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CORRECTION FOR EXTERNAL MASS-TRANSFER RESISTANCE IN DIFFUSIVE SAMPLING

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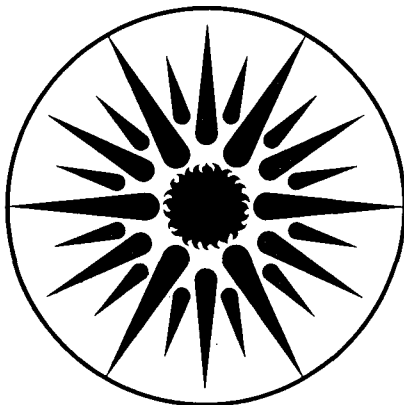
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P. Persoff and A.T. Hodgson

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RESISTANCE IN DIFFUSIVE SAMPLING**

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# CORRECTION FOR EXTERNAL MASS-TRANSFER RESISTANCE IN DIFFUSIVE SAMPLING

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## ABSTRACT

An important source of error inherent in diffusive sampling is depletion of the analyte in the region around the open end of the sampler. Error due to depletion occurs when external mass-transfer resistance is significant compared to the sampler's internal mass-transfer resistance. A simple method to correct for this error is presented. Multiple diffusive samplers of different lengths are simultaneously exposed. A plot of calculated concentration of the analyte at the open end of the sampler versus diffusive flux is extrapolated to a sampler of infinite length, yielding an estimate of concentration in the bulk atmosphere. This method was demonstrated with a diffusive sampler for water vapor by comparing extrapolated concentrations of water vapor to concentrations measured with a dew-point hygrometer.

## Introduction

Diffusive samplers measure the concentration of a gaseous analyte in ambient air or other gaseous environment by measuring the diffusive flux of the analyte through a tube. At steady state, a linear concentration gradient exists from the open end of the tube to the closed end where an efficient sorbent maintains the analyte concentration at zero. Fick's first law gives the flux:

$$J_A = D_A \frac{C_s - 0}{L} \quad (1)$$

where  $J_A$  = flux of analyte along tube, (dimensions  $M/L^2t$ )

$D_A$  = diffusivity of analyte in air, ( $L^2/t$ )

$C_e$  = concentration of analyte at open end of tube, ( $M/L^3$ )

$L$  = length of tube, ( $L$ ).

The sampler is exposed for a measured period of time, and  $J_A$  is determined from the mass of analyte collected by the sorbent. The calculated value of  $C_e$  is taken to represent the concentration of the analyte in ambient air. For a more complete description of the physics of diffusive samplers see Palmes *et al.* (1)

Reliance upon diffusion to deliver the analyte to the sorbent introduces errors into the measurement. The linear concentration gradient takes time to establish; until it is established, the sampler underestimates the concentration.(2) Conversely, if the sampler is not capped with an efficient sorbent material at the conclusion of the sampling period, all of the analyte present in the air within the sampler will diffuse to the original sorbent surface, resulting in an overestimate of concentration.(3) These errors oppose each other, and generally if the sampling time is long enough ( $t \geq 10L^2/D_A$ ), transient effects can be ignored.(2) Another source of error inherent in diffusive sampling, which cannot be eliminated by long sampling times, is depletion of the analyte in the air around the open end of the sampler. This error is commonly termed the "depletion" or "starvation" effect.

For  $C_e$ , the concentration at the open end of the tube, to equal  $C_o$ , the concentration in the bulk atmosphere, the atmosphere must be well mixed. If the atmosphere is not well mixed, the analyte diffusing into the tube is not replaced in the region around the opening, and  $C_e$  will be less than  $C_o$ . The depletion effect is perhaps demonstrated by several experiments in which analyte concentrations determined by diffusive sampling were less than independent measurements of  $C_o$  (4,5) Tompkins and Goldsmith(6) presented the hypothesis that depletion around the opening of a sampler occurs when external mass transfer resistance is significant compared to internal mass transfer resistance. In this paper, we expand on this hypothesis and experimentally demonstrate a simple method to correct for errors in diffusive sampling due to external mass transfer resistance.

