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*Vacuum Thermal Stability  
of Explosives Monitored Electronically*

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**MASTER**

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completed, the final height, temperature, and barometric pressure are measured, and another STP volume is determined. The total volume of gas evolved is calculated as the difference between the start and final volumes.

In order to continuously monitor and calculate the evolution of decomposition gases, a method was developed to do this automatically by electronic monitoring.

## **B. Purpose and Scope**

The purpose of this report is to summarize the development of an electronic transducer manometer tube, a method of calibrating it in place, a method of back pressurizing the manometer to sea level, electronic monitoring of the mercury column, and periodic data acquisition with computer control.

The scope will include a description of the current modular instrument, some of the problems solved in its assembly, conformance to requirements of the DOE Explosives Safety Manual,<sup>4</sup> analysis of errors of measurement, and some typical results. The calibration and experimental procedures are detailed in the Appendices for use as an operating manual.

## **C. Traditional Methods**

Vacuum thermal stability has long been used to determine the stability of explosives alone and to measure their compatibility with other materials. The early methods are described in various ordnance manuals,<sup>1,2,3</sup> reference encyclopedias,<sup>5</sup> specifications, and reports.<sup>6</sup> The test methods described all use some form of ruler to measure the manometer height at the start and end of a test. The gas laws are applied, and the amount of gas evolved is calculated manually.

At Los Alamos National Laboratory (LANL), various improvements were made to continuously collect the data using a strip-chart recorder actuated by a mechanical follower system. The system worked but was subject to mechanical breakdown and the calculation was still done manually. An electronic system was needed to measure the manometer and temperature, and to monitor the system more frequently for intermediate reactions. This objective has been achieved and is reported here.

## **D. Modifications**

Our early versions of the electronic manometer sensing system consisted of a trough in a micarta lath board lined with 10-mil aluminum foil, into which a plain glass manometer was fitted. The foil constituted the outer plate, the mercury was the variable inner plate, and the glass was the dielectric. The assembly acted as a capacitor when an electrode was placed in the mercury pool and another connection was made to the aluminum plate. Each lead was connected to a dedicated power supply/demodulator/meter (Validyne CD-23\*<sup>7</sup>). The capacitance of the manometer was linear with the mercury column height. This capacitor-transducer was made part of a Wheatstone capacitance bridge modulated by the power supply and the signal generated was demodulated to a dc voltage for transfer to a data logger. The output from the CD-23 was a signal in the 0- to 10-V range. This was enough to devise a nonmechanical, all-electronic, nonmoving system.

To make the measurements for calibration, a method was devised to move the mercury up and down the tube with precision.

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\*Mention of a trade name or manufacturer does not imply that Los Alamos National Laboratory or the University of California endorses the brand names and product.

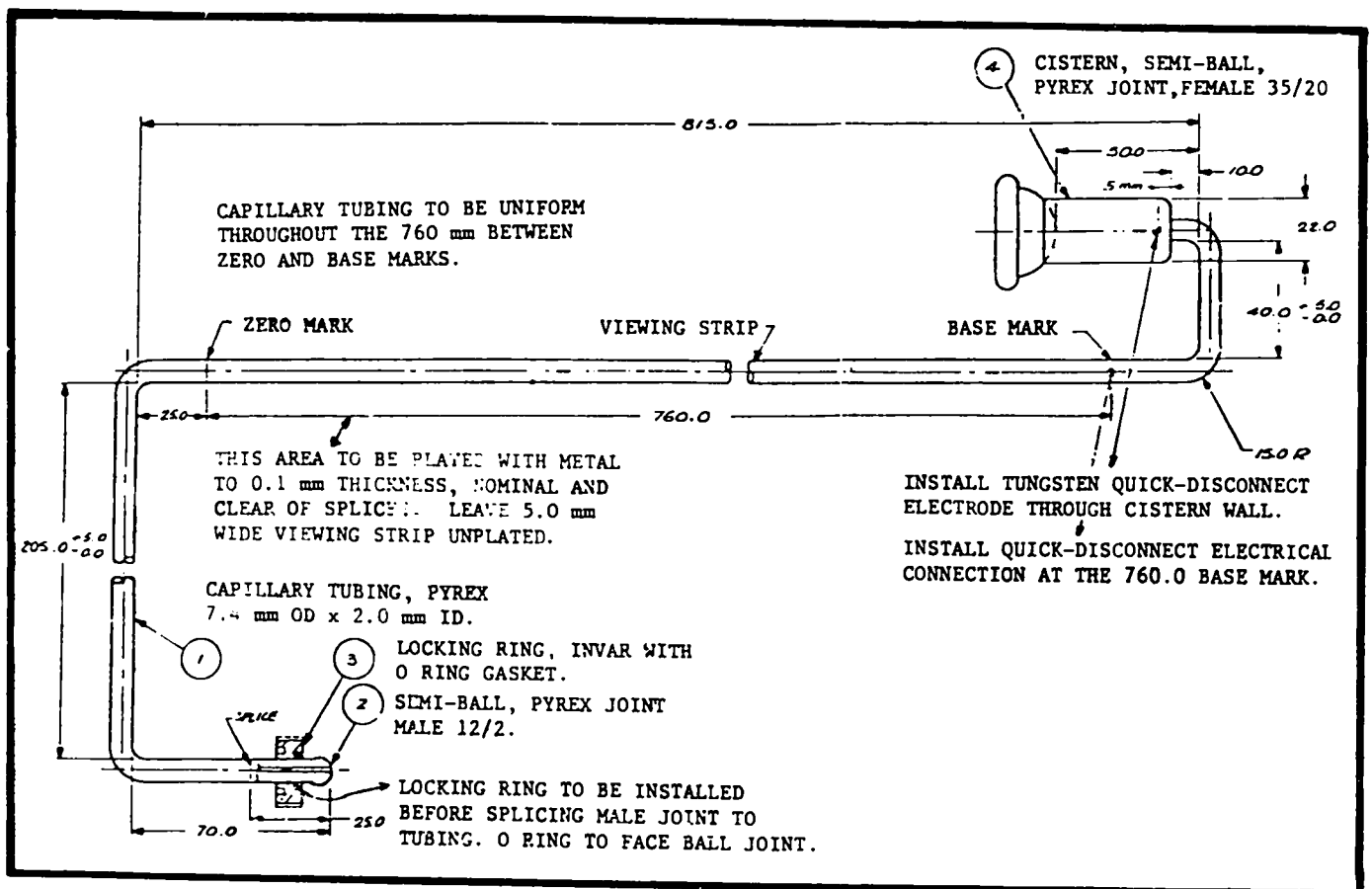
A typical air-pressure-reducing valve (PRV) (Nullmatic pressure regulator, Model 40-30, Moore Products Co.) is sensitive enough to move the mercury by fractions of a millimeter. With the air-back-pressure assembly in an adjustable mode, it was possible to alter the capacitance value when the mercury was forced up or down. This modification made in-place calibration possible.

To get better electrical coupling between the inside and outside plates, aluminum foil was glued to the manometer over the central 760 mm of tube length. This tighter capacitance coupling improved the signal output.

