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CAPTURE COEFFICIENT OF 300°K CO_2 ON A 77°K SURFACE AS MEASURED BY THE ROTATING GAGE TECHNIQUE

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NOTICES

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FOREWORD

The research presented in this report was sponsored by Headquarters, Arnold Engineering Development Center (AEDC), Air Force Systems Command (AFSC), Arnold Air Force Station, Tennessee, under Program Element 65402234.

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ABSTRACT

The capture coefficient of 300°K CO_2 on a 77°K surface is measured using a rotating gage measuring technique. It is concluded that the measured value is 0.99 ± 0.01. However, the experimental data also indicate that the radiation load on the cryosurface may significantly affect the capture coefficient.

CONTENTS

TABLE

ILLUSTRATIONS

NOMENCLATURE

a	Area of gage opening, cm^2
C	Capture coefficient

$\dfrac{dn}{dt}$ Gas flux, molecules/sec

$\left(\dfrac{dn}{dt}\right)_i$ Impinging gas flux, molecules/sec

$\left(\dfrac{dn}{dt}\right)_r$ Rebounding gas flux, molecules/sec

I Ion current

I_i Ion current produced by impinging gas molecules

I_r Ion current produced by rebounding gas molecules

K Constant

M Molecular weight, gm/gm-mole

N_o Avogadro's number

P_i Pressure by gage facing away from hemisphere

P_r Pressure by gage facing toward hemisphere

R Universal gas constant

T Gas temperature, °K

α Fraction of molecules removed by the gage

β Slope of Eq. (6)

δ Error in capture coefficient caused by multiple collisions

SECTION I
INTRODUCTION

The capture coefficient of 300°K CO_2 on a 77°K surface has been reported in previous works (Refs. 1 through 6); however, the capture coefficients of these experiments have varied between 0.5 and 1.0.

This work was initiated in an attempt to make an independent measurement by using a different experimental technique. The method employed was used previously to measure the capture coefficient of H_2 gas on a titanium surface and was referred to as the rotating gage technique (Ref. 7). It was found to avoid many of the experimental difficulties encountered by earlier experimental methods.

SECTION II
EXPERIMENTAL METHOD

Consider a concave hemispherical pumping surface with a capture coefficient C (Fig. 1) and a uniform gas flux $\left(\dfrac{dn}{dt}\right)_i$ impinging on the entrance plane. Situated in the entrance plane of the concave hemisphere is a pressure sensing device, such as a mass spectrometer, which can be rotated toward and away from the pumping surface.
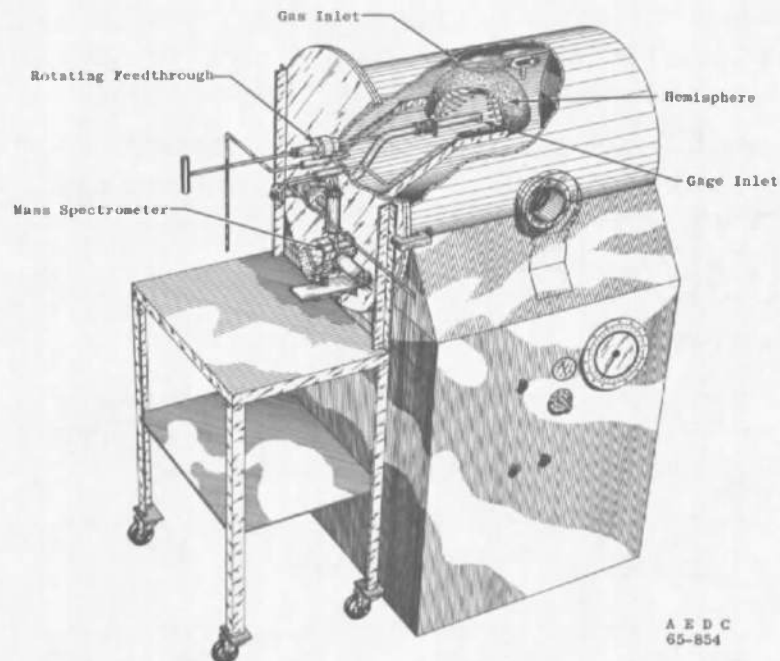

