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Investigation of Thermal Conductivity Gage Calibrations in Condensible Vapors

Leonard Theodore Melfi College of William & Mary - Arts & Sciences

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INVESTIGATION OF THERMAL CONDUCTIVITY GAGE CALIBRATIONS IN CONDENSIBLE VAPORS

A Thesis

Presented to

The Faculty of the Department of Physics The College of William and Mary in Virginia

In Partial Fulfillment

Of the Requirements for the Degree of

Master of Arts

By

Leonard Theodore Melfi, Jr.

May 1964

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APPROVAL SHEET

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the requirements for the degree of

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ABSTBACT

An investigation of the effects of condensible vapors on the calibration of a thermal conductivity pressure gage has been made. A theoretical study is made of thermal conductivity under low pressure and rarified gas conditions. A method using a transfer standard is described for the calibration of thermal conductivity gages in a condensible rapor environment over the pressure range of 10^{-3} to 15 torr. Calibrations are made, using this method, in dry air, water vapor, and acetamide vapor. The gage is also calibrated in the Echo II canister to demonstrate the usefulness of the method in a practical application. The data given in this report show significant differences for the various gas compositions.

SYMBOLS

normalizing factor for a Maxwellian distribution of velocities \mathbf{A} thermal accommodation coefficient \mathbf{a} specific heat at constant volume c_v dE rate of change of the average energy per molecule along x , dх dynes/molecule average energy per molecule at the unit surface, ergs/molecule $E_{\rm g}$ average energy per molecule as a function of x , ergs/molecule $E(x)$ ΔE net heat transfer per unit area per unit time, ergs/cm²sec \mathbf{H} net heat transfer per unit area per unit time, ergs/cm²sec \mathbf{K} coefficient of thermal conductivity \mathbf{L} molecular mean free path, cm molecular weight, gm/mole M mass per molecule, gm/molecule \mathbf{m} Avogadro's number, molecules/mole \mathbb{N}_{Ω} number of molecules per unit volume, molecules/cm² \mathbf{n} n ressure. dynes/ cm^2 \mathbf{P} universal gas constant, $\frac{\text{ergs mole}}{\text{cm molecule}}$ ok \mathbf{R}_{m} T absolute temperature, OK absolute temperature of surface 1, α K ${\bf T}$ ገ absolute temperature of surface 2, α K \mathbf{T}_2 average velocity per molecule, cm/sec molecule $\overline{\mathbf{v}}$

v

- $\overline{\overline{x}}$ average of the squared velocity, cn^2/sec^2
- distance from the unit surface, on \mathbf{x}
- number of molecules crossing unit area in unit time, molecules/cm²sec Γ_{n}
- Maxwellian gas constant, sec/cm β
- γ ratio of specific heat at constant pressure to specific heat at constant volume
- thermal-conductivity gas constant ϵ
- gas density, gm molecules/ cm^3 ρ .
- molecular diameter, cm σ

INVESTIGATION OF THERMAL CONDUCTIVITY GAGE CALIBRATIONS IN CONDENSIBLE VAPORS

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The requirements of in-flight and ground-facility research programs frequently require pressure measurements to be made in the range of 10^{-7} to 15 torr. In the majority of cases the use of gages sensitive to gas composition is required since the few gages that are insensitive to gas composition are, in general, too large for this type work.

The calibration of these gas-composition-sensitive gages is normally determined by comparison with a primary standard (McLeod gage). These calibrations are more difficult if the test gas is a condensible vapor, since the McLeod gage is a compression instrument and cannot be used for this type gas. The calibration of gages in a condensible gas can be accomplished by the use of a gage that is insensitive to gas composition which has been calibrated by a McLeod gage using a noncondensible gas. In this case the gage that is insensitive to gas composition acts as a transfer standard. In a typical procedure the transfer standard is calibrated against a McLeod and the gage to be used is then calibrated against the transfer standard in the gas in question. Three transfer standards were selected to cover the 10^{-3} to 15 torr range.

Because of its wide use in flight and facility work, the gage chosen for calibration was a thermal-conductivity-type vacuum gage system¹. The output of this thermal-conductivity gage is not a linear function of pressure and has asymptotes at approximately 10^{-1} and 10 torr. Its sensitivity to gas composition, therefore, must be determined over the entire pressure range.