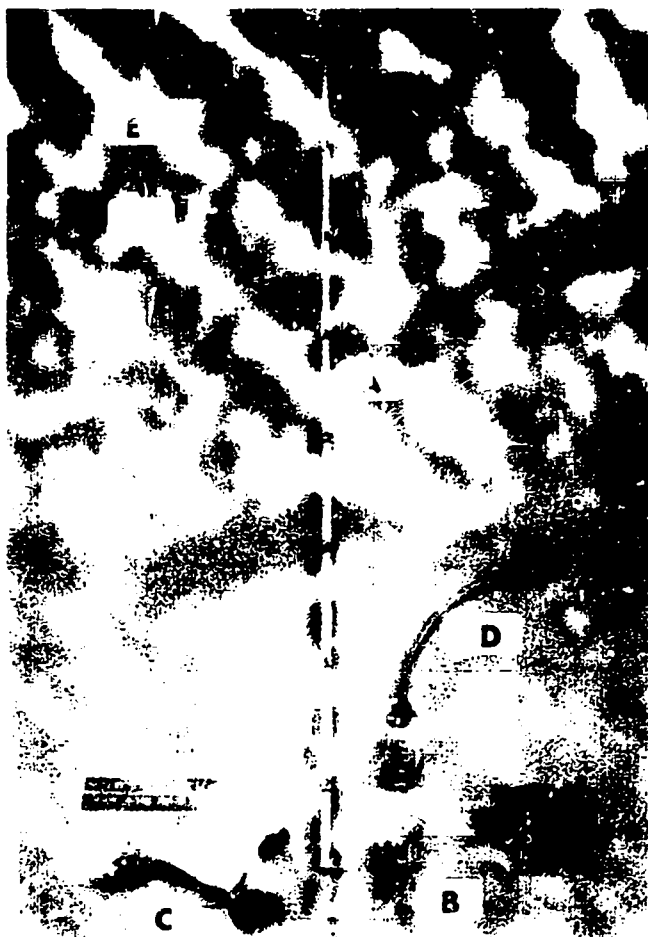
Rather than depend on adhesives to hold the aluminum, we looked for a way to fuse the metal to the glass to make a more reliable

tube with better capacitance coupling. We found that the Ace Glass Company (Vineland, New Jersey) had a proprietary process called "Instatherm" for fusing metal to the outside of glass flasks and large-diameter columns. This fused metal acts as a heating element in their products. A call to their development engineers confirmed that they could fuse metal to custom measurements on a 7-mm tube and make the appropriate connectors so the tube could be used as a capacitor. They custom-built several dozen of these tubes to our specifications (Figs. 1 and 2). These tubes were more rugged and were incorporated into our system.

With these innovations, it was possible to combine the electronic measurements of the



**Fig. 1.** Vacuum stability tube with metal coated on glass, then insulated and electrodes attached.



**Fig. 2.** Disassembled VTS tube (A) including electrodes (B), BNC connector (C), back-pressure assembly (D), test bulb (E), and Invar locking rings (F).

manometer tube with a data control/computer to provide an hourly (or other regular time interval) point-by-point record of evolved gas at ordinary test temperatures and at much higher temperatures for insensitive high explosives.<sup>5,6,8,9</sup>

## II. THE VACUUM THERMAL STABILITY MODULAR INSTRUMENT

The VTS module, shown in Fig. 3, consists of three parts: (1) the heating system and its controls, (2) the manometer and back-pressure assembly, power supply/demodulator signal system, and (3) the data acquisition/control unit and dedicated computer.

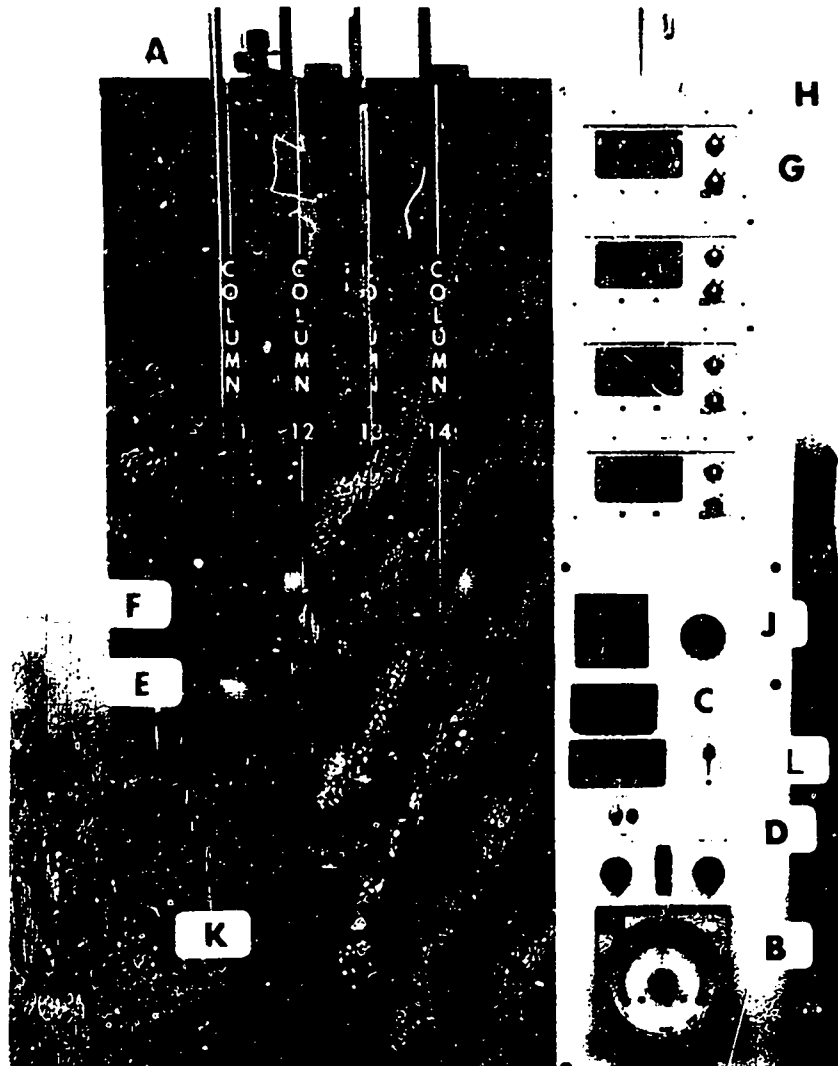
### A. The Heating System

In Fig. 3, an oil-heated bath (A) behind the column panel is controlled by a Dialatrol proportional oil controller (B) in a closed-loop, Chromalox panel thermocouple system. The oil-bath heater can use up to 1100 watts of power. The oil bath is a 4.5-in.  $\times$  12-in.  $\times$  3-in. trough, containing 700 ml of DC-550 silicone heat-transfer oil that is continually stirred. The temperature is displayed on the front panel digital meter (C). To cool the system rapidly, a cooling coil is installed in the heating bath to accept tap water and to return the waste to a drain.