### Mathematical Correction

Mass diffusing from the bulk atmosphere to the sorbent surface of a diffusive sampler is assumed to be transferred through two mass transfer resistances in series, one external to the sampler and one internal. Equation (1) can be rewritten

$$J_A = k_i C_s \quad (2)$$

where  $k_i$  is the internal mass transfer coefficient,  $(L/t)$ . At steady state, the same flux goes through the external mass transfer resistance:

$$J_A = k_e (C_0 - C_s) \quad (3)$$

where  $k_e$  = external mass transfer coefficient,  $(L/t)$

$C_0$  = concentration of analyte in bulk atmosphere,  $(M/L^3)$

Tompkins and Goldsmith<sup>(6)</sup> suggested that  $k_e$  be estimated from the Schmidt and Reynolds numbers. This approach requires that the velocity of air in the atmosphere be known. In typical sampling situations, the velocity is presumed to be constantly changing and cannot easily be measured.

A simpler method to correct for the effects of external mass transfer resistance is to expose multiple samplers, identical in all respects except length, to the same conditions. If only two samplers are exposed, equations (1) and (3) are written for each sampler:

$$J_{A_1} = D_A \frac{C_{s_1}}{L_1} \quad (4)$$

$$J_{A_2} = D_A \frac{C_{s_2}}{L_2} \quad (5)$$

$$J_{A_1} = k_e (C_0 - C_{s_1}) \quad (6)$$

$$J_{A_2} = k_e (C_0 - C_{s_2}) \quad (7)$$

where subscripts 1 and 2 represent the two samplers,  $L_1 \neq L_2$ .

The quantities,  $J_{A_1}$  and  $J_{A_2}$ , are determined by analysis;  $D_A$  is known or estimated<sup>(7)</sup>;  $L_1$  and  $L_2$  are known; and the remaining quantities,  $C_{s_1}$ ,  $C_{s_2}$ ,  $k_e$ , and  $C_0$  are solved by elimination. Inspection of equations (4-7) shows that as  $L$  increases,  $C_s$  approaches  $C_0$ ; that is, longer samplers are less affected by external mass transfer resistance.

Rewriting equation (3) as

$$C_s = C_0 - \frac{1}{k_e} J_A \quad (8)$$

shows that if several samplers of different lengths are exposed, a plot of  $C_s$  versus  $J_A$

will be a straight line with slope  $-1/k_s$  and that the  $C_s$  intercept will be  $C_0$ . This is the concentration that would be measured by an infinitely long sampler with infinitely small diffusive flux.

### Experimental Methods

A diffusive sampler for water vapor was used to demonstrate the method of correction for external mass transfer resistance, because of this sampler's ease of analysis and high precision and linearity of response. This water vapor sampler, which uses molecular sieve as the sorbent, was first described by Palmes *et al.*<sup>(1)</sup> and recently has undergone development and testing in this laboratory.<sup>(8)</sup> The diffusion tube is constructed of cylindrical aluminum tubing with a 9.4 mm ID. Diffusive path lengths of the 15 samplers used in this experiment ranged from 40 to 249 mm. Dried molecular sieve (Type 4A, 1.6 mm pellets, Union Carbide Corp., So. Plainfield, NJ) was held in place in a plastic cap (12.7 mm ID, Protective Closures Co., Inc., Buffalo, NY) at the closed end of the tube by a stainless steel screen with a cloth backing. A removable plastic cap was used to close the open end of the sampler before and after exposure. The mass transfer of water vapor was determined by the weight gain of the sampler during exposure. Exposure of water vapor samplers to an atmosphere of pure CO<sub>2</sub> showed that CO<sub>2</sub> is also taken up by the molecular sieve. Consequently, the mass uptake of CO<sub>2</sub> must be subtracted from the total mass gain of each sampler. (In ambient air sampling, uptake of other potential interfering compounds, such as CO, NO<sub>x</sub>, and SO<sub>x</sub>, is negligible.) A mathematical correction for the mass uptake of CO<sub>2</sub> was made based on the concentration of CO<sub>2</sub> in the atmosphere (see Results).

Samplers were deployed in a 20 m<sup>3</sup> indoor environmental chamber for four separate exposure periods of 139 to 167 h duration. The leakage area of the chamber was approximately 0.4 m<sup>2</sup>. During the first two exposure periods, a small cooling fan (17 W) integral with the data logger located in the chamber 1.4 m distant from the samplers provided some mixing of the air in the chamber. A portable room fan (48 W) located 2.7 m distant from the samplers provided additional mixing during the third exposure period. Both fans were removed from the chamber and no mechanical mixing was used during the final exposure period.

