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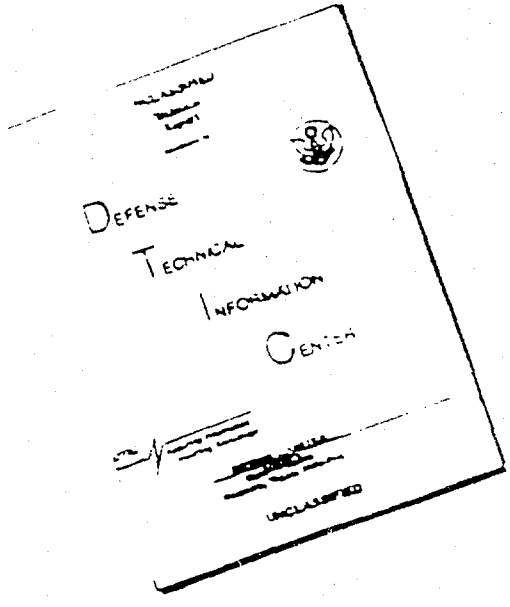

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LOW TEMPERATURE ADSORPTION OF CARBON DIOXIDE

TECHNICAL DOCUMENTARY REPORT NO. ASD-TDR-62-560

September 1962

Flight Accessories Laboratory
Aeronautical Systems Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

PROJECT NO. 6146, TASK No. 6118

(Prepared under Contract AF33(616)8323
by G. Christensen of AiResearch Manufacturing Company,
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FOREWORD

The studies described in this report were performed by the AiResearch Manufacturing Company of Los Angeles, a Division of The Garrett Corporation, under subcontract to the Space and Information Systems Division of North American Aviation, Inc. The Low Temperature Adsorption Study is one of a series of test programs directed toward thermal and atmospheric control systems for manned space vehicles being conducted under Contract AF 33 (616)-8323 sponsored by the Flight Accessories Laboratory of Aeronautical Systems Division. The program was under the direction of A. L. Ingelfinger and Arnold Gross of the Environmental Control Section, Flight Accessories Laboratory. J. P. Allen of the Environmental Control Section acted as monitor of this program.

The Low Temperature Adsorption Study was conducted by G. Christensen and J. R. Wenker. Acknowledgement is given to C. S. Coe and Aaron Shaffer for advice and criticism during the performance of this program.

R. E. Sexton served as program project engineer for the Space and Information Systems Division of North American Aviation, Inc. Acknowledgement is given to Mr. Sexton for advice during preparation of this report.

The studies herein described were performed during the period September 1, 1961 to March 1, 1962. This report is designated AiResearch Manufacturing Company Report SS-715R.

ABSTRACT

A test program was conducted to study low temperature adsorption of carbon dioxide by synthetic zeolites for possible application to space vehicle environmental control systems. Type 5A molecular sieves and a 1 mole per cent carbon dioxide concentration were used for most of the tests.

The range of test conditions included temperatures from 530°R to 300°R, and process air superficial velocities from 30 fpm to 150 fpm. Presented in the report are the results of the tests which include (1) the effect of superficial velocity on adsorption, (2) the effect of bed length on adsorption, (3) the effect of low temperature on adsorption, (4) vacuum and thermal desorption of sieve adsorbed at low temperatures, and (5) miscellaneous data, such as pressure drop, temperature rise, and pellet size. The experimental results are extensively presented and include adsorption curves showing sieve load as a function of time, as well as curves of sieve capacity and adsorption efficiency.

(44 pp)(23 fig)(6 tbls)(0 ref)

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

WILLIAM C. SAVAGE
Chief, Environmental Branch
Flight Accessories Laboratory

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LOW TEMPERATURE ADSORPTION OF CARBON DIOXIDE

SECTION I

INTRODUCTION

General

This report describes the series of tests, their results and conclusions, in the search for a feasible process for regenerable removal of carbon dioxide from space capsule atmospheres. These tests involved the use of crystalline metal alumino silicates, also known as synthetic zeolites, in an adsorption capacity because of their superior capability of adsorbing polar molecules such as water and carbon dioxide. Desorption of the CO_2-loaded synthetic zeolite bed can be accomplished by vacuum, heat, or heat in connection with a non-polar gas. In this way, it is possible to design a regenerable system in which one synthetic zeolite bed is adsorbing CO_2 from the atmospheric gas, while the other is desorbing to space vacuum or by another process.

Synthetic zeolites possess a capacity for CO_2 which depends on temperature. This is illustrated by the curves of Figure 1, published by the Linde Company, Division of Union Carbide Corporation, which show the capacity of Type 5A molecular sieve (a synthetic zeolite) for CO_2 at several temperatures. One purpose of the program discussed in this report was to determine the effect of low temperatures on the adsorption capacity of the synthetic zeolites for CO_2 in a dynamic system which approaches a design which might be used in a space mission. Low temperature adsorption is a possibility in a space mission either through utilization of cryogenic fluids carried in the vehicle for breathing, power, or other purpose, or by means of radiation to space.

Another purpose of the present investigation was to study the desorption of the synthetic zeolites by vacuum, and also thermal desorption by warming the zeolites from the sub-ambient adsorption temperature to ambient temperature.

It is hoped that the information presented herein will be of value to engineers and scientists engaged in the design of carbon dioxide removal systems and the study of the adsorption phonomena. Both literature reviews and past experience have indicated the difficulty of adequately describing or predicting the adsorption phonomena by analytical methods alone. The absence of test data pertinent to a particular design condition, or to a particular adsorbent material, has complicated the problem and frustrated design engineers.

The test data presented herein were obtained using flight type hardware and instrumentation as opposed to a small laboratory type setup. The data is presented in an engineering fashion, and the actual adsorption curves

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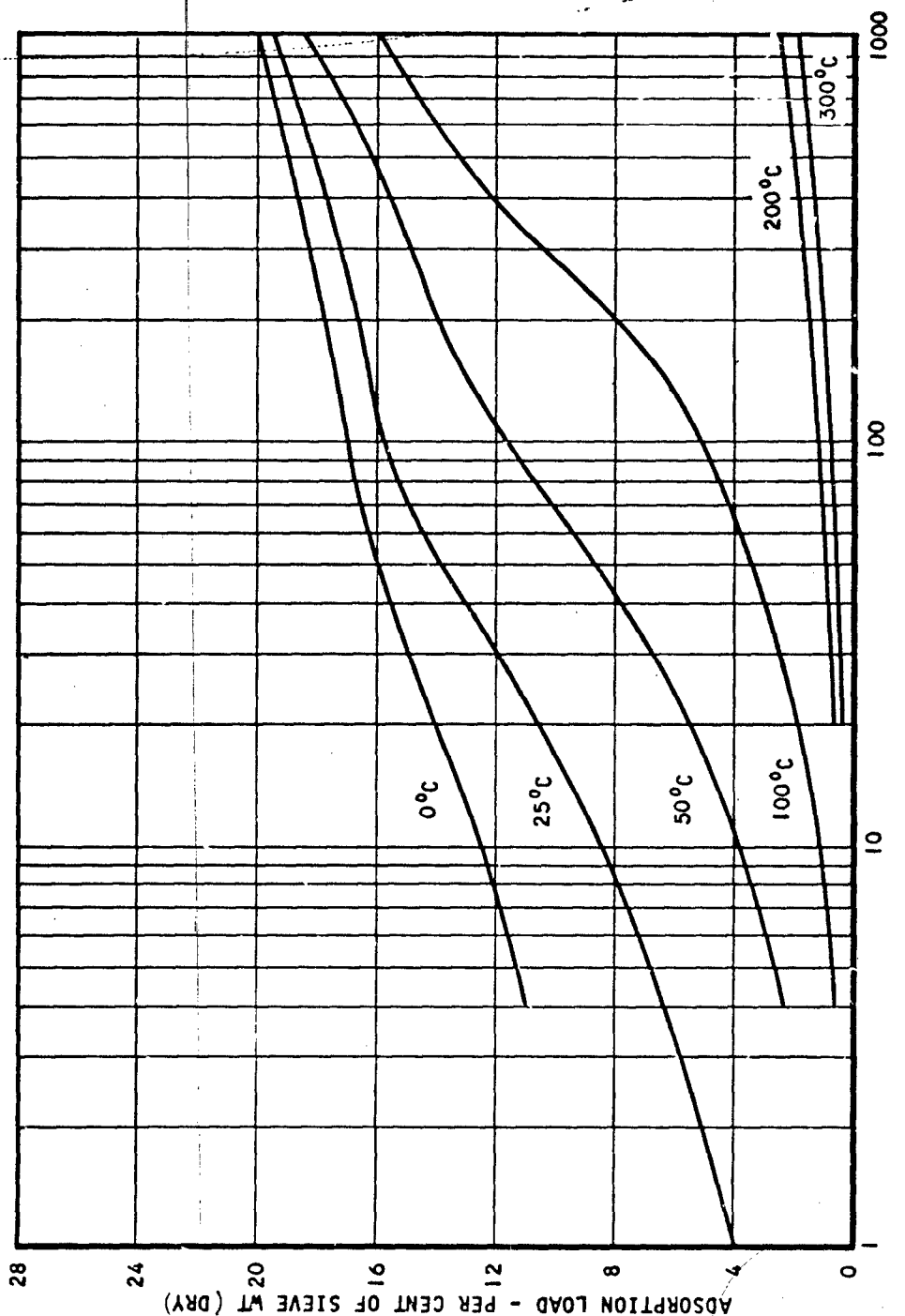


Figure 1. Carbon Dioxide Capacity, Type 5A Sieve (Linde Data)

have been included and are shown as function of the weight load of the carbon dioxide adsorbed. Design engineers should find these adsorption curves extremely useful in sizing and designing molecular sieve canisters for carbon dioxide removal. In particular, since regenerable carbon dioxide adsorption systems are dependent upon the cycling adsorption and desorption of the molecular sieves between load points other than fully desorbed, these intermediate load points and the corresponding carbon dioxide adsorption rate between these points can be readily predicted from the curves presented herein.

Discussion of much of the data presented has been limited to that of a general nature, since particular problems can best be analyzed by a study and comparison of the curves.

Synthetic Zeolites

Based on the property of the natural zeolites to adsorb a variety of gases, the synthetic zeolites were developed to obtain materials of greater selectivity and adsorptive capacity. These materials are crystalline metal alumino silicates, characterized by a large number of uniform pores. Pore size is controlled by varying the nature of the metal ion included in the crystal lattice.

Synthetic zeolites are manufactured by the Linde Company, Division of Union Carbide Corporation, under the name molecular sieves and by the Davison Chemical Company as microtraps. Both materials are stated to be made under the same basic patents. Molecular sieves are supplied in the form of pellets or beads, while the microtraps are supplied in the form of beads and granules. Proprietary surface treatments vary, but in both cases, a clay material is used to bond the crystals into a large particle.

The adsorption properties of the different synthetic zeolites are described by a series of code numbers. These are tabulated in Table I.

TABLE I

ADSORPTION PROPERTIES OF SYNTHETIC ZEOLITES

Pore Diameter, Å	Linde Designation	Davison Designation
4	4A	510-517
5	5A	520-526
10	13X	530-

As expected, the adsorption of various molecules on the synthetic zeolites will be a function of their size. Carbon dioxide is observed to be adsorbed more strongly by synthetic zeolites of pore diameters 5 and 10 angstroms. In addition to physical size, the polarities of the molecule are also very important in controlling adsorption.

SECTION II

TEST APPARATUS

Test Setup

A schematic of the test apparatus is shown in Figure 2. A laboratory air supply, cleaned and dehumidified to a $0^{\circ}F$ dew point, was used as the air source. A low pressure regulator was used to control the air flow into the test system. The air first passed through the air drier, which consisted of a layer of silica gel followed by approximately 50 lb of Type 13X molecular sieve. A low air velocity through the drier was used to insure a minimum dew point. The sieve and silica gel were regenerated regularly to remove the adsorbed moisture. The dry air, after leaving the drier, passed through an orifice flow measurement section. Different orifice sections were used for the different flow rates to insure the best accuracy possible in reading the calibration curves. Each flow section was accurate to approximately ±2.5 per cent. After the flow measurement, the air passed through the various temperature control devices. The dry ice cooler, in conjunction with an electrical heater, was used to obtain the $530^{\circ}R$ and $500^{\circ}R$ temperatures. The liquid nitrogen cooler, which consisted of a series of copper coils immersed in liquid nitrogen in a stainless steel dewar, was used to obtain the lower temperatures. A warm air by-pass was used to regulate the air temperature out of the liquid nitrogen cooler. The carbon dioxide was also metered through a needle valve into the warm air by-pass line to avoid carbon dioxide freeze-out in the injection tube at the low temperatures and to insure proper mixing of the carbon dioxide and process air.

Downstream of the temperature control equipment, the process air dew point was measured, as well as the carbon dioxide concentration and oxygen partial pressure. Temperature and pressure measurements were also made at this point. See the Instrumentation paragraph of this Test section for a description of the instruments used.

A drawing of the test canister is shown in Figure 3. The canister was all aluminum, 3.55 inches in diameter and 18 inches long. Two sleeves of 6 and 12 inches could be inserted in the canister to reduce the bed length to 12 and 6 inches respectively. The inlet and outlet ducts were 1.5-inch O.D. beaded tube. Each end of the bed had a fine mesh screen and a baffle plate with 0.25-inch diameter holes to contain the sieve and to insure uniform flow distribution. During the tests, the canister was covered with 1 inch of insulation to minimize heat transfer. One of the preliminary test canisters is shown in Figure 4. This canister was six inches in diameter and was used for preliminary tests in establishing the final test canister design.

Downstream of the test canister, process air temperature, carbon dioxide concentration, and oxygen partial pressure were measured.

Vacuum desorption of the molecular sieve was accomplished using a Kinney KMB 1200/KDH-130 vacuum pump. This pump is rated at 1000 cfm in the range from 0.5 mm Hg to 0.01 mm Hg and 750 cfm at 0.001 mm Hg.

