containerless processing experiments are normally the experiments performed in the Drop Tube. Some engineering tests can also be performed in the Drop Tube such as the recently completed Tethered Satellite tether vibrational test. For those investigators who are planning to use the Drop Tube Facility, the intent of this document is to explain the facility's structural and electrical features as well as the capability of accommodating experiments. This document outlines the criteria that a user must satisfy in order to prevent detrimental effects to this national facility.

## 1.1 General Information About the Drop Tube Facility

The Drop Tube is 105 meters long and provides a drop time of 4.6 seconds. The facility can be divided into three sections as indicated in Figure 1. They are the belljar section, the drop tube section, and the catch tank section.

The belljar is located at the top of the facility and is connected to the tube through an isolation valve and an adapter flange. The adapter flange contains several vacuum ports which are used in the operation of the materials processing furnaces for purposes such as backfilling, residual gas analysis, and leak testing. The belljar also has several viewports to allow observation of the molten samples or for temperature measurement.

The catch tank section is also connected to the Tube with an isolation valve. This section has detachable catch tanks that mate to the Drop Tube with CAM locks. This arrangement allows for very quick sample removal.

The Drop Tube itself is constructed of schedule 10-S pipe which is 26.6 cm (10.42 inches) internal diameter and extends through the 15 levels of the MSFC Dynamic Test Stand Facility (Building 4550). Six vacuum pumping stations are used to maintain the proper vacuum level in the system. The location of these pumping stations is also shown in Figure 1. Note that four pumping stations are attached only to the tube, while there is a pumping station at both the belljar section and the catch tank section. All six pumping stations consist of turbomolecular pumps (TMPs), and each station can be isolated from the tube with an electropneumatic isolation valve. Levels 15, 7, and 1 also consist of a separate roughing pump (RP) for evacuating the respective section of the Tube from atmospheric pressure. Also noted in Figure 1 are several infrared (IR) detectors which are used to observe the brightness profile of the falling drops. In particular the identification of undercooling recalescence from a solidifying specimen is usually the desired event easily observed with this instrumentation.

The heart of the Drop Tube operation is housed in the control panel on the 15th level. From this panel the system operator controls all pumping stations, isolation valves, and vent valves, and can read the pressure at various locations in the Drop Tube. Pressure determination at each pumping station is monitored by a combination of ion and Pirani gauges. More detail on the Drop Tube Facility hardware and operational procedures can be found in the Operational Procedures and Specifications Document submitted to NASA/MSFC by the University of Alabama in Huntsville (UAH) under contract number NAS8-34530.

The Drop Tube is one of the most flexible methods for performing materials processing experiments in low-gravity. The short sample-to-sample processing time of approximately 20 minutes, the ability to make quick experimental changes due to unexpected results, and the low cost per sample compared to other low-g facilities make the drop tube a valuable tool in the field of materials processing research. The Drop Tube at MSFC is truly a unique national facility not available elsewhere in the United States.

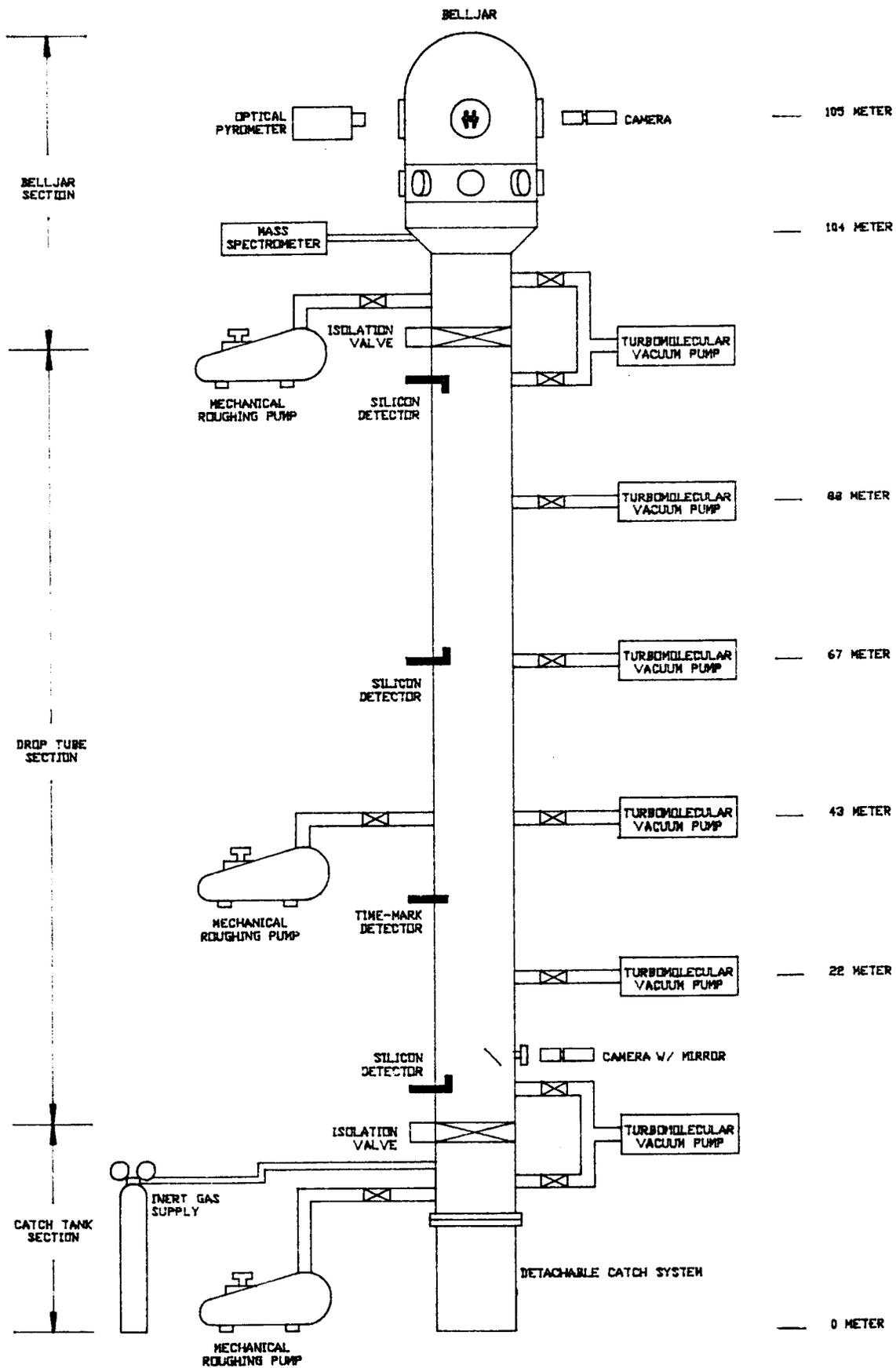


Figure 1. Drop tube schematic.

## 2. EXPERIMENT ACCOMMODATION

### 2.1 Drop Tube Facility Resources

#### 2.1.1 Permanent Hardware

##### 2.1.1.1 Interface/Feedthrough Flanges

The ends of the Drop Tube can be adapted for users who desire to use either a furnace of their design for the top portion of the Tube or a unique sample catch tank apparatus for the bottom section of the Tube. If required, either end can be used as observational ports to monitor drops as long as appropriate hardware is built to satisfy vacuum and furnace/catch tank requirements.

The flange located at the tube bottom end is a standard 16-inch flange using a Leybold ISO-K sealing disc. Four handles are used to hold the mating flange and O-ring assembly against the Tube flange. Figure 2 shows a schematic of this arrangement.

The upper Tube end is shown in Figure 3 and consists of several vacuum rings set atop an inverted belljar to which the Tube is attached. The I.D. and O.D. of each ring is given as well as the approximate O-ring or L-ring gasket location. The cover for the ring assembly is a plate with a centered viewport (for the electromagnetic levitation furnace), a belljar with multiple viewports (for the electron-beam furnace), or any custom hardware the investigator may wish to build.

The inside of the main section of the Drop Tube can be accessed by means of any of the flanges listed in Table 1 which are not being used by turbomolecular vacuum pumps or ion gauges. All flanges are high vacuum flanges and any hardware built to interface to these flanges and to penetrate into the Tube must be constructed of materials and techniques compatible with high vacuum facilities. Figure 4 is a dimensional drawing of the three different sized flanges found on the main section of the Tube.

##### 2.1.1.2 Hardware Propriety

Any hardware designed for the MSFC Drop Tube to be used by the investigator will be held in strict propriety if requested. As with any work performed at a national government facility, it is required that results be published in open journals. Likewise it is hoped that any major contribution by Drop Tube personnel be recognized to some extent by the investigator in his normal reporting or publication actions.

#### 2.1.2 Hardware/Experiment Modification

##### 2.1.2.1 Support Work Areas

There are five different areas within the Drop Facility locality that provide support capabilities for Tube operations. Building 4550 houses three of these areas. The room on level 6 provides an electronic workshop; level 2 provides a small mechanical shop with drill press and acetylene equipment; level 15 itself provides some support via scales and data analysis capability. Building 4551, the adjacent blockhouse, provides a room housing more customary metallurgical lab equipment such as saws, polishers, x-ray diffraction, arc-melting, etc. The basement of building 4551 houses a lathe for custom, on-the-spot machining. Anything not found in these areas can be obtained through MSFC or UAH.

TABLE 1. TYPICAL DROP TUBE FLANGE USAGE

LEVEL	2 3/4 INCH O.D. INSTRUMENTATION PORT	6 INCH O.D. PORT	12 INCH O.D. PORT
15	Above ISO* : Ion tube Below ISO: Beratron	Roughing Pump N/A	TMP TMP
14	Si w/Logarithmic Amplifier	N/A	N/A
13	Si w/Linear Amplifier	Ion Tube	TMP
12	Si w/o Amplifier	N/A	N/A
11	Si w/Linear Amplifier	Window	Blanked
10	Blanked	Ion Tube	TMP
9	Si w/Linear Amplifier	Window	Blanked
8	Si w/Logarithmic Amplifier	N/A	N/A
7	Upper: Si w/Linear amplifier Lower: Ion Tube	Roughing Pump	TMP
6	Ge w/Linear Amplifier	N/A	N/A
5	Si w/Linear Amplifier	Window	Blanked
4	Ion Tube	CaF <sub>2</sub> Window+Mirror	TMP
3	Si w/Linear Amplifier	Blanked Adapter	Blanked
2	Blanked	N/A	N/A
1	Above ISO: Si w/Logarithmic Amplifier Below ISO: Beratron + Ion Tube	Mirror+Camera Roughing Pump	TMP TMP

\* ISO - Abbreviation for vacuum isolation valve used in this Table in reference to the Drop Tube 10 inch valves separating the three sections of the Tube.

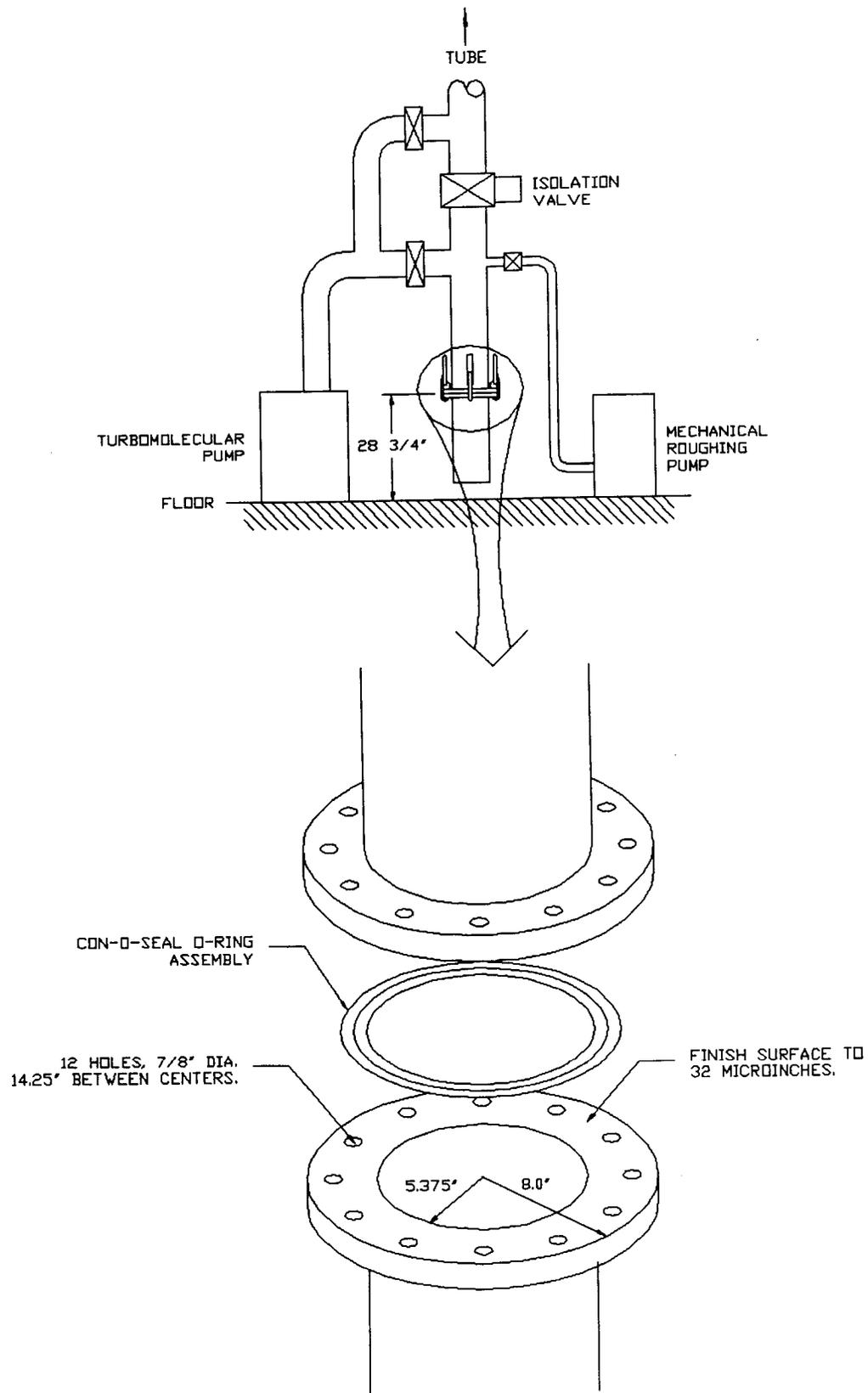


Figure 2. Bottom mating flange.