The samplers were deployed with their open ends down to exclude dust. They were positioned at least 3.8 cm apart in a rectangular grid in haphazard order with respect to length. This spacing has been shown to be adequate to minimize interference among samplers with dimensions similar to those used in this study.<sup>(9)</sup>



The dew point or frost point temperature of the air in the chamber 0.5 m from the samplers was measured with a chilled-mirror hygrometer (Model 911, EG&G Environmental Equipment, Waltham, MA). This is a primary reference instrument consisting of a thermoelectrically cooled mirror and an optical sensing system which measures the reflectance of the mirror. As water condenses or freezes on the mirror, the change in reflectance is sensed, and the dew point or frost point is continuously tracked. This temperature is measured with a platinum resistance thermometer and was recorded by a data logger every 15 minutes throughout each exposure period. The hygrometer has a response time of  $2^{\circ}\text{C s}^{-1}$  and a rated accuracy of  $\pm 0.25^{\circ}\text{C}$ .

Ambient temperature adjacent to the hygrometer probe inlet was measured with a calibrated thermocouple and was recorded every 15 minutes along with the dew point or frost point temperature. Atmospheric pressure in the laboratory was continuously recorded throughout each exposure period.

Six additional samplers ( $L = 70\text{ mm}$ ) were deployed as blanks during each exposure period. These samplers were left capped at both ends. An increase in weight was attributed to diffusion of water vapor around or through the plastic caps. All samplers were weighed to the nearest 0.1 mg immediately prior to and immediately following exposure with an electronic balance (Model AE 163, Mettler Instrument Corp., Hightstown, NJ).

## Results

Data for the four exposure periods are summarized in Table I.

To compute a corrected diffusive flux,  $J_{\text{H}_2\text{O}}$ , for each sampler, the average mass gain of the blank samplers was first subtracted from the total mass gain of each sampler. In all cases, the average blank uptake plus the standard deviation of the blank uptake was less than 1.7% of the mass uptake of exposed samplers of the same length. Next, the mass uptake of  $\text{CO}_2$  was estimated for each sampler using the following method which is based on the assumption that the external mass transfer resistances for  $\text{CO}_2$  and water vapor are equal. (This would be expected if  $k_e$  depended on air motion and not on molecular diffusion). A trial value for  $k_e$  was selected and the mass uptake of  $\text{CO}_2$  was calculated for each sampler using a literature value for  $D_{\text{CO}_2}$  and a  $C_{0\text{CO}_2}$  of  $400\ \mu\text{L L}^{-1}$ . This concentration is reasonable for a low-occupancy building and was confirmed by a single measurement taken during the third exposure period. For each sampler, the calculated mass of  $\text{CO}_2$  uptake was subtracted from the total mass gain and  $J_{\text{H}_2\text{O}}$  and  $C_{e\text{H}_2\text{O}}$  were calculated. The coefficient  $k_e$  was determined by equation (8)

from a linear regression of  $C_{eH_2O}$  versus  $J_{H_2O}$ . The entire procedure was reiterated using new trial values for  $k_e$  until the trial value matched the computed value.

To test the sensitivity of the extrapolated water vapor concentration to the assumption of equal external mass transfer coefficients for  $CO_2$  and water vapor and to the concentration of  $CO_2$ , the data for exposure 2 were recalculated with the assumptions shown in Table II. If  $k_e$  is determined only by molecular diffusion as would be expected in completely stagnant air, then

$$\frac{k_{eCO_2}}{k_{eH_2O}} = \frac{D_{CO_2}}{D_{H_2O}} = 0.582$$

Both this assumption and the original assumption of a ratio of unity yield essentially the same results. Varying  $CO_2$  concentration from the continental average of  $330 \mu L L^{-1}$  to a high of  $600 \mu L L^{-1}$  resulted in a decrease of  $C_{0H_2O}$  of only three percent.