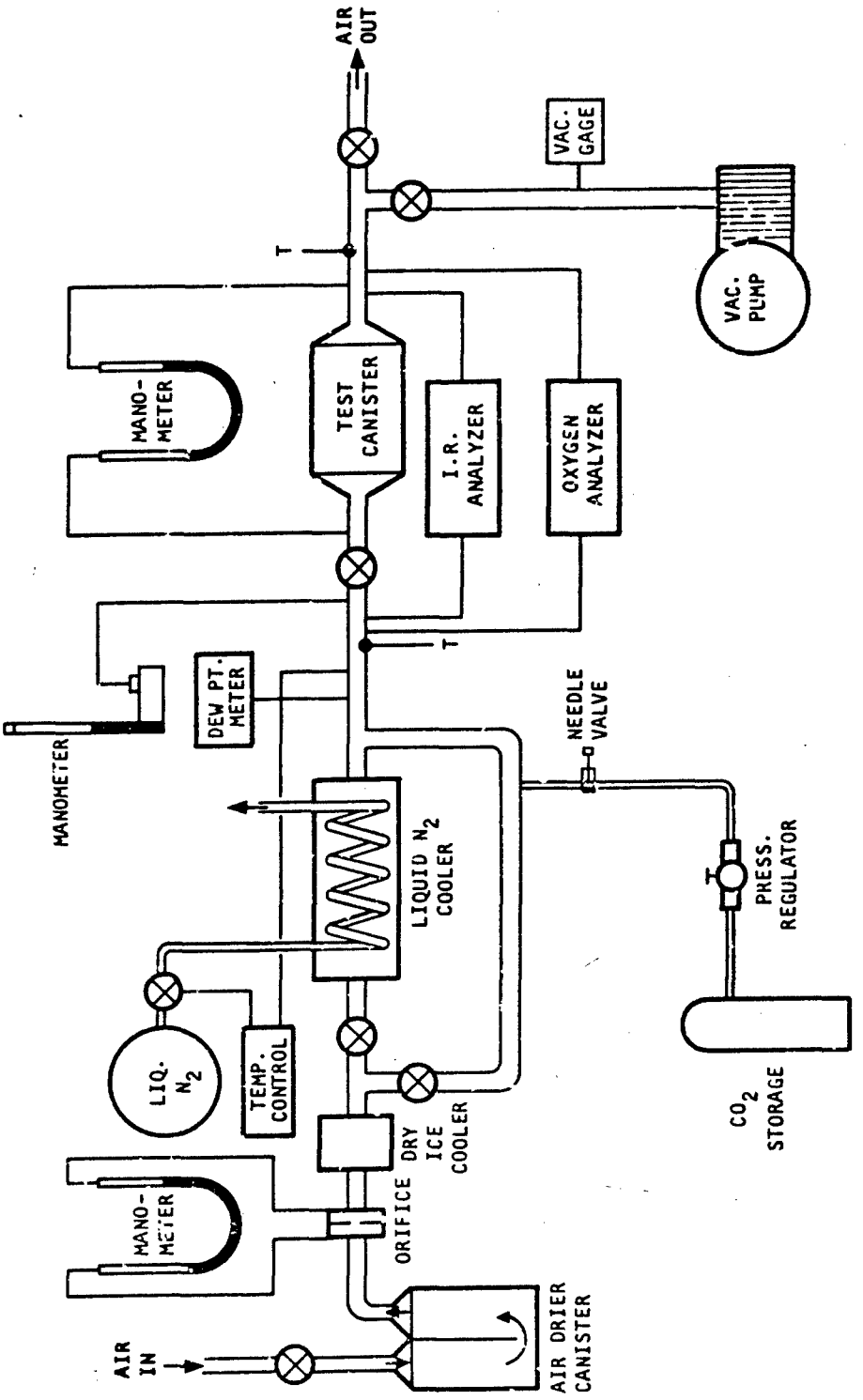


Figure 2. Test Setup, Low Temperature Adsorption Tests

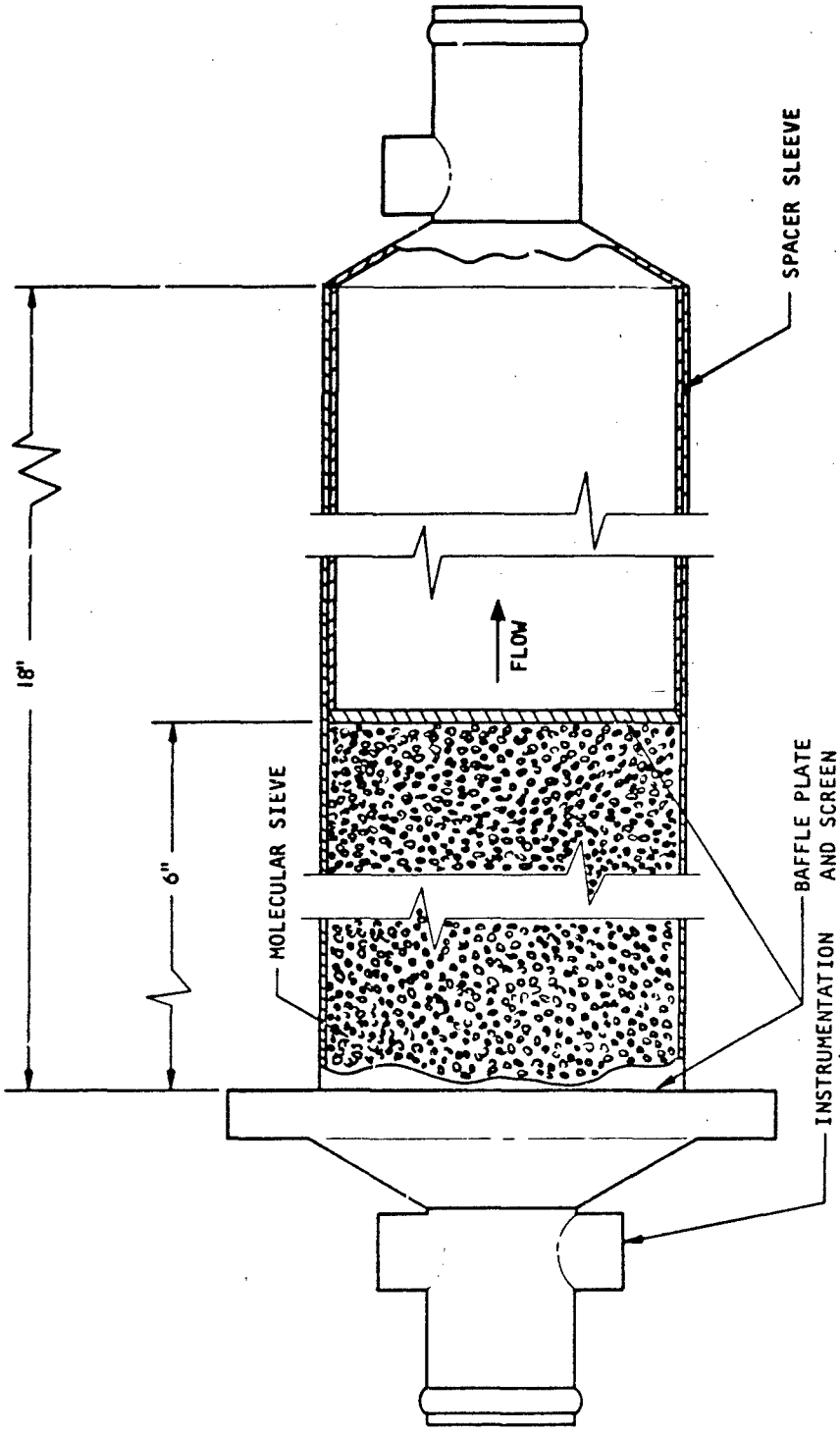


Figure 3. Test Canister Design

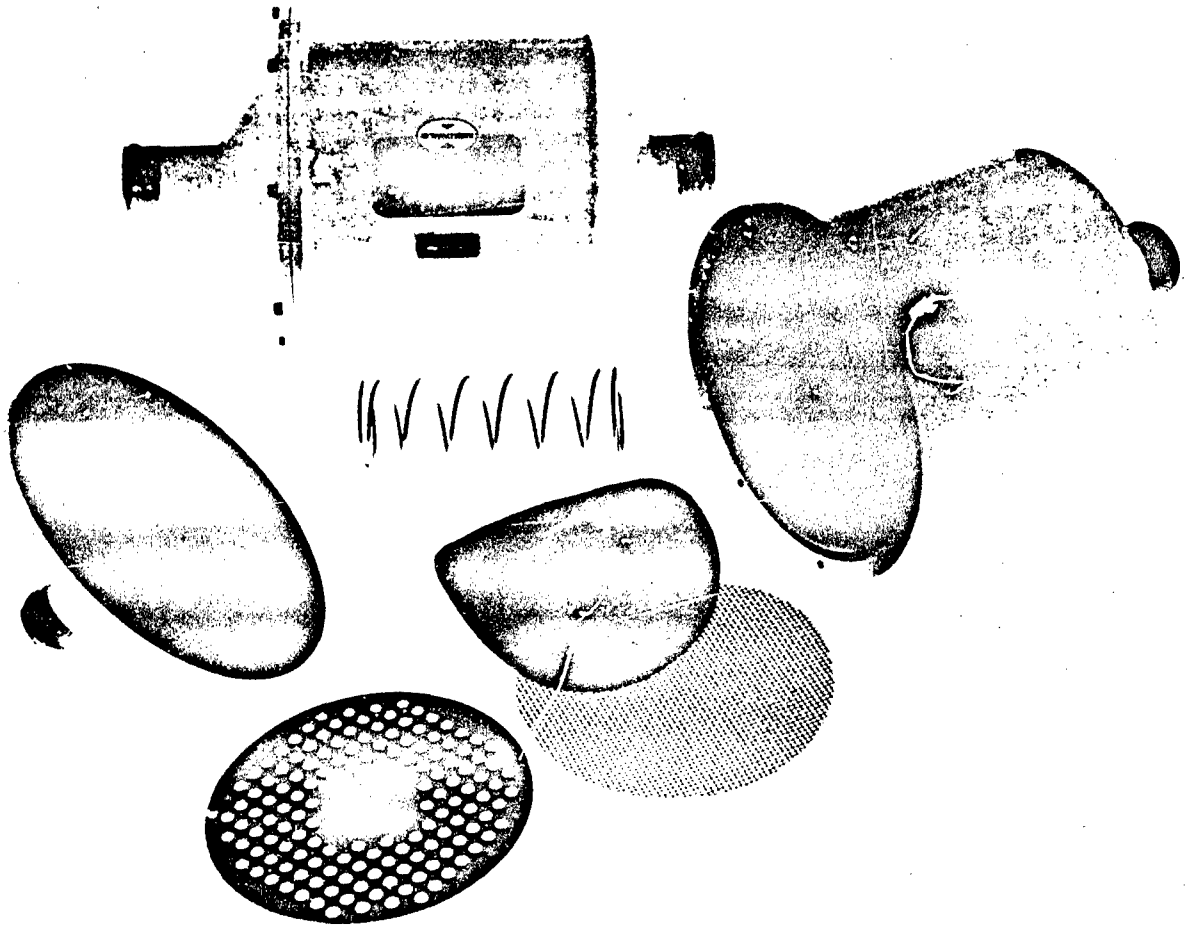


Figure 4. Typical Canister Assembly

High vacuum gate valves were used at each end of the canister, and the vacuum line consisted of a 3 inch diameter silver soldered copper pipe.

Test Instrumentation

The process air dew point indicator was an AiResearch designed instrument capable of accurately measuring dew points to -100°F at 14.7 psia. The principle of the indicator is that the process air sample is passed across a liquid nitrogen cooled mirror in a temperature controlled cell, and the mirror is visually observed through a lense.

A Beckman 15A Infrared Analyzer with a dual range cell for carbon dioxide detection (0-5 per cent and 0-100 per cent) was used to measure the carbon dioxide concentration at the canister inlet and outlet. The analyzer measurement was continuously recorded on a Esterline-Angus Strip Chart Recorder, Model AW (50 microamperes). The combined accuracy of the analyzer and recorder setup was estimated as ±5 per cent (i.e., 1 mole per cent concentration 1.0 ±.05 per cent). The I-R analyzer was calibrated using Beckman calibration gas and was also checked at intermediate concentrations by an Orsat Gas Analyzer.

A Beckman Oxygen Analyzer, Type F3, dual range (0-200 mm Hg and 0-800 mm Hg oxygen partial pressure), was used to indicate whether there was any oxygen or nitrogen adsorption during the low temperature tests. Accuracy was ±1 per cent of full scale.

The vacuum in the vacuum line was measured using a Televac, Model 2AMI-5, Vacuum Gage. A McLeod gage was used to double-check the vacuum readings periodically.

SECTION III

TEST PROGRAM

Approach

The program approach was oriented toward determining the adsorption and desorption characteristics of carbon dioxide by synthetic zeolites at reduced temperatures for possible application to space vehicle environmental control systems.

The test program was to first obtain data for the effects of superficial velocity and canister length on carbon dioxide adsorption by synthetic zeolites at 530°R. The second phase of the program was to determine the low temperature carbon dioxide adsorption by synthetic zeolites for temperatures of 500°R, 450°R, 400°R, 350°R and 300°R. The third phase of the program was devoted to determining the feasibility of desorbing or regenerating the cold zeolite by warming to ambient, and warming to ambient and applying vacuum.

The requirement of the test program was to provide test data of general application rather than data of limited usage to a specific problem statement. The large number of variables possible in the test program (i.e., canister configuration and dimensions, molecular sieve type and pellet size, air flow rates and carbon dioxide concentration) required a streamlining of the program to achieve the objectives and complete the tests within the time limitation.

Linde Type 5A sieves were selected for the bulk of the test program, primarily for the availability of technical data on this type of sieve, and because of its good capacity for carbon dioxide at the lower temperatures. Although Type 13X sieve exhibits a somewhat higher capacity for carbon dioxide at the lower temperatures, Type 5A was selected because of the data and experience available from the use of this type of sieve in ambient temperature carbon dioxide removal systems, and with the idea of correlating this data with that already available to better understand the adsorption phenomena. A pellet size of 1/8-inch diameter was selected in preference over the smaller 1/16-inch diameter pellets because of the lower canister pressure drop.

A straight flow cylindrical canister, as described in the Test Apparatus section of this report, was selected on the basis of the general applicability of the adsorption data obtained from this type canister to other canister types such as a radial flow type configuration. Although the straight flow canister selected provides optimum adsorption data, a disadvantage is the reduced desorption efficiency as a result of the small free surface area of the packed sieve in this type canister. Since the test program did not encompass a detailed study of optimum desorption techniques nor the problems of compromising the adsorption and desorption requirements for a cycling canister design, the canister design was based primarily upon obtaining adsorption data.

A carbon dioxide concentration of 1 mole per cent was used for the majority of the tests, since this concentration has been of most interest

and has been specified as a suitable level for long term exposure during space vehicle missions. Several tests were also conducted at 0.5 mole per cent for comparative purposes.

Procedure

More than 70 adsorption tests were conducted for this program. The test procedure was to first carefully load each canister with minimum exposure of the sieve to the atmosphere. The loaded canister was then weighed to within ±0.5 grams. Each canister was loaded with approximately 650 grams of sieve for the 6 inch length, 1300 grams for the 12-inch length, and 1950 grams for the 18-inch length. Some variation in sieve weights occurred between the different tests, but this variation was insignificant and could not be distinguished when the adsorption load curves were plotted.

The following data was recorded on each test:

Air temperature at canister inlet
Air temperature at canister outlet
Air dew point at canister inlet
CO_2 concentration at canister inlet
CO_2 concentration at canister outlet
O_2 partial pressure at canister outlet and inlet
Canister pressure drop

Both the process air temperature and carbon dioxide concentration were allowed to stabilize for each test before installing the sieve canister in the test setup. Temperature control of ±1°R was achieved for the most part, but a somewhat greater variation occurred at the low temperatures. The carbon dioxide concentration was controlled to ±0.05 mole per cent. The highest dew point for any of the tests was -75°F. On the average, the air dew point was maintained below -80°F for most of the tests.

The carbon dioxide outlet concentration was continuously recorded for each test. Approximately every 5 minutes, the inlet concentration was checked, as well as the recorder zero reading, to insure accuracy of the data trace.

The weight of carbon dioxide adsorbed as a function of time was determined by two methods. First, the canister was weighed periodically during the test run and the weight change noted. A second method was to graphically integrate the curve of the carbon dioxide concentration at the outlet of the canister. For the first tests in the program, the weighing method was extensively employed to cross-check the results obtained by the graphical integration method. Only a very small discrepancy was found between the two methods. Therefore, it was decided to weigh the canister before and after each test and to employ the graphical integration technique to determine the intermediate points of the adsorption load curve. The advantage of this approach was that the weight load of carbon dioxide adsorbed could easily be determined for

any desired time interval without requiring the canister to be removed from the test setup for weighing. Also, temperature stability of the canister could be more easily maintained for the low temperature tests by not requiring removal of the canister from the system for weighing purposes.

All tests were conducted with the test canister insulated so that the test data approached that of adiabatic adsorption. This was particularly necessary for the low temperature tests in order to minimize the heat transfer effects which would otherwise complicate an analysis of the data.

After conducting several preliminary tests at the reduced temperatures, including desorption by vacuum, it became apparent that the most advantageous scheme involved desorption by vacuum at ambient temperatures rather than sub-ambient. Therefore, all adsorption tests conducted at the low temperatures, or sub-ambient temperatures presented in this report, are for a test canister initially at ambient temperature ($530^{\circ}R$) with cooling and adsorption occurring simultaneously until temperature stability is obtained.

SECTION IV

TEST RESULTS

General

Unless otherwise specified, the test results presented below are for Linde Type 5A, 1/8-inch pellet, molecular sieve. All test data presented and the discussion thereof refers to a 1 mole per cent carbon dioxide concentration unless specifically noted.

"Dynamic adsorption capacity" referred to in the discussion of the results is defined as the amount of carbon dioxide adsorbed at the saturation or equilibrium condition for the molecular sieve and is expressed as a per cent of the dry molecular sieve weight.

"Adsorption load" represents the amount of carbon dioxide adsorbed in the molecular sieve at any given time. When the sieve becomes saturated, the adsorption load is equal to the dynamic adsorption capacity. The adsorption load is also expressed as a per cent function of the dry molecular sieve weight contained in the test canister.