To conform to requirements of the DOE Explosives Safety Manual<sup>4</sup> for redundant power controls, two relays, a controller, a fuse and one thermostic switch are in series with the heater. If there is an overshoot in oil-bath temperature, an electrical interruption, or the oil bath cools, the system shuts down and alarm lights (D) go on. It takes a manual reset to restart the system. The thermostic switch is set 5°C above the test temperature for overshoot control. The alarms are set 6°C above and below the test temperature.

### B. The Manometer and Electronic Measuring System

After loading, evacuating, and sealing the manometers (Fig. 2) they are installed in the column space in the rack shown in Fig. 3. In column 11 (leftmost), the manometer is connected electrically to the system through connector pigtailed (E) and back pressurized by the assembly (F), but the test bulb is not inserted into the bath until in-place calibrations are made. In column 13, the test bulb is in the heated bath, and the assembly is ready for start of the test period.



**Fig. 3.** VTS four-column module showing tubes in place, CD-23 digital output displays (G), temperature (C), pressure display (L), and temperature controller (B) with alarm lights (D). The other lettered items are referred to in the text.

The four Validyne CD-23s (G) are mounted on the right side of the panel and are connected electrically to the corresponding manometer transducers. Two back-pressure systems have their own PRV and controlling relays. Three other relays are in line with the system to indicate ambient, variable, and sea-level pressure from the pressure transducer to the display (L). A DPDT switch (H) to the right of each CD-23 controls the back-pressure air for adjusting the mercury column to calibrate in-place voltage as a function of

height, and then to switch the back-pressure air to 760 mm, the sea-level pressure. Each CD-23 has been modified internally with a variable capacitor in the bridge circuit to eliminate crosstalk between columns that occurs because of the modulating frequency. This is the only modification made to the CD-23. Each manometer must be calibrated and mated to a CD-23. An RC circuit to minimize signal noise is installed at the output of the CD-23 circuit before the signal enters the control unit.

For any column, when the air-pressure switch (H) is in the adjustable mode, the operator can check the output of the system in place by adjusting PRV (J) to move the mercury column to the top (the zero mark) and by adjusting the span on its corresponding CD-23 to the specified voltage. Dropping the mercury 100 mm, and then, 200 mm, and reading the corresponding voltage confirms that the system conforms to the original calibration.

The trough (K) catches any spills or breakage. A clear plastic safety shield (not shown) protects the tubes from accidental bumping.

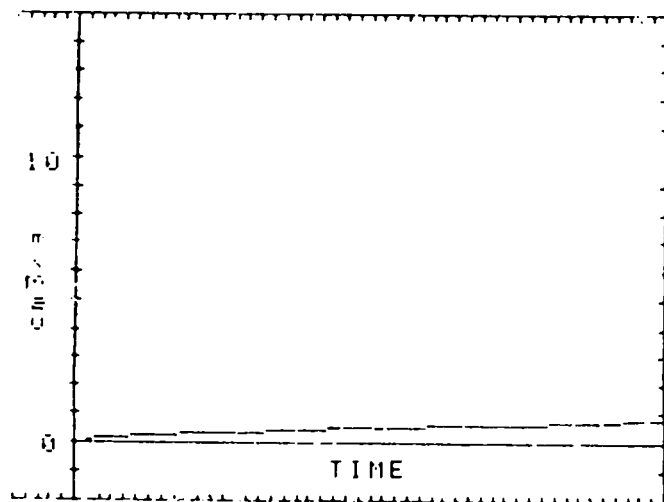
### C. The Data Acquisition/Control Unit, and Computer/Plotter

For data acquisition, a Hewlett-Packard HP3497A Data Acquisition/Control Unit is coupled to a HP85B Personal Computer (PC) (Fig. 4). An Inmac UPS 1000 back-up power module for saving data and operating the computer during power failures is also part of the system.

The HP3497A is the signal processor and timer for the voltage from the CD-23. The input is transferred to the PC by program command. The PC accepts the timed voltages and events from the HP3497A under program



Fig. 4. The data acquisition/control unit and HP85B computer.



SAMPLE ID = CB318  
 PO-PC = CN-20  
 LAB NO. = 20362  
 REQUESTOR = ELR  
 OPERATOR = JON  
 TUBE NO = 3  
 BULB NO = 3  
 SAMPLE WT. = 2058  
 TEST START TIME = 11-03 14:26:46  
 TEST END TIME = 11-05 14:26:47  
 TIME RUN (HOURS) = 48  
 BATH TEMP = 120 Deg C  
 GAS EVOLVED = .77 cm<sup>3</sup>/g

Fig. 5a. Typical printout of a VTS experiment showing an hour-by-hour amount of gas evolved on the plot and a printout of the parameters for the test and the calculated amount of gas evolved.

control, calculates the results at the specified time periods and holds them in memory.

At the conclusion of a run, the PC prints the operating parameters and results in a programmed format, and constructs a plot of gas evolution as a function of time (Figs. 5a and 5b). A PC software program has been written at Los Alamos to monitor the instrument and to solve the gas-evolution equation. This PC program can monitor as many as 14 channels from three modules, store the data, and transfer the data to tape for later analysis. The software consists of a 456-statement program (872 lines) with provisions to store and recall calibration data and to recall specific calibration data for the calculations. Although the program generally works well, it periodically needs to be debugged because of unforeseen malfunctions and operator miscues. Therefore, no detailed listing is given in this report.

As computers and program languages frequently become obsolete, the next generation computer for this system could be a general small personal computer with a signal-control unit that can use any of the better commercial software packages available on the market today.

Sample ID = CB318  
 Test start time = 11/03 14:26 46

Time (hrs)	Gas Evolved (cm <sup>3</sup> /g)
1 00	8806
2 00	1169
3 00	1448
4 00	1670
5 00	1913
6 00	2118
7 00	2254
8 00	2479
9 00	2700
10 00	2906
11 00	3103
12 00	3251
13 00	3350
14 00	3484
15 00	3586
16 00	3728
17 00	3842
18 00	3985
19 00	4174
20 00	4342
21 00	4504
22 00	4651
23 00	4804
24 00	4991
25 00	5085
26 00	5193
27 00	5250
28 00	5335
29 00	5416
30 00	5553
31 00	5672
32 00	5763
33 00	5888
34 00	5944
35 00	5977
36 00	6069
37 00	6154
38 00	6253
39 00	6326
40 00	6440
41 00	6520
42 00	6731
43 00	6951
44 00	7067
45 00	7235
46 00	7369
47 00	7564