Plots of  $C_{eH_2O}$  versus  $J_{H_2O}$  are shown for all four exposure periods in Figure 1. From equation (8), the linear regressions describing the relationship have  $C_e$  intercepts of  $C_0$  and slopes of  $-1/k_e$ . Confidence limits of the regression estimates are shown as dotted lines representing the 95% bounds of the slopes.<sup>(10)</sup> The coefficients of determination,  $r^2$ , for the linear regressions are presented in Table I. Also shown in Figure 1 are the water vapor concentrations,  $C_{0H_2O}$ , calculated from the dew-point hygrometer data along with the absolute errors of the measurements determined from the rated accuracy of the instrument.

## Discussion

The data for all four exposures plot as straight lines (Figure 1), with coefficients of determination greater than 0.97. In addition, near agreement was obtained between extrapolated and measured values of  $C_0$  in exposures 1, 2, and 4 (Exposure 3 is discussed in more detail below). These observations support the model of diffusive sampling expressed in equations (1-3) and (8) and the conclusion that mass uptake by diffusive samplers is determined by internal and external mass transfer resistances in series as proposed by Tompkins and Goldsmith<sup>(6)</sup> Consequently, all diffusive samplers are in error. This error will be significant when samplers are so short that external mass transfer resistance ( $1/k_e$ ) is substantial compared with internal mass transfer resistance ( $L/D_A$ ). For example, in exposures 1, 2, and 4, samplers shorter than 100 mm underestimated the average measured concentration by more than 10%. Because the length of most diffusive samplers is less than 100 mm due to practical

considerations, these devices may often significantly underestimate analyte concentration. This error can be eliminated by the method of correction presented here. This new method does not require that  $k_g$  be known in advance or estimated from air velocity data. Accurate  $C_0$  values are obtained simply by exposing two or more samplers of different diffusive path lengths and extrapolating the quantitative data to a sampler of infinite length.

Palmes and Gunnison<sup>(4)</sup> in their pioneering work on diffusive sampling also observed that relatively short samplers underestimate concentration. In one experiment, they exposed water vapor passive samplers of equal cross-sectional areas and varying lengths and found that collection efficiency (defined by them as the ratio of observed mass uptake to predicted mass uptake; in the present nomenclature, collection efficiency =  $C_g/C_0$ ) ranged from 1.04 for a sampler 4 cm long to 0.68 for a sampler 0.5 cm long. For another experiment, in which they exposed  $SO_2$  samplers of various lengths and diameters to the identical atmosphere for the same length of time, it is possible to calculate  $J_{SO_2}$  and  $C_g$  values from the published data. These values are plotted in Figure 2. Although the data are scattered ( $r^2 = 0.65$ ) because of error inherent in the colorimetric method used to analyze the samples, the trend is correct. Longer samplers have smaller  $J_{SO_2}$  values and higher and more accurate  $C_g$  values, and the extrapolated value of  $C_0$  ( $0.48 \text{ nmol cm}^{-3}$ ) is close to the true value ( $\sim 0.45 \text{ nmol cm}^{-3}$ ). No trend with sampler diameter is evident. From the slope of the regression line,  $k_g$  for the experiment is calculated to be  $1.35 \text{ cm s}^{-1}$ , which is approximately one order of magnitude greater than  $k_g$  calculated for the present experiments. This difference is demonstrated by the fact that in Palmes and Gunnison's experiments, samplers 3 cm long had accurate  $C_g$  values, while in the present experiments, a sampler length of 25 cm was needed for accurate results.

More recently, Lindenboom and Palmes<sup>(5)</sup> varied both sampler length and atmospheric pressure using  $NO_2$  diffusive samplers. Collection efficiency was less for short samplers than for long samplers at all pressures and was lowest for the shortest samplers at the lowest pressures. Diffusive sampler response would be expected to be unaffected by pressure, because in equation (1)  $C_g$  is directly proportional to pressure while  $D_A$  is inversely proportional to pressure. The results, therefore, must be explained by external mass transfer resistance. At either short sampler length or low pressure, internal mass transfer resistance is reduced; external mass transfer resistance is not affected, and consequently becomes more important relative to internal mass transfer resistance.