Fig. 1 Vacuum System

1

If it is assumed that:

1. The gas entering the hemisphere is uniformly distributed across the entrance plane of the hemisphere,

2. The capture coefficient of the gas molecules not removed by one collision is unchanged by subsequent collisions with the pumping surface, and

3. The ion current produced in the mass spectrometer is directly proportional to the pressure.

Then it was shown (Ref. 7) that:

1. The gas flux emitted from the hemisphere $\left(\dfrac{dn}{dt}\right)_r$ is uniformly distributed across the exit plane.

2. The ratio of pressures when the gage is rotated is

$$\frac{P_r}{P_i} = \frac{1 - C}{1 + C} \tag{1}$$

where P_i and P_r is the pressure produced in the mass spectrometer by the impinging and rebounding gas flux.

This method of measuring capture coefficients can be shown to have the following advantages.

1. It does not require a calibrated flow system and allows the measurement to be made at any desired pressure in the free molecular flow regime.

2. During the measurement, it is not necessary to isolate additional pumps from the chamber. As a result, these pumps can be used to prevent accumulation of noncondensable gases during the measurement.

3. The substrate area need not be known as long as the gage views only the substrate.

4. It is not necessary that the gage be calibrated, nor will a change in gage sensitivity affect the measurement if a linear gage is used.

5. The capture coefficient measurement is independent of gas temperature.

Although the rotating gage technique has several advantages, some experimental difficulties still exist.

2

It has been assumed that the gas flux to the hemisphere is uniformly distributed across the entrance plane. In practice, this ideal condition may require care to achieve, since the flux will be influenced by the cell's contents, location of gas source, and pump inlets. All of these things will contribute to nonuniformities in the flux.

Secondly, as a result of multiple collisions with the pumping surface, the gas temperature may change and therefore alter the capture coefficient. This would, of course, be contradictory to assumption 2. This will be discussed in more detail later. Further, Eq. (1) does not account for molecules which are re-evaporated from the pumping surface (those caused by the vapor pressure of the surface). It has also been derived on the assumption that the ion current produced in the pressure sensing device is directly proportional to the pressure. However, there is generally a nonzero indication experienced in such instruments, i.e., an ion current is measured even at zero pressure.

The ion current produced by the impinging pressure on a linear response pressure sensing device can be written

$$P_i = K_1 I_i + K_2 \tag{2}$$

where K_2 is the zero indication.

The pressure caused by the rebounding flux can be written in terms of the ion current as

$$P_r = K_1 I_r + K_3 + K_4 I_v \tag{3}$$

Where I_v is the ion current produced by the molecules being re-evaporated from the surface. This current is assumed to be constant and independent of the impinging gas flux.

Equation (1) can now be written

$$\frac{K_1 I_r + K_3 + K_4 I_v}{K_1 I_i + K_2} = \frac{1-C}{1+C} \tag{4}$$

$$I_r = \left(\frac{1-C}{1+C}\right) I_i + \frac{1}{K_1}\left[K_2\left(\frac{1-C}{1+C}\right) - (K_3 + K_4 I_v)\right] \tag{5}$$

Since the last term on the right is a constant, Eq. (1) can be written

$$I_r = \left(\frac{1-C}{1+C}\right) I_i + K^1 \tag{6}$$

If the experimental currents are now plotted, a straight line should result. The slope of this line will be equal to $\frac{1-C}{1+C}$.

The above derivation also demonstrates why it is not necessary for the gage to be calibrated, since it is not necessary that the calibration constant K_1 be known.