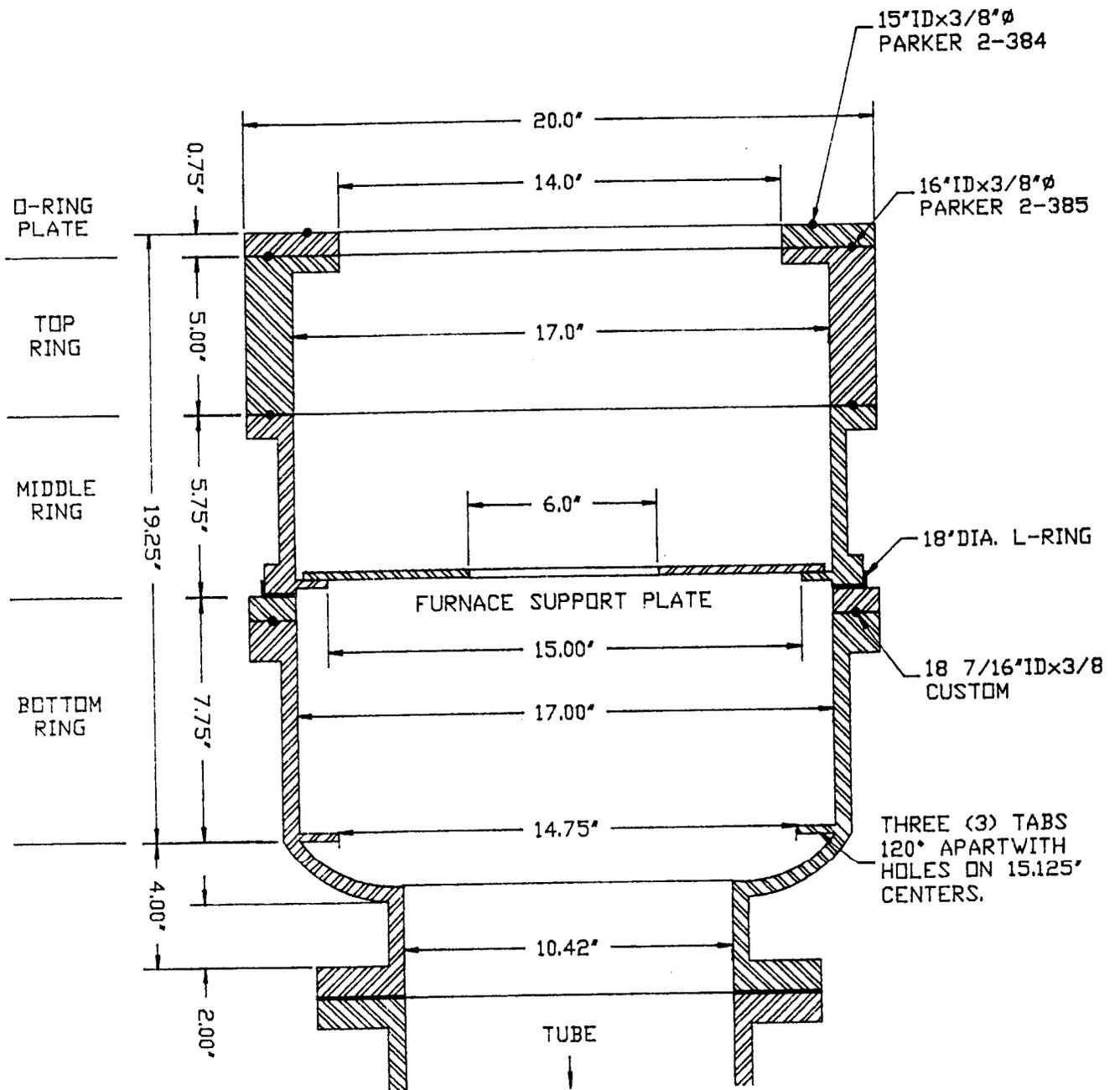
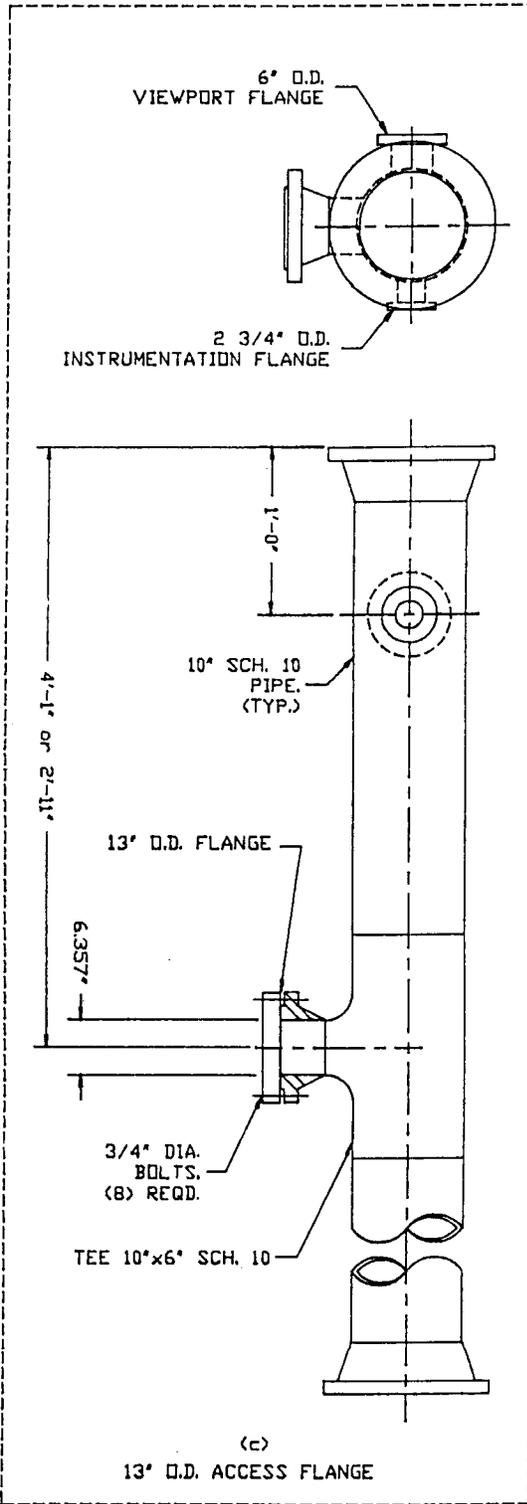
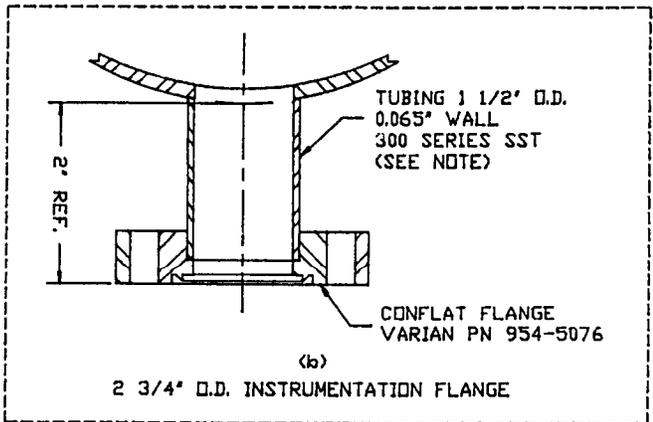
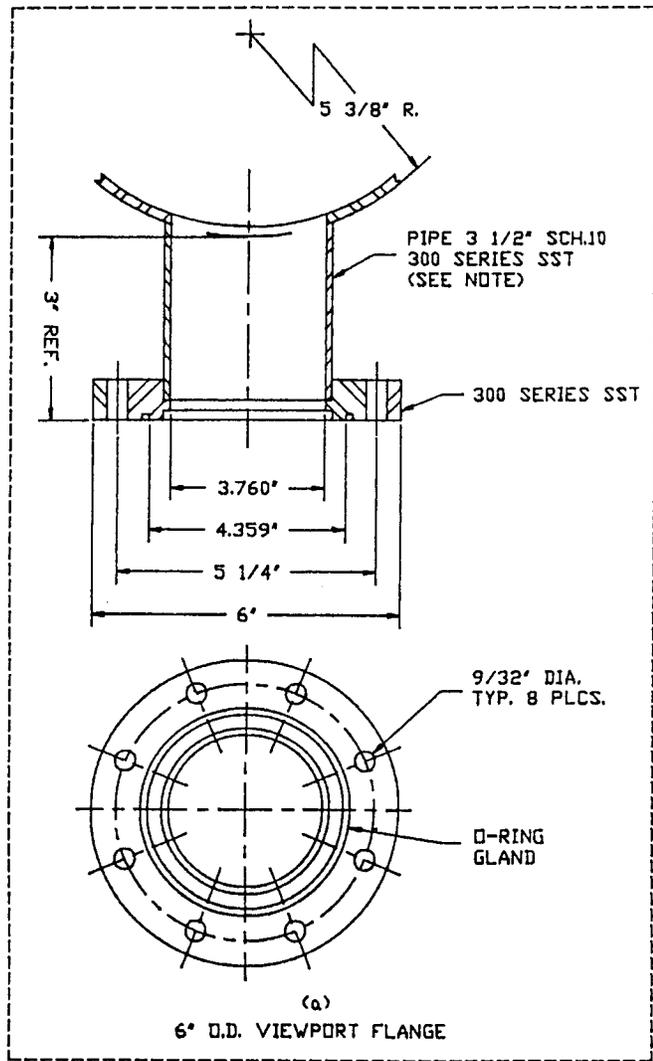


Figure 3. Level 15 belljar schematic.



NOTE: ACTUAL I.D. WILL VARY DUE TO WELD BEAD. 1/4" SHOULD BE SUBTRACTED FROM THE I.D. TO ACCOUNT FOR THE WELD BEAD.

Figure 4. Flange configuration of Drop Tube main section.

## 2.1.2.2 Characterization Work Areas

Some characterization capabilities can be accomplished in Building 4551 using available optical microscope and x-ray diffraction units. Arrangements can be made to use whatever facilities are available at either UAH or MSFC if scheduled in advance. It is strongly advised that the investigator obtain analysis of his samples at his employer's facilities. The purpose of Building 4551 equipment is to provide a quick-look assessment of the specimens to determine if scientific objectives are being met in the Tube and whether the processing conditions should be altered.

## 2.2 Furnace Resources

### 2.2.1 Structural/Mechanical

#### 2.2.1.1 Available Furnaces

At present, there are two functional furnaces: an electromagnetic levitation/RF heating (EM) furnace and an electron-beam (EB) furnace (Figures 5 and 6, respectively). The EB furnace consists of a 0.020-inch-diameter tungsten wire looped into a 1-inch-diameter filament. This filament is surrounded by an electron deflecting ellipsoidal grid. The grid is attached to a well-insulated and sturdy support structure as shown in Figure 7. The grid and filament are kept at negative 4000 V d.c. relative to the grounded sample. The grounded samples are lowered into the loop via another structure which can rotate and vertically move a sample carousel. The -4 kV potential strips electrons by thermionic emission from the filament and accelerates them into the sample; the electron kinetic energy is converted into heat when decelerated by the sample.

The EM furnace uses a coil constructed from 1/8-inch copper tubing as shown in Figure 8. This tubing connects to the furnace interface flange described in section 2.2.2.3. Seven turns comprise the bottom coil which is made at a 30° conical angle. This construction produces a magnetic field gradient that enhances the levitating capabilities. The bucking top coil is made of two turns which constrains the sample to reside in the potential well of the bottom coil thus producing stability in the levitated specimen. The heating is produced by RF inductive coupling between the sample's conduction electrons and the oscillating magnetic field of the coil. The samples are inserted into the coil via a carousel device which allows rotation and vertical motion within the vacuum chamber.

#### 2.2.1.2 Viewports and Vapor Barriers

The support structure and sample insertion devices of both furnaces rest on a plate inside the inverted belljar as was shown in Figure 3. This plate and the location of the furnace above this plate are not changeable with the present hardware. When the furnaces are covered with their appropriate lid, the furnaces are optically aligned so that the lid viewports allow visual and pyrometric observation of the process. Investigators using custom-built furnaces and desiring visual data must give this viewport alignment criteria high priority in their designs.

To prevent vapor deposition from the samples onto the quartz vacuum viewports, vapor barriers/windows are used between the sample and viewport. Enough windows are available to provide quick exchange if a window becomes undesirably dirty. For very vaporous samples, an assembly is available to provide in situ changing of the dirty window by rotating a clean section of a piece of glass into the optical line-of-sight. The clean glass is protected behind a vapor mask until needed.

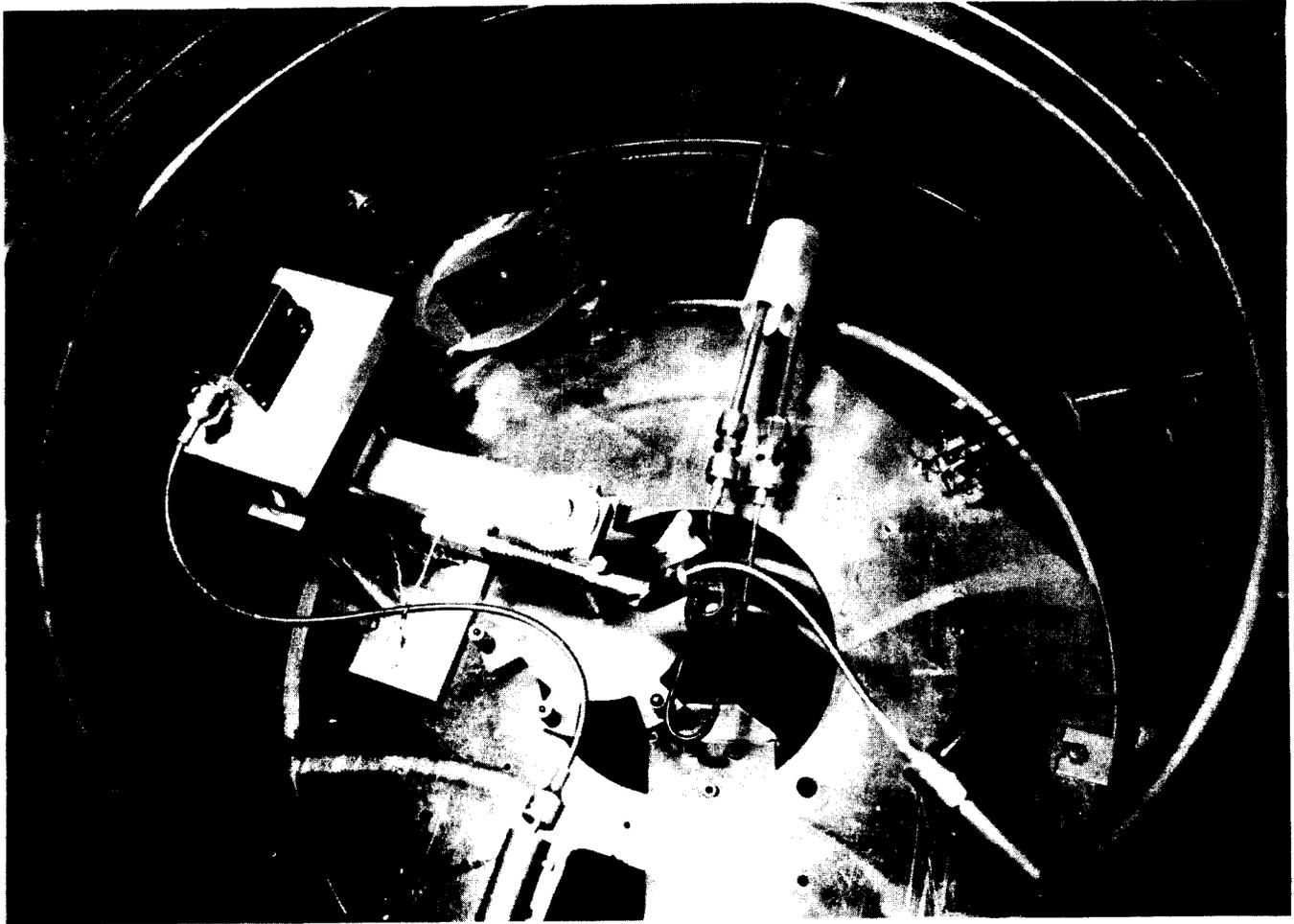


Figure 5. View of electromagnetic furnace with sample carousel.