 \mathbf{z}

If thermal conductivity is considered as a transport of kinetic energy between two surfaces at different temperatures as in the kinetic theory of gases, then its variance with pressure can be divided into three regions. The first region is a pressure range in which there are enough molecules to support a temperature gradient. In this region

 $H = -K$ grad T

In the one-dimensional case

$$
H = -K \frac{dP}{dx}
$$
 (1)

If a surface of unit area is placed between two plates, then the number of molecules that cross the surface in unit time is

$$
\Gamma_{\rm n} = \frac{1}{4} \; \mathbf{n} \overline{\mathbf{v}} \tag{2}
$$

A derivation of Γ_n is given by Kennard². The energy at station χ $(x = 0$ taken at the unit surface) for positive x is

$$
E(x) = ES + x \frac{dE}{dx}
$$

If no persistence of velocity and no transfer of mass are assumed, then the average energy per molecule that crosses the surface in the positive x-direction is

$$
\overline{E} = E_B + x \frac{\partial E}{\partial x}
$$
 (3)

Since E_{g} is fixed at $x = 0$ and $\frac{dE}{dx}$ is constant then

$$
\overline{L} = E_{\text{S}} + \overline{x} \frac{\text{d}E}{\text{d}x}
$$

Hence the total flux of energy that crosses unit area in unit time in the positive x-direction is

$$
\frac{1}{4} \pi \overline{v} \left(E_B + \overline{x} \frac{dE}{dx} \right)
$$

Similarly for particles crossing in the negative x-direction, the energy flux is $\frac{1}{4}$ $n\overline{v}$ $\left(E_5 - \overline{x} \frac{dE}{dx}\right)$. The negative sign appears in order to keep \overline{x} on essentially positive quantity.

The net flux for all molecules per unit area in unit time is

$$
H = -\frac{1}{4} n\overline{v} \left(2\overline{x} \frac{d\overline{B}}{dx}\right)
$$

$$
= -\frac{1}{2} n\overline{v} \overline{x} \frac{d\overline{B}}{dx}
$$
 (4)

however, if we assume that the mean free path is not a function of speed and the collision rate is Maxwellian, then after Kennard 3

$$
\overline{\mathbf{x}} = \frac{2}{3} \mathbf{L} \tag{5}
$$

Using the value of \bar{x} in equation (5) and the chain rule of differention, equation (4) becomes

$$
H = -\frac{1}{2} m\overline{v} \left(\frac{2}{3} L\right) \frac{d\overline{E}}{d\overline{x}} \frac{d\overline{v}}{dx}
$$

$$
= -\frac{n\overline{v}L}{3} \frac{d\overline{v}}{d\overline{x}} \frac{d\overline{v}}{dx}
$$

By definition

$$
c_v = \frac{1}{m} \frac{q\overline{m}}{dT}
$$

Therefore

$$
H = -\frac{nm}{3} \overline{v}Ic_v \frac{dT}{dx}
$$

But for a Maxwellian gas Kennard⁴ shows that

$$
\overline{\mathbf{v}} = 2 \left(\frac{2 \mathbf{R}_{\underline{\mathbf{m}}} \mathbf{T}}{\pi \mathbf{M}} \right) \frac{1}{2}
$$

and

$$
L = \frac{1}{\sqrt{2}} \frac{1}{\text{mod}^2}
$$

Therefore

$$
H = -\frac{2\pi m}{5} \left(\frac{2R_m T}{\pi M}\right)^{1/2} \frac{1}{\sqrt{2}} \frac{c_v}{\pi a^2} \frac{dT}{dx}
$$

But

$$
m = \frac{M}{N_O}
$$

Therefore

$$
H = -\frac{2}{3} \left(\frac{R_m T M}{\pi}\right)^{1/2} \left(\frac{1}{N_Q}\right) \frac{c_V}{n\sigma^2} \frac{dT}{dx}
$$

In order to correct for molecular collisions, vibrational, and rotational energy of complex molecules, a constant is needed which is derived by Eucken⁵.

$$
\epsilon = \frac{2T-2}{4}
$$

Therefore

$$
H = -\left(\frac{2V-2}{6}\right)\frac{1}{N_0}\left(\frac{R_{\text{m}}TM}{\pi}\right)^{1/2}\frac{c_V}{m^2}\frac{dT}{dx}
$$
 (6)

It can be seen from equation (6) that the net heat transfer is independent of pressure. Comparing equations (1) and (6) leads to

$$
\mathbf{K} = \left(\frac{9\mathbf{Z} - 5}{6}\right) \frac{1}{N_{\rm O}} \left(\frac{\mathbf{R}_{\rm H} \mathbf{f} \mathbf{M}}{\pi}\right)^{1/2} \frac{c_{\rm v}}{\pi \sigma^2}
$$
 (7)

which is the well-known coefficient of thermal conductivity.