SECTION III
APPARATUS

The vacuum system used in this study is shown in Fig. 1. This chamber is 2 ft in diameter and 3 ft long. Its pumping system is composed of a 6-in. oil diffusion pump backed by a mechanical forepump. The pumping system was separated from the chamber by a 6-in. sliding gate valve. A LN_2 cold trap was located between the valve and diffusion pump.

The 5-in.-radius hemispherical LN_2 pumping surface was located as shown in Fig. 1 with the entrance plane in the center of the chamber.

The gas inlet consisted of a 1/4-in. stainless tube formed in a 5-in.-diam ring. For gas to enter the cell, 80 20-mil holes were drilled in the top of the ring.

A mass spectrometer was mounted externally to the chamber with a 1-in.-diam tube penetrating the chamber to the entrance plane of the hemisphere. The tube could be rotated by means of a rotating feed-through, located in the end flange.

SECTION IV
PROCEDURE

Each experimental run began by pumping the cell to a pressure in the 10^{-7} torr region by means of the diffusion pumps. The hemisphere was then cooled by passing LN_2 through the coils. Carbon dioxide gas was next admitted to the cell through the gas addition system. The hemisphere was then precoated with a CO_2 cryodeposit at a cell pressure of approximately 10^{-5} for a period of 2 hr. This resulted in a deposit thickness of about 30,000 monolayers. Next, the leak rate into the chamber was adjusted to result in a cell pressure in the 10^{-5} or 10^{-6} torr region.

The partial pressure analyzer tubulation was rotated so as to view away from the hemisphere, and the readings were recorded. The tubulation was then rotated 180 deg so as to view into the hemisphere, and the reading was again recorded. The gas leak rate was then adjusted to give a different gas flux to the pumping surface and the same procedure repeated.

4

SECTION V
RESULTS AND DISCUSSION

The ion current in the mass spectrometer produced by the gas flux entering and leaving the hemisphere is shown in Fig. 2. A straight line has been fitted to this curve by the method of least squares. From Eq. (6), the slope of this line should be equal to $\frac{1 - C}{1 + C}$. From Fig. 2, the slope is found to be 0.0035 with a standard deviation of 0.00073. This results in a capture coefficient of from 0.996 to 0.997.
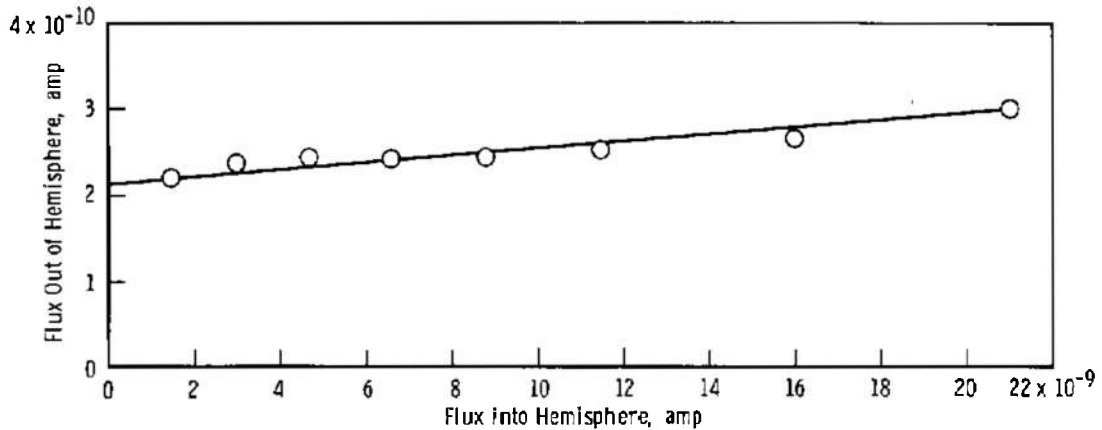
Fig. 2 Mass Spectrometer Ion Currents with Rotating Gage Inlet

4.1 ERROR ANALYSIS

4.1.1 Nonuniform Flux

If the gas flux entering the hemisphere is not uniformly distributed, an error will result, since this was a necessary assumption in deriving Eq. (1). To determine if any nonuniformities existed, the entrance tube of the mass spectrometer was moved across the entrance plane of the hemisphere, both while facing in and out of the hemisphere. No variation in reading was detected.