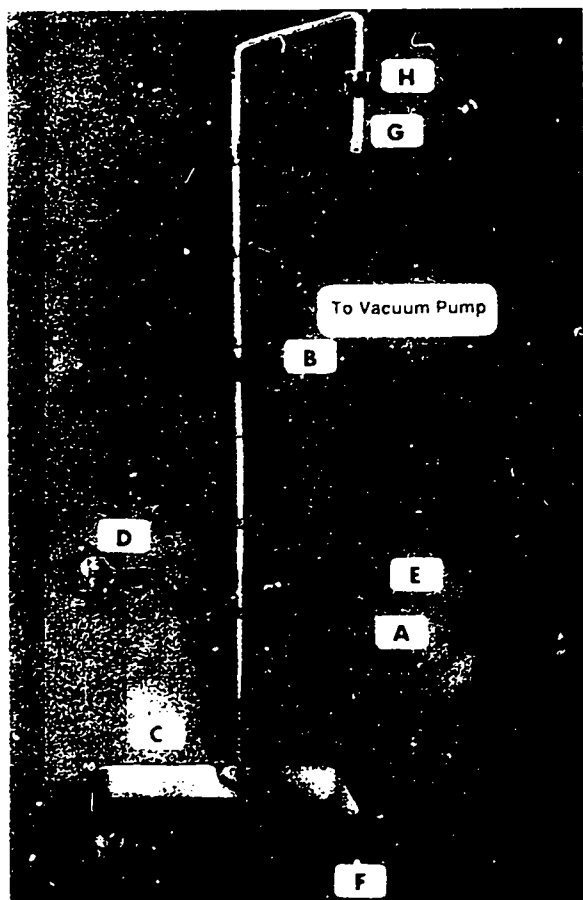
Fig. 5b. Typical listing of the gas evolved hourly from which the plot in Fig. 5a is made.



## D. The Vacuum Pumping System

To prepare a VTS manometer, a manifold fixture (A) (Fig. 6) is mounted on a lab rack connected with tubing (B) to a vacuum pump. The manifold has provisions to clamp the manometer at the cistern (C) end. The system is pumped down to 25- $\mu$ m Hg before the flask containing 6 ml of mercury (D) is turned 90 degrees to allow the mercury to flow into the cistern, thus sealing the sample from the ambient atmosphere. By turning the stopcock (E) and allowing ambient air into the cistern, the column of mercury will rise to indicate the ambient barometric pressure.

The tube is removed from the system by declamping at (C). Then pigtail electrodes are attached along with the back-pressure assembly for installation on the apparatus.



**Fig. 6.** Vacuum pumping system manifold (A), hose (B) to pump (not shown), tube ball-socket clamp (C), mercury flask (D), stopcock (E), drip pan (F), sample bulb (G), and locking ring (H).

## III. DISCUSSION

### A. Gas Evolution Equations and Variable Experimental Data

Because of the fixed test parameters, the equation for calculating the gas evolved for a 48-hour test at a set temperature has been simplified and adapted for computer operation. The volume of the gas in the tube is calculated at the start of each test (time zero) when the system is equilibrated. Equilibration takes five minutes. At the end of the test (48 hours), the volume is calculated using the same equation. The gas evolved is the difference between the calculated volumes at finish and at start, normalized to unit weight and STP.

The equations are:

$$V_A = a (c/d) + e (c/f),$$

$$V_B = a (g/h) + i (g/j), \text{ and}$$

$$G = ((V_B - V_A) (0.359)) / b,$$

where

$V_A$  = volume of gas at start of test after equilibration ( $\text{cm}^3$ ),

$V_B$  = volume of gas at end of test or an intermediate time ( $\text{cm}^3$ ),

$a$  = volume of sample test bulb ( $\text{cm}^3$ ),

$b$  = weight of sample (g),

$c$  = height of column, zero to mercury level at start (mm),

$d$  = oil-bath temperature at start (K),

$e$  = tube volume to mercury level at start ( $\text{cm}^3$ ),

$f$  = room temperature at start (K),

$g$  = height of column, zero to mercury level at end of test or an intermediate time (mm),

$h$  = bath temperature at end (K),

$i$  = tube volume to mercury level at end ( $\text{cm}^3$ ),

- $j$  = room temperature at end (K),
- $G$  = gas evolved in time of test at temperature ( $\text{cm}^3/\text{g}$ ). and
- 0.359 = conversion constant (273/760) to STP.

The correction for sample volume is deleted from the equation because it is nearly constant and added or generated only very small errors. By back pressurizing the tube to 760 mm one mathematical adjustment was eliminated. Since most of the parameters are fixed, the only variables are room temperature and height of column. Calibration constants are required to determine the height of the mercury column and the volume displaced from a voltage reading. The calibration constants do not change from test to test and are stored in the memory of the computer. The calibration is described in Appendix A.

### B. Temperature Variations in the Oil Bath

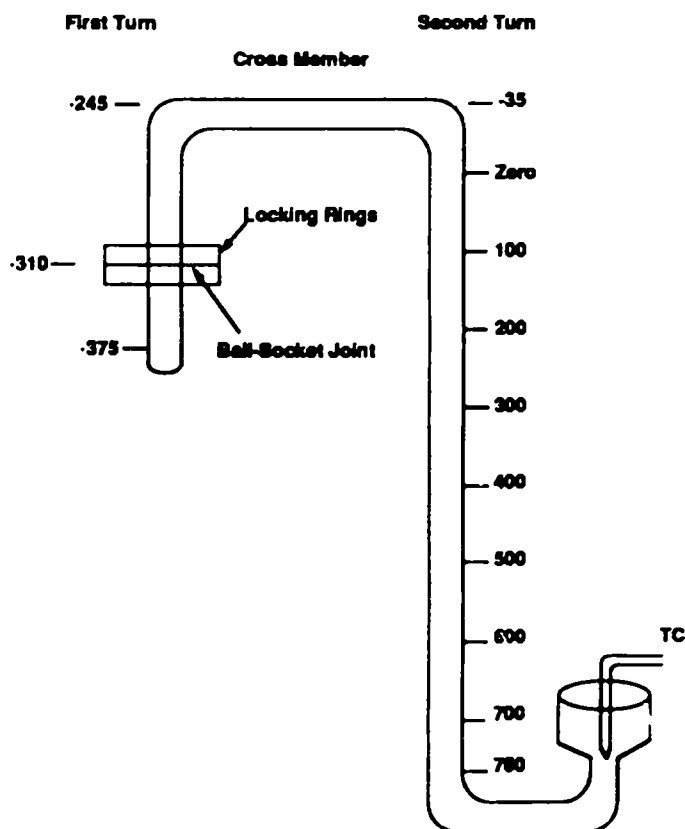
The stirred oil-bath temperature was determined by monitoring the bath with thermocouples calibrated to a platinum resistance thermometer traceable to a National Institute of Standards and Technology standard. The temperature was recorded on a strip chart and showed a typical cycle-correction curve. At both temperatures of 100°C and 120°C, the feedback controller kept the temperatures within 0.1°C over the test period of 48 hours. This was the optimum control for heating in the normal laboratory atmosphere.