The "dynamic adsorption efficiency" is an expression for the weight per cent of carbon dioxide removed from the air stream at any time.

$$\text{Dynamic Adsorption Efficiency} = \frac{Y_{in} - Y_{out}}{Y_{in}}$$

where Y_{in} = CO_2 concentration of process air at canister inlet (mole per cent)

Y_{out} = CO_2 concentration of process air at canister outlet (mole per cent)

Canister Length Tests

Tests were conducted to determine effect of canister length upon the dynamic adsorption characteristics of the molecular sieve. Canister lengths of 6-, 12-, and 18-inches were used for the tests. The diameter of each canister was 3.55 inches.

Figures 5, 6, and 7 and corresponding Table 2 contain the test results for the canister length study. Figures 5, 6, and 7 show the adsorption load curves for each canister as a function of the adsorption time. Figure 5 shows the adsorption loads for a process air temperature of 530°R and a mass flow of 0.308 lb air per minute. Figure 6 shows the load curves for a process air temperature of 530°R and a mass flow of 0.71 lb air per minute. Figure 7 presents the load curves for a process air temperature of 450°R and a mass flow of 0.308 lb air per minute.

Table 2 indicates the adsorption load value for dynamic removal efficiencies of every 10 per cent.

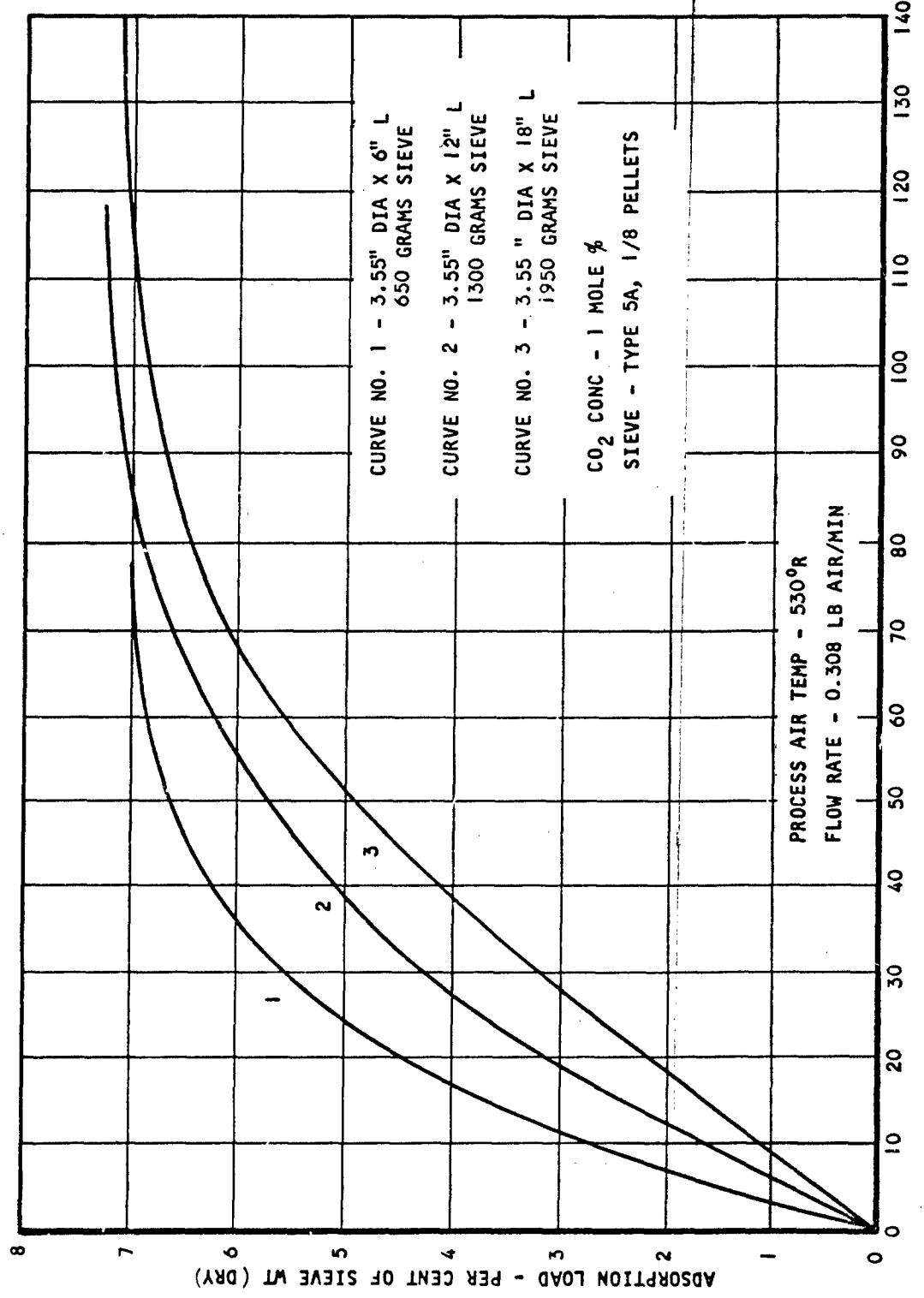


Figure 5. Effect of Canister Length on Adsorption (530°R, 0.308 lb air/min)

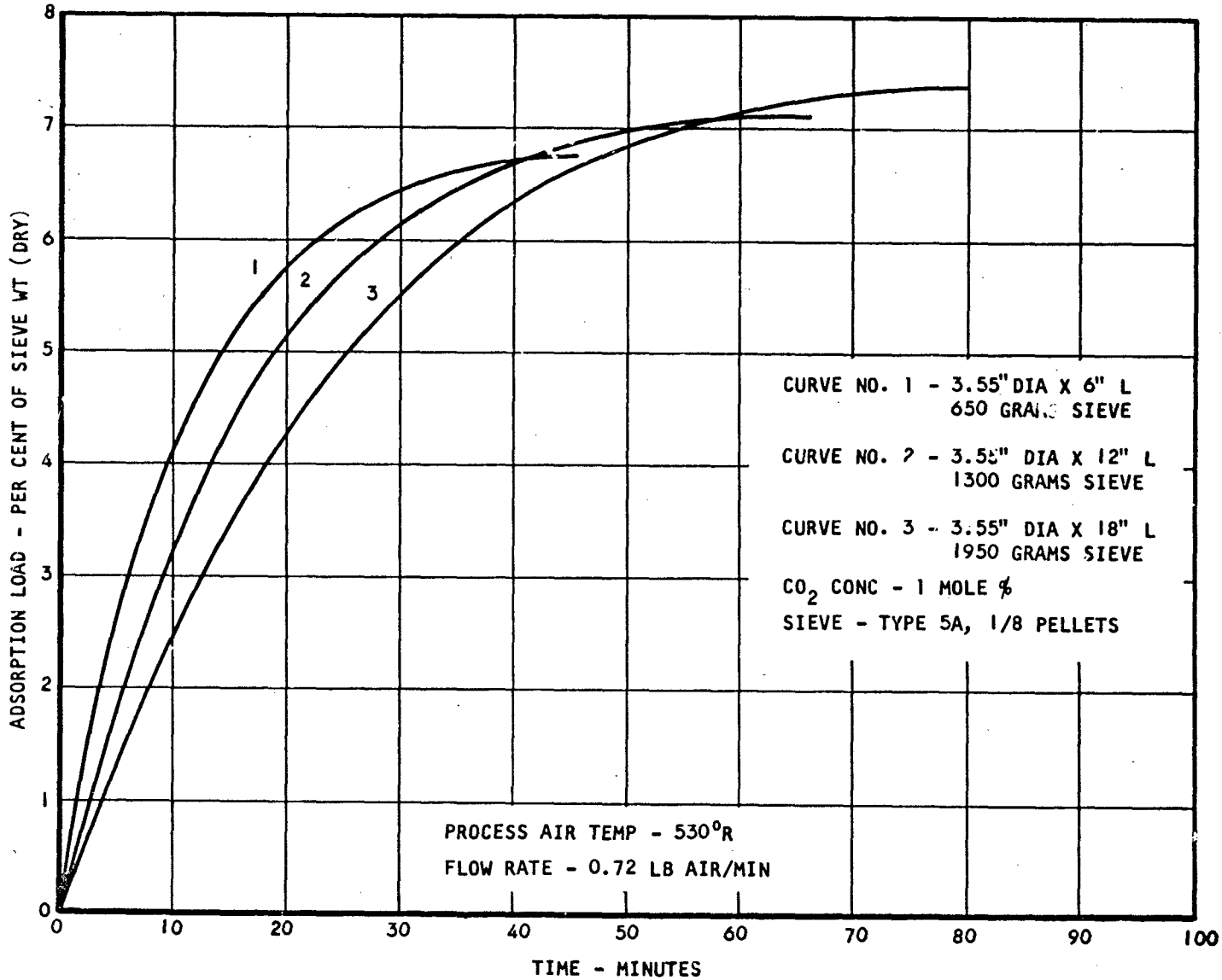


Figure 6. Effect of Canister Length on Adsorption (530°R, 0.72 lb air/min)

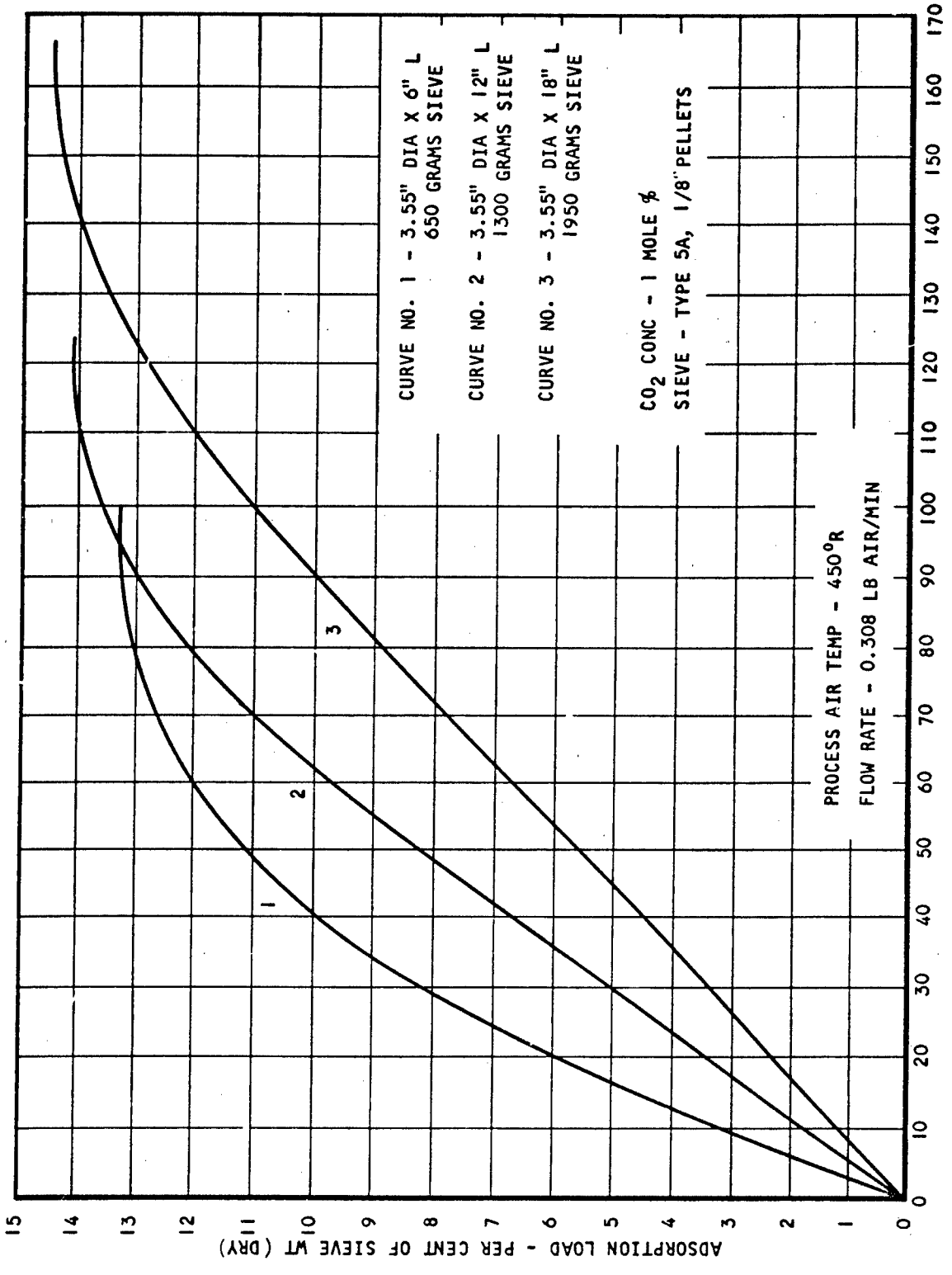


Figure 7. Effect of Canister Length on Adsorption (450°R, 0.308 lb air/min)

TABLE 2

EFFECT OF CANISTER LENGTH ON DYNAMIC ADSORPTION EFFICIENCY

DYNAMIC ADSORPTION EFFICIENCY	Adsorption Load - Per Cent of Sieve Wt.								
	(See Figure 5)			(See Figure 6)			(See Figure 7)		
	6"	12"	18"	6"	12"	18"	6"	12"	18"
100% Break-through	.9	1.7	2.5	.6	1.0	1.4	2.9	7.1	10.6
90%	1.6	2.7	3.5	.9	1.6	2.18	4.9	9.5	12.3
80%	2.2	3.3	4.2	1.3	2.3	3.0	6.4	10.8	12.6
70%	2.9	4.0	4.9	1.9	2.9	3.8	7.7	12.0	13.1
60%	3.6	4.5	5.4	2.4	3.8	4.4	8.8	12.4	13.4
50%	4.3	5.0	5.8	3.1	4.3	5.0	9.6	12.8	13.7
40%	5.0	5.9	6.2	4.0	5.1	5.6	10.6	13.2	13.9
30%	5.6	6.3	6.5	4.8	5.6	6.1	11.6	13.6	14.2
20%	6.2	6.8	6.8	5.4	6.1	6.7	12.4	13.8	14.4
10%	6.7	7.2	7.1	6.4	6.6	7.2	13.0	14.0	14.5
0% Saturation	7.0	7.3	7.2	6.8	7.2	7.4	13.4	14.1	14.6

Comparing the figures and table, it can be seen that the longer canister lengths for any test condition results in greater adsorption in the higher dynamic removal efficiency range (100%, 90%, etc.). The tests also indicated that the dynamic adsorption capacity (saturation) was slightly higher for the longer canisters. Therefore, when canister length and its effect on adsorption is alone considered, the longest possible canister length yields the best adsorption results. However, other considerations, such as canister pressure drop, method of desorption, and process air superficial velocity effects, must be balanced with the canister length considerations before a successful canister design can be achieved.

Superficial Velocity

Tests were conducted to determine the effect of superficial velocity on the dynamic adsorption characteristics of carbon dioxide and molecular sieves. The canister selected for this series of tests was 3.55 inches in diameter and 6 inches in length and contained approximately 650 grams of Linde Type 5A, 1/8-inch pellets, molecular sieve. Process air flow rates were chosen to correspond approximately to superficial velocities in the canister of 30 fpm (0.154 lb/min), 60 fpm (0.308 lb/min), 90 fpm (0.463 lb/min), 120 fpm (0.616 lb/min), and 140 fpm (0.72 lb/min) at 530°R. These same mass flows were also used for the low temperature adsorption tests at process air temperatures of 500°R, 450°R, 400°R, 350°R. Carbon dioxide concentration in the process air was 1 mole per cent for all tests.

Figures 8, 9, 10, 11, 12, and 13 show the adsorption load curves as a function of time for the selected mass flow rates and process air temperatures.

Figure 8, which shows the adsorption curves at 530°R for the selected mass flows, indicates that a maximum velocity can be found for a given canister design. For mass flows higher than 0.463 lb per minute (90 fpm), there is only a small increase in the carbon dioxide adsorption rate. The same characteristic can also be observed in the other figures. When comparing the figures, it should be remembered that the mass flows are constant, and the velocities are smaller for each drop in temperature. Comparison of the curves indicates that a maximum mass flow is obtained for a given canister and is independent of temperature. Maximum velocity or maximum mass flow for a canister is important in balancing the adsorption rate with respect to the fan or compressor power requirements.