4.1.2 Capture Coefficient, Change Caused by Multiple Collisions

It is shown (Ref. 7) that Eq. (1) can be written in the form

$$P_r = P_i \left[(1 - C_1) 0.5 + (1 - C_2)^2 (0.5)^2 + (1 - C_3)^3 (0.5)^3 + \ldots (1 - C_n)^m (0.5)^n \right] \quad (7)$$

5

The first term is the pressure produced in the gage by the molecules that have had one collision with the wall. The second term is the pressure produced by molecules which have had two collisions, etc. Since the capture coefficient is probably a function of gas temperature, the molecules which make more than one collision with the surface will have a different temperature and thereby a different capture coefficient from those which strike the surface only once. This will, of course, produce an error in Eq. (1). The worst possible case is when the accommodation coefficient is sufficiently large, so that the capture coefficient is one, for the molecules which make two collisions.

This maximum error, as a result of condensation on the second strike, is the difference between the two expressions

$$\frac{P_r}{P_1} = \frac{1 - C_1}{1 + C_1} \qquad \text{for } C = \text{constant} \qquad (8)$$

$$\frac{P_r}{P_1} = \frac{1 - C_2}{2} \qquad \text{for } C = 1.0 \text{ on second collision} \qquad (9)$$

equating Eqs. (8) and (9), and solving for C_2 in terms of C_1,

$$C_2 = \frac{3C_1 - 1}{1 + C_1} \qquad (10)$$

The error δ will be

$$\delta = \frac{C_2 - C_1}{C_2} \qquad (11)$$

Substituting for C_2 from Eq. (10) gives

$$\delta = \frac{C_2 - C_1}{C_2} = \frac{2C_1 - C_1^2 - 1}{3\,C_1 - 1} \qquad (12)$$

Equation (12) is shown graphically in Fig. 3. From this plot, the maximum possible error caused by a change in capture coefficient as a result of gas temperature change can be determined from the measured capture coefficient C_1. It can be seen that if the measured capture coefficient is greater than 0.75, less than a 5-percent error will result. For smaller values, the error increases rapidly. Possible methods of handling this error are discussed in Ref. 7.

4.1.3 Gage Pumping or Cracking

The pumping of test gases by the pressure sensing devices may have an effect on the pumping speed or capture coefficient measurement. If a mass spectrometer is used, then the "cracking" of molecules has the same result as if the gas were pumped. With the rotating gage technique,