The second region is a pressure range in which the gas between the surfaces is so rarefied that no interaction between molecules exists; therefore, there can be no temperature gradient. Then the number of molecules of velocity v per unit area per unit time is

$$
\Gamma_{n_v} = \frac{1}{4} n_v v
$$

The energy of these Is

$$
= \frac{1}{4} n_v v \left(\frac{mv^2}{2} \right)
$$

$$
= \frac{1}{2} m_v v^3
$$

If a Maxwellian gas is assumed, then the average energy \overline{u} per unit area per unit time is

$$
= \frac{ma}{3} \mu_{m} \int_{0}^{\infty} v^{5} e^{-\beta^{2} v^{2}} dv
$$

$$
= \frac{\beta}{2} \frac{\beta^{3}}{\sqrt{\pi} \beta^{5}}
$$
(3)

Since

$$
P = \frac{1}{5} \rho v^2
$$

where

 $\overline{v^2} = \left(\frac{3}{2} \frac{1}{\beta^2}\right)$

Then

 $P = \frac{1}{3} \rho \left(\frac{3}{2} \frac{1}{\beta^2} \right)$

$$
=\frac{p}{2g^2}
$$

and

 $\overline{v} = \frac{2}{\sqrt{\pi}} \left(\frac{1}{\beta}\right)$

whence

$$
\frac{1}{2} P \overline{v} = \frac{\rho}{2 \sqrt{\pi} \beta^2}
$$

which is identical to equation (8) ; in consequence, the average energy per unit area per unit time for the second region is

$$
=\frac{1}{2} P\overline{v}
$$

If we assume the energy of the molecule adjusts to that of the surface, then

$$
\Delta E = \frac{P\bar{V}}{2} \frac{T_1}{T_2} - \frac{P\bar{V}}{2}
$$

or

$$
\Delta E = \frac{P\vec{v}}{\Delta T_2} (T_1 - T_2)
$$

$$
P\overline{v} = \frac{P}{2\sqrt{\pi} \beta^2}
$$

where

$$
\overline{v} = 2 \left(\frac{2R_n T}{\pi M} \right)^{1/2}
$$

Therefore

$$
\Delta E = P \left(\frac{2R_{\rm H}T}{\pi M}\right)^{1/2} \left(\frac{T_1 - T_2}{T_2}\right)
$$

If the energy does not adjust, then a correction factor must be included. This constant \mathbf{a}^n is called the thermal accommodation coefficient. Then

$$
\Delta E = a \left(\frac{2R_{\rm m}T_2}{\pi M} \right)^{1/2} \left(\frac{T_1 - T_2}{T_2} \right) P \tag{9}
$$

where the slope Is

$$
B = a \left(\frac{2R_m T_2}{\pi M} \right)^{1/2} \left(\frac{T_1 - T_2}{T_2} \right)
$$
 (10)

It can be seen from equation (9) that there is a linear relation between heat transfer and pressure.

The third region is a transition pressure range that exists between the first and second regions. No successful attempt at a theoretical treatment has been made for this region because of the difficulties involved in defining the transition. The net flux is usually represented by fitting a smooth curve taken from the experimental data between the first and second regions. Since this is the region in which the thermal conductivity gage operates with maximum sensitivity, the need for an experimental method of calibration becomes readily apparent.

The theoretical curves for the first two regions are given in figure 1. Since no adequate theory of the third region exists, the experimental data for this region are also given.

Pressure

Figure 1.- The three theoretical regions of thermal conductivity for low pressures.

EXPERIMENTAL EQUIPMENT

The equipment consists of a high-vacuum pumping system; a McLeod gage, as a primary standard; three diaphragm-type pressure transducers, as the transfer standards; and the gas-sensitive thermal-conductivity gage system.

