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CARBON DIOXIDE ABSORPTION CHARACTERISTICS OF HIGH PERFORMANCE S--ETC(U)
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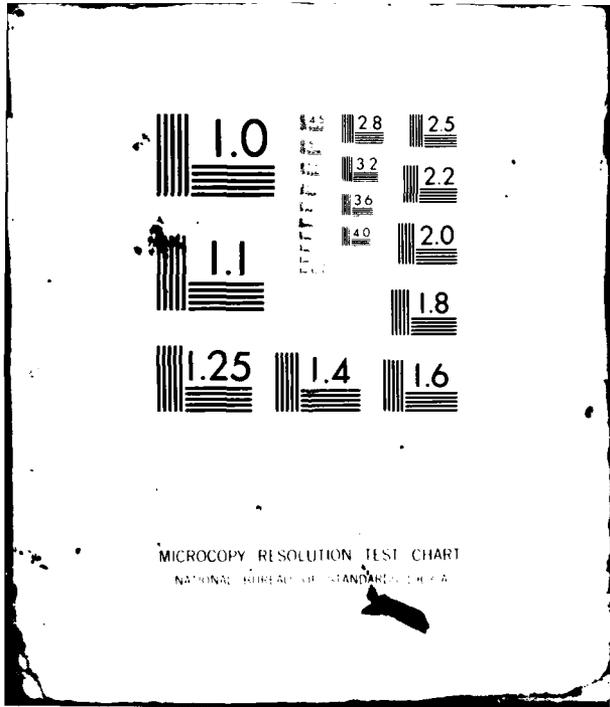
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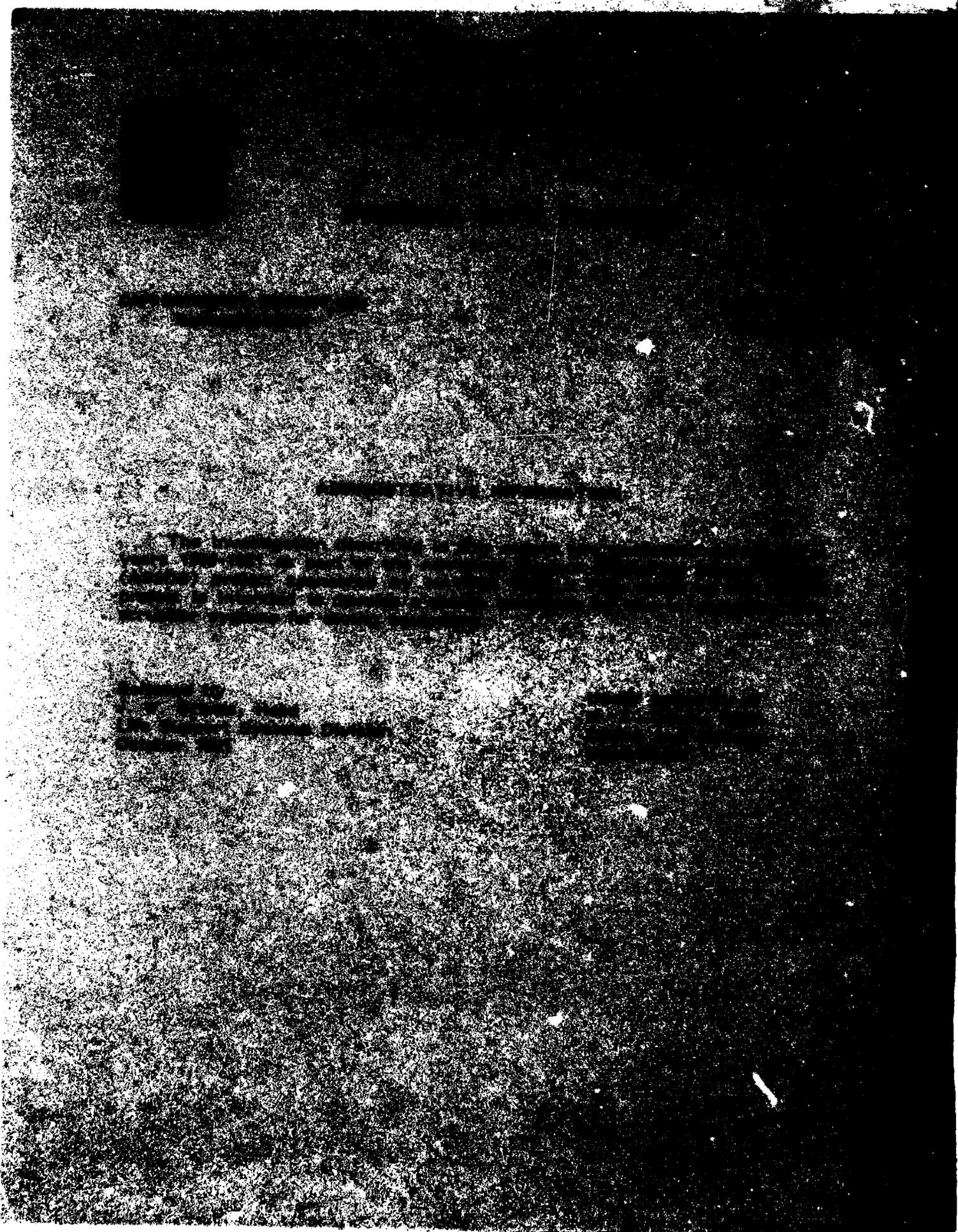
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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER TM 327-81	2. GOVT ACCESSION NO. AD-A107 488	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) Carbon Dioxide Absorption Characteristics of High Performance Sodorb at 1 Atmosphere	5. TYPE OF REPORT & PERIOD COVERED FY 1980-81	
	6. PERFORMING ORG. REPORT NUMBER	
7. AUTHOR(s) A. Purer, G. A. Deason, M. L. Nuckols, and J. F. Wattenbarger	8. CONTRACT OR GRANT NUMBER(s)	
9. PERFORMING ORGANIZATION NAME AND ADDRESS Naval Coastal Systems Center Panama City, FL 32407	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS	
11. CONTROLLING OFFICE NAME AND ADDRESS	12. REPORT DATE October 1981	
	13. NUMBER OF PAGES 22	
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)	15. SECURITY CLASS. (of this report) UNCLASSIFIED	
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE N/A	
16. DISTRIBUTION STATEMENT (of this Report) This document has been approved for public release and sale; its distribution is unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Sodorb; Carbon Dioxide; Absorption; Diving Systems; Temperature; Flow Rate; 1 Atmosphere Pressure; CO ₂ Scrubber		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Standardized methods have been developed to evaluate the efficiency of different carbon dioxide absorbents. Effects of moisture, absorbent cell dimensions, linear velocity, and temperature have been included in this investigation. The use of such standardized procedures will allow data comparisons which have not been possible. The engineering data should also be directly applicable to future canister design.		

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INTRODUCTION

In order to achieve maximum time from semi-closed and closed circuit breathing systems, the highest efficiency must be obtained from the carbon dioxide absorbents. Some of the various parameters that determine the efficiency of such systems are absorbent mesh size, type of absorbent, water content of carbon dioxide containing gas, water content of absorbent, temperature, gas density, thermal properties of gas, residence time, linear velocity, flow type, carbon dioxide injection rate, and packing density.

This report presents the results from the first series of carbon dioxide absorbent testing. The effects of temperature, flow rate, and gas moisture content at 1 atmosphere pressure are investigated. This is an initial report on a continuing Navy task to investigate techniques for carbon dioxide absorption from diver breathing systems and to establish a data base for a consistent systematic engineering approach to carbon dioxide absorbent canister design. The ultimate objective is to obtain useful relationships between the variables for consideration in future carbon dioxide scrubber designs.