Another important consideration of the velocity effect on adsorption is the effect of velocity on the dynamic removal efficiency for a given temperature. Figures 14 and 15 contain adjusted curves of the test data showing the dynamic removal efficiency as a function of the superficial canister velocity at 530°R and 400°R. The curves represent the best curve that could be drawn through the test points. Some variation was observed in comparing the adsorption load values for intermediate values of dynamic removal efficiency for any set of test conditions; however, these figures are representative compilations of the test data for illustrative purposes. As shown in Figures 14 and 15, the adsorption load value for the 100 per cent dynamic adsorption efficiency is higher for the lower velocities. It should also be noted that

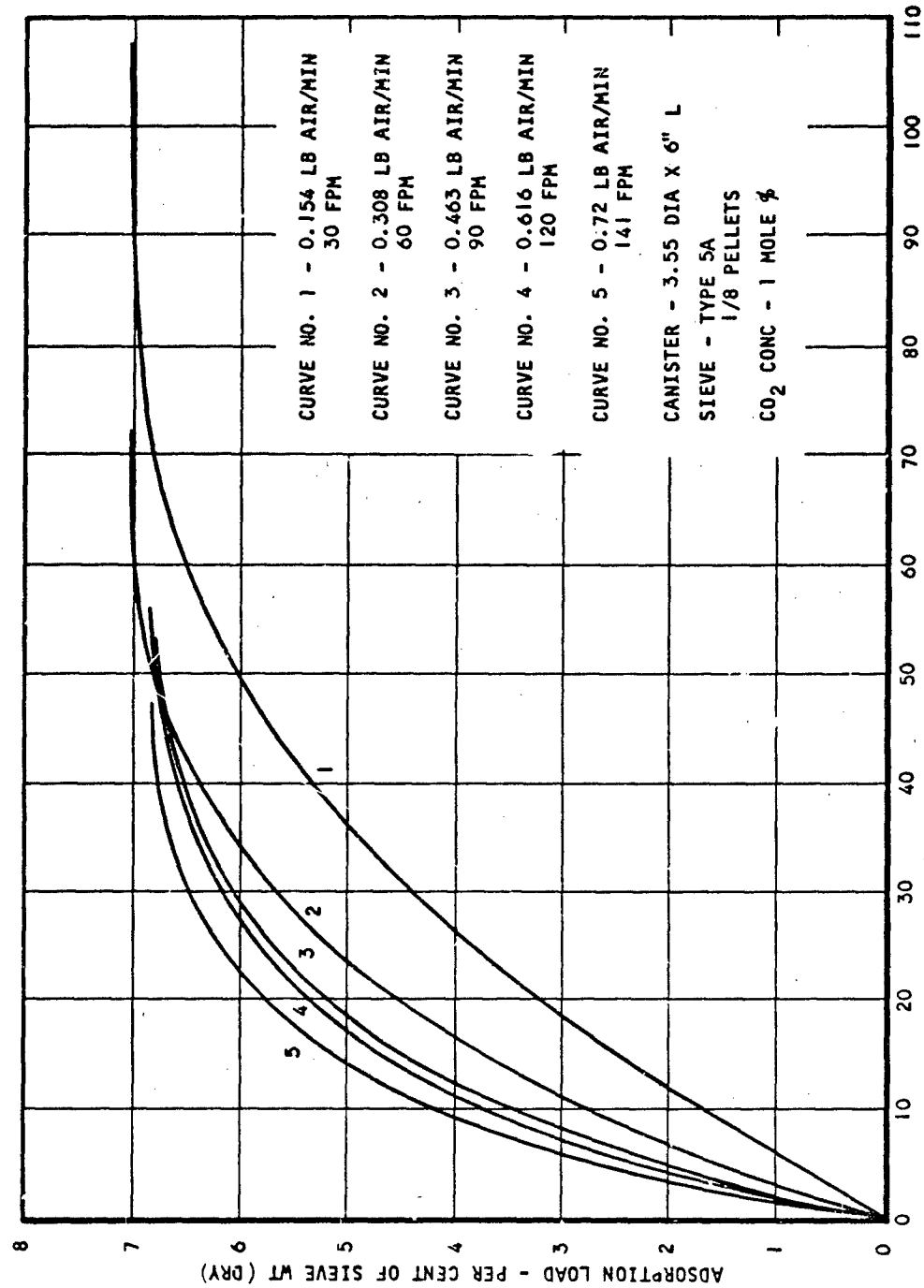


Figure 8. Effect of Superficial Velocity on Adsorption, 530°R

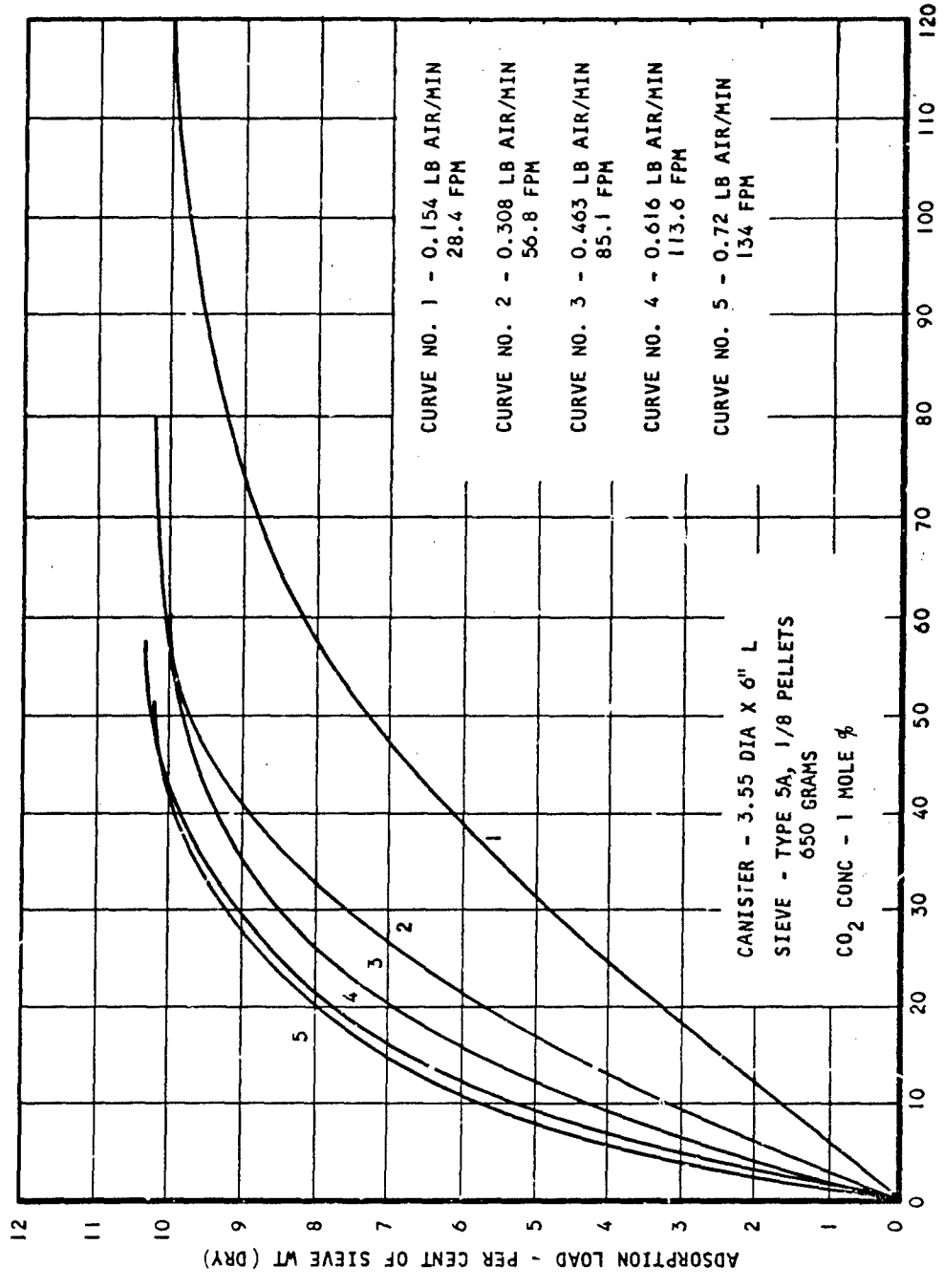


Figure 9. Effect of Superficial Velocity on Adsorption, 500°R

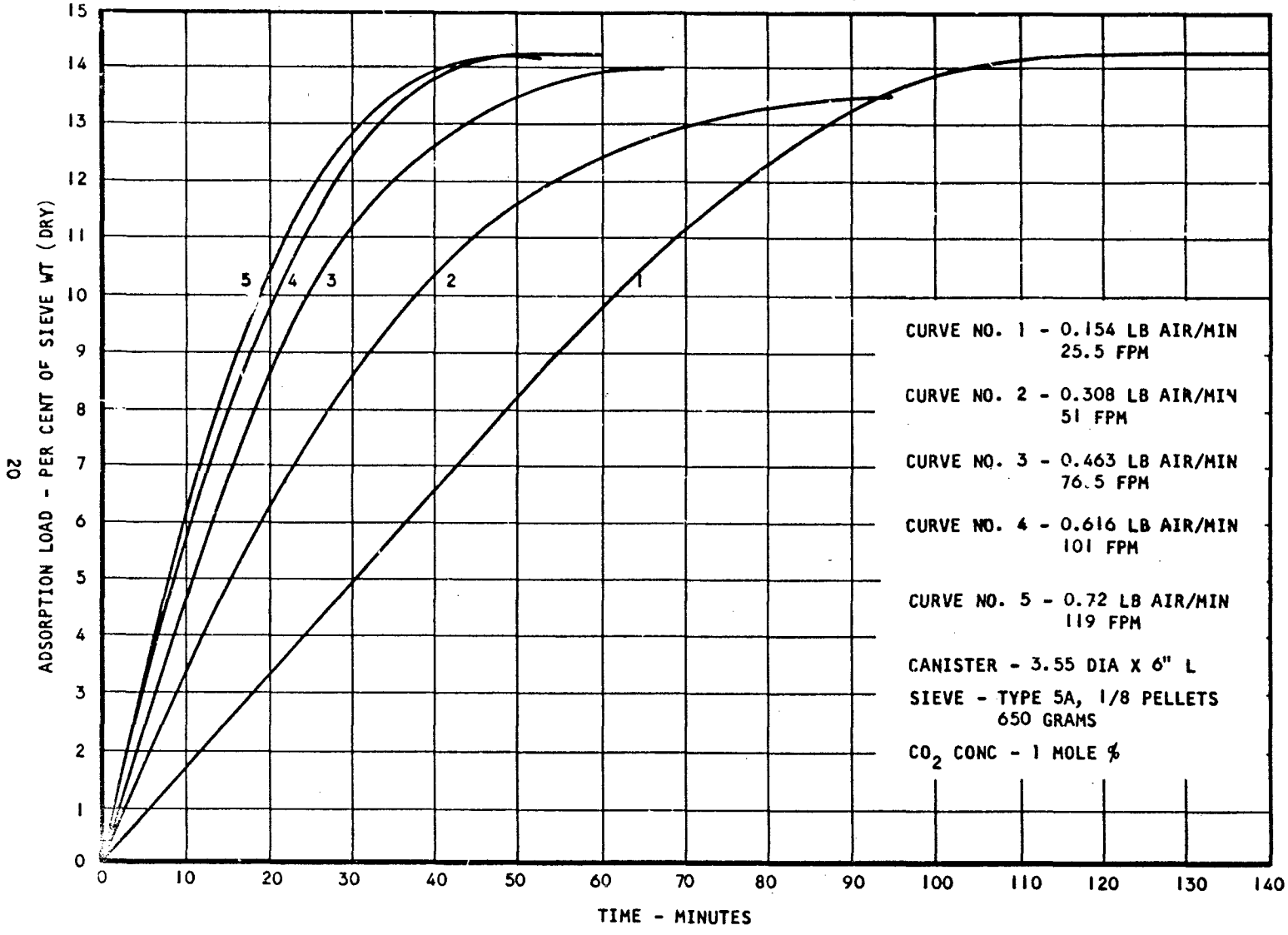


Figure 10. Effect of Superficial Velocity on Adsorption, 450°R

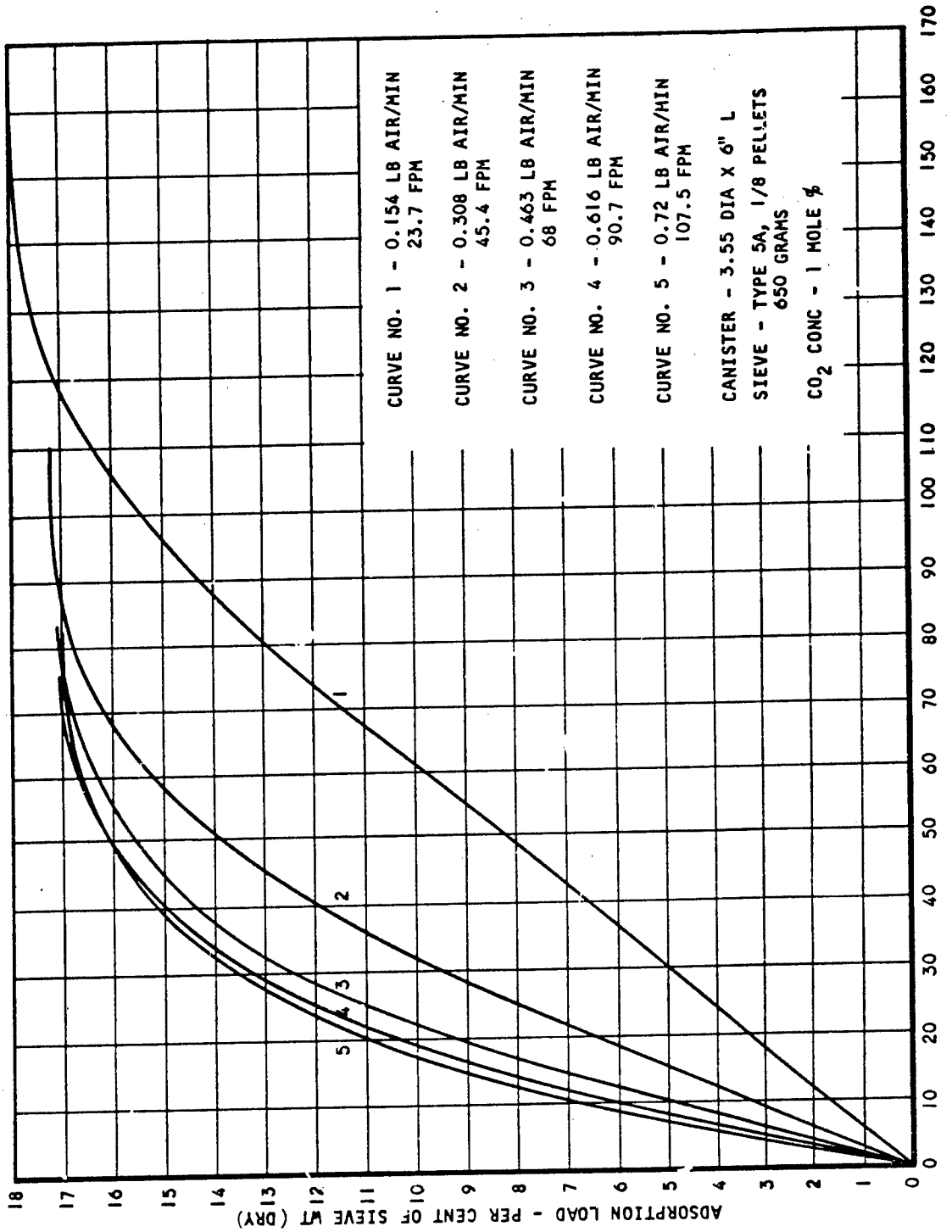


Figure 11. Effect of Superficial Velocity on Adsorption, 400°R

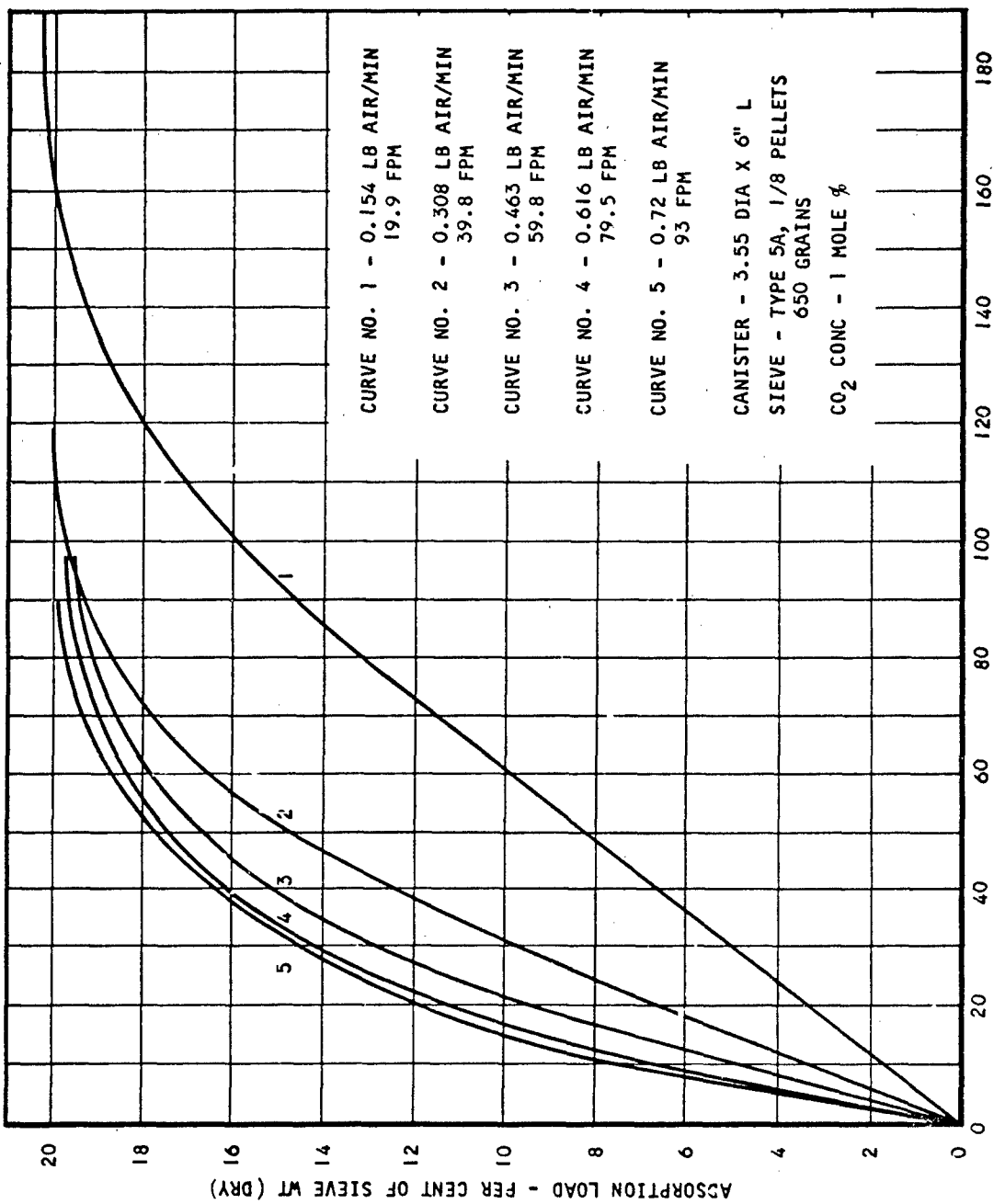


Figure 12. Effect of Superficial Velocity on Adsorption, 350°R