A standard vacuum system 7 containing a 2-inch oil-diffusion pump and a mechanical forepump was used to attain a pressure in the 10^{-4} torr range. This system (figs. 2 and 3) can cover a workable pressure range of 10^{-5} to 17 torr, with an ultimate pressure for outgassing of 10^{-7} torr. An air dryer and a mercury McLeod gage are included in order to complete the system as a calibration unit. The system is protected from the McLeod gage mercury vapor by a liquid-nitrogen cold trap. Suitable connections are available for connecting the gages to be calibrated.

The McLeod $\boldsymbol{\mathit{g}}$ age 3 used in this study was a multiple-range instrument having one quadratic scale from 3×10^{-5} to 1 torr, and three linear scales covering the range of 0.3 to 1.6 torr, 0.8 to 5.0 torr, and 3.5 to 17.0 torr.

In order to read true pressure in a test gas environment, it is necessary to choose pressure transducers which are not sensitive to gas composition. A resistance strain gage, 0-50 torr transducer⁹; a variable reluctance, $0-5$ torr transducer¹⁰; and a variable-capacitance micromanometer¹¹ were chosen. These gages allowed operating ranges of 5 to 30 torr, 0.1 to 5 torr, and 0.001 to 0.125 torr, respectively. The gages use a thin metal

Figure 2.- Block diagram of the calibration unit.

ł,

Figure 3.- Vacuum calibration unit and thermal conductivity gage system. Figure 3.- Vacuum calibration unit and thermal conductivity gage system.

diaphragm which move with a change in pressure, unbalancing an a-c bridge and giving an output that was proportional to pressure.

i The operation of the thermal-conductivity gage¹ is based upon the changes in thermal conductivity of residual gases in a vacuum. The noble metal thermocouples, which are directly heated by a 20-kc power supply, are cooled by an amount which varies with the pressure of these gases. The output is a function of the temperature of the thermopile and is amplified and recorded. The instrument (figs. $3, 4$, and 5) consists of a control unit, a thermopile gage tube, a low pass filter and a d-c amplifier. The control unit provides the thermopile gage tube with 20-ke heating voltage. It receives its power from a highly regulated 28 -volt d-c power supply. The thermopile gage tube contains the noble metal thermopile and gives an output of approximately 10 mv below 10^{-3} torr and 1 mv at atmospheric pressure. The output of the gage tube is returned through the balance circuit in the control unit. The low pass filter was used to filter out the residual 20-kc heater voltage appearing on the d-c output. The d-c amplifier was used to give an output of approximately 5 volts below 10^{-3} torr and 0.5 volt at atmospheric pressure. The thermal conductivity gage has an operating range of 0.010 to 10 torr in air.

Figure 4.- Experimental thermal conductivity gage system.

Figure 5.- Electrical block diagram for the thermal conductivity gage system.

EXPERIMENTAL PROCEDURE AND RESULTS

The experimental work has been divided into three parts: water vapor, acetamide vapor, and the practical application. A procedure and a discussion will be given for each part.

Water Vapor

Procedure

A system (fig. 6) was designed to compare water-vapor and dry-air calibrations of the thermal-conductivity gage. This system was built around the calibration unit and contained a cylinder of water, the 0-50 torr resistance strain gage transducer, the necessary plumbing and valving, and the gas-sensitive thermal-conductivity gage.

With valve 2 closed and valve 1 open, a calibration of the resistance strain-gage transducer (fig. 7) and the thermal-conductivity gage (fig. 8) in dry air was made by using the McLeod gage as the standard. To calibrate the thermal-conductivity gage in water vapor, a mechanical pump, through valve β , was used to lower the pressure above the water below its vapor pressure. Sufficient pumping time was allowed to insure that all the absorbed air was removed from the water. Valve 3 was closed and the pressure above the cylinder was allowed to rise to its liquid-vapor equilibrium. After outgassing the gage at 10^{-7} torr, a number of data points were taken by bleeding water vapor into the gages through valve 2 . Using the diaphragm transducer as a transfer standard, the output of the thermal-conductivity gage was then compared to pressure. This calibration (fig. 8) is displayed

Figure 6.- Water vapor calibration system.

on the same figure as the dry-air calibration to show a comparison between the two gases. A plot of true pressure versus indicated pressure (fig. 9) was made from figure 3 for water vapor, using dry air as a standard.