$$\delta = \frac{C_2 - C_1}{C_2} \quad \text{(Reference Eq. (12))}$$
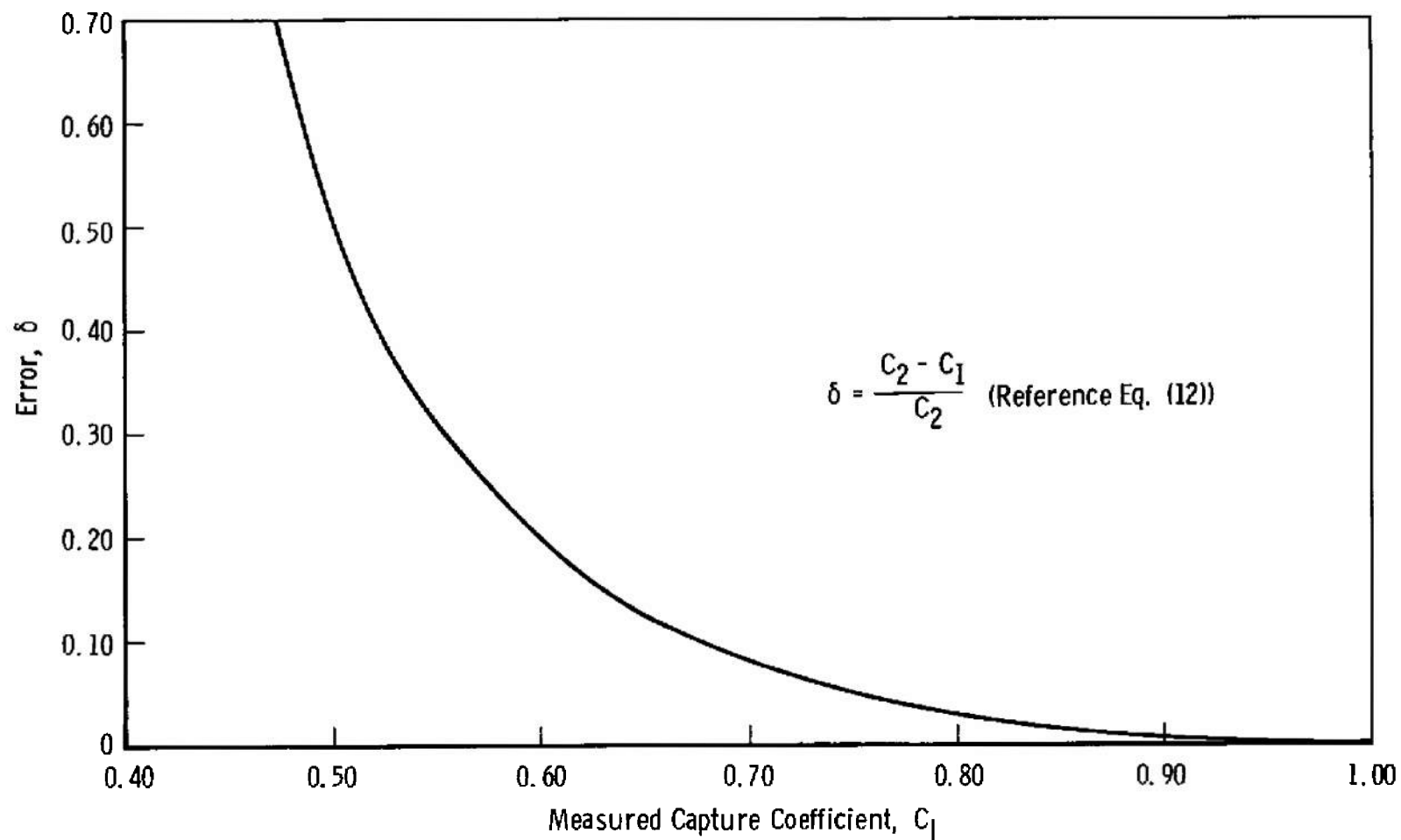
Fig. 3 Maximum Error in Eq. (1) Caused by Multiple Collisions

the removal of test gas by the pressure sensing device will not affect the measurement if the removal rate is proportional to strike rate during the measurement. This can be seen as follows: If α is the fraction of the molecules $\left(\dfrac{dn}{dt}\right)_c$ entering the gage that is removed, then the rate of removal $\left(\dfrac{dn}{dt}\right)_p$ by the gage is

$$\left(\frac{dn}{dt}\right)_p = \alpha \left(\frac{dn}{dt}\right)_c \tag{13}$$

The number flux indicated by the gage is then

$$\left(\frac{dn}{dt}\right)_g = \left(\frac{dn}{dt}\right)_c - \left(\frac{dn}{dt}\right)_p \tag{14}$$

$$\left(\frac{dn}{dt}\right)_g = \left(\frac{dn}{dt}\right)_c (1 - \alpha) \tag{15}$$

This is the same effect as changing the gage sensitivity; however, since the gage sensitivity cancels out of the expression, the result is unchanged.