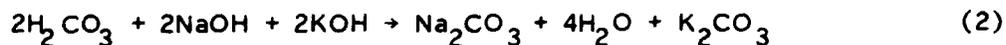
The high performance Sodasorb used in this investigation was manufactured by W. R. Grace and Company. This high moisture type Sodasorb had a reported water content of 14 to 19 percent. Actual analyses revealed a water content of 16.8 percent. The mesh size was reported to be 4-8 Tyler. Actual measurements were:

Larger than 4 mesh	1.1 percent
4-8 mesh	86.3 percent
8-12 mesh	10.5 percent
Smaller than 12 mesh	2.1 percent

Sodasorb contains about 80 percent calcium hydroxide, 14 to 19 percent water, and about 5 percent activators which consist mostly of sodium, potassium, and barium hydroxide. A pH type indicator, ethyl violet,¹ may be incorporated into the mixture. As the caustic NaOH is consumed, the resulting pH change causes the indicator to change from colorless to purple. This color change often represents only surface pH of the Sodasorb particles and thus, under certain canister conditions, is not a reliable indicator of absorbent use.

¹Grace, W. R., "The Sodasorb Manual of Carbon Dioxide Absorption," Library of Congress Catalog, Card No. 62-14923, pp. 33-34, copyright 1962.

The chemical absorption of carbon dioxide by Sodasorb may be represented by the following three equations:



The controlling factors which establish the rate of carbon dioxide absorption are the surface area of the Sodasorb particles, the liquid film present, and Equation (3). The moisture content can change during a run. Gas saturated with water normally would result in an increase in moisture; however, if the heat of reaction (13,500 calories per gram molecular weight, 44 grams) is not dissipated, high temperature areas will result in the removal of water from certain sections of the canister thus rendering such zones ineffective for carbon dioxide removal.

APPARATUS AND PROCEDURE

The laboratory apparatus is displayed in Figure 1. Inlet gas normally containing 1 percent surface equivalent carbon dioxide in helium was used for all runs except those in which carbon dioxide level was the variable of concern. Helium was used as the major component because its high thermal conductivity, 5.8 times that of air,² would dissipate the heat of absorption faster than air and thus assure a more uniform temperature across the absorption cell. The 1 percent carbon dioxide level allowed the heat of reaction to be dissipated over a longer time than a higher carbon dioxide percentage and thus reduced the temperature differential throughout the cell.

Inlet gas flow was controlled with a needle valve and a five-tube rotometer which had been calibrated with helium. The rotometer was also used to ensure a constant gas flow during a run. Gas next flowed through a temperature equilibrating coil fabricated of 8 feet (2.44 metres) of 1/8-inch copper tubing. The coil was submerged in a constant temperature bath before being split into two streams prior to one passing into a bubble tower. By adjusting the ratio of the two gas streams, humidity of the re-united gas stream could be controlled. Absolute humidity of this re-united gas stream was obtained with an EG&G Model 660 Dew Point Hydrometer. This instrument displayed dew point temperature obtained by cooling the gas to its dew point; thus, this instrument was its own standard. These dew point temperature readouts were then converted to

²Weast, R. C. (Editor), "Handbook of Chemistry and Physics," CRC Press, Cleveland, 56th Edition, p. E-2, 1976.

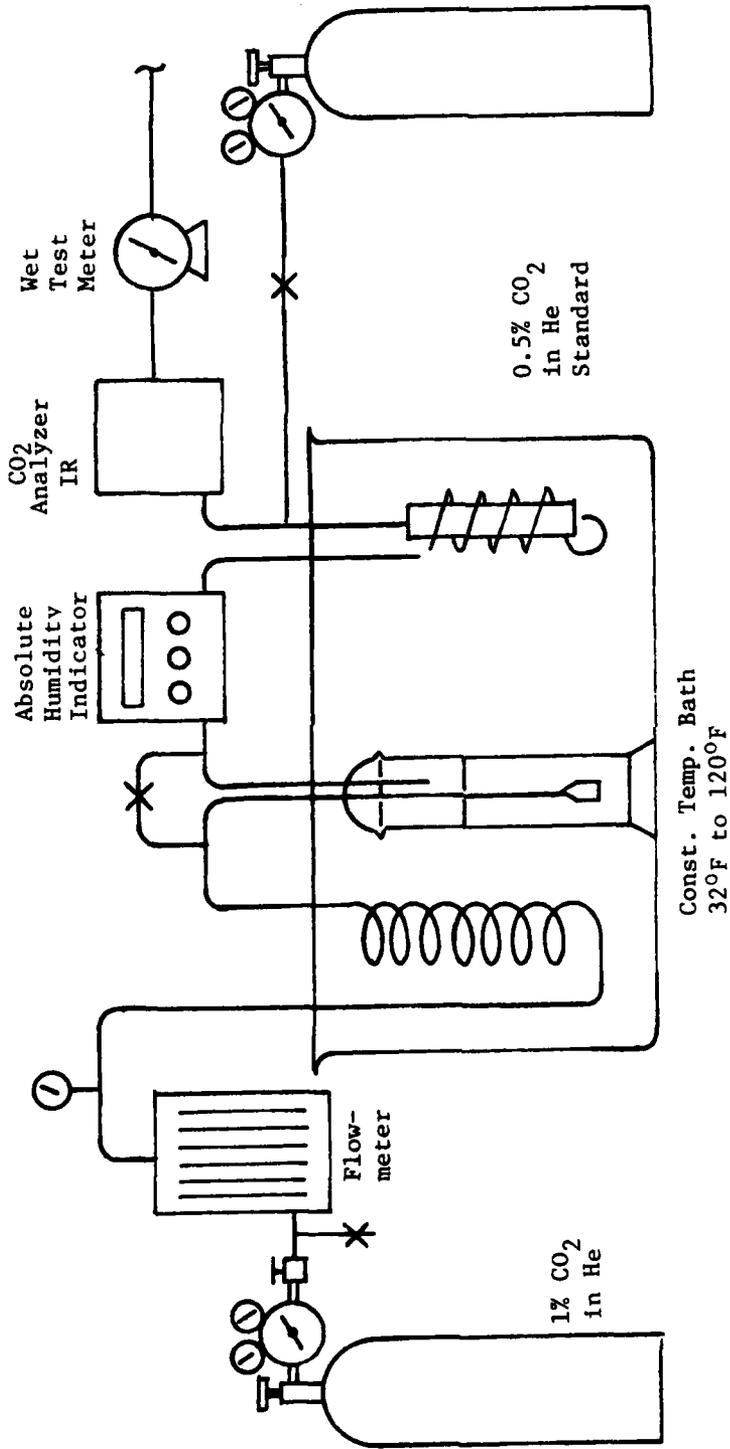


FIGURE 1. CO₂ ABSORBENT MATERIALS STUDY TEST APPARATUS

relative humidity and ppm water. The carbon dioxide containing gas then passed through a temperature equilibrating coil, fabricated of 6 feet (1.83 metres) of 1/8-inch copper tubing, before entering the absorption cell. The absorption cell had an internal diameter of 9.78 millimetres and a length of 12.50 centimetres. A maximum distance of 4.89 millimetres that a gas molecule could be from the constant temperature-maintained copper walls assured rapid dissipation of the heat of reaction. This small cell diameter ensured the near isothermal conditions desired for this parametric investigation.

The absorption cell was equipped with CPV O-ring type connectors. These connectors allowed easy access to the cell while ensuring a gas-tight seal during testing. The flat ends of the CPV connectors facilitated volume calculations. A 5-gram charge of an absorbent of known absolute density was normally used.

Absolute density of the absorbent material was obtained by expanding a known volume of dry helium gas into a container of known empty volume. The container was filled with a weighed amount of the absorbent under study. The need of corrections for manifold volume was eliminated by pressurizing the manifold system to the pressure obtained in the previous expansion. After a few expansions at the starting pressure, the manifold system was at the final pressure before the last expansion and therefore does not contribute to the final pressure. Operating conditions are such that corrections for compressibility factors, temperature, and container volume changes due to pressure are avoided.