### C. Variation Due to Temperature of Gas in Bore of Tube

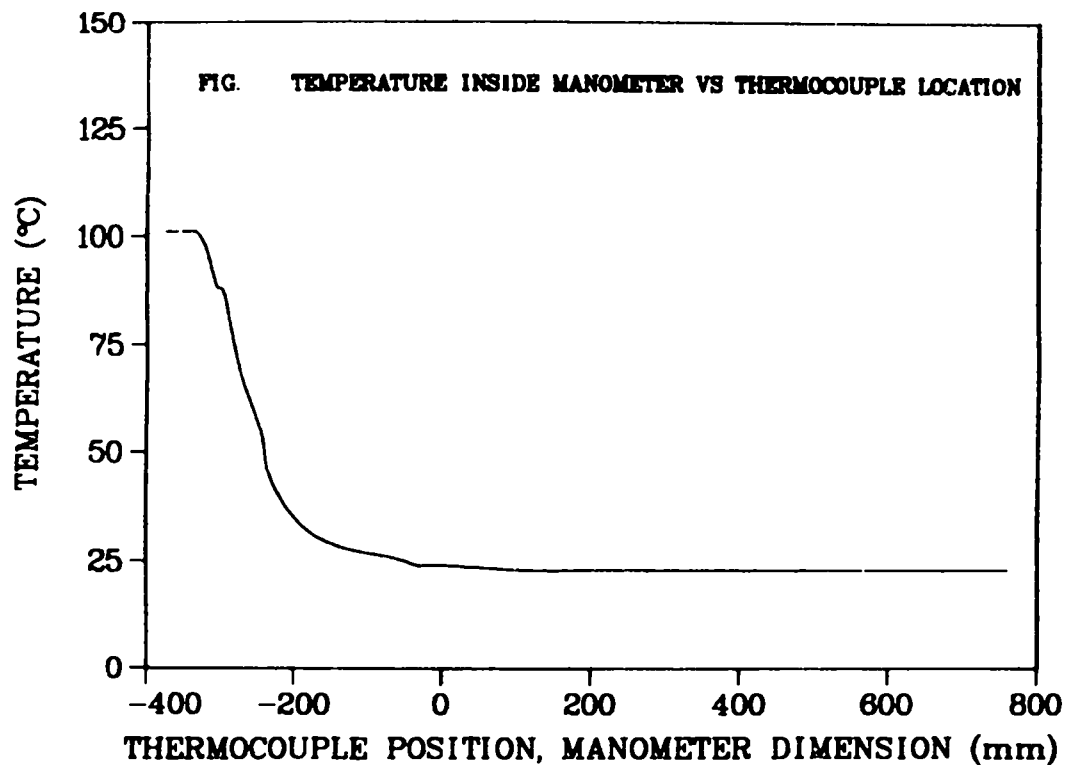
In calculating the decomposition of samples from gas evolved in the vacuum stability tests two assumptions are made: (1) the temperature of the sample bulb is at test temperature and (2) the temperature of the rest of the manometer tube is at room temperature.

To determine if there is an error because of temperature gradients, temperature profiles of the manometer tube and bulb were determined. By inserting a thermocouple in the bore of the manometer, the temperature was measured every 10 cm along its bore (Fig. 7). A plot of temperature versus thermocouple location shows a fast drop to room temperatures for both the 100°C and 120°C conditions. The profiles are shown in Figures 8 and 9.

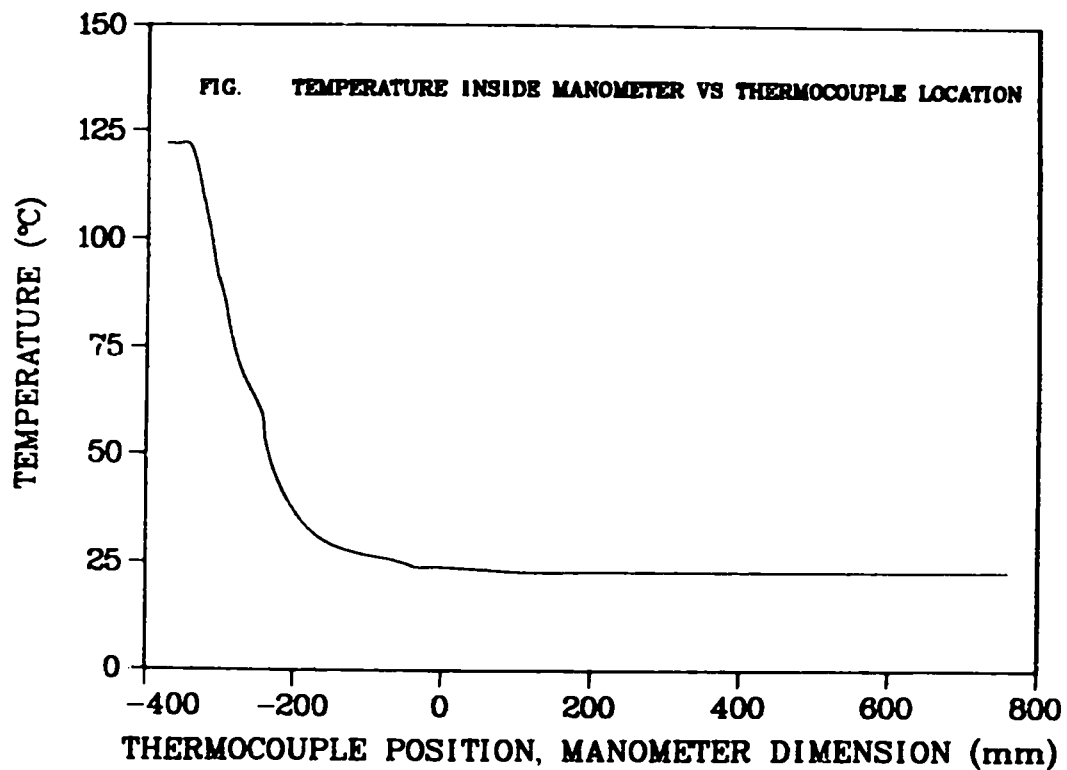
Knowing the volume per unit length, temperature profile, and incremental length, the volume of gas was integrated over the length of the tube from the socket to an arbitrary start value, and again from the socket to an arbitrary finish value. Using the same



**Fig. 7.** Profile of VTS tube and distance from zero mark for determining temperature inside the bore.



**Fig. 8.** Temperature inside the capillary tube with respect to distance from zero mark, for a bath temperature of 100°C.



**Fig. 9.** Temperature inside the capillary tube with respect to distance from zero mark, for a bath temperature of 120°C.

arbitrary variables, the volumes were calculated according to the routine procedure for both 100°C and 120°C conditions.

In the worst case, the difference in volume between the integrated and straight calculations was 0.02 cm<sup>3</sup> at the start and 0.08 cm<sup>3</sup> at the finish for an overall error of 0.06 cm<sup>3</sup>. This translates into a 0.1 cm<sup>3</sup>/g error in a 7-cm<sup>3</sup>/g gas evolution; that was a 1.5% error for both 100°C and 120°C tests.

In the best case (low gas evolution), the difference in volume between the integrated and straight calculations was 0.005 cm<sup>3</sup> at the start and 0.006 cm<sup>3</sup>/g at the finish for an overall error of 0.001/cm<sup>3</sup>. This translates into a 0.003-cm<sup>3</sup>/g error, which was lost in the noise of the measurement.

Therefore, for all practical purposes, the error from calculating the gas evolved with assumptions noted above can be neglected.