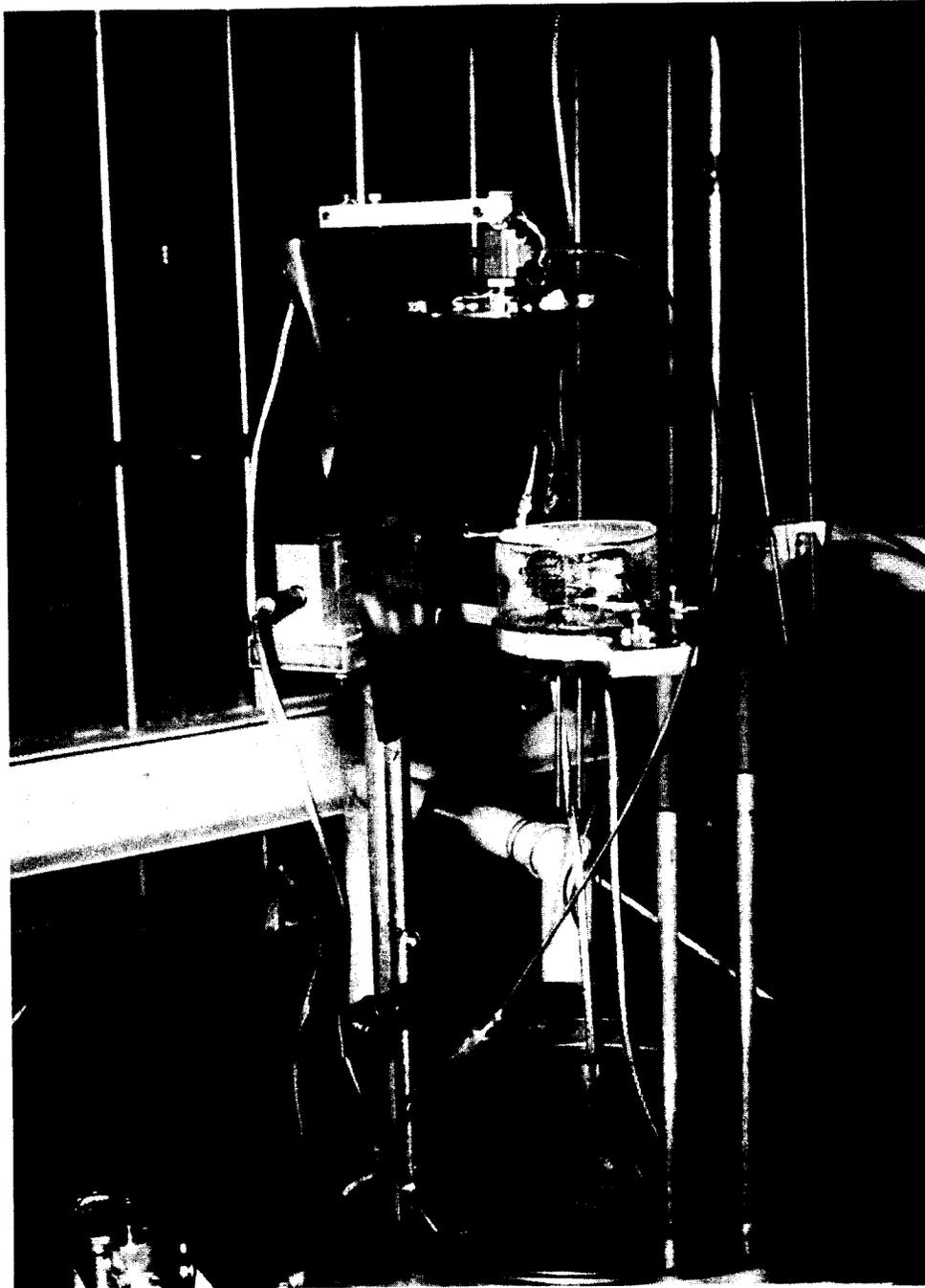


Figure 6. View of electron-beam furnace with sample carousel.

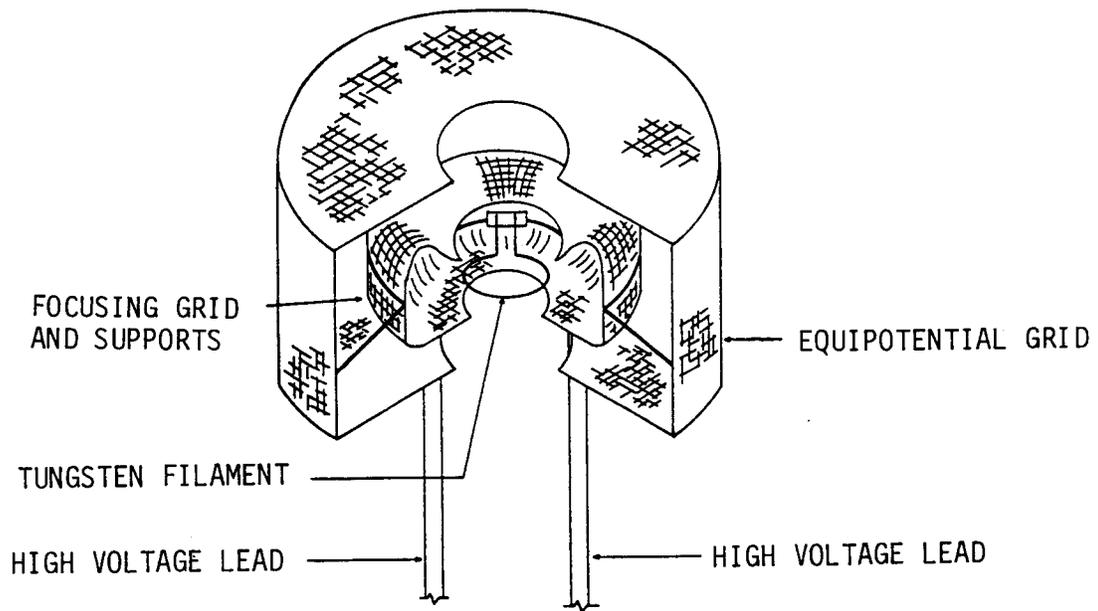


Figure 7. Electron-beam furnace three-dimensional illustration.

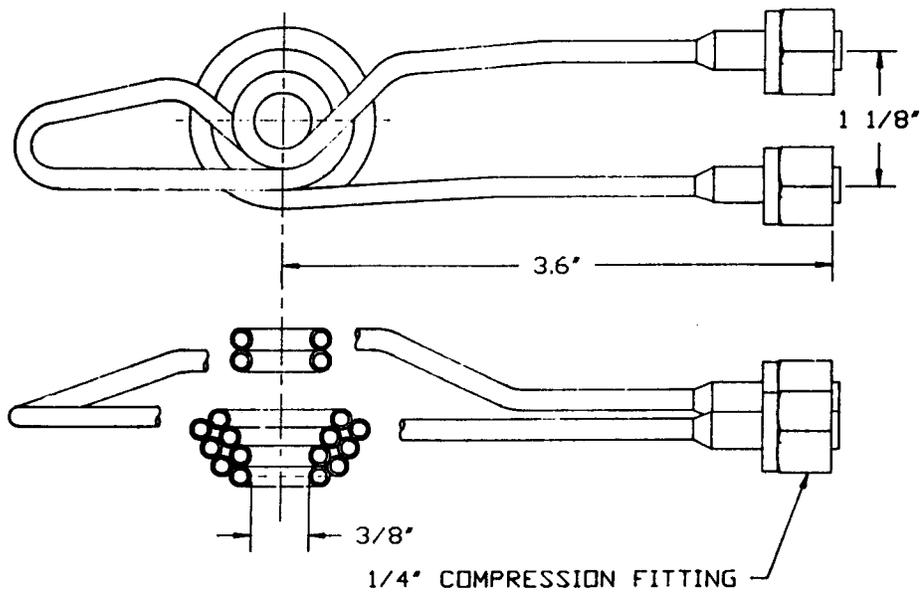


Figure 8. Schematic of electromagnetic furnace coil.

### 2.2.1.3 Sample Mounts and Carousels

#### 2.2.1.3.1 Sample Carousels

The mounts to which the samples are attached are themselves mounted to a carousel-type device. The two available carousel devices are shown in Figures 9 and 10. These are for the EM and EB furnaces, respectively. The carousels can traverse vertically for sample insertion or rotate in a horizontal plane to bring a new sample into the furnace. The carousel motion is controlled by the operator via rotatable feedthroughs which are coupled to vacuum-compatible gear boxes on the carousels.

#### 2.2.1.3.2 Sample Mounts

The EM sample mounts are of two types: ceramic pedestals or alligator-clips. The ceramic pedestals are typically 3/8-inch diameter with the top surface countersunk into which samples are set. The pedestal carousel can accommodate a maximum of seven samples. Each pedestal holds a freely sitting specimen. The pedestal is quickly moved down and out of the EM coil as levitation begins. The pedestal is then rotated from under the coil to give an unobstructed path to the sample when it is released.

The EM alligator-clip carousel is used to hang samples from wires. These samples, like those of the pedestal carousel, are inserted into the EM coil before power is applied. The carousel can then be moved upward to remove the wire from the levitating sample or not moved at all if the samples are known to melt and fall off of the wire and out of the coil.

The EB sample holders are depicted in Figure 10. The holder for cylindrical samples is available in various diameters (maximum of 1/2 inch) with two opposite set screws that can grip intermediate size sample rods or wires. These holders are held by the attachment blocks found on top of the carousel. For odd-shaped samples, custom-built holders can be made but must still be able to interface to the four attachment blocks atop the carousel. The block hole is 1/16 inch in diameter and with only four blocks; the furnace can accommodate only four samples. The sample holders must be made from good electrically conducting materials. The samples are rotated above the EB furnace and lowered into the tungsten heating loop. The maximum vertical travel distance and thus sample length is 9 inches.

#### 2.2.1.4 Vibration

Since the Drop Tube Facility sits atop a building 110 meters in height, both furnace assemblies will be susceptible to vibrations caused by winds, elevator, Army test explosions, crane operations, etc. These vibrations are particularly noticeable in the EB furnace whose samples must be melted by the pendant drop technique, or a variation thereof. Any significant horizontal vibration of low frequency may impart enough horizontal momentum to the molten drop to cause the drop to hit the Tube wall as it falls.

The EM furnace is not as sensitive to building vibrations because the sample is "contained" within the electromagnetic field created by the coil as it is being levitated. However, any instability of the sample in the coil will have the same affect as vibrations if horizontal momentum is imparted to the sample upon release.

To circumvent this vibration problem, drops will only be made when the Drop Tube Operator determines that conditions are favorable.

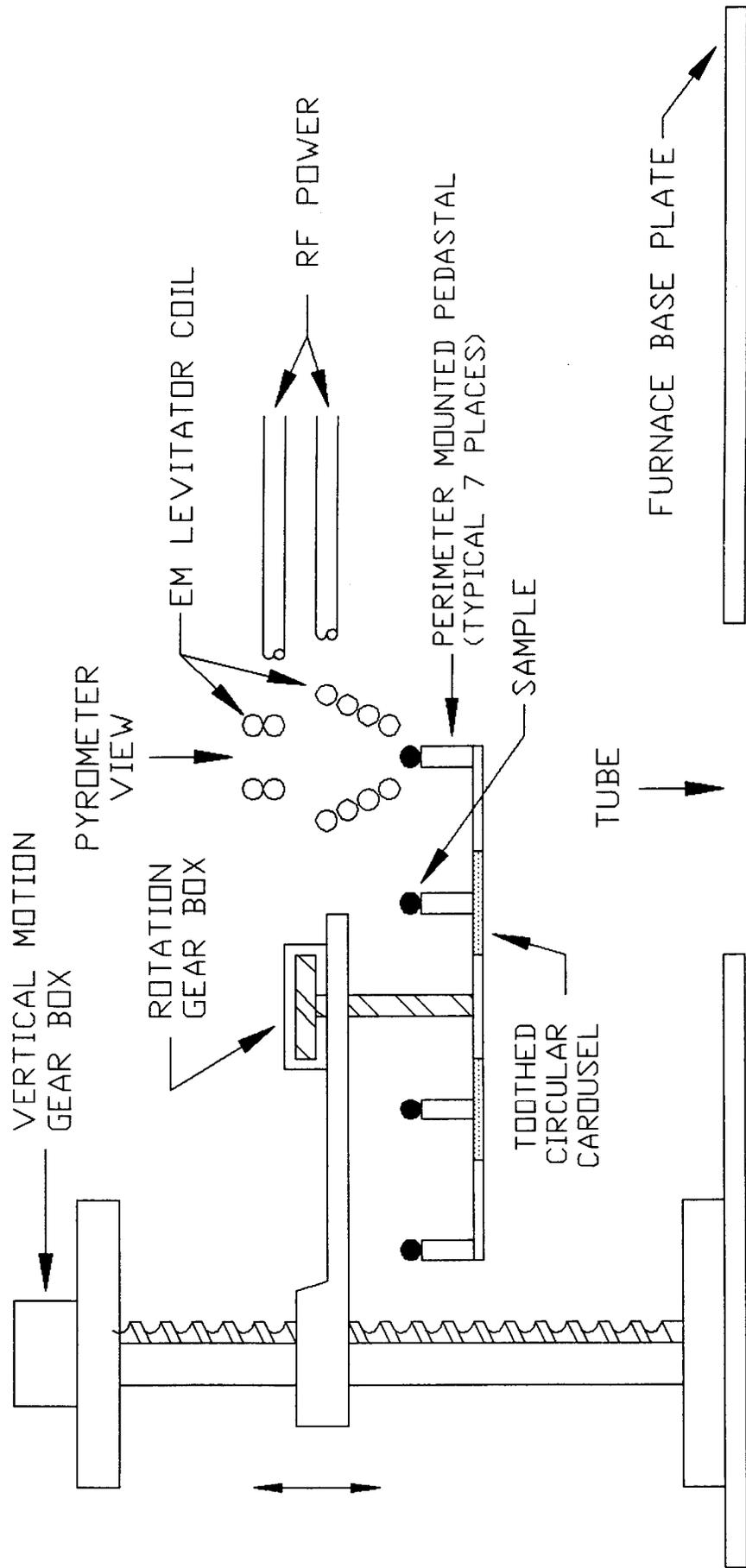


Figure 9. Electromagnetic sample carousel.