The constant temperature bath was capable of maintaining temperature at $\pm 0.2^{\circ}\text{C}$. Temperatures below ambient were obtained using an external cooling system.

The gas stream passed from the absorption cell into the carbon dioxide analyzer. A Beckman Acculab 4 equipped with a 10-centimetre gas cell was used for carbon dioxide detection; this was later updated to a Beckman Model 865 Infrared Analyzer. The instrument was set in a non-scanning mode at a wave number of 2350. Instrument calibrations were made both prior to and after each run. If a calibration changed during the run, the standard obtained immediately after breakthrough was used in the final calculations. Total flow was recorded with a Precision Instruments wet test meter. Each run was terminated when the carbon dioxide level reached 0.5 percent surface equivalent in the outlet gas.

CALCULATIONS

FLOW CALCULATIONS

Flow was monitored by a rotometer before entry into the absorption cell; however, the accuracy of such units is not suitable for total flow

measurements into the cell. The rotometer was only used as a rough indication of flow rate. The wet test meter was used for final flow measurements. However, the values recorded by this unit needed to be adjusted to represent correct flow volumes through the test cell. A temperature correction was first made. The base temperature was 72°F (22.2°C) ambient temperature. Therefore, the volume of gas passed through the cell corrected for temperature was:

$$V_t = V_m \left(\frac{273 + 22.2}{273 + T_1} \right)$$

where

V_t = Total flow corrected for temperature (litres)

V_m = Reading from wet test meter (litres)

T_1 = Temperature of wet test meter (°C).

The gas volume, V_t , was measured at water saturation. The operation of the wet test meter is such that the volume, V_t , included water vapor in addition to the flow being measured.

As dry gas passed through the wet meter, it would become saturated with water; therefore, this water vapor would have to be subtracted from V_t to obtain the dry gas volume passing out of the absorption cell. The corrections are obtained by:

$$V_s = V_t \left(\frac{760 - P_v}{760} \right)$$

where

V_s = Total flow corrected for vapor pressure of water (litres)

P_v = Vapor pressure of water (mm Hg).

The volume of gas, V_s , represented the gas leaving the absorption cell. However, this did not represent the amount of gas entering the cell since about 1 percent of the gas consisted of carbon dioxide. Therefore, the gas entering the cell would be represented by

$$V = V_s + V_s \frac{(\%CO_2)}{100}$$

where

V = Total flow into absorption cell (litre).

The value, V , still contained a small error because the cutoff at 0.5 percent was not abrupt. However, for this engineering-type evaluation, this small error was overlooked.

The flow rate was then determined by:

$$F = \frac{V}{T} \times (1000)$$

where

F = Corrected flow rate into cell (cm^3/min)

T = Total time of flow (min).

The next consideration was that of data presentation. The most convenient presentation would be to divide the total volume, V , of gas passed through the cell prior to the 0.5 percent breakthrough by the weight of the absorbent. This method was not chosen since it tends to favor the lightweight materials. The amount of absorption would be related to the number of moles present and whether one or two moles of carbon dioxide would react with each mole of absorbent. Since many absorbents are mixtures rather than pure materials, it would be difficult to base efficiency on the number of moles present.

A second possibility of presenting efficiency relates absorbed carbon dioxide to canister residence time. However, this calls for a knowledge of the absolute density of the material and the total volume occupied by the bulk material. The disadvantage of this presentation is that residence time of the canister does not represent linear velocity. A canister residence time of 1 second would yield a high linear velocity with a large canister and a low linear velocity with a small canister. The efficiency of an absorbent is related to the linear velocity of a carbon dioxide molecule passing over the absorbent. If the linear velocity is too high, there will not be time for mass transfer. A compromise between residence time, flow rate requirement of the system, and linear velocity will have to be made to allow time for mass transfer and time-related secondary chemical reactions.

A third data presentation method and that used in this report plotted gas linear velocity against litres of gas which passed through the test cell prior to the 0.5 percent carbon dioxide breakthrough level per gram of absorbent in the test cell (1/g). The total volume of the canister occupied by the absorbent, the weight of absorbent, its absolute density, and gas residence time in the test cell must be known to obtain such data. A knowledge of canister geometry allows calculating the volume occupied by the absorbent. The weight of the absorbent is easily obtained by either

weighing the canister before and after filling or weighing the actual material placed in the canister. The absolute density of the material is required to calculate the absolute volume of the absorbent, a necessary parameter in the linear velocity calculation.

ABSOLUTE DENSITY

It is necessary to know the total volume of the canister, the weight of the absorbent, and the absorbent's absolute density to obtain the residence time of a gas molecule passing through a carbon dioxide absorption canister. The dead volume of the system, i.e., volume not occupied with solid absorbent, would then equal total volume minus total weight divided by absolute density of the absorbent. Due to the porous nature of most absorbent materials, it was necessary to develop a special measurement technique for recording their absolute densities. The absolute density of the absorbent was obtained by filling a container of known empty volume with a weighed amount of absorbent. The dead volume, gas volume between the irregularly shaped absorbent particles including small open voids in the particles, is obtained by expanding a known volume of helium at a known pressure into the absorbent-filled canister.³ Low pressures of 0 to 15 psig were used to avoid problems with compressibility factors and temperature changes. A unique procedure was developed which eliminated the need for corrections to the pressure obtained in the previous expansion due to the manifold system. After a few expansions at the same starting pressure, the manifold system is at the final pressure before the last expansion and therefore does not contribute to the final pressure. The gas laws adapted to this application are:

$$P_1 V_1 = P_2 (V_1 + V_2) - I (V_2)$$

solving for V_2

$$V_2 = \frac{V_1 (P_1 - P_2)}{P_2 - I}$$

where

P_1 = Initial absolute pressure (ata)

P_2 = Absolute pressure after expansion (ata)

V_1 = Known volume (litres)

V_2 = Unknown volume (litres)

³Purer, A., Hoffman, C. A., and Smith, D. R., "Chromatographic Determinations of Column Dead Volume and Absolute Density of Absorbents at Cryogenic Temperatures," J. Gas Chromatography, Vol. 6, March 1968.

then substituting

$$P_1 = P_1^1 + 1.0$$

$$P_2 = P_2^1 + 1.0$$

where

P_1^1 = Initial gauge pressure (ata)

P_2^1 = Expansion gauge pressure (ata).

The equation becomes

$$V_2 = \frac{V_1 (P_1^1 - P_2^1)}{P_2}$$

A test was made to check the validity of the above system by measuring the density of a short length of copper tubing of known density. The reported density of 8.92 compared favorably with the determined density of 8.85. Improved agreement could be obtained by the use of more precise instrumentation in the above system such as the use of a fused-quartz pressure gauge in place of a 4 1/2-inch diameter, 0 to 15 psig gauge used in this evaluation.

The absolute density of various absorbents was found to be:

Component	Absolute Density (g/cc)	Bulk Density (g/cc)	Mesh Size
Baralyme	2.15	0.937	4-8
LiOH	1.54	0.456	8-20
Sodasorb (low moisture) 002% H ₂ O	2.54	0.686	4-8
Soda lime, Fisher (4.6% H ₂ O)	2.19	0.703	4-8
Soda lime, VWR (14-19% H ₂ O)	1.96	0.803	4-8
Protosorb	1.90	0.841	12-20
Sodasorb high performance (14-19% H ₂ O)	2.49	0.745	4-8

RESULTS

EFFECTS OF TEMPERATURE, FLOW RATES, AND GAS MOISTURE CONTENT AT 1 ATMOSPHERE PRESSURE

Experimental runs were made at 80°F (26.7°C), 70°F (21.1°C), 50°F (10°C), and 35°F (1.7°C) at various flow rates and saturated, 50 percent saturated, and dry gas moisture contents. The absolute moisture content originally based on relative humidity changed for each temperature; thus, seven different absolute moisture contents were used.