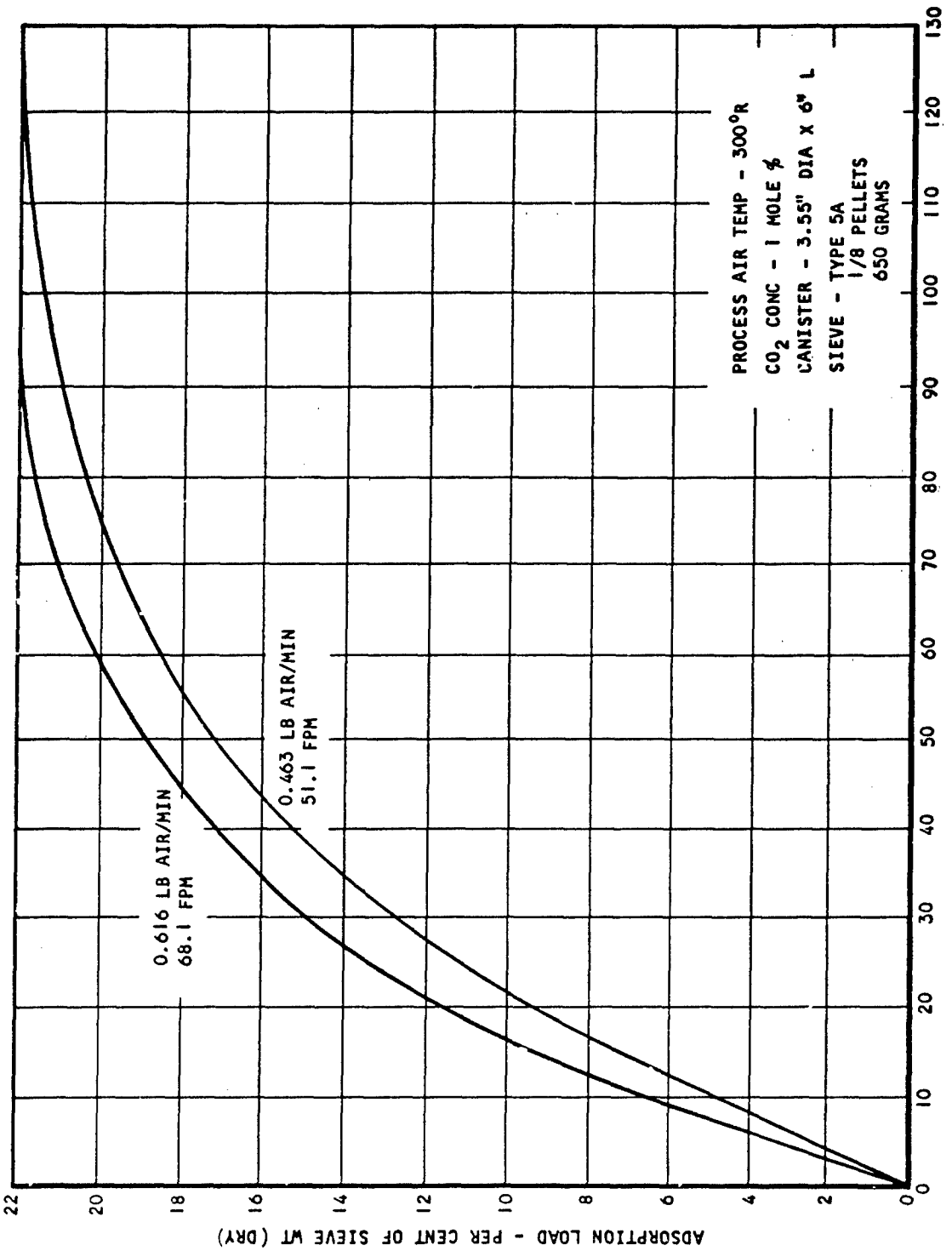


Figure 13. Effect of Superficial Velocity on Adsorption, 300°R

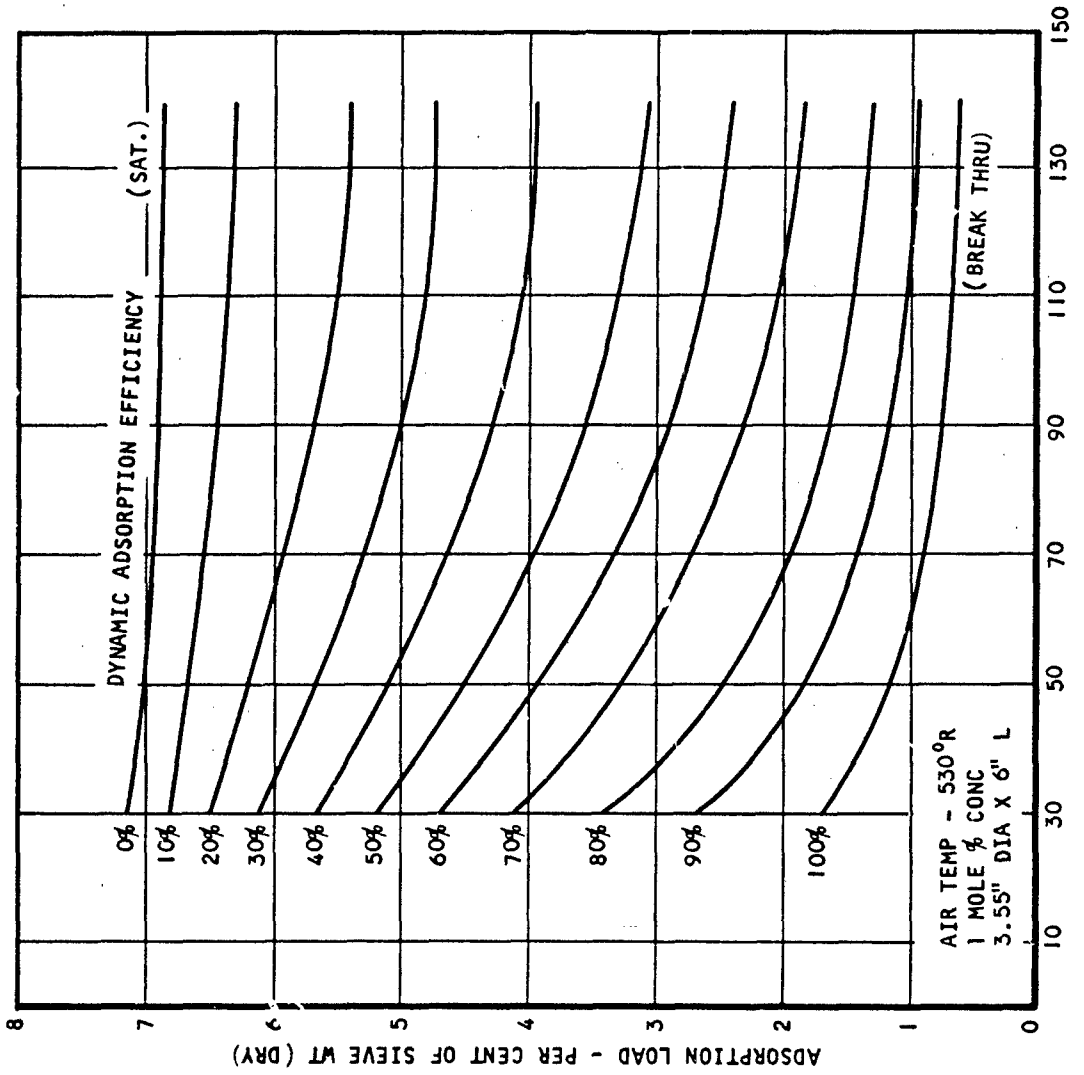


Figure 14. Effect of Superficial Velocity on Dynamic Adsorption Efficiency (Air Temperature 530°R)

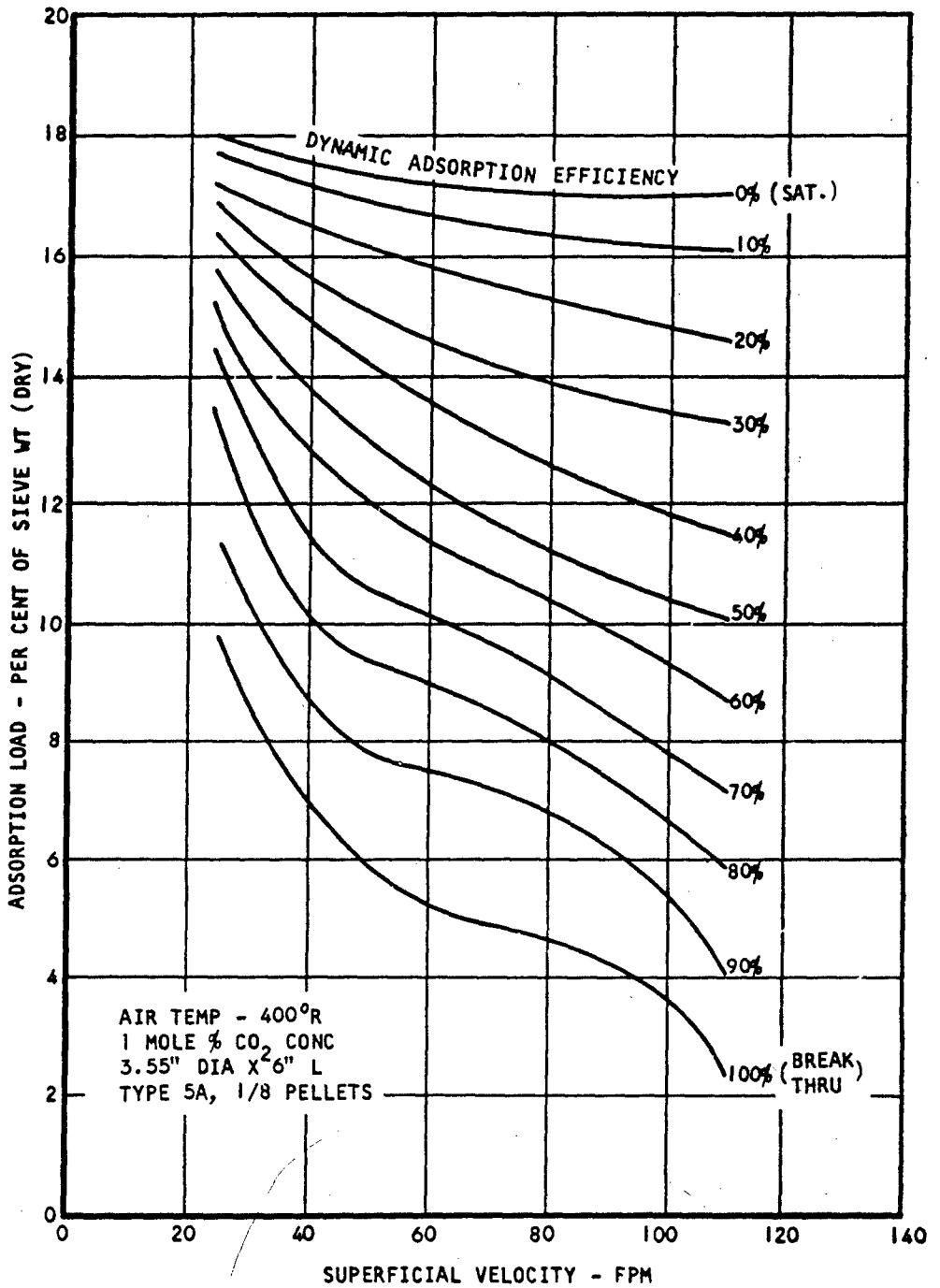


Figure 15. Effect of Superficial Velocity on Dynamic Adsorption Efficiency (Air Temperature 400°R)

at low velocities, a greater portion of the adsorption occurs at the higher efficiencies while at the higher velocities, a greater part of the adsorption occurs at the intermediate and lower efficiencies. On the average, the dynamic adsorption capacity also decreased slightly with increased velocity, as shown by the zero per cent line. It is believed that the decrease is primarily the result of the adsorption tests being cut short while the bed adsorbed at less than 2 per cent efficiency.

Low Temperature

The low temperature adsorption tests consisted of a series of tests of constant air flow rates (0.154 lb/min, 0.308 lb/min, 0.463 lb/min, 0.616 lb/min, and 0.72 lb/min) run at selected temperatures of 530°R, 500°R, 450°R, 400°R 350°R, and 300°R. On the basis of previous tests, the 6-inch canister was used for the tests. The data presented is obtained for a canister initially at 530°R placed into the system and cooled by the air stream while adsorption occurs.