4.1.4 Sensitivity of Measurement

One method of determining the sensitivity of the measurement is to find the change in the capture coefficient that would result from a change in the slope from Eq. (6).

$$\frac{dI_r}{dI_i} = \beta = \frac{1 - C}{1 + C} \tag{16}$$

$$C = \frac{1 - \beta}{1 + \beta} \tag{17}$$

$$dC = \frac{-2}{(1 + \beta)^2} \, d\beta \tag{18}$$

From Eqs. (17) and (18)

$$\frac{dC}{C} = \frac{-2\beta}{1 - \beta^2} \frac{d\beta}{\beta} = \frac{C^2 - 1}{2C} \frac{d\beta}{\beta} \tag{19}$$

Equation (19) is shown graphically in Fig. 4 for a 10-percent error in measuring the slope. It can be seen that for large values of the capture coefficient, 0.9 or greater, that an error as large as 10 percent in the measured slope will only result in approximately a 1-percent error in measuring the capture coefficient.
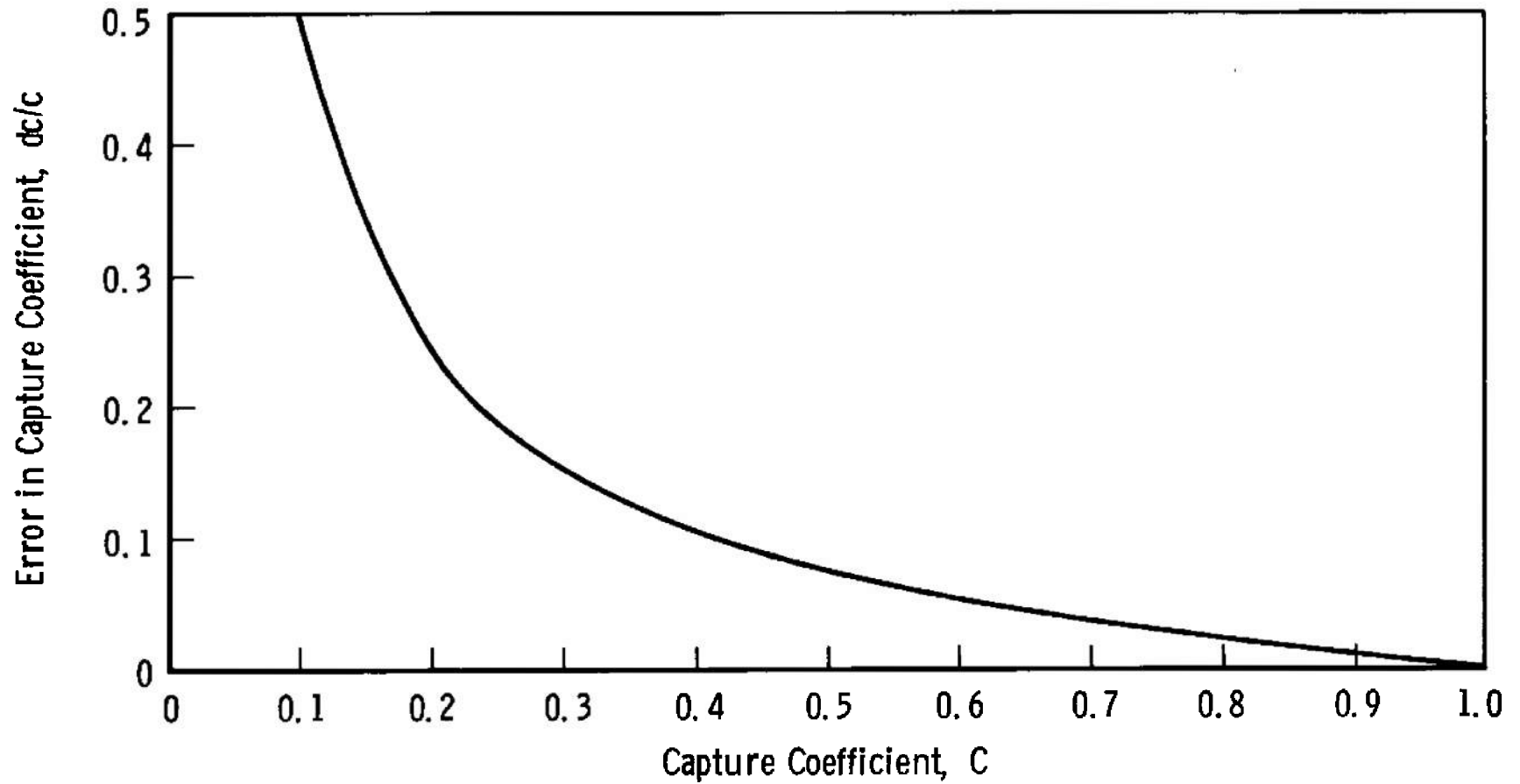
Fig. 4 Error in Capture Coefficient for a 10-percent Error in Slope