The results of this study are displayed in the 1 atmosphere performance curves of Figure 2. These curves indicate that absolute moisture content of the gas stream, not relative humidity, has the greater effect on efficiency. The moisture content of the gas stream also appears to have more effect than temperature. The effect of temperature is somewhat secondary in that temperature controls humidity. The lower temperatures do slow down chemical reactions, but this may not be the major controlling factor. The controlling factors may vary depending on the linear velocity of the gas molecules, the temperature, and the moisture content of both the gas and absorbent. This is illustrated by the groupings according to water content. The 0 to 6150 ppm H₂O represents a lower group of high performance curves which includes temperatures ranging from 35°F (1.7°C) to 80°F (26.7°C). A second grouping of the curves occurs at a moisture content of around 12,450 ppm H₂O which includes both 50°F (10°C) and 70°F (21.1°C) temperature runs. The third area of efficiency is 25,000 ppm H₂O at 70°F (21.1°C) and 34,600 ppm H₂O at 80°F (26.7°C).

The vapor pressure of water at a specified temperature is a fixed value; therefore, as the total pressure of the system increases, the ppm water concentration will decrease. This is illustrated in Figure 3. At 1 atmosphere of pressure and 70°F (21.1°C), the ppm water content of a saturated system is 24,690 ppm. The same saturated conditions at a depth of 2000 feet sea water reduces the water content to about 400 ppm. This reduction in water concentration could tend to decrease canister performance at higher pressures.

It is interesting to note that at linear velocities greater than 20 cm/sec there is not much difference in the efficiency regardless of temperature or moisture content of the gas stream.

The 50°F (10°C), 100 percent saturated, 12,300 ppm water curve shows a very abrupt change at the 3 to 4 cm/sec linear velocity. This deflection is more related to the selection, or definition, of breakthrough than it is to a sudden increase in absorption efficiency. Under these conditions, the carbon dioxide effluent tends to increase until it approaches the 0.4 to 0.6 percent range. At some point in this range, depending on linear velocity, the absorbent's ability to take up carbon