Figure 16 presents the dynamic adsorption capacities at various temperatures for Linde 5A, 1/8-inch pellets, and carbon dioxide concentration of 0.5 and 1.0 mole per cent in the process air. These curves represent average values of dynamic adsorption capacity for all tests conducted at each temperature and do not represent the maximum values obtained. The dynamic capacity was determined by measuring the weight increase of the molecular sieve when the carbon dioxide concentration at the outlet of the canister equaled that of the inlet. As can be seen from Figure 16, there is a considerable increase in the adsorption capacity for a decrease in process air temperature. The dynamic capacity ranged from 7 lb of CO_2 per 100 lb of sieve at 530°R to approximately 22 lb of CO_2 per 100 lb of sieve at 300°R for a 1 mole per cent concentration.

The adsorption load curves for constant mass flow at the various temperatures are presented in Figures 17, 18, 19, 20, and 21. As a result of limitations in the test apparatus, temperature stability of the air stream for the 300°R tests could not be achieved except for two of the flow conditions. At the lower temperatures of 350°R and 300°R, some nitrogen adsorption was observed at the beginning of each run prior to the breakthrough condition. However, the nitrogen adsorbed was later displaced as a result of the preferential adsorption of carbon dioxide by the sieves. The adsorption rate of the nitrogen was low, and attempts to measure the amount adsorbed were inconclusive.

The effect of temperature on the dynamic removal efficiency is shown in Figures 22 and 23. Lower temperatures increase the amount of adsorption occurring at the higher dynamic adsorption efficiencies. Test results indicated that considerable variation in the adsorption load values for the intermediate dynamic adsorption efficiencies could be expected for identical test runs. However, the breakthrough point was repeated with fair consistency. At the intermediate and low dynamic adsorption efficiencies for the low temperature tests, the adsorption load value was found to be extremely sensitive to temperature variations of even ±1°R. As a result, an accurate determination of the dynamic efficiency was not possible.