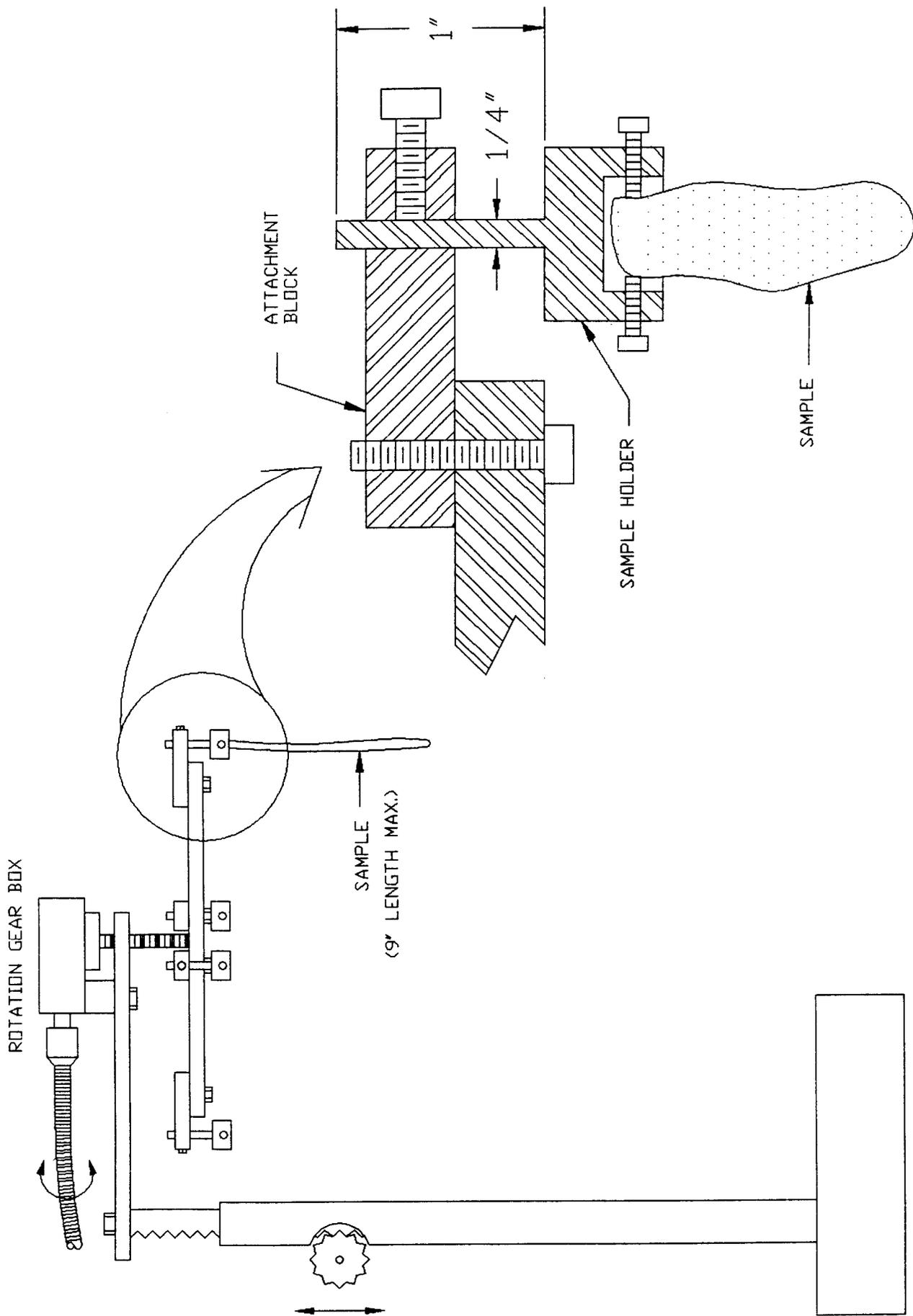


Figure 10. Electron-beam carousel and sample holders.

### 2.2.1.5 Feedthroughs

Two vacuum rings sit atop the inverted belljar as described in section 2.1.1.1. One ring has eight and the other ring has six 2 3/4 inch conflat flanges arranged circumferentially. These flanges are used for feedthroughs to the furnace environment. Hardware that is available for use with the feedthroughs include Cajon fittings, rotary and thermocouple feedthroughs, and viewports; any other device for mechanical, electrical, or instrumentation necessities can be used. Figure 11 shows the present usage of these belljar ring flanges.

### 2.2.2 Electrical Power

#### 2.2.2.1 Power Availability and Connectors

The facility power available to operate furnaces consist of the following a.c. circuits operating at 60 Hz :

<u>POWER</u>	<u>CONNECTOR TYPE</u>
120 V, 15 A, 1 $\phi$	Duplex
120 V, 30 A, 1 $\phi$	Hubbell
208 V, 30 A, 1 $\phi$	Hubbell twist-lock
208 V, 30 A, 3 $\phi$	Hartlock L-14-30
208 V, 60 A, 3 $\phi$	Crousehinds APJ6485 3W4P

The power available from secondary supplies, which themselves operate from facility power, provide the following maximums:

EB supply: Thermionics Laboratory, model 150-0030  
-4,000 V d.c., 750 mA; 26 V a.c., 30 A, 60 Hz

EM supply: Lepel RF generator, model T-10-3-DF-RP-H  
10,000 V a.c.,\* 300 A,\* 250 kHz-8 MHz

Tube solenoid valve supply: Lambda, model LK-351  
40 V d.c., 30 A

A 3:1 or 12:1 transformer can be used to provide more current and less voltage to the working coil. With the 1/8-inch-diameter tubing used for the working coil, there is a limitation in experiment run time due to the flow restrictions of the cooling water in the coil.

#### 2.2.2.2 Sample Heating Circuitry

For the EB furnace, the W-filament is normally kept at -4000 V d.c. relative to the grounded sample. A 26 V a.c., 60 Hz variable current source is used on the W-filament loop to generate thermally emitted electrons. The electron cloud emitted from the filament is quasi-focussed onto the sample by an ellipsoidal-shaped screen/grid at the same potential as the filament. The thermionic electrons kinetically heat the sample at its surface.

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\*The power available to the EM coils from the Lepel generator depends strongly on the impedance of the working coil.

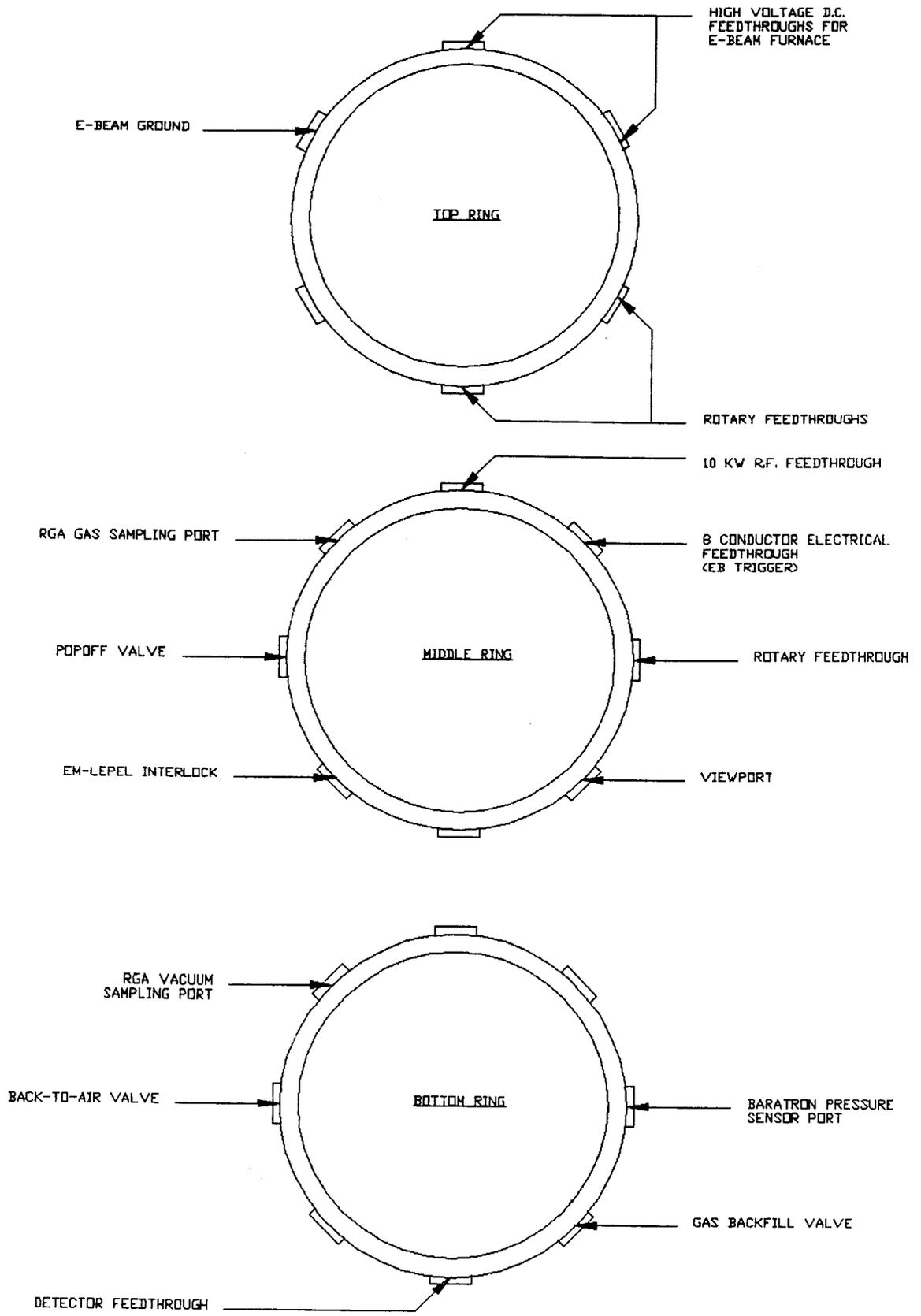


Figure 11. Belljar vacuum ring feedthrough arrangement.

The EM furnace requires that the sample be an electrical conductor either before or after heating to allow levitation. The user controls the Lepel power to the coil to change the amount of heating and levitation. The sample heating capability is very dependent on the coil design which is typically a slight variation of the design described in section 2.2.1.1.

### 2.2.2.3 Furnace Interface Connectors

The EB electrical connectors are those that accompanied the power supply. These connectors mate to a male copper rod high voltage vacuum feedthrough. The flange is Huntington's PN EF-151.

The EM coils attach to a coaxial RF vacuum feedthrough (Varian PN 954-5017 or Ceramaseal PN 808C6164-02-CF) via a 1/4 inch to 1/8 inch Swagelok adapter. The RF feedthrough is connected to the transformer which is connected to the Lepel generator via a 10-foot coaxial cable.

## 2.2.3 Thermal Control

### 2.2.3.1 Liquid Cooling

For the EM furnace, a Neslab model HX-500 water chiller is available to provide distilled, deionized water as low as 15 °C, 65 psi, and 12 gal/min. This water cools both the Lepel generator and the working coil; however, the water is available for other uses if desired.

### 2.2.3.2 Touch Temperature

Any furnace constructed for use at the Drop Tube must be constructed so that any surfaces exposed to human contact shall not exceed 115 °F.

## 2.2.4 Process Environment

### 2.2.4.1 Vacuum

As alluded to in section 1.1, there are three sections of the Drop Tube which can be isolated from each other by high vacuum gate valves. In any one section of the Tube, 400 liters/s Sargeant-Welch turbomolecular vacuum pumps can typically achieve a vacuum in the low  $10^{-6}$  Torr range. No special hardware is used (such as titanium getters) to achieve this range; but, these can be attached as well as heat tape or other hardware if so desired by the user.

### 2.2.4.2 Gases

There are presently four gases that are available to the Drop Tube: 5-nines pure argon, helium, helium-6 atomic percent (a/o) hydrogen, and grade B, 4-nines pure facility-provided nitrogen. Other gases can be obtained if given an appropriate lead time. Hydrogen gas can only be used at 6 a/o or less in dilution with an inert gas because of the safety requirements imposed by its flammability limits. All gases but the nitrogen can be gettered of oxygen using a Centorr model 2A-100-SS gettering furnace. Any subsequent dust particles injected into the gas stream from the oxidation breakdown of the titanium gettering charge are filtered by 140  $\mu\text{m}$  and a 2  $\mu\text{m}$  Nupro in-line filters. The gettered gases, except those like Ar which will freeze, are then passed through a liquid nitrogen cold trap.

The gases used to backfill the Tube are tested bimonthly by MSFC to determine the moisture, organic, and particulate distribution content. These gas samples are taken at the purge nozzles located at the hand backfill valves. Typical values are 1 ppm of moisture and organics while the particulate content is 3 particles of 100  $\mu\text{m}$  or less per 30 cubic feet.

#### 2.2.4.3 Contamination

All instruments or furnaces that are placed inside the belljar or Drop Tube must adhere to high vacuum standards. Nothing shall be inserted into the process environment that may produce extraordinary outgassing, particulate emission, high intensity radiation, or other facility contaminants during the sample processing unless it is evolved from the sample itself. If contaminants are evolved from the sample, the Drop Tube operator must be notified in advance.

### 2.3 Sample Accommodation

This section deals with all the aspects relevant to the processing of specimens: data management, pre-processing requirements, sample integration and checkout, retrieval hardware/techniques, and typical process data.

#### 2.3.1 Data Management

##### 2.3.1.1 Pyrometer

There are two, two-color Ircon model R optical pyrometers for acquiring the temperature of the samples as they are being heated and melted in the furnace before dropping. Each pyrometer covers a different temperature range and has a temperature resolution as follows: 1500  $^{\circ}\text{C}$  to 3500  $^{\circ}\text{C}$ , 0.4  $^{\circ}\text{C}$ ; 700  $^{\circ}\text{C}$  to 1400  $^{\circ}\text{C}$ , 0.07  $^{\circ}\text{C}$ . All data are gathered by the data acquisition computer for later converting, graphing, and storing. It is gathered at a rate of 35 readings per second or slower for a corresponding experiment time limit of 8 minutes or longer.

Even with such good resolution, the accuracy of the pyrometer is quoted by Ircon at 1%, but can be determined more accurately by processing a sample having a known melting temperature. This melting temperature provides only one calibration point. All other temperature readings must rely on the assumption that the sample emissivity ratio for the wavelength ranges of the detectors used in the two-color technique does not deviate from the calibration curve provided by Ircon.