From the sources of error considered in the preceding error analysis, it can be concluded that, since no appreciable error was incurred from (1) a nonuniform flux, (2) multiple collisions, or (3) gage pumping and since the slope could be measured to within 10 percent, then the value measured for the capture coefficient is within one percent for large capture coefficients, i.e., greater than 0.75.

SECTION VI
COMPARISON OF RESULTS WITH OTHER INVESTIGATIONS

Some of the variations in the reported value of capture coefficients (0.5 to 1.0) can be attributed to experimental errors, although it is unlikely that such large variations can be entirely a result of experimental error. However, some uncontrolled experimental variables may be the source of the observed differences.

One such parameter, suggested by Haygood (Ref. 8), might be the radiant heat load on the cryosurface. In this investigation, while the gas temperature was 300°K, only half of the radiation from the 300°K surface was incident on the cryosurface. The remaining radiation was emitted from a 77°K surface (self-radiation from the hemisphere).

A preliminary check of this hypothesis was made by adding to the heat load on the cryosurface. Since the experimental apparatus was not well adapted for this purpose, a systematic investigation was not attempted. However, the initial results indicated a significant effect, and further research appears to be warranted.

The object of the investigation was to supply sufficient radiation to the surface of the hemisphere to produce a radiant heat load equivalent to that on a convex cryosurface surrounded by a 300°K surface, with an emissivity of one. Such a surface receives about 0.05 w/cm^2. In this investigation, the surface was only receiving about 0.025 w/cm. It was necessary then to supply an additional 0.025 w/cm^2 uniformly on the hemisphere.

In order to accomplish this, a commercially available "point source," tungsten filament was located one radius below the entrance plane of the hemisphere (Fig. 5). The filament was then resistance heated to supply the necessary power to the filament corresponding to 0.025 w/cm^2 ±0.005 w/cm^2.