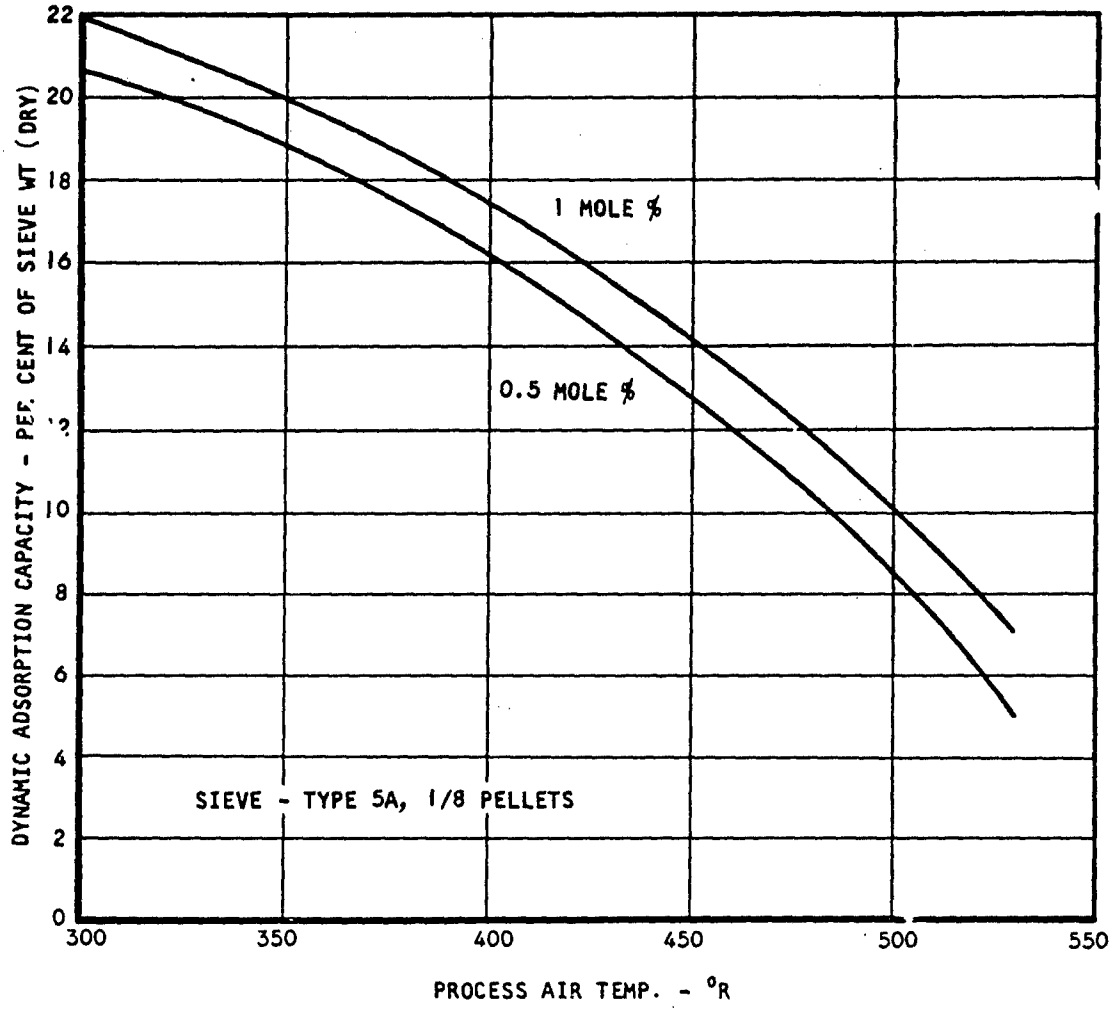


Figure 16. Effect of Temperature on Dynamic Adsorption Capacity

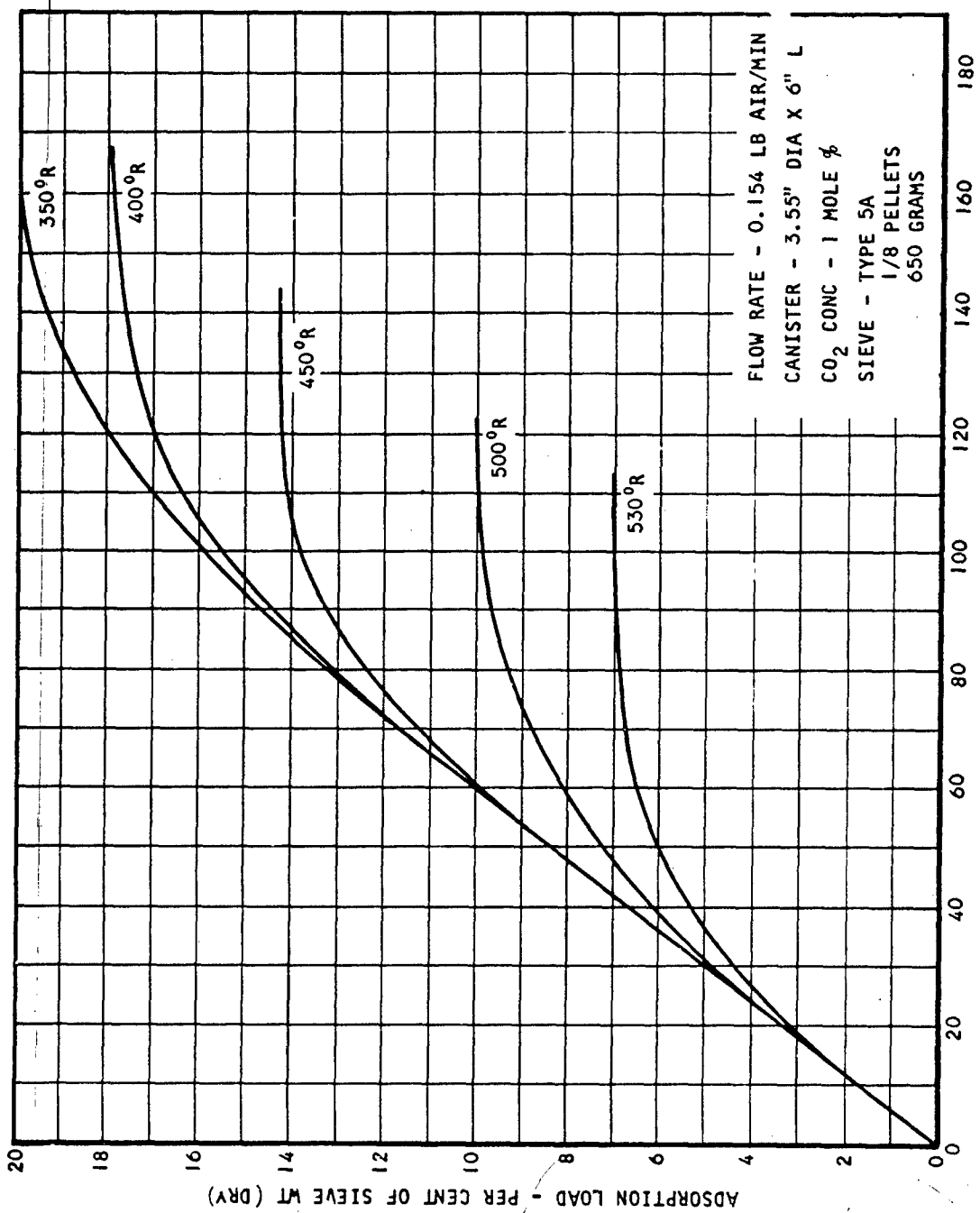


Figure 17. Effect of Process Air Temperature on Adsorption, 0.154 lb air/min

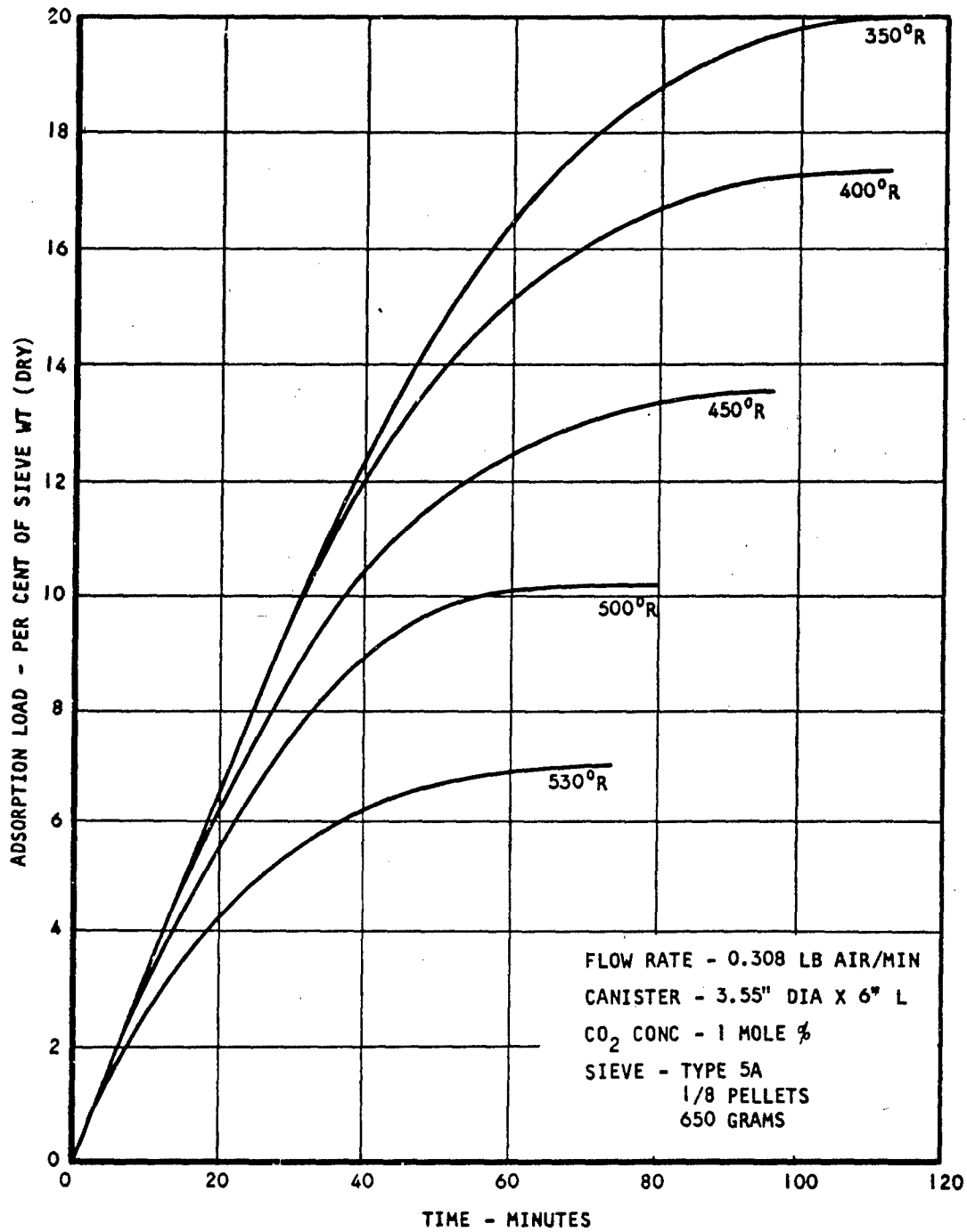


Figure 18. Effect of Process Air Temperature on Adsorption, 0.308 lb air/min

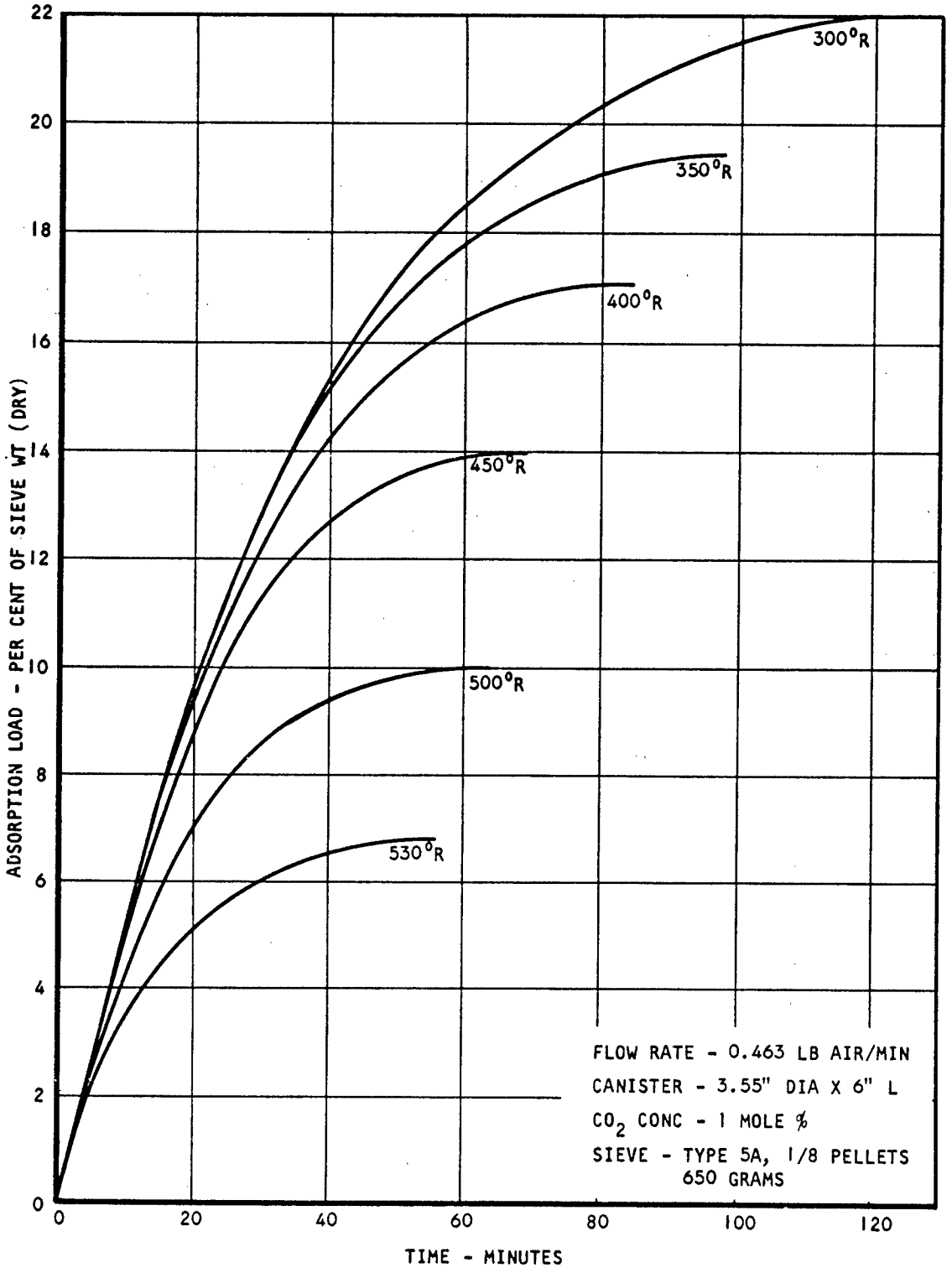


Figure 19. Effect of Process Air Temperature on Adsorption, 0.463 lb air/min

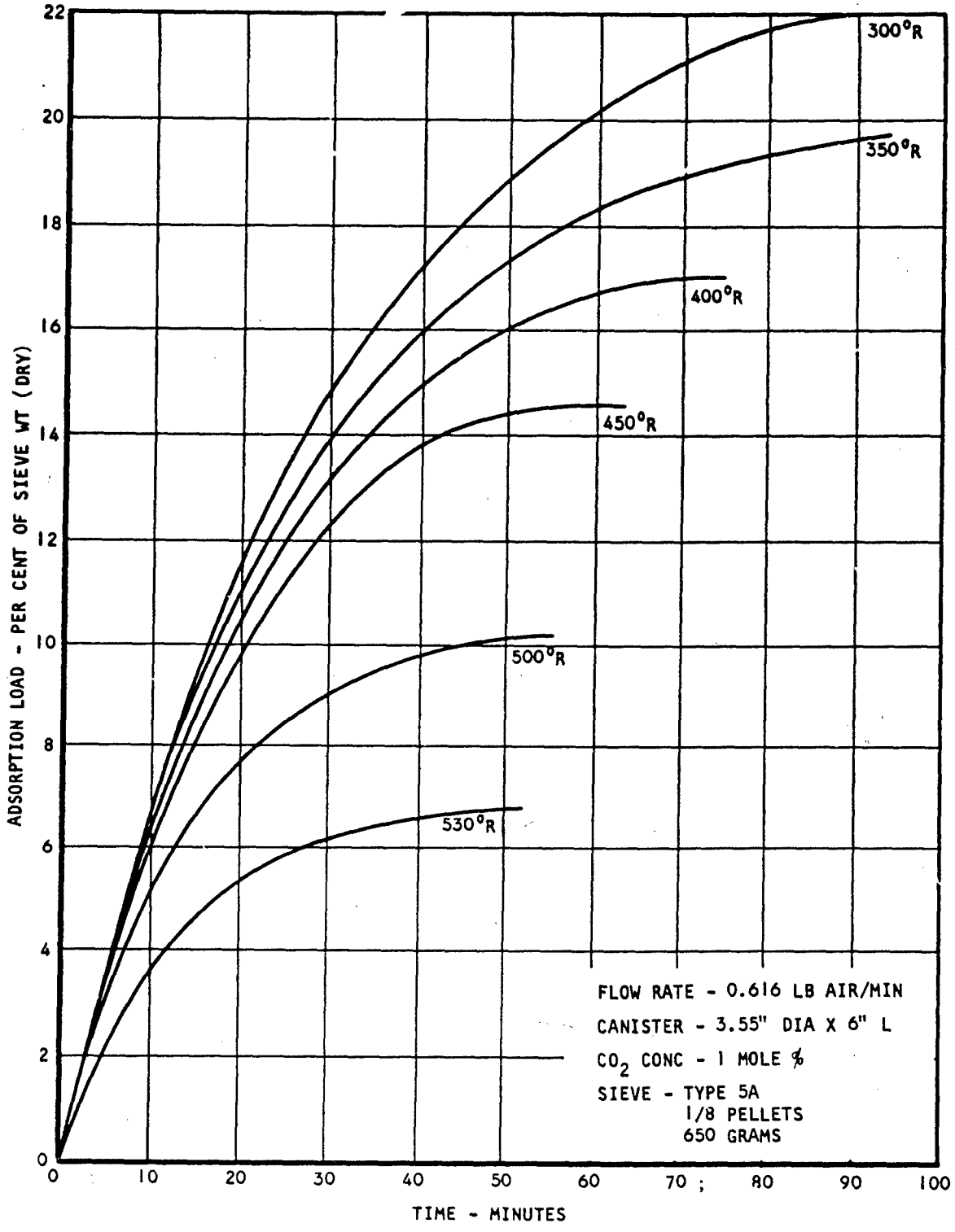


Figure 20. Effect of Process Air Temperature on Adsorption, 0.616 lb air/min

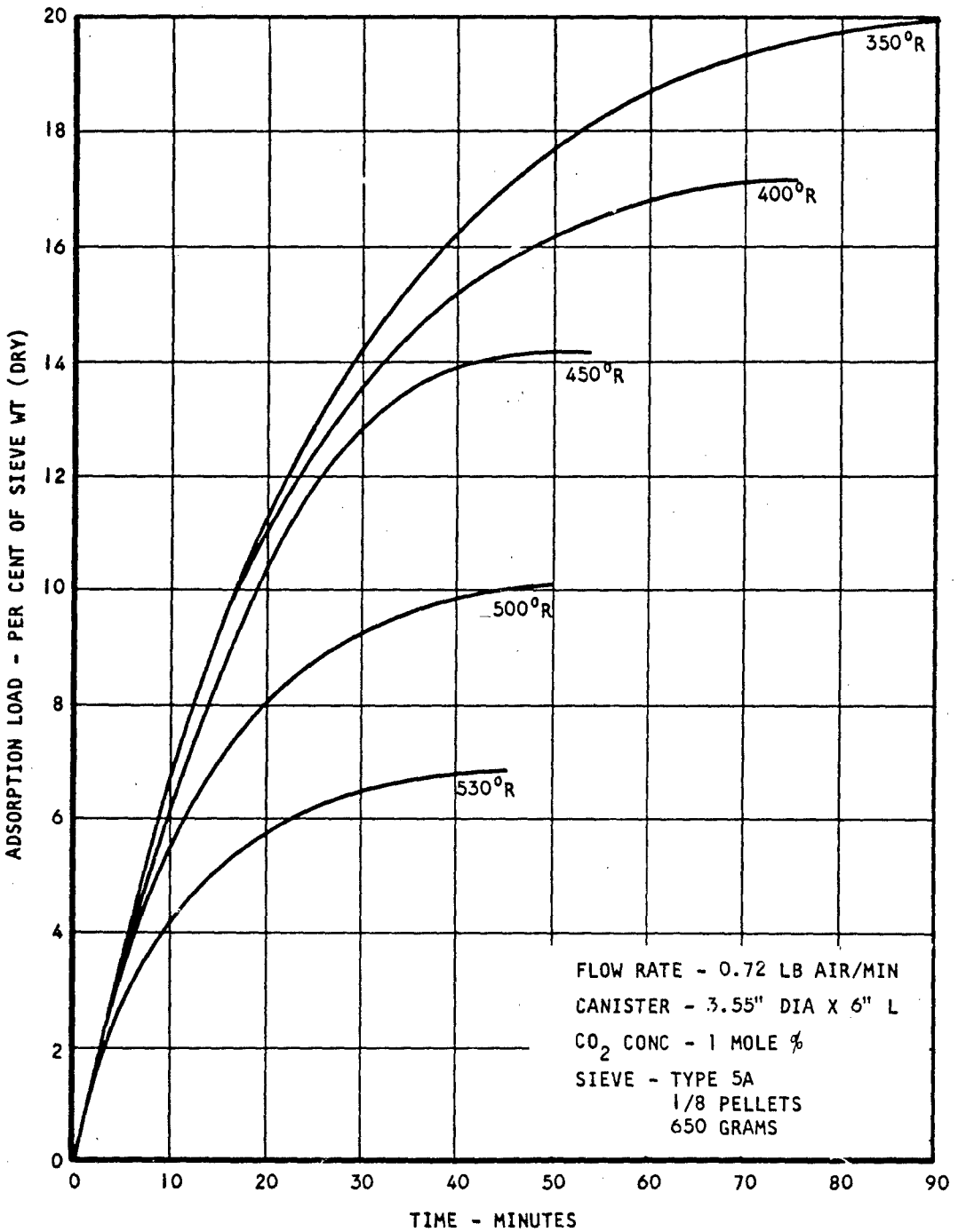


Figure 21. Effect of Process Air Temperature on Adsorption, 0.72 lb air/min

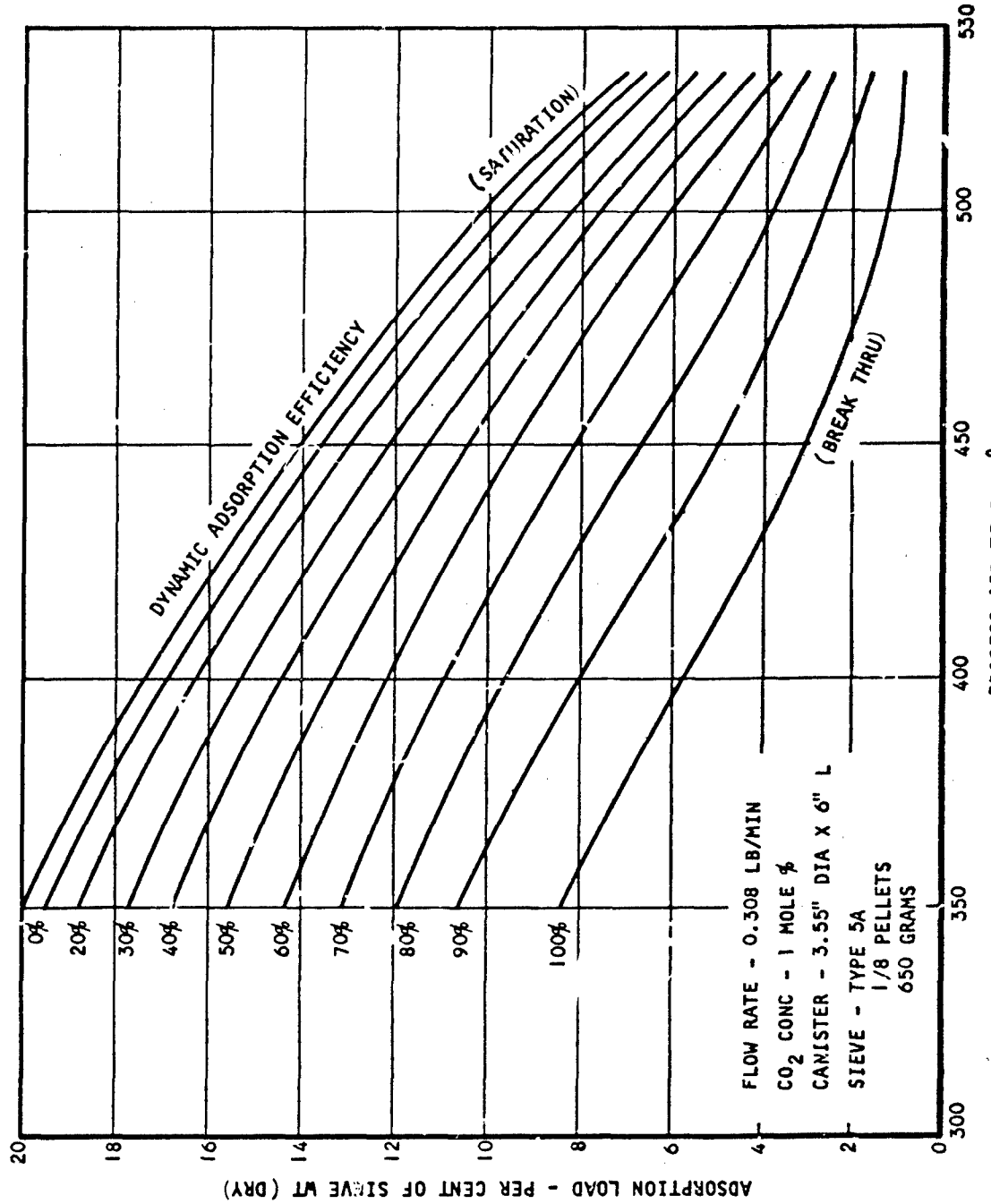


Figure 22. Effect of Process Air Temperature on Dynamic Adsorption Efficiency

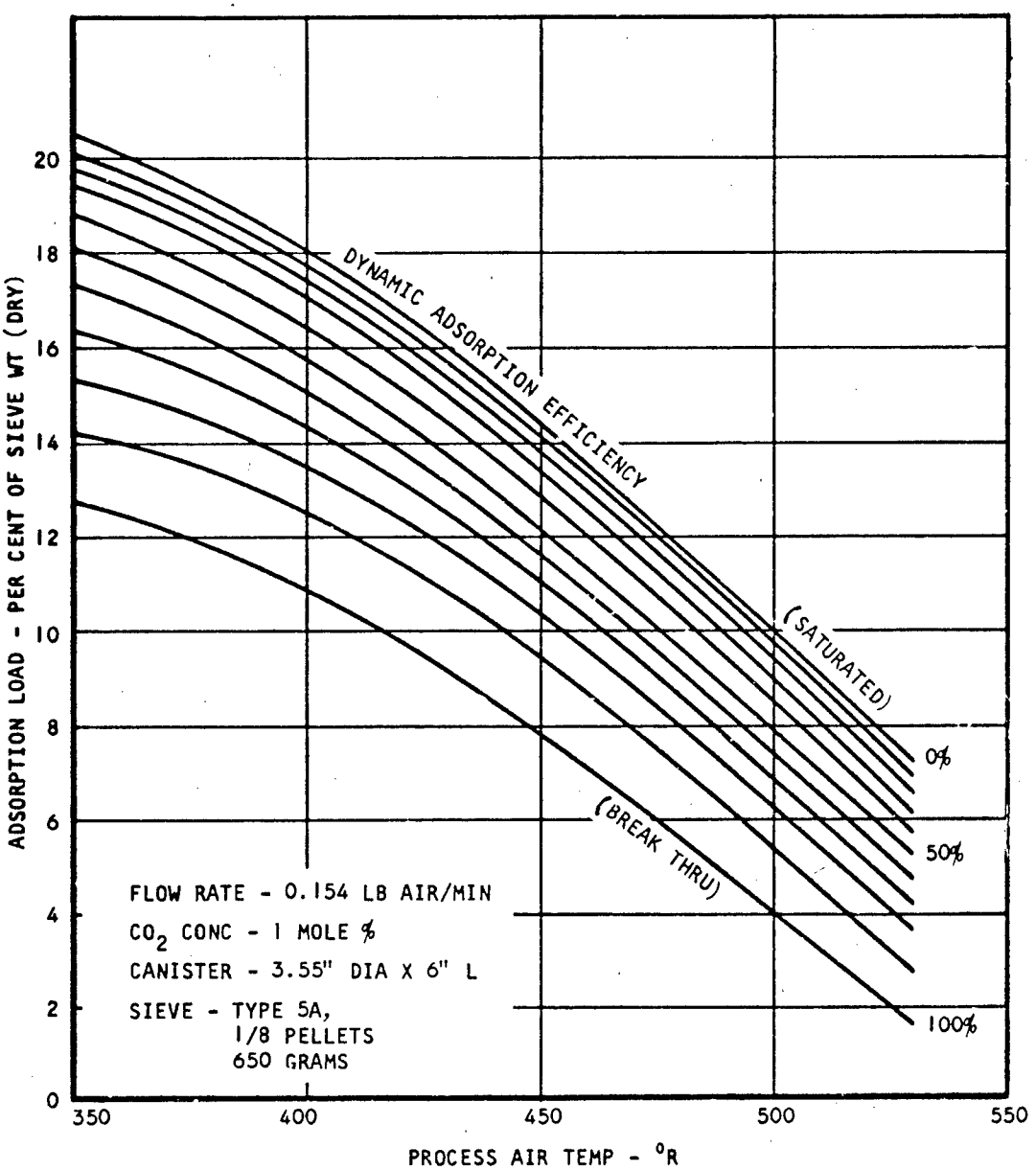


Figure 23. Effect of Process Air Temperature on Dynamic Adsorption Efficiency

Desorption Tests

Desorption of the molecular sieves followed each adsorption test. The first desorption tests consisted of applying vacuum to the canister immediataly following the adsorption run. This meant that for the low temperature test, the canisters were not warmed before desorption but were desorbed cold. The only warming of the molecular sieves occurred by the heat leak through the canister walls and the insulation blanket. A preliminary desorption test at 350°R confirmed a prediction that vacuum alone was a poor method for desorbing molecular sieves at low temperatures. The results of this test compared with the desorption data for the 520°R adsorption test are as follows:

	Adsorption Load 350°R	Adsorption Load 530°R
Sieve Capacity	20%	7.1%
20 minutes vacuum	17.3%	3.9%
40 minutes vacuum	15.9%	3.0%
60 minutes vacuum	14.0%	2.5%

Comparing the adsorption load values, it can be seen that less carbon dioxide is desorbed at the lower temperature than at the ambient 530°R test. This was expected since vacuum desorption has the effect of cooling the molecular sieves as the desorption takes place.