##### 2.3.1.2 Detectors

During the free-fall period of the sample, brightness measurements are made using various detectors and optics. The working detectors are silicon; research is still progressing to implement germanium or indium antimony for lower melting temperature materials. The access flanges available on each level allow combinations of viewangles (up or down), interdetector distances, and thus amplifier gains necessary to optimize the signal. The silicon detectors used in the past have been United Detector Technology, model UDT555D made with a built-in linear amplifier. These attach to a Heraeus Amersil T18 Infrasil quartz rod which is inserted through a Cajon O-ring seal (Figure 12) which isolates the electronics from the vacuum. Also used are logarithm amplifiers which allow a continuous view of a drop the entire length of the Tube. Response times are typically 10-15  $\mu\text{s}$  for both, but a 20 ms decay time for the logarithmic amplifiers.

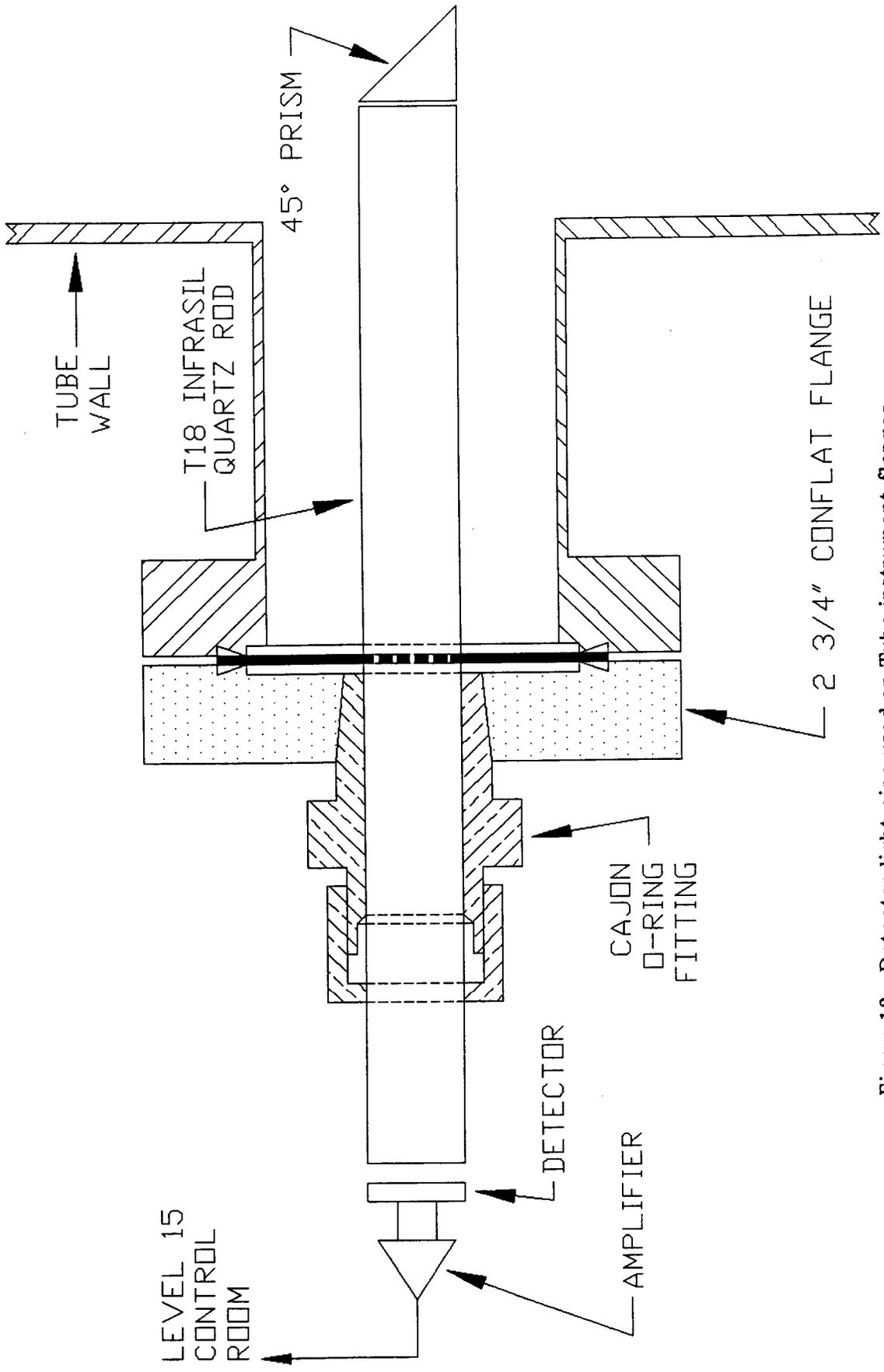


Figure 12. Detector light-pipe used on Tube instrument flanges.

All the electrical lines between the detectors and the 15th level control room are tied along an I-beam running the length of the building. At all the detectors, there is one line that can be activated from the control room that provides power to control relays which turn on low-noise d.c. power supplies for the detectors' amplifier. The amplifiers' d.c. output signal is transmitted via coaxial RG-58 cable to the 15th level data acquisition system. The signal is then acquired at any rate up to 10 MHz.

#### 2.3.1.3 Residual Gas Analyzer (RGA)

To control, monitor, and analyze any contamination in the belljar volume, a Dycor Electronics, series M100 residual gas analyzer (RGA) is attached to the side of the inverted belljar. It can operate with the belljar in either a vacuum or a pressurized state. A schematic of the RGA station is provided in Figure 13. It consists of a 200 liter/s Varian model V200 turbomolecular pump attached to a Varian cross in the center of which is situated the RGA filament. A straight-through valve provides a direct line-of-sight to the filament for molecules from the belljar chamber when it is under high vacuum. For pressurized belljar operations, the straight-through valve is closed and the gas molecules are pumped across the filament as a Granville-Phillips, series 203 variable leak valve is opened.

#### 2.3.1.4 Video

An important facet of the data management is the video data taken of both the heating and melting process before dropping, and the cooling and recalescence during free-fall. The data are essential to the determination of a successful drop; i.e., that the sample melted and fell the length of the Tube without contact with the Tube wall.

The video of the heating/melting from the control room camera (Cohu, model 4815) is mixed with the video image of the falling sample from the catch room camera (Cohu, model 4815) by a Panasonic special effects generator, model WJ-5500B. The equipment configuration is shown schematically in Figure 14. Also, a Panasonic time-mark generator superimposes date/time and stop-watch characters over the images. The images are then displayed on overhead monitors for ease of viewing. The video data is recorded in VCR VHS format for replaying in slow motion or for making copies for the investigator.

#### 2.3.1.5 Time Sequencing

Until such time that continuous temperature measurements can be performed on the falling drops, the temperature of the drop at any location in the Tube can only be modeled mathematically. What is needed from the pyrometer and detector measurements is the release temperature, initial time ( $t = 0$ ), and the final time. From these three pieces of data, the final temperature can be calculated (see Appendix).

To obtain the final time, the instrumentation timing uncertainty must be known from the moment of sample release to the moment of detecting the event of interest. The biggest uncertainty in the final time is caused by the method of triggering the DAS system to start taking detector data at the moment of sample release. Different triggering methods are used for the EM and EB furnace. Table 2 lists the sequence of events for both furnaces that occur from time  $t = 0$  until the DAS is triggered.

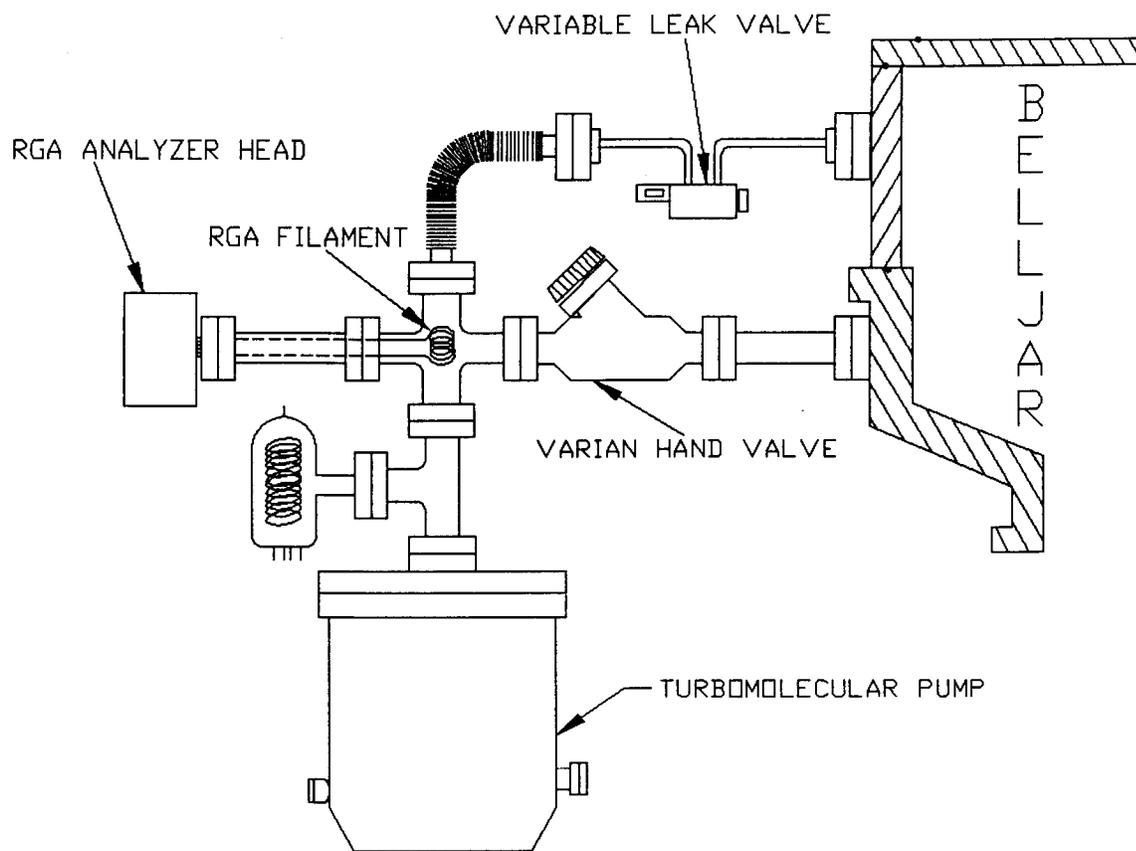


Figure 13. Residual gas analyzer system.

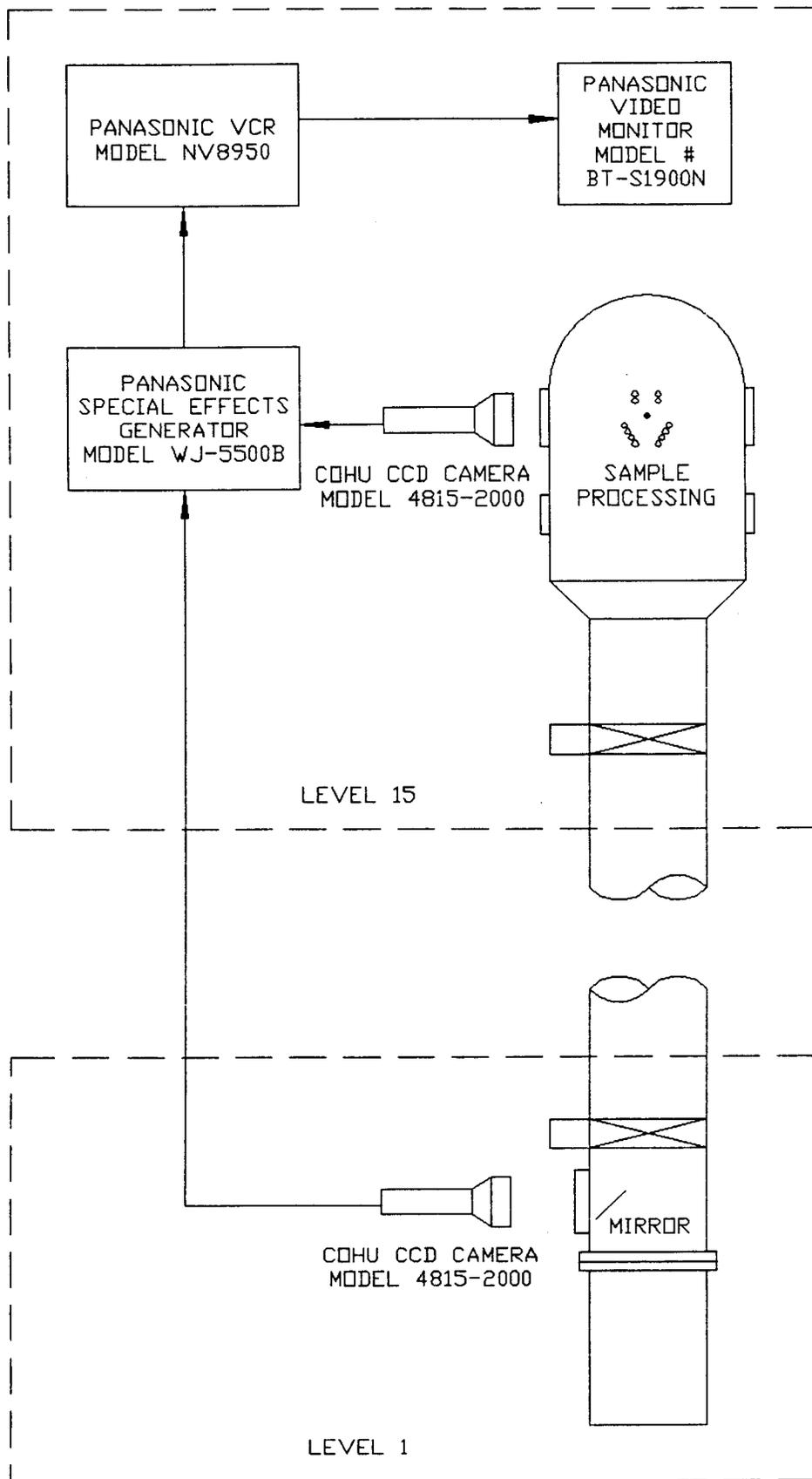


Figure 14. Video equipment configuration.

**TABLE 2. TIME DELAYS OF EVENTS INITIATING DROP RELEASE DETECTION**

Events	Time Delay
EM Furnace	
Power OFF switch pressed	0
Trigger relay activated	50 ms
Soltec activated	100 ns
EB Furnace	
Drop falls off of wire	0
Drop arrives at Schmidt trigger detector	360 ms
Schmidt trigger/detector activated	50 $\mu$ s
Trigger relay activated	50 ms
Soltec activated	100 ns

In the data above, the delay time due to the signal time-of-transmission within the interconnecting coaxial cables has been neglected. For all practical purposes, the trigger relay which was measured at 50 ms is the only hardware item whose delay time is significant. The total time delay of 50 ms (plus 360 ms for EB) is the time needed to be added to the detector data due to the delay from the moment the drop is released until the detectors are being read by the computer.