Discussion and Results

In order to run water-vapor calibrations it was necessary to take extreme care in maintaining a constant temperature in the entire vacuum system. Any small decrease in temperature can cause a condensation of the water into its liquid form.

Calibrations in water vapor were made from 17 to 0.5 torr. From the indicated-true pressure curves, it can be seen that the sensitivity of the thermal-conductivity gage is affected by the presence of water vapor. The following table gives the percentage error for water vapor in the above range assuming dry air as the standard:

It can be seen that for water vapor at 0.3 torr there will be an error of $+54$ percent and at 8 torr an error of -32 percent, as calculated from the following relation:

Percent error =
$$
\frac{(\text{True pressure - Indicated pressure})}{\text{True pressure}}
$$
 \times 100 percent

Figure 9.- True versus indicated pressure curves for the thermal conductivity system.

These calibrations also show a crossover at 6.5 torr where the sensitivity for water vapor and dry air is the same.

If the gas constants for air and water vapor are substituted into equation (**6**), the net heat flux can be calculated in the area in which it is independent of pressure. If we assume that the temperature gradient is the same for air and water vapor, then a comparison can be made between each gas. In the second region where a linear pressure dependence exists, the slope of this dependence can be calculated from equation (10) . These values are tabulated and plotted in the following tables and figure.

 N_{0} - Avogadro^ts number - 6.021 x 10^{23} c.g.s.

 R_m - Universal gas constant - 83.15×10^6 c.g.s.

- Temperature of the walls - **300**° K

 T_2 - Temperature of the thermopile in region I - 575° **K**

It can be seen from this figure that the net heat flux in region I is higher for air than for water vapor and that the slope in region II (assuming the thermal accommodation coefficient of water vapor is greater than 10.9/13.9 for air) is smaller for air than for water vapor. If these areas are connected by a smooth curve, as experiment verifies, then a crossover should be expected.

Acetamide CH₃CONH₂

Procedure

For comparison of acetamide vapor to dry air, the system (fig. 10) was modified to contain a variable capacitance gage as the transfer standard.

Figure 10.- Acetamide vapor calibration system.

Since the sample side contained the capacitance plates, it was necessary to reverse the sides for these tests before calibration in acetamide. For the rest of the report all reference to these sides will be opposite to that recommended by the manufacturer.

To begin the test procedure the transfer standard was calibrated against the McLeod in dry air. The acetamide was cured in a vacuum oven at 80° C for a period of 48 hours and then was placed in an evacuated chamber for 24 hours by pumping through valve 4 with valve 3 closed (fig. 10). This was done to insure as pure an acetamide vapor as possible. With valve 2 open and valves 1 and 3 closed (fig. 10), the pressure was raised with dry air in the reference side of the gage to $0.1/0$ torr. Valve 2 was then closed and dry air was bled in through valve 1 in 0.025 torr steps. The outputs from the micromanometer balance circuit and the thermalconductivity gage were recorded to give a dry-air calibration of output versus pressure for both gages. The pressure in the chamber (fig. 10) was reduced through valve 4 and acetamide vapor was bled into the gages through valve 3 in 0.025 torr steps as determined by the calibrated micromanometer. The thermal-conductivity gage output was then recorded. All data for the acetamide vapor portion are recorded in the following table:

Discussion and Results

It can be seen from this table that there is no readable difference in output between dry air and acetamide vapor, the small variations in output being well within the accuracy limits of the gage. We can therefore conclude that the introduction of acetamide vapor in any vacuum system, measured by the thermal-conductivity gage, causes no appreciable error. It was not expected that the output of gage for dry air and acetamide vapor generally would coincide. The apparatus for the test was checked and the test was repeated giving identical results. Since the test data were apparently accurate, it followed that the reasons for the similarity in response for air and acetamide vapor should be investigated.