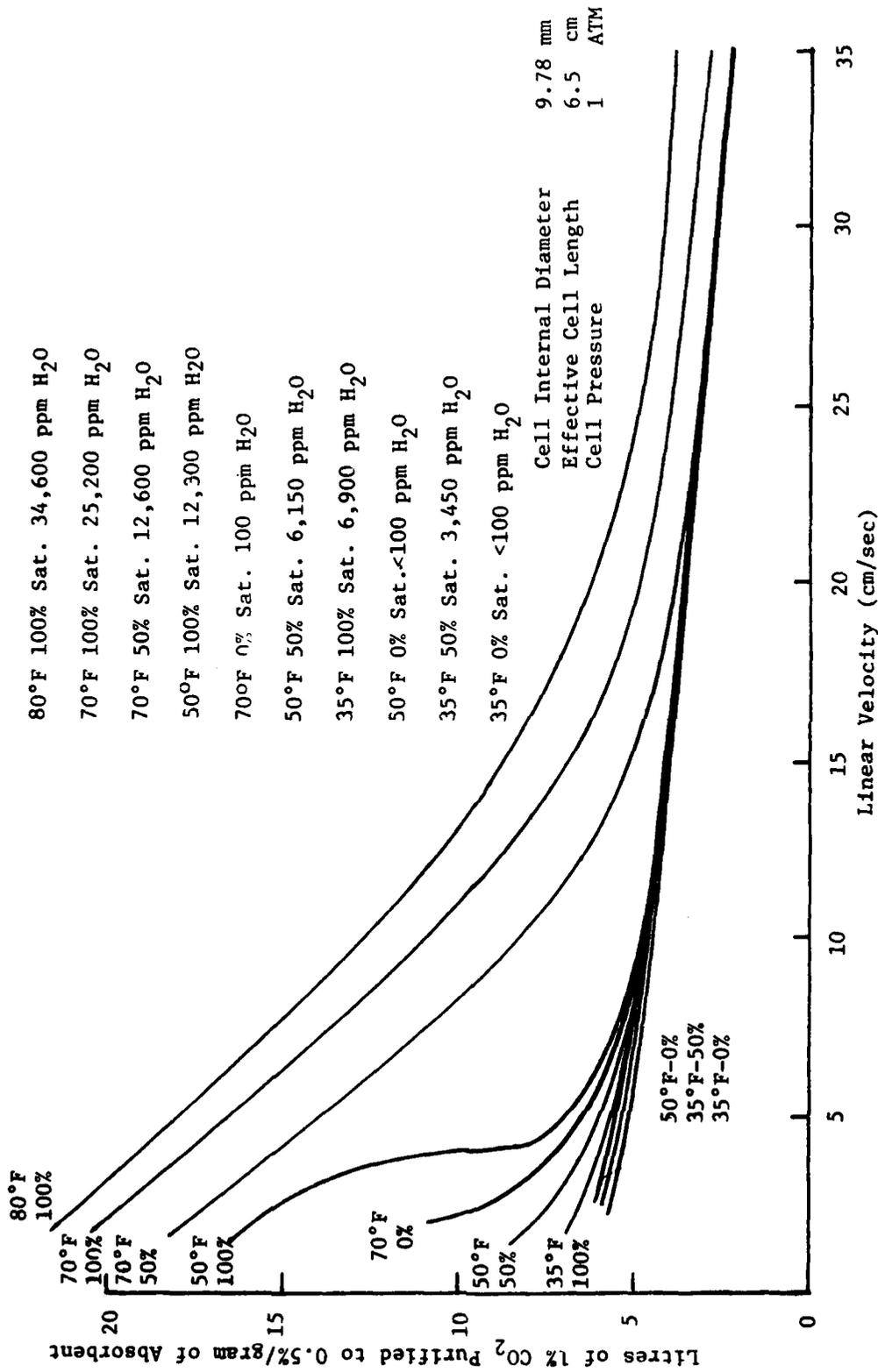


FIGURE 2. HIGH PERFORMANCE SODASORB EFFICIENCY

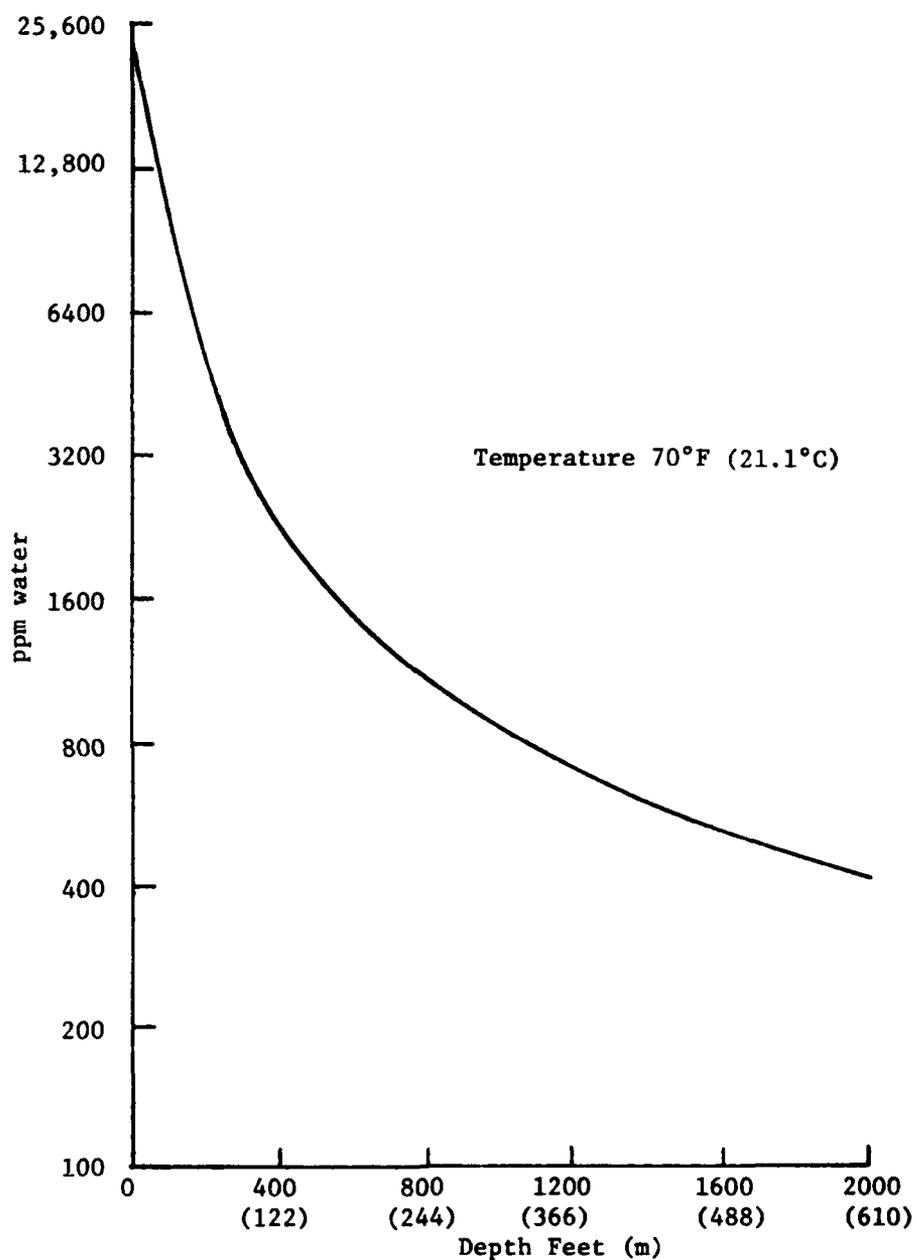


FIGURE 3. PPM WATER CONCENTRATION VERSUS DEPTH AT 70°F (21.1°C)

dioxide increases. This is noted by a drop, up to 15 percent, in the carbon dioxide content of the effluent gas. If this break occurs slightly below breakthrough, 0.5 percent carbon dioxide, it will require 60 to 80 percent more flow of 1 percent carbon dioxide inlet gas to reach the 0.5 percent breakthrough level.

At low flow rates, this breakover occurs before 0.5 percent. However, as flow rates are increased, the first upswing will cross the 0.5 percent carbon dioxide concentration level-off and then drop sometimes below 0.5 percent. However, by definition of breakthrough, the first upswing must be used to terminate the run. This effect is graphically illustrated in Figure 4. It is difficult to obtain reproducible data near the 0.5 percent breakthrough when the carbon dioxide concentration curve is bending over in the same area; therefore, Figure 4 represents the general trend. It is believed that this increase in absorption efficiency is due to accumulation of moisture in the absorbent.

EFFECTS OF CARBON DIOXIDE CONTENT ABOVE 1 PERCENT

The effects of carbon dioxide injection levels above 1 percent at 1 atmosphere pressure were evaluated by holding all variables such as cell geometry, humidity, and temperature constant. The operating conditions chosen were 70°F (21.1°C) and 100 percent moisture saturation (water content 23,200 ppm) for the incoming helium-carbon dioxide mix.

Increasing the carbon dioxide content from 1 to 2 percent would be expected to yield about one-half the L/g of purified gas; however, actual runs showed the value to be much lower than this. Actual data obtained are displayed in Figure 5 for 1 percent, 2 percent, and 4 percent carbon dioxide in helium mixes. Figure 6 represents a normalization of these data; this was achieved by plotting the actual volume of carbon dioxide absorbed for each of the three inlet gas concentrations. If the same total volume of carbon dioxide was absorbed, same efficiency, for each of the three inlet carbon dioxide concentrations, the three curves would have been identical; however, this was not the case. The data show that there is a large reduction in the absolute amount of carbon dioxide that can be absorbed as the inlet carbon dioxide concentration increases. The graphs in Figures 5 and 6 are again based on the 0.5 percent carbon dioxide level as the definition of breakthrough.

THE EFFECTS OF INCREASED ABSORPTION CELL LENGTH

In order to evaluate the effect of increased absorption cell length, absorption cell diameter, carrier gas velocity, and carbon dioxide content were kept constant. Operating conditions were chosen which yielded low absorption efficiency (L/g) to avoid long runs. It was also desirable to avoid flow rates of regions where absorption efficiency was changing rapidly. Thus, small variations in flow rates during a series of runs would have minimum adverse effects on the final results. The efficiency