The video data that are available have chronological information superimposed on them. In particular, there is a stopwatch mode with 10 ms resolution that is initiated by the same triggering method used for detector acquisition. This video timer allows correlation between visual observations and detector signals.

#### 2.3.1.6 Data Acquisition System (DAS)

The DAS consists of those items schematicized in Figure 15. The detector d.c. voltage is gathered using a waveform digitizer from Soltec, model SDA 2000. This is a 12-bit, 8-channel, 10 MHz machine which is programmable from the host computer. The Ircan pyrometer d.c. voltage is digitized for the host computer by a Hewlett-Packard 3472A digital multimeter. Both digitizers require an IEEE488 interface to the host computer: an Iotech and a National PCII host computer card are used.

The host computer is an IBM/AT whose hardware consists of the two IEEE488 cards mentioned above, an Intel Inboard 386 with piggyback RAM for a total of 3 Mbytes, a Sysdyne EGA graphics board, and a parallel/serial output board. The software to control the hardware is provided by Quarterdeck's Desqview and their QEMM 386 device-driver software. This multi-tasking software provides windows to the Soltec operating software, the pyrometer software written in Asyst language, and Reflex database software for storing managerial and technical drop information for later statistical analysis.

Peripheral equipment consists of an Epson serial printer, an Axiom TX2000 video printer, a Sysdyne EGA color monitor, and an N/Hance write-once-read-many (WORM) optical mass storage drive.

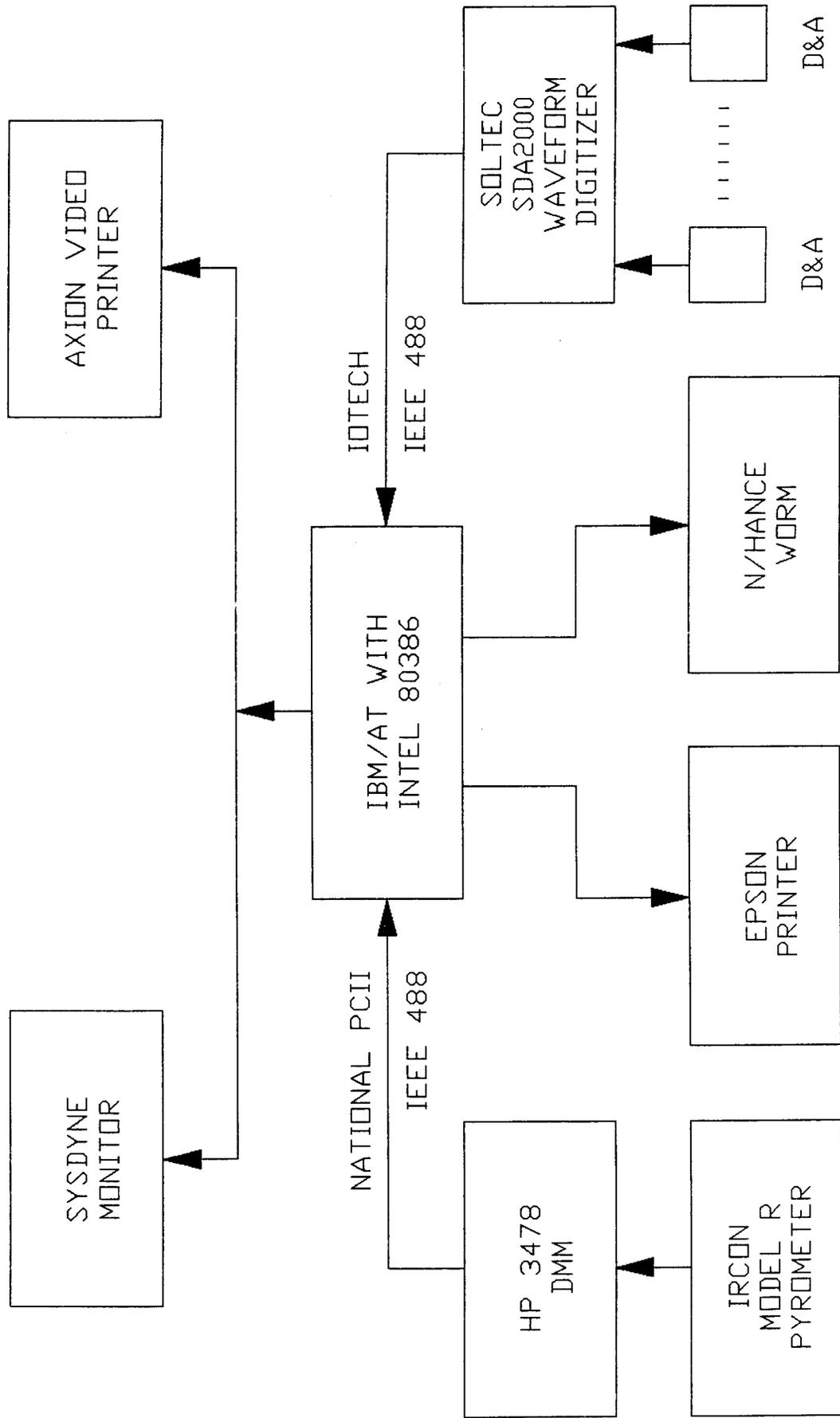


Figure 15. Data acquisition system schematic.

D&A - DETECTOR AND AMPLIFIER

### 2.3.1.7 Data Display/Analysis

Immediately after the drop, analysis can be performed on the pyrometer and detector data as they are displayed on the IBM's monitor. The Soltec software allows simultaneous viewing of each detector's data as well as automatic calculations (such as ratioing two channels) and data manipulation. The capability exists to import the detector data to the Asyst software for further analysis such as FFT. The pyrometer data are similarly displayable on the monitor using the Asyst software. These plots are then hard-copied on either the slow Epson printer (5-minute plots) or the Axiom high speed video printer (5-second plots). Typical plots of the pyrometer data and the detector data are shown in Figures 16 and 17, respectively.

### 2.3.1.8 RGA Display

The residual gas analyzer has four display modes from which to choose: analog, bar, tabular, and degas. The analog mode is typically used to transmit output to a screen from which a hard copy can be obtained. A typical spectrum of the RGA chamber and the belljar is shown in Figure 18. Typical features of the RGA include background subtraction and monitoring specific species of gas if quantitative analysis for those particular species is needed.

### 2.3.1.9 Data Storage/Availability

There are several media available for externally storing experimental data: 356 K and 1.2 Mbyte floppies, and a 240 Mbyte WORM optical cartridge. The magnetic media provide compatibility with most investigator's computers in their labs where further data analysis can be performed. The optical storage media provides a means of archiving the data at the Drop Tube for later retrieval. If required, a modem link can be established to allow more immediate transfer of the data.

### 2.3.1.10 Data Propriety

All the data gathered at the Drop Tube will be proprietary if requested by the investigator.

## 2.3.2 Pre-Processing Requirements

### 2.3.2.1 Sample Size

The EM levitator has a minimum coil I.D. of 3/8 inch. This is the limitation imposed by the 1/8-inch-diameter copper tubing used to wind the coils. Thus, the maximum drop diameter must be less than 3/8 inch for this smallest coil size. However, there is no upper limit to the size of a sample that can be processed if larger copper tubing is used to make larger coils; the only practical limitation is the cooling capability of a large drop in the 4.6 seconds of free-fall time. The smallest sample size is typically 2 mm diameter. However, much depends upon coil configuration and the type of material being processed. In this regard, the EM levitation technique is still a research furnace. In general, the best sample size for levitation and heating is one that fills the coil cross-sectional area as much as possible.

Cooling considerations for a sample in free-fall are left to the investigator and are highly recommended before making drops. This action will limit the choices of processing variables and thus save much time and samples when processing begins in the Drop Tube. Similar cooling considerations are left to the investigator for the EB furnace

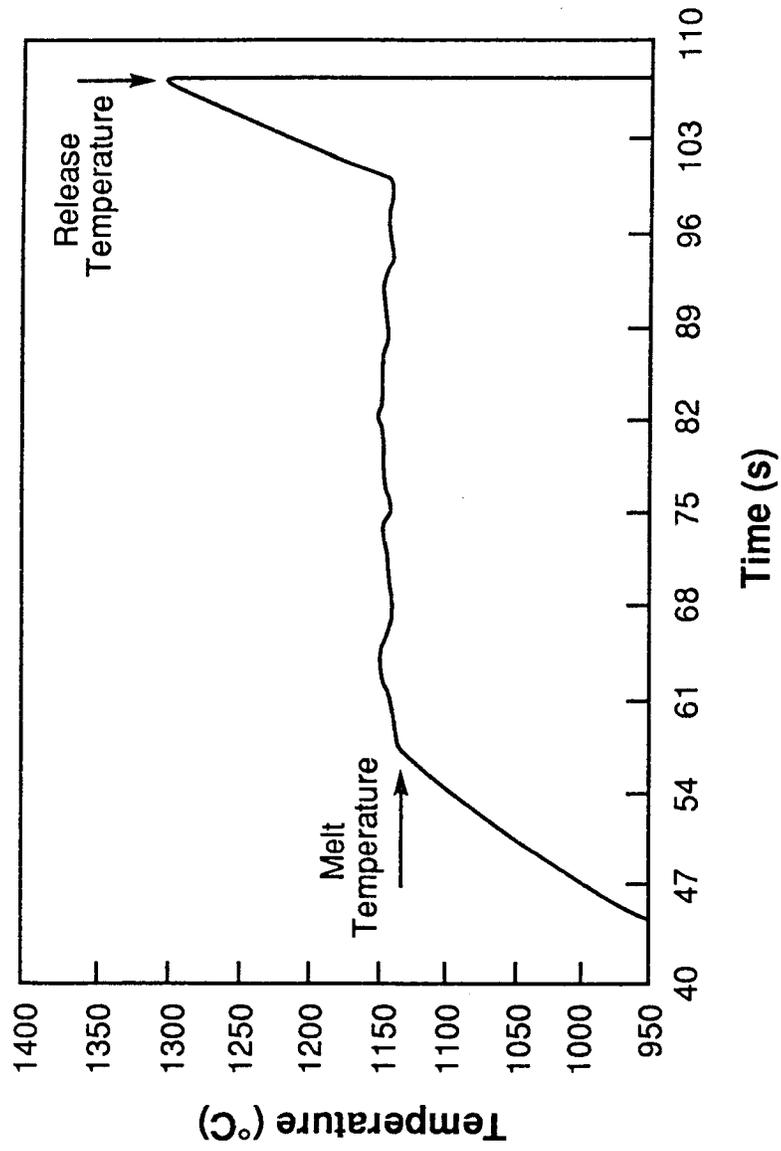
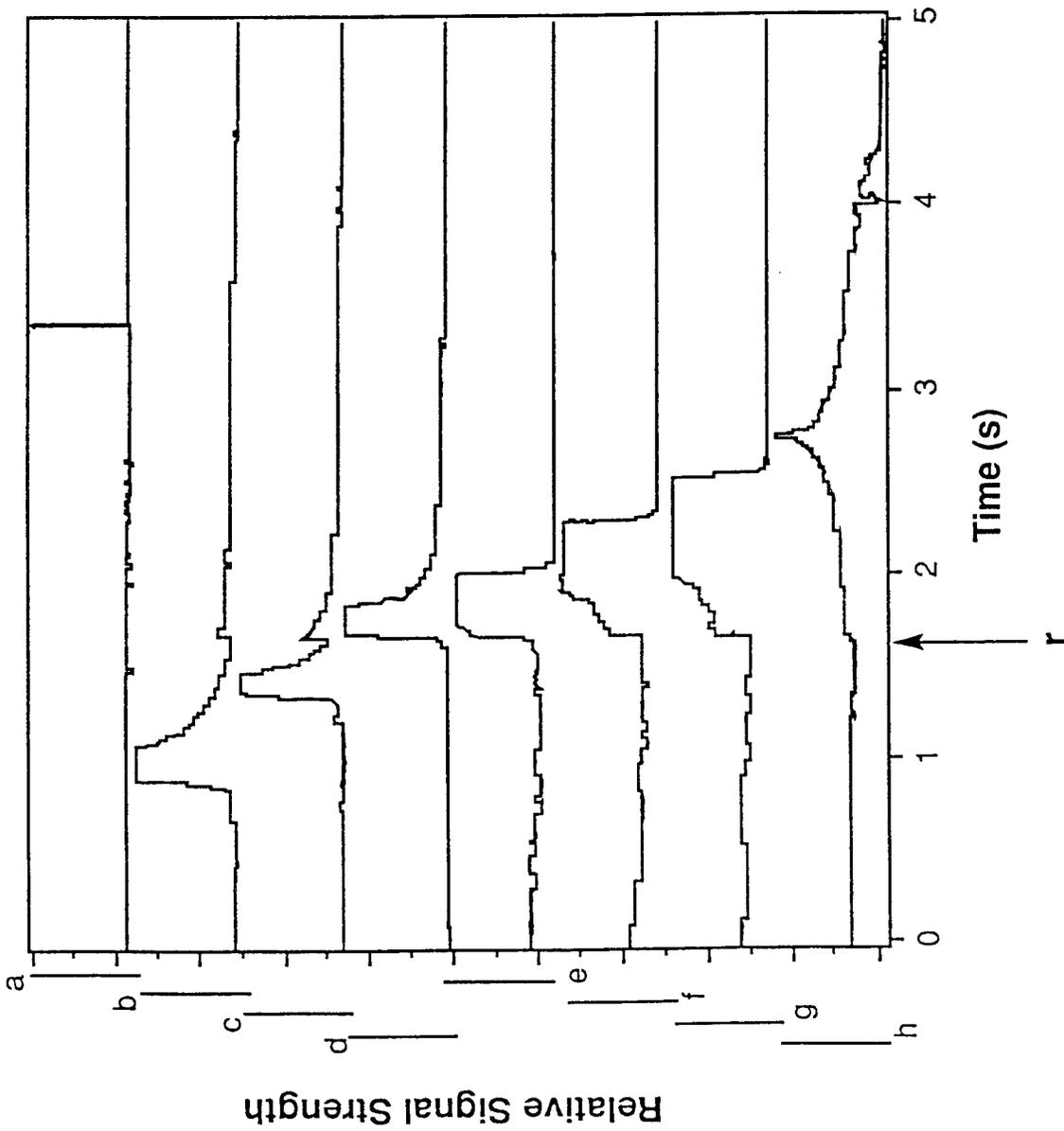


Figure 16. Typical pyrometer data plot.