The present and previous experiments demonstrate that the effect of external mass transfer resistance in reducing the collection efficiency of diffusive samplers is not dependent upon the analyte being sampled or the sorbent. The effect has now been observed for SO<sub>2</sub>, NO<sub>2</sub>, and water vapor uptake, with three different sorbents for the latter (molecular sieve, concentrated H<sub>2</sub>SO<sub>4</sub>, and silica gel).

In well-mixed air, external mass transfer resistance ( $1/k_e$ ) is zero by definition. Therefore, it was expected that  $k_e$  would vary with the amount of mixing power in the four exposures. Calculated  $k_e$  values, however, were found to be almost constant for the three exposures with zero or low mixing power and slightly lower for the exposure with high mixing power. This suggests that  $k_e$  was not determined by air mixing, but rather by an unidentified cause such as a possible sampler mouth effect. The lower  $k_e$  for the exposure with high mixing power may possibly be explained by turbulence effectively reducing the diffusive path length since the simulated subtraction of 0.27 cm from the path length of all samplers in that experiment results in a calculated  $k_e$  of 0.16 cm s<sup>-1</sup> which is approximately equal to the  $k_e$  for the other exposures. The nonagreement between the extrapolated and the lower measured  $C_0$  for the exposure is not, however, explained by this effect since the  $C_0$  intercept varies only slightly with small uniform changes in diffusive path length.

#### Acknowledgement

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TABLE I

## Experimental Conditions and Results

Parameter	Exposure			
	1	2	3	4
Duration (h)	167	139	166	139
Avg. temperature ( $^{\circ}$ K)	296	293	296	299
Avg. pressure (torr)	743	742	739	738
Avg. $D_A$ ( $\text{cm}^2 \text{s}^{-1}$ ) <sup>a</sup>	0.263	0.258	0.263	0.269
Mixing power (W)	17	17	65	0
$C_0$ , measured ( $\text{g m}^{-3}$ )	6.01 $\pm$ 0.13	8.32 $\pm$ 0.14	7.76 $\pm$ 0.13	10.35 $\pm$ 0.17
$C_0$ , extrapolated ( $\text{g m}^{-3}$ ) <sup>b</sup>	6.31 $\pm$ 0.08	8.60 $\pm$ 0.13	8.60 $\pm$ 0.13	10.76 $\pm$ 0.21
Coef. of determination, $r^2$	0.985	0.983	0.986	0.974
$k_e$ ( $\text{cm s}^{-1}$ ) <sup>c</sup>	0.170	0.162	0.137	0.159

a Average of diffusivities determined for 15-min intervals using

$$D = \left( \frac{0.26}{p} \right) \left( \frac{T}{298.2} \right)^{1.81}$$

where  $p$  = pressure (atm) and  $T$  = temperature ( $^{\circ}$ K)

b Determined from diffusive sampler data corrected for  $400 \mu\text{L L}^{-1} \text{CO}_2$

c External mass transfer coefficient, determined by linear regression

TABLE II

Sensitivity of  $C_0$  and  $k_e$  for Water Vapor to Values Used  
in Correcting for  $CO_2$  Uptake (Data for Exposure 2)