The following is offered as a likely explanation: The gage tube dimensions are small compared with the mean free path of dry air and acetamide in the pressure range in which the data were taken. The second region, therefore, is chosen to calculate the data. It is obvious from equation (10) that the slope varies as the square of the molecular weight if we assume that the thermal accommodation coefficient is the same, which would make the sensitivity for air larger than that for acetamide vapor. This, however, is not the case. A number of factors can account for this discrepancy. First, the gage is operating on its lower asymptote which makes its sensitivities to pressure a small part of its output in this region. Second, the thermal accommodation coefficient could just balance out the difference in slope. The table of accomodation coefficients given in reference¹² indicates that, for a given geometry, the coefficient increases with molecular weight. It is the opinion of the author that this increase in "a" for acetamide corrects the slope of acetamide vapor calibration just enough to obscure the difference in the asymptote of the gage.

Practical Application

Procedure

In order to show the applicability of the method to a particular problem an actual test will be discussed. It should be noted that this test was not done under laboratory conditions, and the problems involved are not necessarily typical but are shown to give some idea of the complexity of making such measurements.

Since flight packages are minimized in weight, it was necessary to choose a physical small gage for measuring the pressure in the Echo canister. The gage chosen was a thermal-conductivity gage and a transfer standard was used in order to give true pressure in the unknown vapor environment of the canister. Since the canister contained acetamide powder as an inflation material, and since the canister could not be baked, the presence of water vapor and acetamide vapor was strongly suspected.

The transfer standard chosen was a variable reluctance pressure transducer. The thermal-conductivity gage and the reluctance gage were calibrated in dry air against the McLeod gage (figs. 11 and 12) on the calibration unit. Since the tests involved a number of days in which the transfer standard could not be calibrated, it was necessary to determine its shifts in zero and sensitivity on a day-by-day basis. Experiments showed that when the gage was balanced before each run, the sensitivity did not change during the tests. In order to accomplish the calibrations in the canister and balance the gage at the same time, a valving system was designed. The two gages and the valving system (fig. 13) were moved to the canister and the entire system was evacuated to 0.2 torr. The system had a pressure rise of approximately 0.2 torr/day, and it was not known if this was caused by

Figure 11.- Calibration of the variable reluctance pressure transducer.

leakage or outgassing. It was, therefore, necessary to perform two calibrations to determine the causes of the pressure rise. First, the canister was allowed to rise in pressure and data points were taken for 7 days. This gave a calibration of the thermal-conductivity gage using the reluctance gage as a standard. This curve, called the outgassing calibration, is shown in figure 12 . The thermal-conductivity gage was then calibrated in the canister by bleeding in air. This is shown in the same figure as the outgassing curve (fig. 12). In order to compare the error due to gas composition a true pressure versus indicated pressure curve was plotted using dry air as a standard $(fig, 14)$. This figure consists of the dry-air curve, the water-vapor curve, the outgassing curve, and the inbleed curve.

Discussion and Results

It can be seen from figure 14 that there is very little difference between the inbleed and outgassing curves and that these curves lie between the dry-air and water-vapor curves. Also, the two curves approach the water-vapor curve at low pressure. Since water is a known component of unbaked vacuum systems in this pressure range, it is safe to state the gas in the canister at the lowest pressure is mainly water vapor and approaches dry air as the pressure increases. Since the inbleed and outgassing curves are the same, this indicated that the canister pressure rise was due to leakage. The percent difference between the unknown gas and dry air is appreciable, and these errors are given in the following table:

33-

Indicated pressure, torr

Figure 14.- True versus indicated pressure curves for the Echo II canister.

It can be seen from this table that that error varies from a $+20$ percent error at 0.3 torr to a 0 percent error at 6.5 torr. No theoretical approach will be taken in this section since the unknown gas is a comvination of gases. It should be noted, however, that the composition of the gas changed with pressure. The gage must therefore be calibrated under the conditions in the container in which it is used.

$CONCLUSTONS$

An investigation of the effects of condensible vapor was made using dry air, water vapor, and acetamide vapor. The results were compared to the theory of thermal conductivity as a transport phenomenon in kinetic theory of gases. In comparing water vapor and dry air, a crossover occurred at approximately 6.5 torr. This crossover was quantitatively explained by a similar crossover in the low pressure and rarified energytransport condition in the theory. The effects of acetamide vapor were minimized because of its low vapor pressure equilibrium at room temperature. These results were explained in the rarified gas condition. The application in the Echo II canister indicated appreciable error and the presence of water vapor. The data given in this report show significant differences for various gas compositions.

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VITA

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