#### **D. Voltage to Height and to Volume Measurements**

Voltage output from the CD-23 is measured with mercury at the zero mark (Fig. 7) and every 100 mm down to the base at 760 mm. At the base, the voltage is adjusted to 0.0 V using the zero potentiometer on the CD-23. At the zero mark, the voltage is adjusted to 10 V with the span potentiometer. Coefficients for a linear least-squares fit are calculated for height as a function of voltage output. This is the electronic calibration. The heights are used in the gas-evolution calculation, terms c and g.

From the volume calibration using mercury, described in Appendix A, the volume of the tube at any mercury level is calculated from coefficients from another least-squares fit, where the height determined electronically is used as the independent variable. The volumes calculated for the gas-evolution equation are the terms e and i.

In operation, the linear coefficients for the two equations are entered into memory by the calibration subroutine as calibration constants. Each numbered tube is calibrated at its respective numbered column in the system for both height as a function of voltage and volume as a function of mercury height. The voltage output is linear for the first 400-mm height and the regression equation is valid for this portion only. It would require a spline-curve equation to cover the entire curve and this would make the program more complicated. In the interest of simplicity only the linear portion is considered. If gas evolved is greater, the sample fails the test anyway and is rejected.

When the column height is calculated from the voltage, the results show an error of less than 0.5% compared with height measured by a ruler. In 50 routine experiments run at 120°C, the height of the mercury column measured with a ruler was compared with the computerized calculation of regression coefficients. The data were within 1 mm of each other, indicating stability of the system.

The conversion of height into volume is based on direct measurements with mercury as described in Appendix A. Repeated calibrations give the same coefficients, but this operation need be done only once when the tube is put into operation.

#### **E. Sealing of Tube to Bulb for Vacuum**

The stability of the system depends on how tightly the bulb is attached to the tube at the ball-socket joint by the Invar locking ring. (Fig. 1). A Teflon O-ring is installed at each component of the locking ring to cushion the ball on one side and the socket on the other. Invar has a low coefficient of thermal expansion compared to Teflon. When the ball-socket is sealed with silicone grease and the locking ring secured to compress the ball-

socket, the sample bulb is sealed to the tube for vacuum operation without leakage.

After mercury has been poured into an evacuated tube and the assembly allowed to stand at ambient pressure, the column of mercury varied with the change in barometric pressure (about  $\pm 5$  mm). The seal was secure at room temperature.

Several tubes were run without sample at 120°C as if each run were a test, and back pressure was applied at the cistern end. There was no change in the mercury level during the test. Output was 0.0 cm<sup>3</sup>/g.

To make the test severe, the experiment was run at 300°C. Again, the locking ring exposed to the high temperature did not allow leakage at the ball-socket. The Teflon O-rings expand with heat more rapidly than the Invar locking ring and therefore, will increase the pressure to seal the ball-socket joint.

Tests were run with water alone as a sample. At 100°C and 120°C, all the water vaporized and condensed on the cold part of the cross member, and sometimes overflowed into the upright part. The drop in the mercury column was not measurable and the gas evolved was reported as less than 0.01 cm<sup>3</sup>/g. If one of the decomposition products in a real sample is water, the VTS test will not account for it. If a gas is soluble in water, it will not show as a change in mercury level. This is reported as water condensation in the tube, with an estimate made for the amount.

#### IV. TYPICAL RESULTS

To check the repeatability of the VTS tests over a period of time, an inert sample with a high gas evolution (1900-10)\* and another with a low gas evolution (1900-24)\*\* were run over a period of three weeks in different tubes and columns.

The 1900-10 contains nitrocellulose (NC) as the gassing decomposing ingredient. At 120°C over a 48-hour period, the mean amount of gas evolved over eight experiments was 4.5 cm<sup>3</sup>/g with a standard deviation of 0.2 cm<sup>3</sup>/g. Figure 10 is a typical computer record.

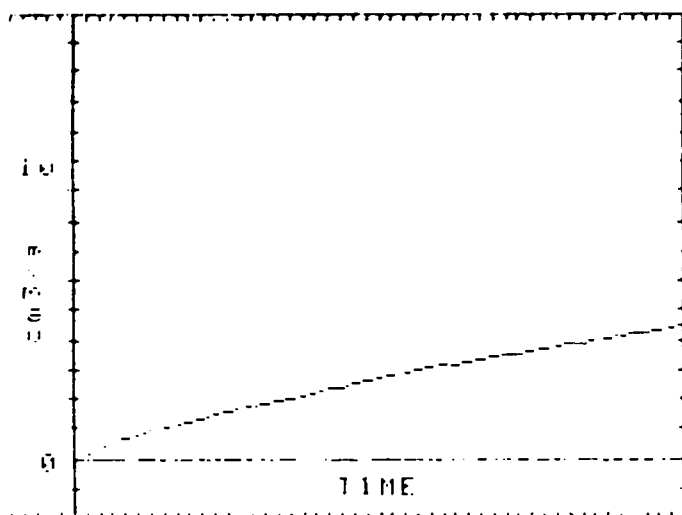
The 1900-24 has no known gassing ingredient and any gas evolution is due to impurities. For this inert sample, the mean amount of gas evolved was less than 0.1 cm<sup>3</sup>/g with a standard deviation of 0.1 cm<sup>3</sup>/g. Figure 11 shows a typical curve and record.

These tests show the stability of the system. For periodic check of the calibration, the NC-containing inert material used as a standard repeats the above gas-evolution result within a standard deviation.

Over the years of operation of VTS, explosives have been run and typical results established. Results for about 18 cast, plastic-bonded, and unprocessed explosives are given in Table I. The results from these tests are compiled in *LASL Explosive Property Data* by Cibbs and Popolato.<sup>10</sup>

\* 1900-10—46.0/48.0/2.8/3.2 wt%—Barium nitrate/Pentek/NC/CEF

\*\* 1900-24—60.9/74.1/15.0 wt%—Cyanuric Acid/Talc/Kel-F 800

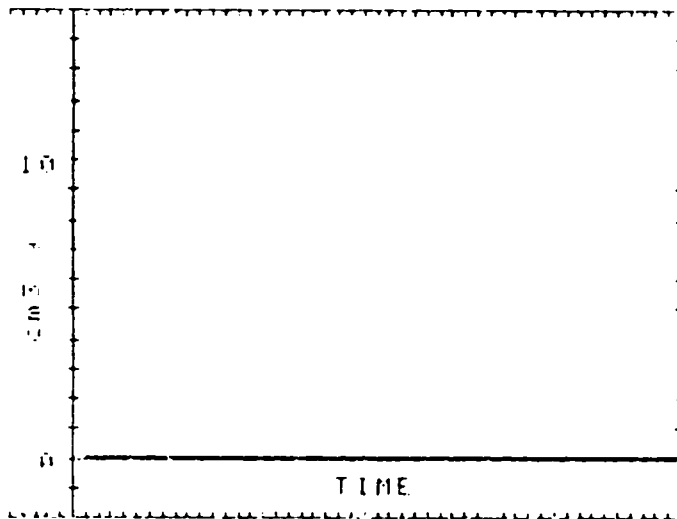


SAMPLE ID = I10010  
 POL PC = 555B  
 LAB NO. = 31077  
 REQUESTOR = HC  
 OPERATOR = JOM  
 TUBE NO = 10  
 BULB NO = 7  
 SAMPLE WT = 2026  
 TEST START TIME = 04 09 11 23 17  
 TEST END TIME = 04 11 11 23 18  
 TIME RUN (HOURS) = 48  
 BATH TEMP = 120 Dea C  
 GAS EVOLVED = 4.53 cm<sup>3</sup> g