This amount of radiation, although small, was sufficient to reduce the capture coefficient approximately 15 percent. Such an effect might
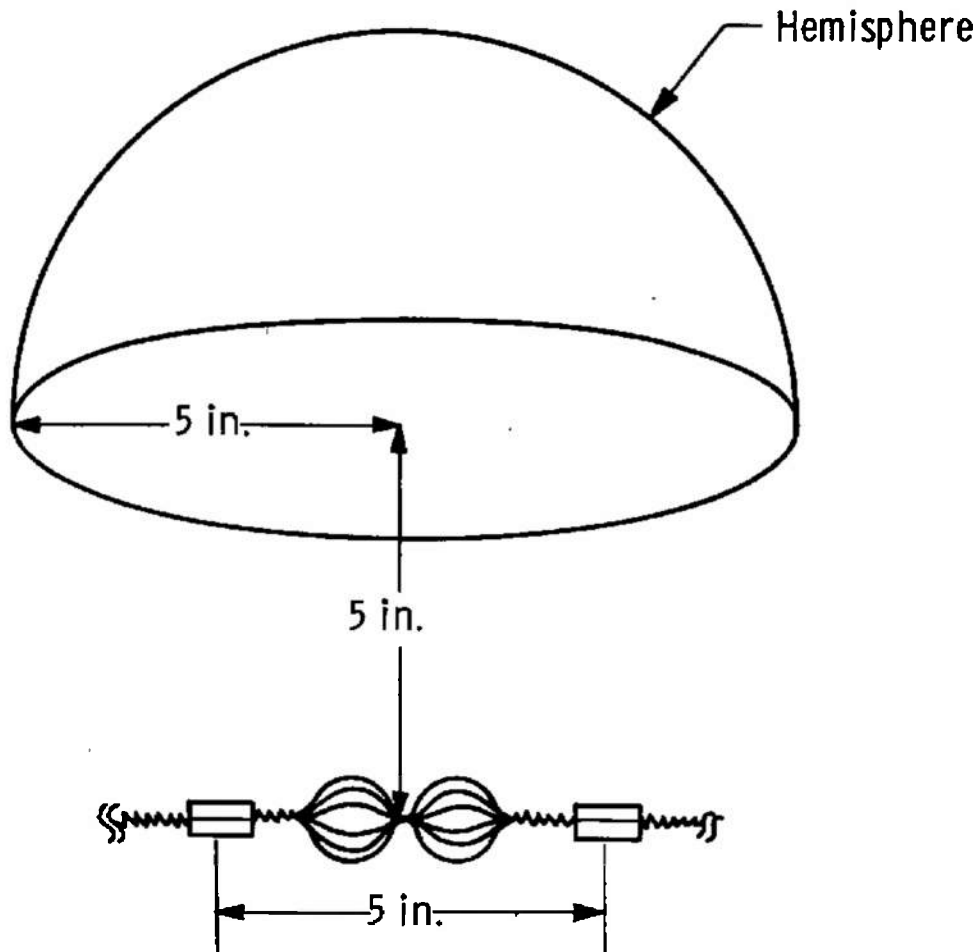
Hemisphere

5 in.

5 in.

5 in.

Fig. 5 Arrangement of Hemisphere and Supplementary Radiation Source

easily explain the discrepancies in the data of previous investigations. Further, if this can be shown to be a general effect, that is, for other gases and cryosurface temperatures, then it might be expected to affect the design of future cryopumps as well as past cryopumping data. For example, Dawson (Ref. 6) has reported the effect of gas temperatures on the capture coefficient of CO_2 on a 77°K surface. These experiments were performed by maintaining a shroud around the cryosurface at the desired gas temperature and assuming that the test gas assumed the temperature of the shroud. It might be possible, however, that the different radiation loads from the shroud had a significant effect on the capture coefficient, rather than the gas temperature.

Table I shows the radiation load assuming an emissivity of one and measured capture coefficient for both Dawson's and this investigation. It should be noted that although the values of the capture coefficient at a radiation load of 0.05 w/cm^2 do not agree, Dawson's values did increase as the radiation load decreased. In fact, Freeman (Ref. 4) has reported that, using the same experimental chamber as Dawson, but a different experimental method, the capture coefficient of 300°K CO_2 surrounded by a 300°K surface is 0.82 to 0.85 which would be in good agreement with this work.

TABLE I
RADIATION LOADS

Investigator	Radiation Temperature, °K	Watts/cm^2 on Cryo-surface Assuming an Emissivity of One	Capture Coefficient
Moody	256	0.023	1.0
Moody	320*	0.060	0.85
Dawson	400	0.145	0.49
Dawson	300	0.046	0.64
Dawson	195	0.00817	0.80
Freeman	300	0.046	0.82 and 0.85

*Heated filament equivalent

SECTION VII
CONCLUSION

It is concluded that the capture coefficient for 300°K CO_2 on a 77°K surface is 0.99 ±0.01 for the geometry used. The results of the cursory investigation of the effect of a small radiation load on the cryosurface indicated that the capture coefficient may be a strong function of the surrounding radiation. Such an effect could explain the discrepancies among previous investigators.

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