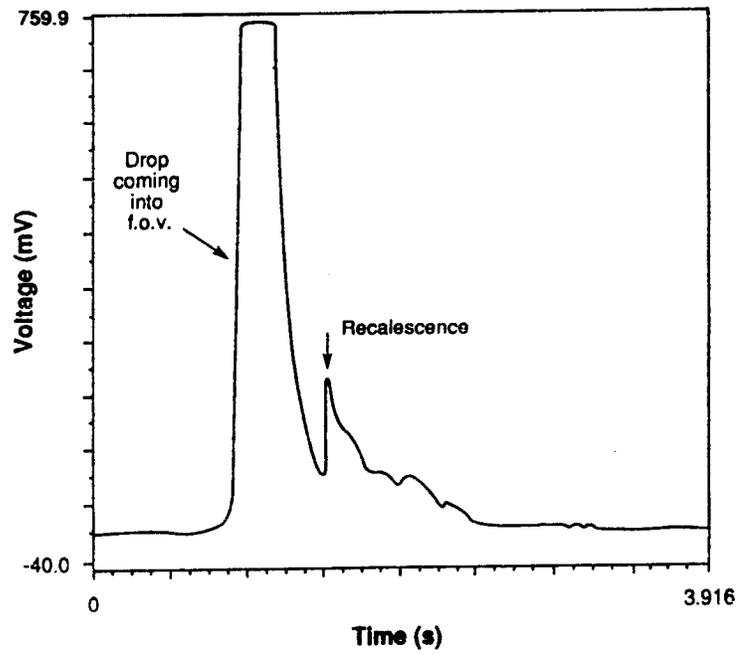


(A)

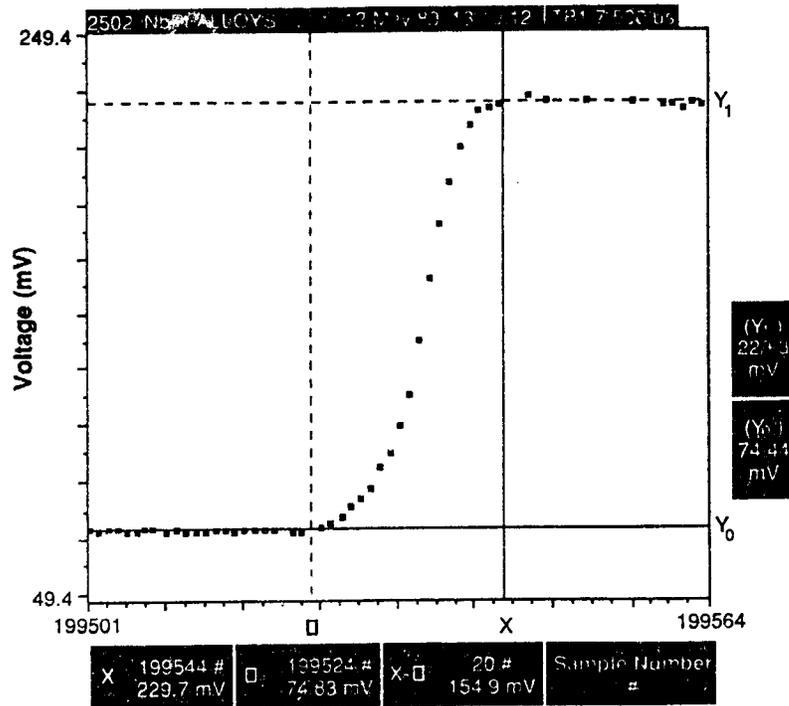
Legend:

- a = Timer Detector on level 5.
- b, c, d = Down-Looking Detectors on levels 14, 13, 12.
- e, f, g = Up-Looking Detectors on levels 11, 10, 9.
- h = Up & Down-Looking Logarithmic Amplifier/Detector on level 8.
- r = Recalescence Event

Figure 17(a). Typical detector data plot showing signal on each channel.



(B)



(C)

Figure 17(b). Expanded view of channel c; (c) expanded view of recalescence showing the initiation ( $\square$ ), duration ( $x - \square$ ), and signal strength ( $y_1 - y_0$ ) of undercooled solidification. The x-axis is in units of number of data points and the time base is  $7.5 \mu\text{s}$  per point.

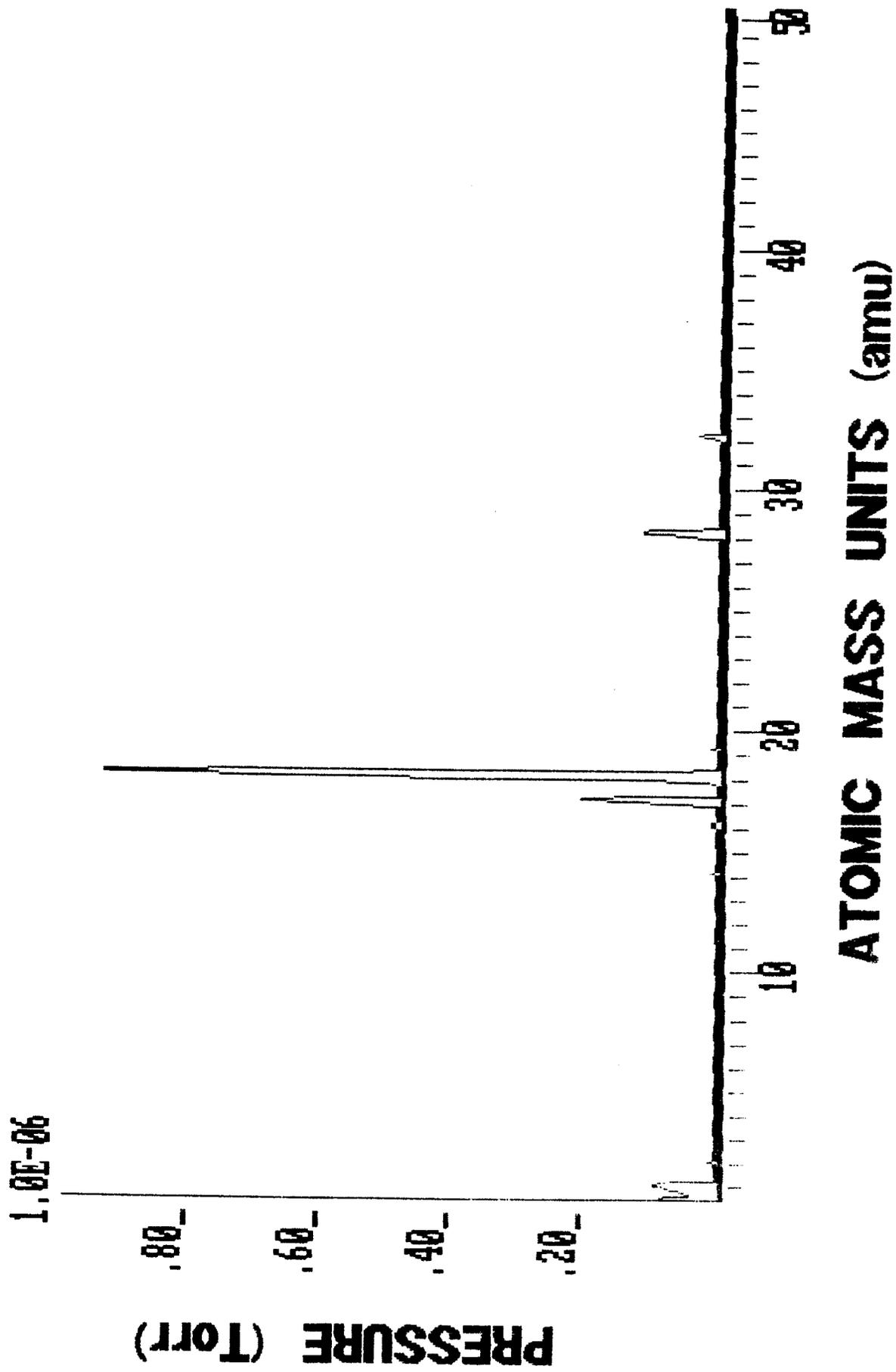


Figure 18. Typical RGA data output.

which must always process samples in a vacuum. If samples are melted directly from the ends of wires or rods, then the size of the drop will be determined by the liquid-to-solid surface tension and the mass of the drop. If samples are hung from wires, there is no limitation on sample size as long as reactivity of wire and molten sample is considered.

The reactivity consideration is also true for the EM levitation using wires as the sample insertion technique instead of the pedestal arrangement. Care must be taken to assure that the sample is either large enough to fall off of the wire when the power is shut off or that the wire will extract manually from the levitated molten sample. In both the EB and EM wire techniques, the investigator must be reasonably sure of sufficient overheat to assure there are no solid particles in the sample to act as nucleation sites which would prevent undercooling, unless so desired.

The EB furnace samples are limited to less than 1 inch diameter because of the 1-inch tungsten-filament loop used for electron-irradiating the sample. The sample size will also be limited by the heating limitations of the EB power supply. When the sample/wire technique is used, the stiffer the wire the better; this will help prevent electrostatic attraction between the sample and the grid/filament which will pull the two together and short out the power supply.

#### 2.3.2.2 Sample Shape

There is no requirement for sample shape for the EB furnace except that the wire-to-sample attachment point should be as close to the top of the sample as possible since the sample melts from the bottom upward. A commonly used technique is to use an electrodischarge machine (EDM) to drill a hole in the sample and to suspend the sample from the hole with a wire.

The EM furnace has a more stable levitation with samples that have aspect ratios as close to one as possible; spheres are preferred. However, one disadvantage to EM samples with no distinguishable edges is the lack of a visual indicator when melting occurs.

#### 2.3.2.3 Sample Vapor Pressure

The sample vapor pressure is very important to the processing procedure no matter which furnace is used. If processing is done in a vacuum or near-vacuum, vaporization of the sample will coat optics and insulators. The latter could cause voltage breakdown and arcing within the coil (EM) or between the filament/grid (EB). If vapor coating of optics is a problem, a rotary window device can be placed inside the belljar to allow rotating a fresh piece of glass in the optical path of any instrumentation.

At partial pressures in the belljar, a sample that exudes lots of vapors will form a plume in the EM coil. Since the plume will rise upward from convection, the optical pyrometer will have its line-of-sight blocked by the smoke. This is a problem that cannot be avoided unless another pyrometer optical path is constructed.

#### 2.3.2.4 Sample Cleanliness

Samples are expected to be cleaned by the investigator to his requirements before being processed in the Tube. Most surface organic contaminants will be burned off the surface as the sample is heated. If required, various acids and solvents are available at the Drop Tube for a final cleaning.

### 2.3.2.5 Sample Physical Properties

Both furnaces require samples to be metallic for their electrical conductivity. However, it has been reported that semiconductors and some ceramics have been made electrically conducting by a secondary heating source until the usual furnace methods (EB, EM) can be applied. One material in particular that has always been a problem for stable levitation in the EM furnace is ferromagnetics because of the strong coupling between the sample and the EM field and the changes of this property as the temperature of the sample rapidly increases.

Knowledge of the sample's spectral directional emissivity is only important for the Ircon pyrometer if the ratio of the emissivity in the range of 0.7-1.8  $\mu$ m to the emissivity in the region of 1.8  $\mu$ m changes drastically during the course of the processing. These wavelength ranges are those used by the two detectors in the pyrometer. The total hemispherical emissivity, however, is important for cooling calculations. The accuracy of the pyrometer can be increased if a calibration sample having known thermophysical properties is used to correct any pyrometer uncertainties.

### 2.3.2.6 Safety

All aspects of the processing procedure should be considered with safety in mind. Particular aspects of the process to consider are those involving the power supplies of the furnaces, the toxicity of the sample residual vapors, and the eye-damaging brightness of high temperature samples.

## 2.3.3 Sample Integration and Checkout

### 2.3.3.1 Laboratory Test Bench

A 15 kW Lepel, model T-15-3-DF-E-S is available in the laboratory adjoining the Drop Facility building for testing EM coils and levitation techniques. This facility has the same vacuum and backfill capabilities as the Drop Tube belljar so that problems such as vaporous samples can be solved before attempting actual drops in the Tube. New furnaces and/or sample insertion techniques can be developed with minimal impact on the Tube resources.

As useful as this test bench is, it must be remembered that there are enough significant differences in the impedances and power levels between the laboratory Lepel and the Tube Lepel to affect the operating frequencies, levitation/stability, and heating capability when attempting to convert from one to the other. It is thus recommended that the laboratory Lepel be used only as a rough approximation tool and that the Tube Lepel be used to make fine-tuning adjustments.

### 2.3.3.2 Equipment Test Specimens

Because detector amplifiers and the processing atmospheric conditions are usually unique to the particular samples being studied, it is prudent to have many calibration/test specimens available. These specimens should be identical to or close to the actual experimental materials. These test samples can then be used to assure the furnace's capability to process experimental samples as expected and, when dropped, to assure the triggering and detector/amplifier settings of the system. If possible, these test specimens should be a pure element, preferably one of the components of the alloy system under study. This elemental sample would then also serve as a pyrometer calibration point when monitoring the melting temperature equilibrium point. Some elemental

materials are available at the Tube Facility and can be used if requested.

#### 2.3.4 Sample Retrieval Hardware

Once a drop is made, there are various devices that can be used to stop and retrieve the sample at the bottom of the Drop Tube. These devices are shown in Figure 19. Each device is made for specific quenching requirements and for expediency of retrieval. All of these devices fit into a flanged catch tank similar to that of Figure 2.

##### 2.3.4.1 Solid Samples (Spheres)

###### 2.3.4.1.1 Quick-Retrieval Catch Tank (QRCT)

One mechanism for retrieving a whole, solid sample from the bottom of the Tube is the QRCT. This device fits into a flanged catch tank and consists of a funneled copper plate which directs the solid specimens into a 1 3/8-inch diameter by 2 1/2-inch long stainless steel cup. The cup is then isolated from the entire Tube by a 2 3/4-inch conflat flange mounted butterfly valve. The QRCT allows a 5-minute retrieval time.

###### 2.3.4.1.2 Ceramic Cushioned Catch Tank (CCCT)

If the impact at the Tube bottom causes the samples to shatter or induces unwanted stress in the samples, there are various ceramic cushioning materials that can be added to the catch tank. Care must be taken to avoid post-processing contamination from the cushioning ceramic. Niobium foil can be laid on top of the felt to provide some protection from the felt contamination and also to provide some impact shock absorbency.

##### 2.3.4.2 Liquid Splat Samples

A catch tank is available which uses a polished copper plate upon which to splat samples. The plate is a flat plate cut into an elliptical shape so that it will fit into the catch tank at a 45° angle.

To minimize the loss of materials upon splatting, a funnel-shaped copper plate is also available to direct all splatted and fragmented pieces into a small copper cup. The entire catch tank must be removed to retrieve the sample, just as in the case of the angled flat copper plate described above. Because the funnel-shaped copper plate is not flat, the sample foils tend to have a curvature to them when retrieved. This curvature should be considered if post-processing analysis such as TEM or SEM is to be performed.

##### 2.3.4.3 Liquid-Quenched Samples

Some investigators may require a liquid into which to drop their samples for greater quenching rates. A previously used liquid was Dow Corning 704 silicone diffusion pump fluid. Much greater pump-down times are required between drops to degas the oil, unless a quick-retrieval mechanism were to be built which avoided the removal of the entire catch tank. Other quenching liquids could be used, such as room temperature liquid metals. Once again, sample contamination by the liquid or its breakdown by-products should be considered. Additional facility problems such as liquid splashing up the Tube walls will occur also.