**Fig. 10.** Typical computer record of an Inert I900-10 high-gas-evolution sample used as a calibration check.

**TABLE I.** Acceptable limits of gas evolution of explosives by VTS at LANL.

Explosive	Gas Evolved After 48 hours at 120°C (cm <sup>3</sup> /g at STP)
Baratol	0.1 - 0.4
Composition B	0.2 - 0.6
Cyclotol	0.4 - 0.5
DATB	0.1 - 0.3
HMX	0.1 - 0.4
Nitroguanidine	0.2 - 1.7
Octol	0.1 - 0.4
PBX 9011	0.3 - 0.9
PBX 9404	1.3 - 4.0
PBX 9407	0.1 - 0.2
PBX 9501	0.4 - 0.7
PBX 9502	0.0 - 0.5
PETN	0.2 - 0.5 (at 100°C)
RDX	0.1 - 0.3
TATB	0.0 - 0.5
Tetryl	0.4 - 1.0
TNT	0.0 - 0.5
XTX-8003	0.0 - 0.2 (at 100°C)
XTX-8004	0.1 - 0.3



```

SAMPLE ID = 124152
PO PC = CES1
LRE NO = 20974
REQUESTOR = MS
OPERATOR = JOM
TUBE NO = 10
BULB NO = 10
SAMPLE WT. = 2034
TEST START TIME = 05 01 10 39 07
TEST END TIME = 07 01 10 39 07
TIME RUN (HOURS) = 48
BATH TEMP = 120 Deg C
GAS EVOLVED = 0.05 cm3

```

**Fig. 11.** Typical computer record of an Inert 1900-24 low-gas-evolution sample used as a calibration check.

Although most explosives will decompose partially at 120°C, the new insensitive high explosives (IHE) give no gas evolution at this temperature. To characterize this test at a high temperature, a remote system based on this electronic monitoring was developed. By varying temperatures from 150°C to 300°C, different decomposition rates were determined as a function of time at a fixed temperature or as a function of temperature at a fixed time. In this system, the important feature was a graphite-lined furnace tightly coupled to the sample bulb for efficient heat transfer. Oil could not be used, as it vaporizes at 200°C and above. The remote control system and results for some IHE were reported at an Engineering Faculty Meeting at LANL.<sup>8</sup>

The VTS system, as described here, can determine stability and compatibility of explosives from gas evolution by VTS at the ordinary temperatures of 80°C, 100°C, 120°C, and 150°C. With a modified remote system with special heaters, VTS gas evolution can be run up to 300°C to get reproducible results.

## ACKNOWLEDGMENTS

I thank F. W. DuBois who helped establish that an aluminum-foil-clad tube can be made into a capacitor. I thank D. Stephens for securing the various peripheral instruments, for assembling the apparatus, and for negotiating the customized manometers from Ace Glass Company. Technicians of Group WX-12 put the system together. I thank J. Laux, Group WX-3, and H. H. Cady, Group M-1, for writing the software and debugging it to make the monitoring and reporting automatic.

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## APPENDIX A: CALIBRATIONS

### A. CALIBRATION OF SAMPLE BULBS FOR VOLUMES

1. Place a numbered bulb in the holder and onto the pan of an electronic balance. Then tare and record the weight on a calibration form.
2. Add mercury to the bulb—to the bottom of the socket and no higher.
3. Allow the mercury to equilibrate to the temperature of the weighing chamber and adjust the amount of mercury as necessary to maintain its level to the bottom of the socket.
4. Take the temperature of the weighing chamber.
5. Record the net weight of the mercury and the temperature of the weighing chamber on the calibration form.
6. Calculate the absolute volume of bulb using the density of mercury at the recorded temperature, as specified by a handbook, and using the following equation:

$$\text{Volume (cm}^3\text{)} = \text{Weight (g)} / \text{Density Hg (g/cm}^3\text{)}.$$

7. Enter the volume as a calibration constant into the computer.

### B. CALIBRATION OF MANOMETER TUBE FOR VOLUME AND MODULUS

1. Invert a numbered manometer tube and clamp it in a pivoted burette fixture on a ring stand above a catch pan.
2. Place a short piece of rubber tubing over the socket, then attach a clamp and a small funnel to the rubber tube.
3. Add mercury to the capillary through the funnel to fill the cross member to the zero mark.

4. Swing the tube sideways until the zero mark and socket (Fig.1) are in the same horizontal plane. Tap the tube to remove all bubbles—do not allow bubbles in mercury column.
5. Clamp the rubber tube when the desired level of mercury is achieved, then remove the excess mercury.
6. Remove the rubber tubing. Add a drop or two of mercury as needed so the zero mark and socket are still in the same plane.
7. Swing the tube so that mercury flows from the cross member into the upright member.
8. Remove the manometer tube from the clamp.
9. Lay the manometer tube on a clean flat bench so that the mercury flows somewhere between zero and base, and stops.
10. Take a metric ruler and measure the length of the mercury column in millimeters.
11. Record this length on the calibration form. The bore of the glass is considered constant.
12. Take the temperature of the manometer tube and mercury by laying the thermometer alongside; record the temperature on the calibration form.
13. Empty the mercury into a tared 25-ml beaker.
14. Reweigh the beaker with mercury and calculate the net weight of the mercury in grams.
15. Record this value on the calibration form.
16. Find the density of mercury for the test temperature in a handbook table and record it on the calibration form.
17. Calculate the volume of the cross member from socket to zero, and the modulus of the tube as follows:

- A = weight Hg(g)/density of Hg at recorded temperature ( $\text{g}/\text{cm}^3$ ) = the volume of the cross member ( $\text{cm}^3$ ),
- B = A ( $\text{cm}^3$ )/length of Hg column (mm) = modulus or volume per unit length ( $\text{cm}^3/\text{mm}$ ),
- C = B ( $\text{cm}^3/\text{mm}$ )  $\times$  760 mm = volume of capillary, zero to base ( $\text{cm}^3$ ), and
- D = A + C = volume of capillary, socket to zero to base ( $\text{cm}^3$ ).

These physical parameters give a linear equation for volume at any mercury level.

$$V(\text{cm}^3) = A (\text{cm}^3) + [B (\text{cm}^3/\text{mm}) \times \text{height (mm)}],$$

where height is the measured mercury level in the test experiment.

- Record A and B on the calibration form for later entry into the computer as calibration constants in the calibration routine at the proper address.

### C. CALIBRATION OF ELECTRONIC TRANSDUCER TUBES

This procedure calibrates height as a function of voltage. The tube needs to be calibrated electronically to the system. It is calibrated in place for the same numbered tube to same number channel.