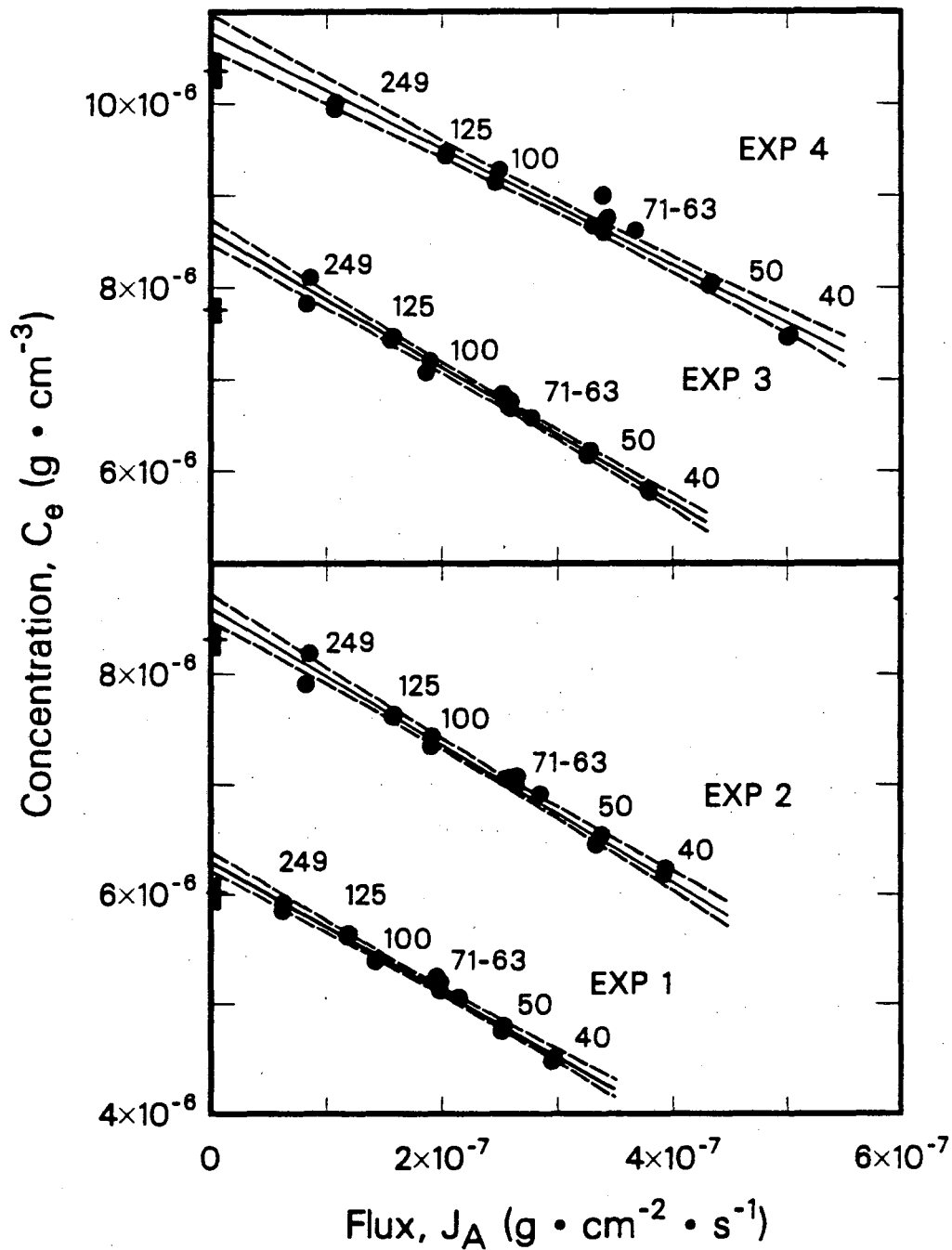
CO <sub>2</sub> concentration ( $\mu\text{L L}^{-1}$ )	Assuming			
	$\frac{k_{eCO_2}}{k_{eH_2O}} = 1.00$		$\frac{k_{eCO_2}}{k_{eH_2O}} = 0.582$	
	$k_{eH_2O}$ ( $\text{cm s}^{-1}$ )	$C_{0H_2O}$ <sup>a</sup> ( $\text{g cm}^{-3}$ )	$k_{eH_2O}$ ( $\text{cm s}^{-1}$ )	$C_{0H_2O}$ ( $\text{g cm}^{-3}$ )
0 <sup>b</sup>	0.166	$9.02 \times 10^{-6}$	0.166	$9.02 \times 10^{-6}$
330 <sup>c</sup>	0.162	$8.67 \times 10^{-6}$	NC <sup>d</sup>	NC
400	0.162	$8.60 \times 10^{-6}$	0.166	$8.60 \times 10^{-6}$
500	0.161	$8.49 \times 10^{-6}$	NC	NC
600	0.160	$8.39 \times 10^{-6}$	NC	NC