Figure 19. Various catch tank devices: (a) flat plate tank, (b) quick-retrieval tank, (c) silicone oil quench, (d) copper splat funnel, (e) centered viewport tank.

#### 2.3.4.4 Sample Containers

Small 1 1/2 inch O.D. by 1/2 inch depth Poly-cons plastic containers are available and normally used for storing the retrieved samples. Other containers can be supplied if desired.

### 2.4 Operational Aspects

#### 2.4.1 Support Personnel

Available to the user for Drop Tube operations are on-site personnel from MSFC and the UAH contractors. Both parties have access to supplies, equipment, or services from their respective institution or from local businesses. Drop Tube personnel and thus the operational daily hours are typically from 8:00 a.m. to 4:00 p.m. Monday through Friday. Longer daily work hours need to be requested well in advance to allow authorization of overtime.

#### 2.4.2 Scheduling

Use of the Tube can usually be scheduled 2 weeks in advance at the minimum. Formal communications should be carried out between the investigator and Dr. Mike Robinson of Marshall Space Flight Center (205-544-7774). Informal operational and scheduling communications are between the user and the UAH Facility Manager. The Facility Manager will determine when the best time is available for at least a week's length of uninterrupted processing, thus maximizing the successful production of drops by operating the facility while everything is "up and running."

Occasionally, the adjacent Drop Tower Facility will have experiments needing to be performed in the same timeframe as the Drop Tube. Because the support personnel run both facilities and because the Tower drops are much more involved, some down-time of the Tube may occur to accommodate the Tower drops.

#### 2.4.3 Investigator/Operator Communications

A prerequisite for any successful experiment in the Tube is good communications between the investigator and the Drop Tube operator. If questions still arise after reading this document as to the appropriate processing conditions necessary for an experiment, the first person to contact is the MSFC resident Drop Tube science advisor, Dr. Michael Robinson (205-544-7774). After the appropriate process gases, furnace, carousel, temperatures, etc. have been chosen, the Drop Tube operator is to be informed (205-544-1409). As the experiments are first being performed, it is advisable that the investigator be on-site to witness the actual procedures and to make appropriate suggestions/comments. Thereafter, if desired, the operations can be performed by the Tube personnel and any questions, samples, or data can be transmitted to the user at his/her research center. A responsibilities flow chart is presented in Figure 20 to help the investigator understand the typical scenario of getting an experiment performed.

#### 2.4.4 Sample Preparation

All samples are to be prepared by the investigator at his/her research center. Some facilities exist within MSFC for preparing samples, but this course of action is solely the responsibility of the investigator and his MSFC contact.

# Drop Tube Experiment Flowchart

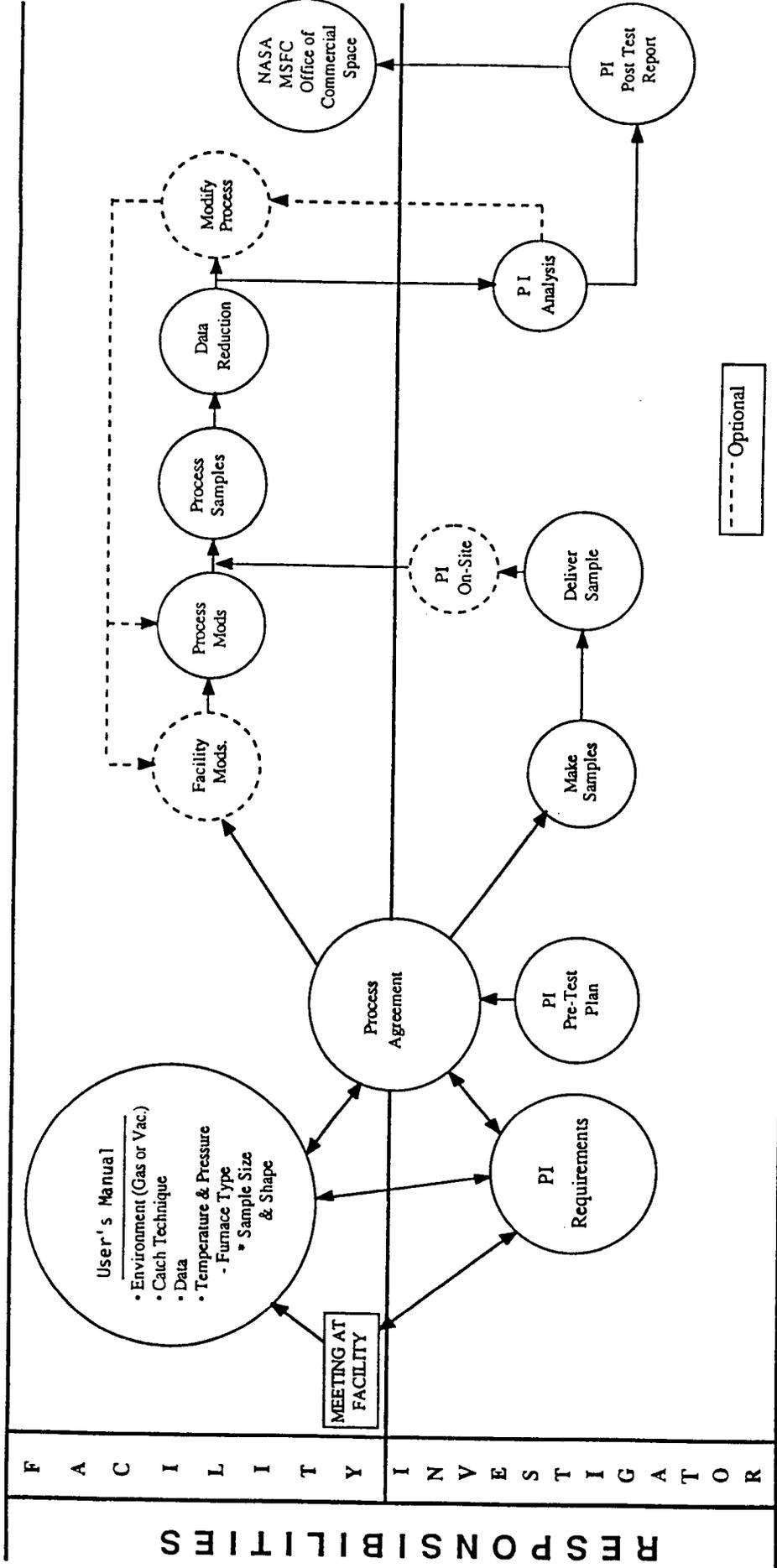


Figure 20. Drop Tube experiment flowchart.

#### 2.4.5 Maintenance and Repair

The Drop Tube support personnel have the responsibility for maintaining and repairing all facility-provided hardware. These people will be available to work on problems with user provided hardware if desired.

#### 2.4.6 Processing Operations

The user is encouraged to participate in any and all aspects of the processing operations at the discretion of the Tube operator. This may include operating the sample carousels, the optical pyrometer, the DAS, the furnace, the retrieval process, etc. The only operation not available to the user is operation of the isolation valve panel in the level 15 control room. More information on specifics can be found in the Drop Tube Operational Procedures and Specifications Document mentioned in section 1.1.

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## APPENDIX

### DROP TUBE SAMPLE TEMPERATURE DETERMINATION

Users of the MSFC Drop Tube Facility who perform materials processing experiments require the temperature of their material as it is being processed. Since the Drop Tube provides containerless processing capabilities, the temperature of the samples must be obtained via a noncontact measurement, i.e., infrared optical pyrometry. The processing technique consists of two stages: heating/melting and cooling/solidification. Each stage has its own temperature versus time data acquisition technique.

The heating/melting stage uses an Ircon two-color optical pyrometer to acquire the temperature data. These data are acquired with relative ease: the IRCON voltage output is fed into a voltmeter which digitizes the information for later computer comparison to calibration curves and eventual plotting. Any errors associated with the experimental arrangement such as windows, sample emissivity, or electronic problems can be greatly reduced by observing the melting temperature of a known material, normally pure elements. Because the sample is restrained from falling down the Tube until the heating/melting stage is complete, the Ircon pyrometer can be focussed on the sample at a fixed distance and acquire temperature data until the moment the sample is released into the Tube. Figure 14 is an example of typical data.

The cooling/solidification stage does not presently have a means to directly measure the temperature of a sample as it is falling somewhere within the 10 1/2 inch cross-section of the Tube. The Drop Tube is also presently limited to samples with melting temperatures above 1200 °C because of the lack of operational photo-sensitive detectors attached to the Tube for samples having a free-fall temperature below 800 °C. These photo-sensitive devices provide an output voltage that is proportional to the incident radiation upon them from the falling sample. Because the temperatures cannot be extracted directly from these voltages due to unpredictable Tube wall reflections, Tube wall intrinsic background radiation, and other optical filtering problems, the investigator must depend upon calculated values of the temperature of the droplet during free-fall.

As with any heat transfer calculation, an initial time ( $t_o$ ) and temperature ( $T_o$ ) and a final time ( $t_f$ ) must be given to be able to calculate a final temperature ( $T_f$ ). The initial conditions are found from the last temperature data point measured by the Ircon from the heating/melting stage. This last data point is time  $t_o = 0$  as the sample begins its fall into the Tube. The final time needed for the calculation can be picked to be any time up to and including the moment of impact at the bottom of the Tube. The final times used by investigators are the time of impact, the time of recalescence, and/or the time from a timing detector located at a measured distance from the release point in the belljar (see Figure 1). The recalescence time is that moment when an undercooled liquid begins solidification whereupon it rapidly releases its latent heat of fusion causing the drop temperature to rise and thus give off a flash of light. Figure 15 shows a typical computer output of this infrared detector data. Knowing  $t_o$ ,  $T_o$ , and  $t_f$ , the final temperature  $T_f$  can be calculated from the standard heat loss equation:

$$dQ/dt = -\epsilon A\sigma(T^4 - T_o^4) - hA(T - T_o) \quad (1)$$

This equation includes both radiation and convective heat losses for cases when a user may wish to use a gas to help cool the sample while it is falling in the Tube. A closed-form solution to this equation has been described by Robinson<sup>\*</sup>; a more detailed iterative solution can be obtained upon request. A typical set of curves for pure niobium are shown in Figure A-1. As with all calculations of this type, large uncertainties are inherent due to the values of measured thermophysical properties of the materials under consideration.

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<sup>\*</sup>Robinson, M. B., "Radiative and Gas Cooling of Falling Molten Drops," NASA TM-78189, 1978.

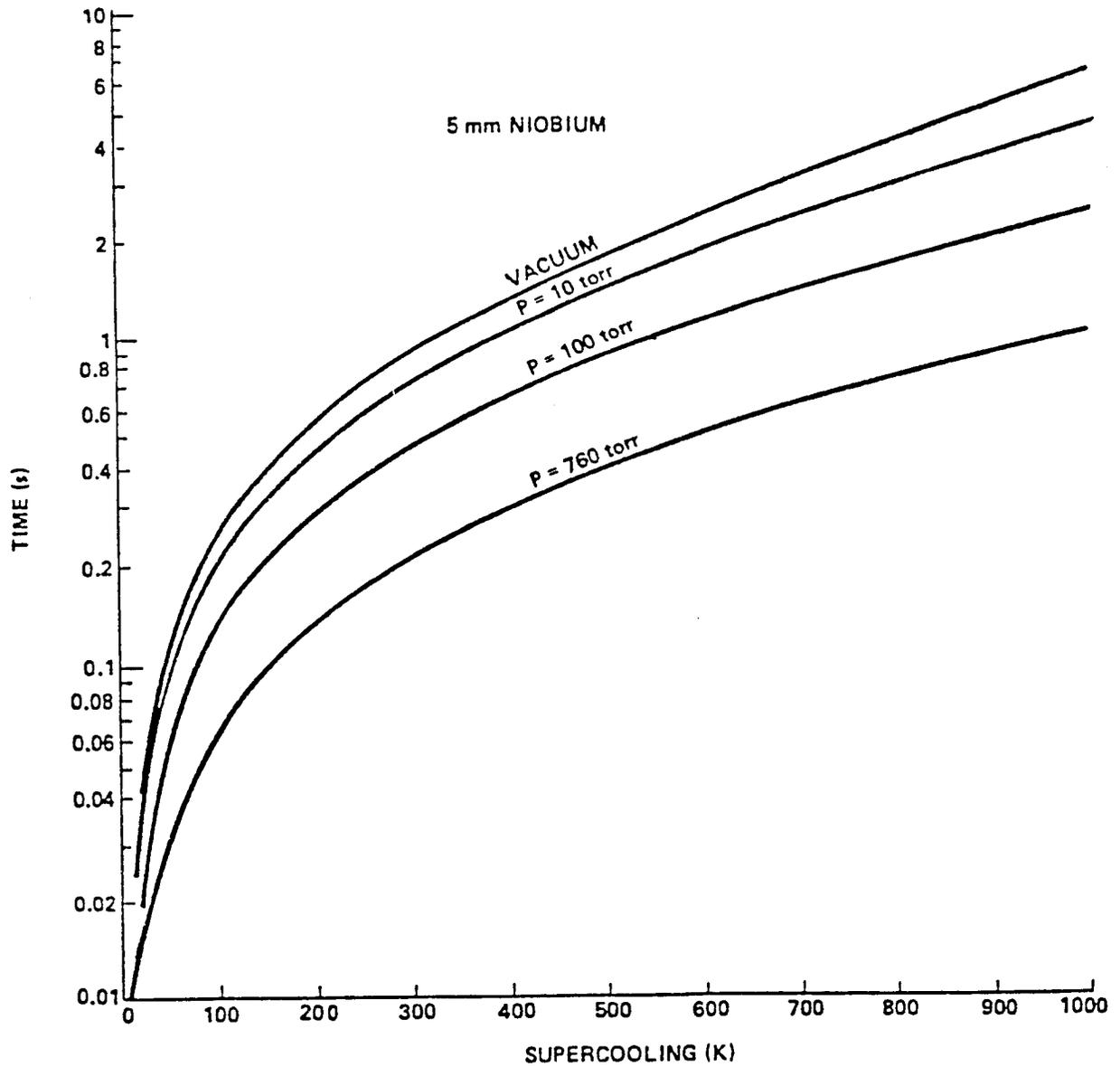


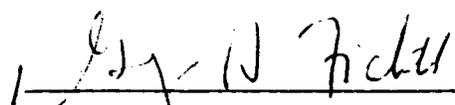
Figure A-1. Amount of supercooling for a 5-mm-diameter niobium sphere plotted as a function of time before nucleation. (The calculations are for a molten drop falling in a vacuum or helium gas at different pressures.)

APPROVAL

THE NASA/MARSHALL SPACE FLIGHT CENTER DROP TUBE USERS MANUAL

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The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

  
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