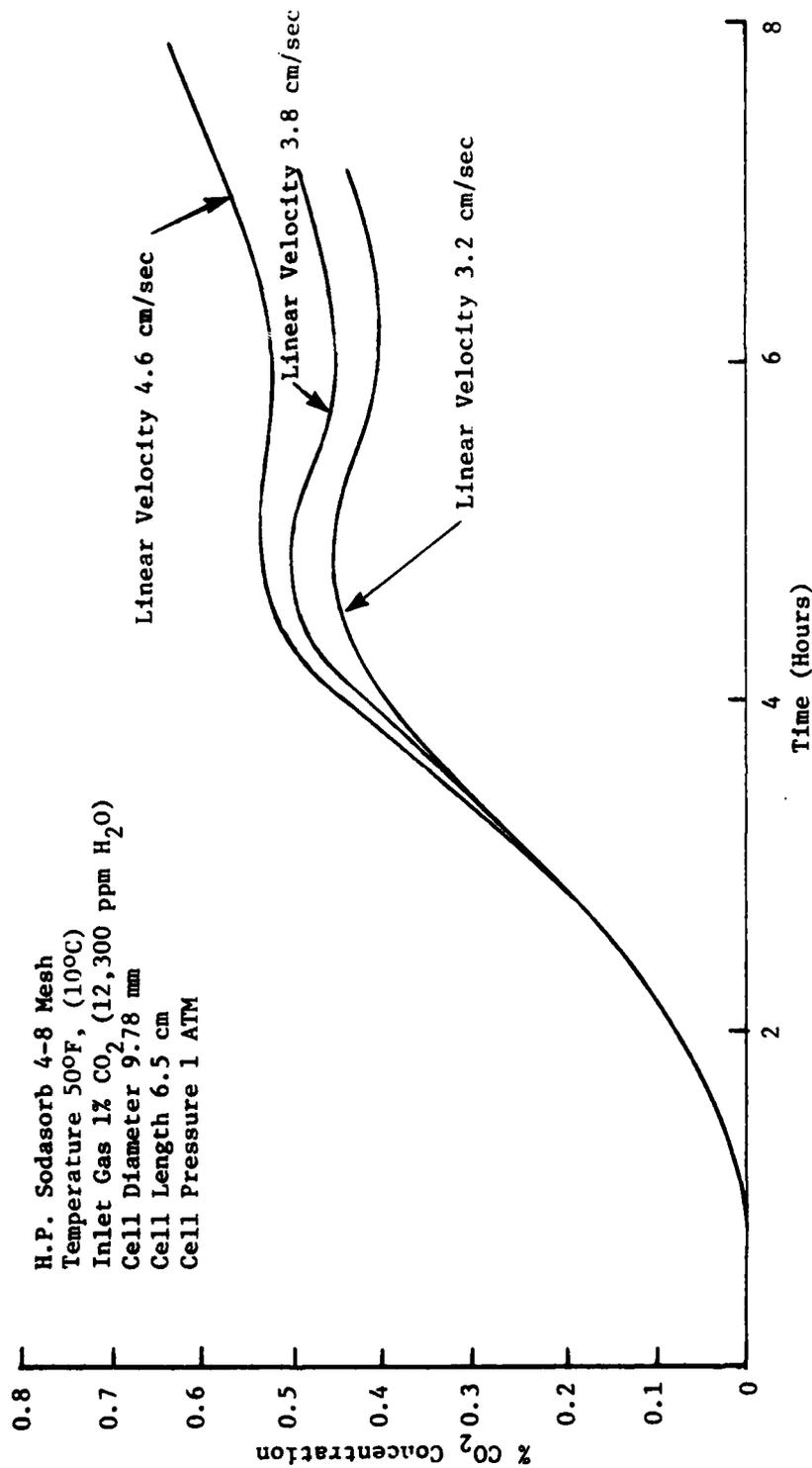


FIGURE 4. CO₂ CONCENTRATION VERSUS TOTAL TIME AT VARIOUS LINEAR VELOCITIES

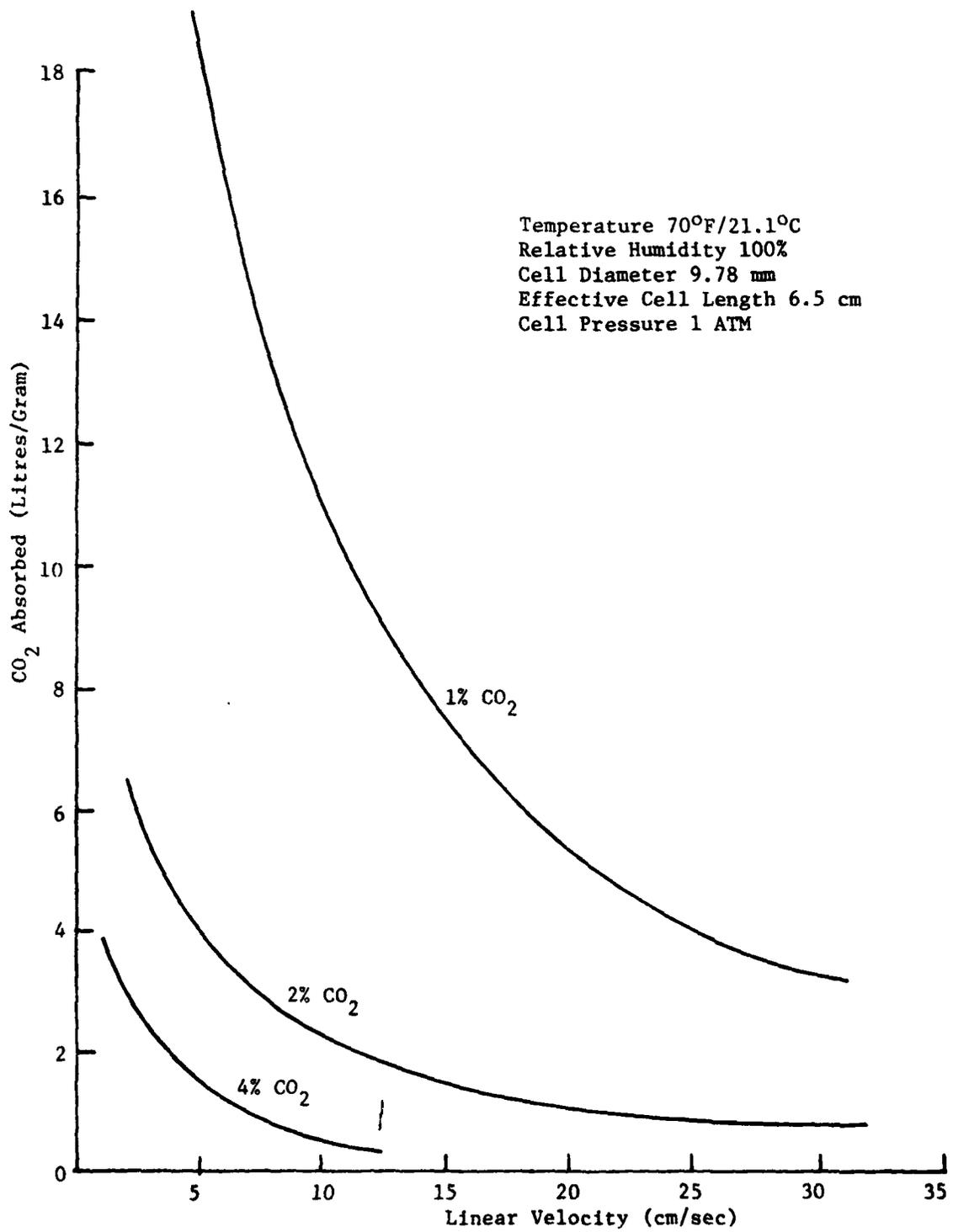


FIGURE 5. EFFECTS OF INCREASED CO₂ INJECTION

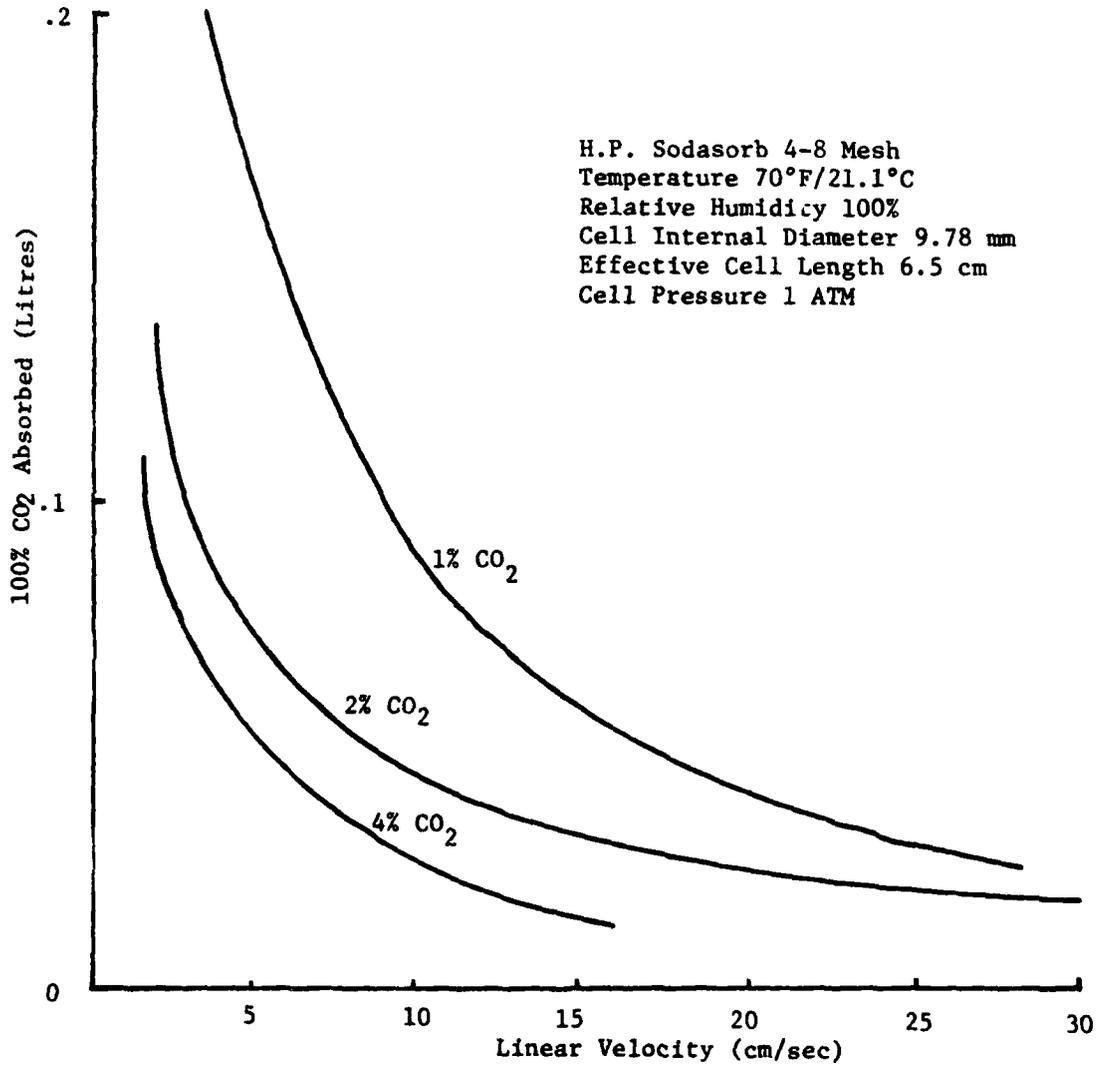


FIGURE 6. NORMALIZED EFFECT OF INCREASED CO₂ INJECTION RATE ON ABSORPTION EFFICIENCY

curves, Figure 2, revealed that these conditions exist at 70°F (21.1°C), 0 percent humidity, and a flow rate which was represented by a linear velocity of about 6 cm/sec to 8 cm/sec, 125 to 190 cm/min. The efficiency curve was somewhat flat over this region. A series of seven runs was made with absorption cell length varying from about 6.5 to 40 centimetres. The results of these studies are represented in Figure 7. The increased efficiency due to increased column length would be expected to change in a similar manner for other locations on the efficiency curves (Figure 2).

THE EFFECTS OF INCREASED ABSORPTION CELL DIAMETER

The effective cell length, temperature, gas composition, and humidity were kept constant in order to evaluate the effect of increased absorption cell diameter on absorbent efficiency. Fixed operating conditions used were cell temperature 70°F (21.1°C), gas mix 1 percent carbon dioxide in helium gas mix, 0 percent humidity, and absorbent bed length of 6.5 centimetres. The variables were cell diameter and linear velocity. Cell diameters used were 0.978, 4.76, 6.45, and 9.37 centimetres with a gas flow linear velocity of about 1 to 18 cm/sec. A series of 28 runs was used in this investigation to obtain the results displayed in Figure 8. For low linear