#### On Bench:

- Clean the calibrated and numbered manometer tubes and dry them in a  $60^\circ\text{C}$  oven.
- Remove a tube from the oven, place it upright on a loading stand, and allow it to equilibrate to room temperature before calibration.

- Place a large test tube in the bath opening to catch any mercury overflow from the socket during calibration. For ease of calibration, the socket is not connected to a bulb.
- Add 6 ml of mercury to the cistern of the tube, which is in place on the loading stand.
- Attach the electrical leads and back pressure assembly to tube.

#### In Place on VTS Apparatus:

- Mount the manometer tube in the appropriate numbered column. Hook up the BNC electronic pigtail fitting and connect the LRL-air fitting to the controlled-air outlet.
- Turn the pressure-adjust knob (J in Fig. 3) fully counterclockwise. Adjust the ZERO potentiometer on Validyne CD-23 readout to read 0.000 V when mercury level is at base.
- With the air pressure switch (H in Fig. 3) on ADJ position, slowly raise the mercury level to zero mark (top of tube) with the pressure-adjust knob. This is 0.0-mm level for mercury. Gas evolution is measured with respect to this level.
- Adjust the SPAN potentiometer on the Validyne CD-23 to equal 10.000 V.
- Drop the mercury level to 100 mm with the pressure-adjust knob.
- Read the voltage and record it on the calibration form.
- Drop the mercury level to 200 mm, read the voltage, and record it on the calibration form.
- Do the same for 300, 400, 500, 600, 700, and 760 mm. At the base, 760 mm should read 0.000 V.
- Repeat steps 3 to 8 and recheck values.

10. Remove and dismantle the setup and clean the manometer tube.
11. Pick another manometer tube and channel, and repeat from Step 1.

#### D. CALCULATION OF REGRESSION COEFFICIENTS

1. With voltage/height data (only pairs from 0 to 400 mm are needed) do a linear regression of the data where  $x = \text{voltage (V)}$  and  $y = \text{height (mm)}$ .
2. The equation to be fitted is  $Y \text{ (mm)} = A \text{ (mm)} + [B \text{ (mm/V)} \times X \text{ (V)}]$ . Record these A and B coefficients on the calibration form. These are electronic parameters for determining height of fall of mercury. This Y, when applied to volume meter equation as the height, gives the volume of the manometer at any voltage.
3. Key the four values entered on the calibration form as constants into the HP85B CALIB routine at their proper addresses.
4. The VTS system is ready for automatic data logging and calculation.

## APPENDIX B: EXPERIMENTAL PROCEDURES

### A. PROCEDURE FOR SETUP OF SAMPLE IN MANOMETER TUBES IN VTS TESTS.

1. Turn the Validyne CD-23 power on, set the range to HIGH, and set the air pressure switch to OFF.
2. After the manometer tube with a sample is prepared on the vacuum manifold and removed, attach the back-pressure ball joint assembly and the electronic BNC pigtail connectors.
3. Set up the manometer tube in the matching column in the upper rest post slot.
4. Connect the BNC connector to the panel plug at the left of the column.
5. Attach the air supply LRL tubing to the outlet that corresponds to the column being tested.
6. While the manometer tube is in a rest slot, enter the experimental data into the HP85B at SET UP.
7. On the Validyne CD-23, set the air pressure switch to OFF.
8. With the pressure-adjust knob raise the mercury to top of the zero mark.
9. Adjust the SPAN on the CD-23 to 10.000 V showing on its digital readout. This is 0.0 mm height.
10. With the pressure adjust knob lower the mercury to the 100-mm mark.
11. Check that the digital readout corresponds to the recorded calibration value for 100 mm; they should agree within 0.020 V.
12. With the pressure-adjust knob lower the mercury to the 200 mm mark.
13. Check that the digital readout corresponds to the recorded calibration value for 200 mm. It should agree within 0.020 V. Adjust the pressure-adjust knob to zero pressure.
14. Set the air pressure switch to OFF.
15. Set the air pressure switch to the 760-mm position.
16. The mercury should rise to reflect the sea-level back pressure.
17. Take the manometer tube assembly off of the rest slot and lower the sample bulb into the heated bath. Secure the manometer tube in its slot.
18. Equilibration starts after START is keyed on the computer. The computer counts time after the 5-minute equilibration period.
19. For each manometer tube setup, go to Step 3 above and repeat the procedure.

### B. DATA LOGGING

1. The computer data logger will automatically record, store, and print data and calculate hourly. At the end of the 48-hour run, it will do a printout and plot of results.
2. In the event of a computer failure, data could be recovered manually. After 48 hours, take the 48-hour voltage reading from the printout, take the ambient temperature, and record these values in the 48-hour columns of the worksheet. Then do Step 3 below.
3. Calculate the height and volume of the mercury column from calibration forms for both the start and the end of the test, and record in proper spaces. Then calculate the gas evolved by using the back-up program of the gas equations on the work sheet. Record answer on work sheet.

### C. END OF TEST

1. To remove the manometer tubes from the bath, first set the air pressure switch to OFF. Lift the bulb out of the bath, and place the manometer tube on its rest slot.
2. Disconnect the AIR fitting and the electronic BNC connector from panel.
3. Remove the manometer tube with its attached fittings and place it on a bench rack to cool.
4. When the manometer tube is cool, remove its fittings, dismantle the equipment, recover the mercury, and clean the tube and bulb by the usual laboratory procedure.
5. Dry the tubes in the 60°C air oven, so they are ready for reuse.

### D. COMPUTER MANIPULATION

1. The HP85B PC program is menu driven (Fig. B-1).
2. Before any run, the calibration data need to be entered and edited through the CALIB routine.
3. Before lowering the sample bulb into the heated oil, key SET-UP, and answer all questions. The data requested are needed to calculate the gas equation.
4. Drop the test bulb into the heated oil and secure the manometer tube in position.
5. Key START. The equilibration period of 5 minutes begins and is timed by the program. An "S" will appear above the channel number on the screen, and a start-up message will be printed.

6. After 5 minutes, the zero-time data will be printed and entered in memory, and the zero-time volume will be calculated and stored in memory. This value is used for all subsequent calculations. An asterisk appears in place of the "S" No further attention is required.
7. At each one-hour time period, a reading is taken and the incremental change is calculated with respect to time zero, and stored in memory. The hours number appearing above column number on the screen indicates elapsed time of test.
8. At the end of the test time, a printout is produced (Fig. 10). All data are then stored on the tape.
9. Copies of the printout of the test results can be made using the PLT/LST routine.
10. The other user-definable keys are annotated in Fig. B-1.

```

                                WACSTOP
SETUP--Set up new run
START--Start run
DIRECT--Print directory
IMMED--Take immediate reading
ABORT--Abort run in progress
SAVE T--Save memory to tape
PLT/LST--Plot or List data
CALIB--manipulate calibration
                                data
1  2  3  4  5  6  7  8  9 10 11 12 13 14
-----
ABORT  SAVE T  PLT/LST  CALIB
SETUP  START  DIRECT  IMMED
```

**Fig. B-1.** Menu-driven computer program display for VTS calculation and monitoring.