a Measured value is  $8.32 \pm 0.14 \times 10^{-6} \text{ g cm}^{-3}$

b No correction for  $CO_2$  uptake

c Continental average  $CO_2$  concentration

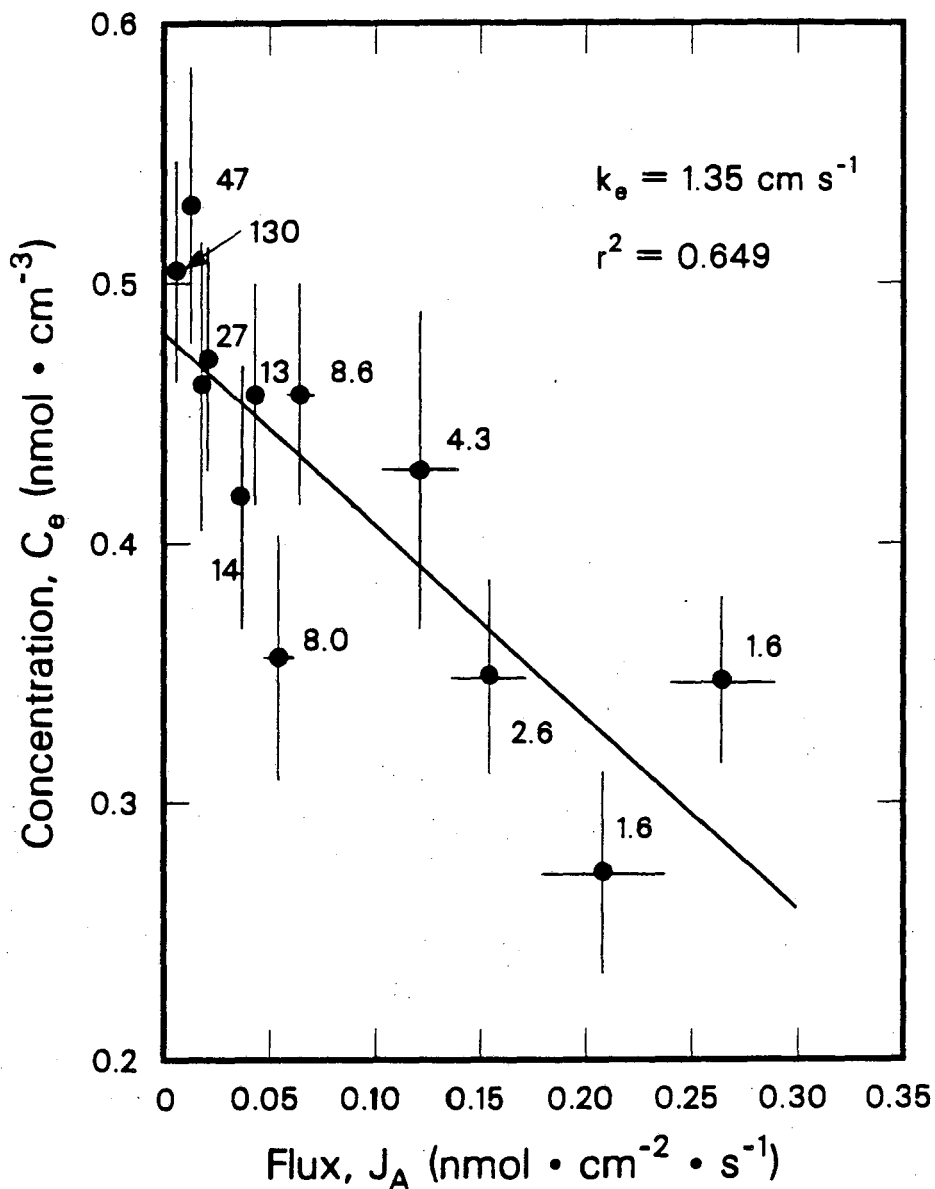
d Not calculated



XCG 849-13259

Figure 1 -- Extrapolation of  $C_e$  and  $J_A$  data for 15 water vapor samplers of various lengths. Data are for four exposures. Dashed lines show 95% confidence limits of the slopes. Heavy bars on left axis are measured values of  $C_0$  (See Table I). Sampler lengths in mm are shown near data points.





XCG 849-13260

Figure 2 -- Extrapolation of  $C_e$  and  $J_A$  data for  $\text{SO}_2$  samplers of various lengths and diameters. Data are calculated from data presented in Table II of Palmes and Gunnison.<sup>(4)</sup> Actual  $\text{SO}_2$  concentration was approximately  $0.45 \text{ nmol cm}^{-3}$ . Error bars are  $\pm$  one standard deviation for measurements made using 18 to 55 samplers. Sampler lengths in mm are shown near data points.

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