velocities, maximum efficiency is obtained from the smallest diameter cell. This was not unexpected since this cell is similar to a chromatographic column. It is a well-established fact that the smaller the diameter of a chromatograph column, the higher its efficiency.

As linear velocity (Figure 8) increases, the curves converge until at about 4 to 6 cm/sec they cross over. From this point on, the larger cells yield higher efficiency. One reason for this may be that the interior of the larger cells is more thermally removed from the 70°F (21.1°C) constant temperature bath; thus, due to the heat of reaction, may be operating at slightly higher temperatures (removed from the isothermal environment) which would result in improved efficiency. This was confirmed (Figure 9) by placing a temperature probe 8 millimetres from the top of the Sodasorb in the 9.37 centimetre canister. The canister was operated in a 70°F (21.1°C) constant temperature bath. Temperature readings at two different linear velocities showed that the thermally removed portion of the canister was operating at temperatures above 70°F (21.1°C).

CONCLUSIONS

The effects of temperature, flow rates, gas moisture content, increased carbon dioxide content, increased absorption cell length, and increased cell diameter at 1 atmosphere pressure for a non-pulsating system were investigated. It is expected that the efficiency tables and method of data presentation will lead to methods for time duration predictions of future canisters. If the weight of the absorbent, the volume of a proposed canister, the length, diameter, absolute density of the absorbent,

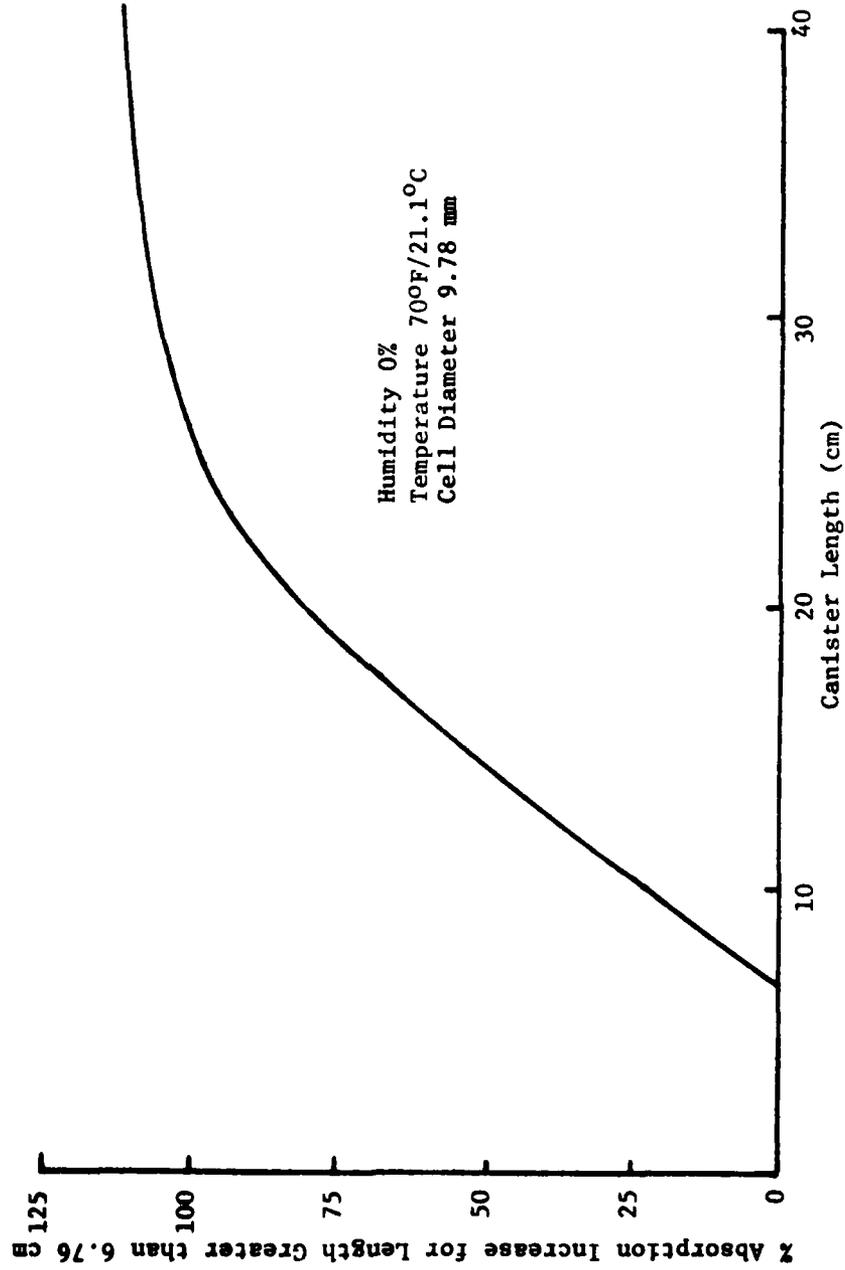


FIGURE 7. EFFECT OF INCREASED CELL LENGTH

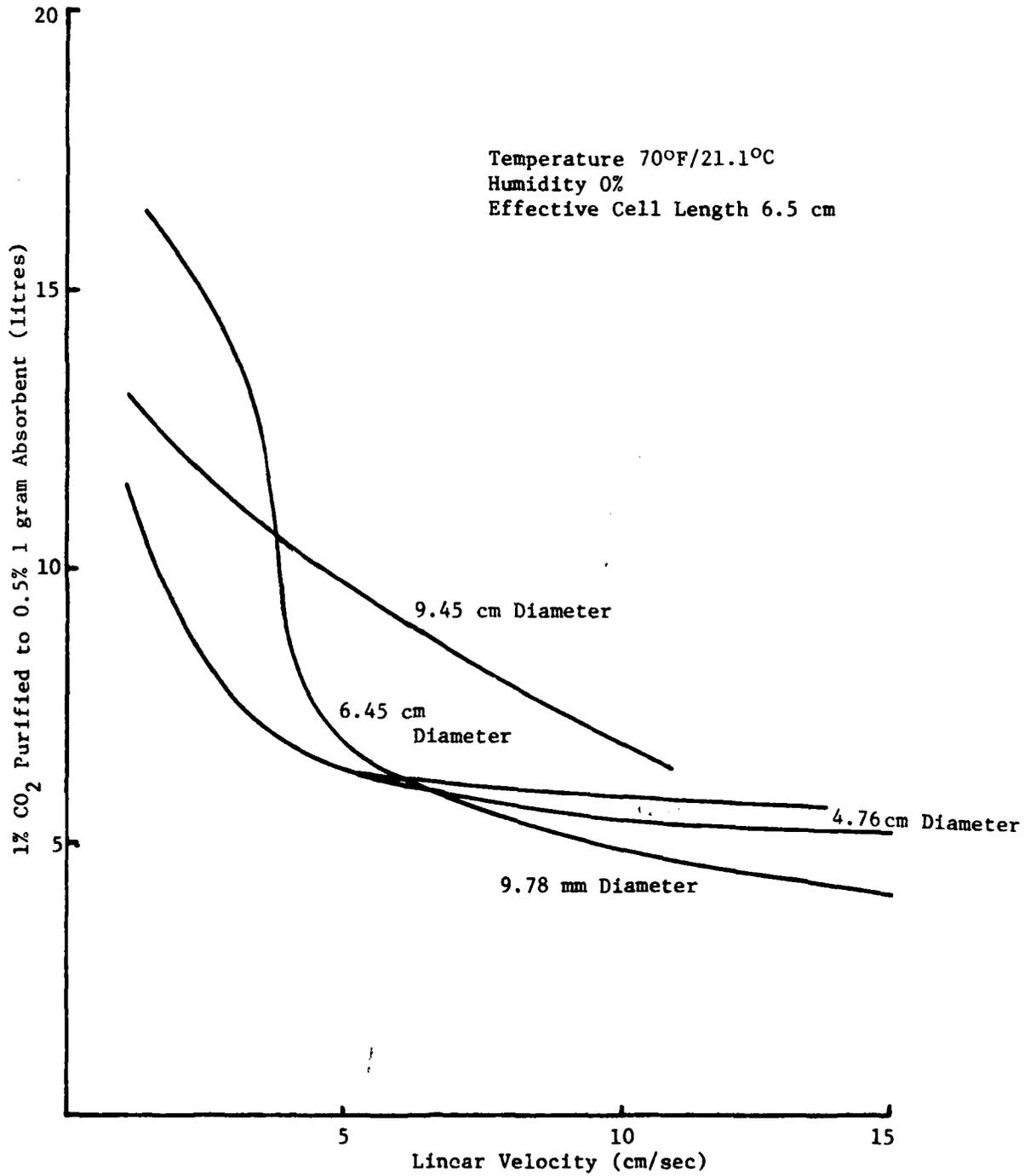


FIGURE 8. EFFECTS OF INCREASED CELL DIAMETER

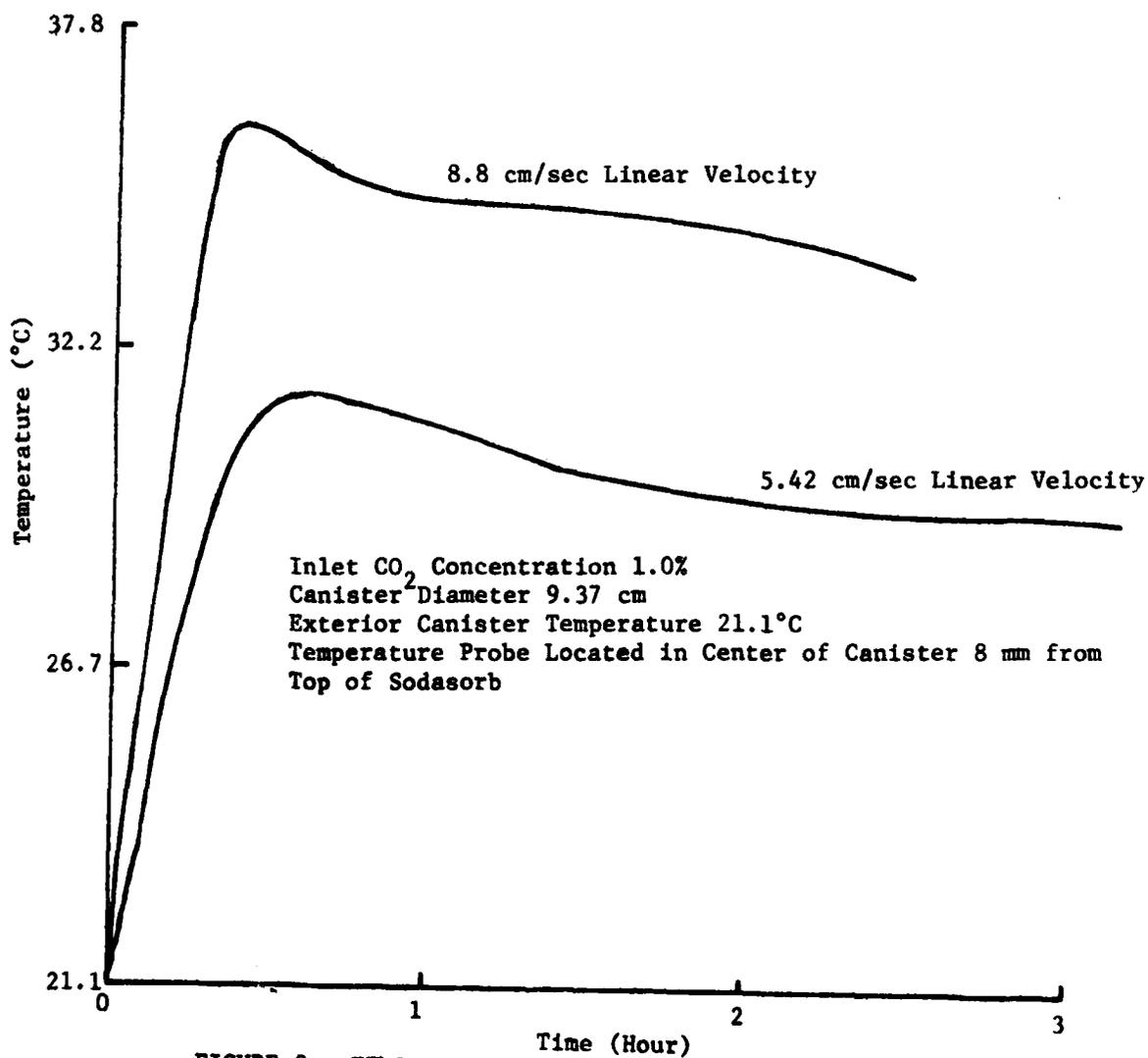


FIGURE 9. TEMPERATURE INCREASE DUE TO HEAT OF REACTION

and the required flow rate are known, one should be able to calculate the approximate time a canister would operate at 1 atmosphere pressure for a steady flow. Future studies now under way will cover changes in efficiency tables due to pressure, absorbent, mesh size, and the effects of pulsating flow.

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