The second method of desorption tested was to allow the canisters to warm to 530°R before applying vacuum. The rate of warming was not measured, since no consideration was given to heat transfer rates in the design of the canister. Figure 24 presents the results of these tests. Curve No. 1 shows the adsorption load of the sieves after warming to 530°R. Curve Nos. 2, 3, and 4 represent the adsorption loads after 20 minutes, 40 minutes, and 60 minutes of vacuum desorption respectively. As the figure indicates, a much greater amount of carbon dioxide can be desorbed in a given time from the sieves adsorbed at low temperatures. This means that the test canister adsorbed at 350°R and desorbed at 530°R was capable of removing the required amount of carbon dioxide from the process air with 1/4 the amount of sieve required for adsorption at ambient (530°R) temperatures. Or, if the reduction of canister weight is not important, the cycle time can be increased by a factor of four for a canister containing the same amount of sieve as an ambient temperature canister.

The vacuum desorption rate was limited by the canister design and pumping speed in these tests. The 1 1/2-inch diameter duct acted as a restriction between the canister and the 3-inch diameter vacuum line. Also, the thinner bed with a larger free surface area would improve the desorption rate. Therefore, the results presented are not considered optimum but rather representative of the desorption feasibility.

Pressure Drop

Some variation was observed in the canister pressure drop data for the various tests. This variation can probably be accounted for in the packing of

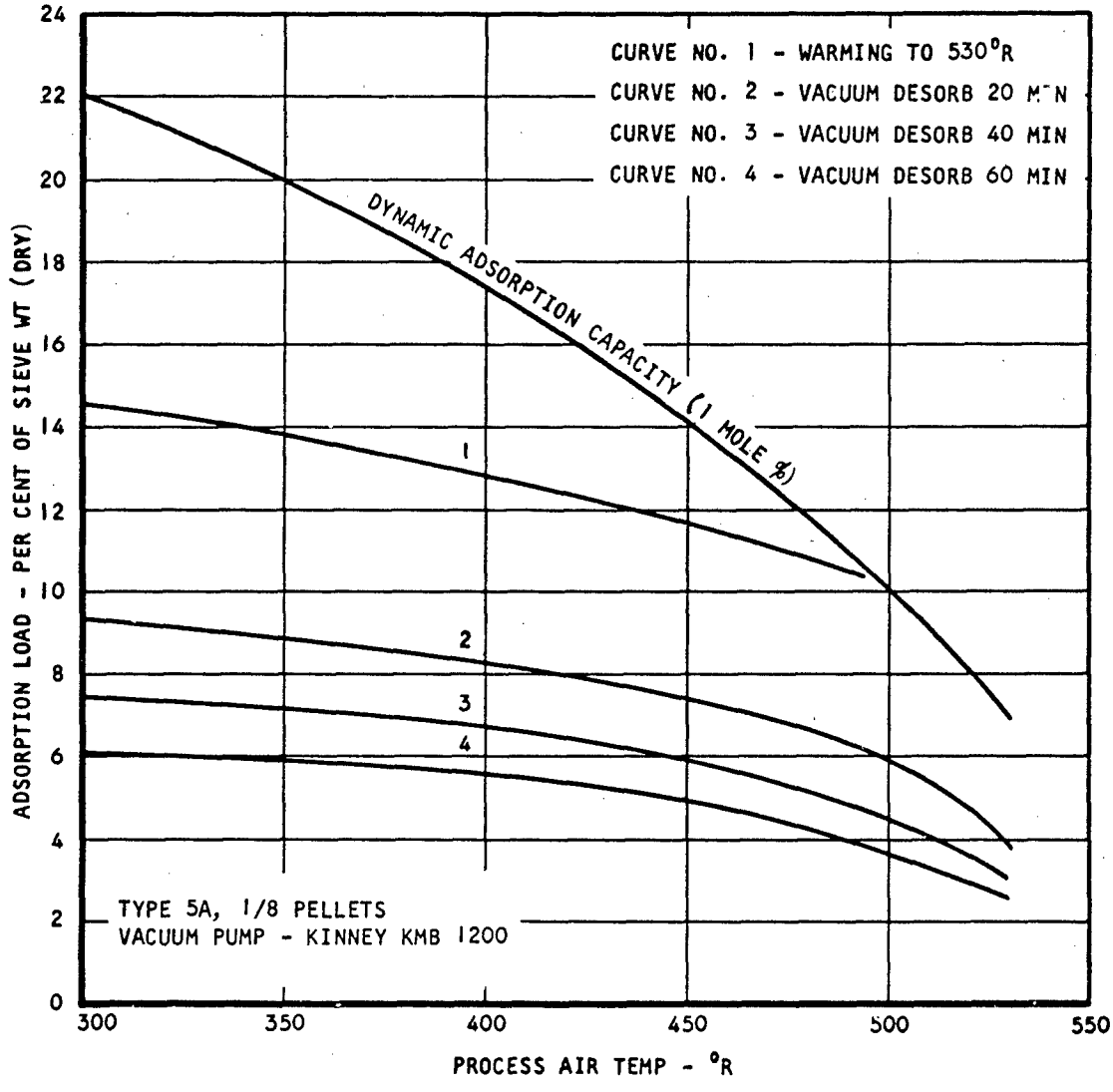


Figure 24. Desorption Data

the two molecular sieve pellets. Figure 25 presents the pressure drop (temperature corrected) across the canister as a function of the process air mass velocity. The area between the two lines in the figure indicates the range of data excluding those points believed to be experimental error.

Pellet Size

In addition to the 1/8-inch Type 5A pellets tested, a few tests were conducted with 1/16-inch Type 5A pellets. The 1/16-inch pellets exhibited a somewhat higher dynamic adsorption capacity. The values of dynamic adsorption capacity for the 1/16-inch pellets were from 0.25 per cent to 0.5 per cent of the sieve weight higher than the 1/8-inch pellets. The pressure drop for the same canister using 1/16-inch pellets was approximately 2.2 times higher than for the 1/8-inch pellets.

Temperature Increase

The temperature increase of the process air was measured during each adsorption test. Figures 26, 27, and 28 show the temperature increase (T) of the process air through the 6-inch length canister for the various flow rates at the 530°R inlet condition. The maximum temperature rise for each flow rate tested was approximately the same varying between 21° and 23°F. The maximum temperature rise occurred shortly after the start of each test, as expected. However, the maximum temperature rise was of only a short duration and was followed by a fairly rapid decrease in the temperature differential.

Tests of Type 13X and 4AXW

Several tests were conducted to determine the dynamic adsorption capacity for Type 13X sieve. The results are shown in Figure 29. The broken portion of the curve indicates uncertainty of the data with respect to the weight of carbon dioxide adsorbed. The dashed line represents the capacity as determined by the weight measurements before and after the test. The recording of the carbon dioxide concentration at the canister outlet indicated that a smaller amount of carbon dioxide was adsorbed, and the values obtained by integrating the curve are shown by the dotted line in the figure. The test points were repeated for the dashed region of the curve and the same results obtained. No definite conclusions were reached, but it is possible that some nitrogen and/or oxygen was adsorbed at these low temperatures and was not displaced by the selective adsorption of carbon dioxide after the breakthrough condition of the sieve.

Linde Type 4AXW, a new type sieve, was also tested. Very few tests were conducted using this material, and the results indicated that the sieve was unsuitable for carbon dioxide removal purposes in environmental control systems. The sieve exhibited a rapid breakthrough and prolonged adsorption at low dynamic removal efficiencies.

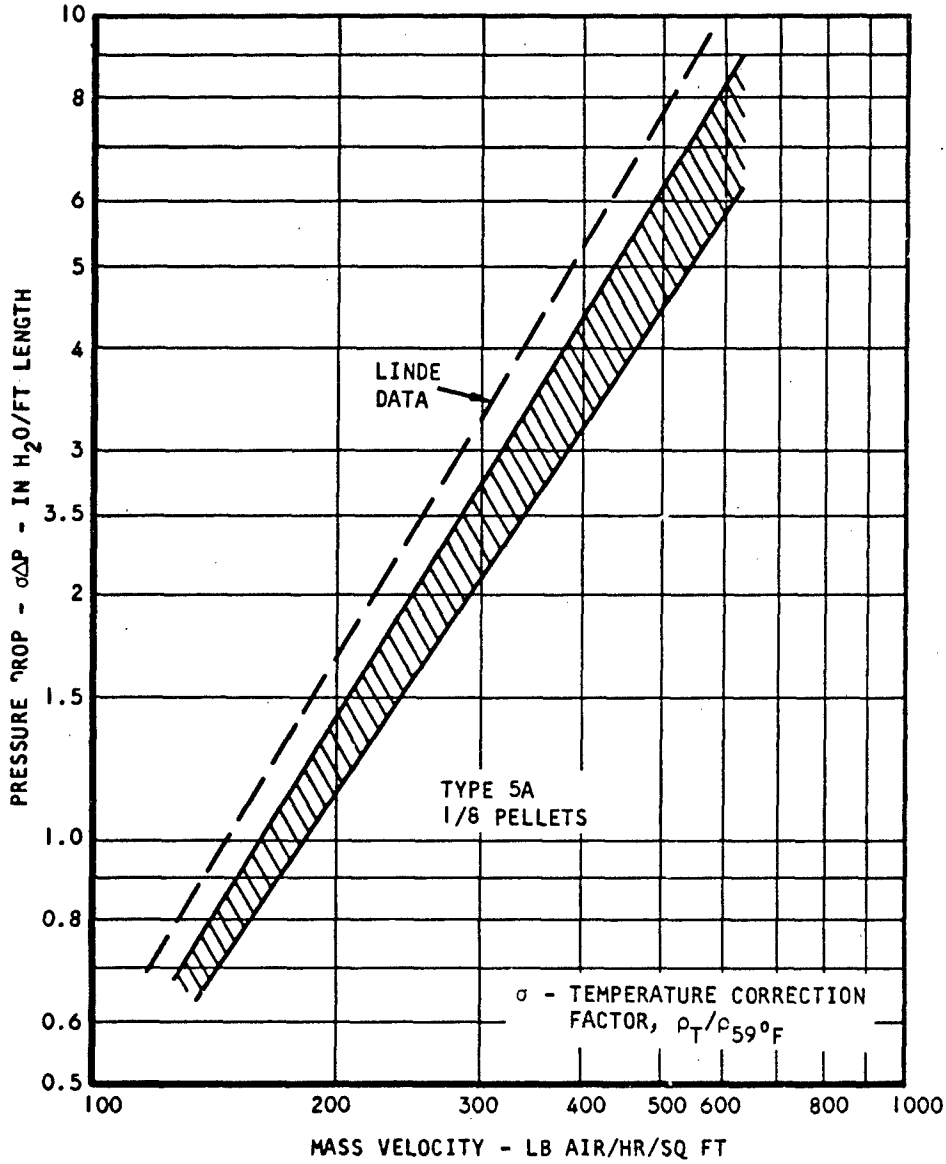


Figure 25. Pressure Drop (59°F)

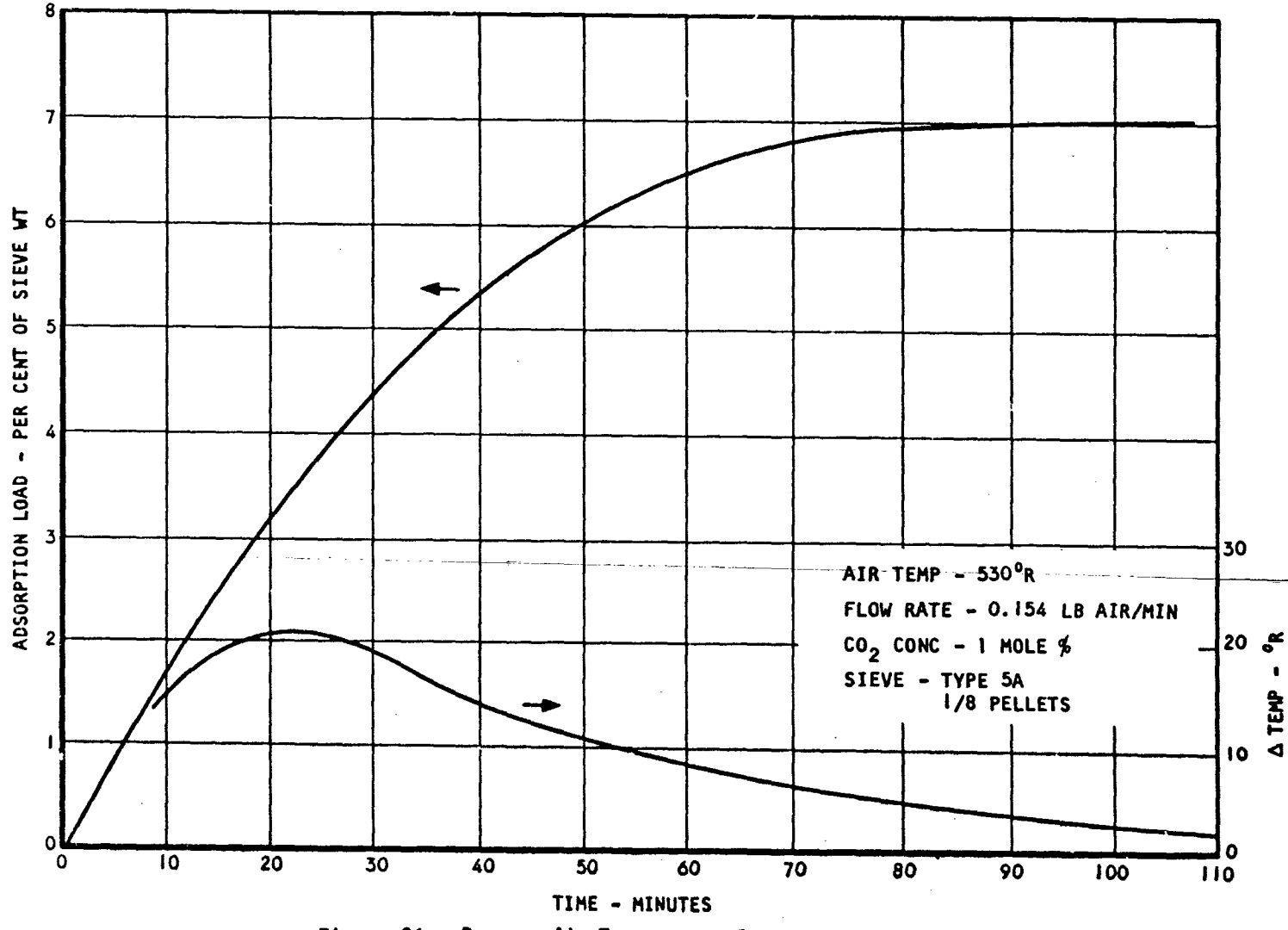
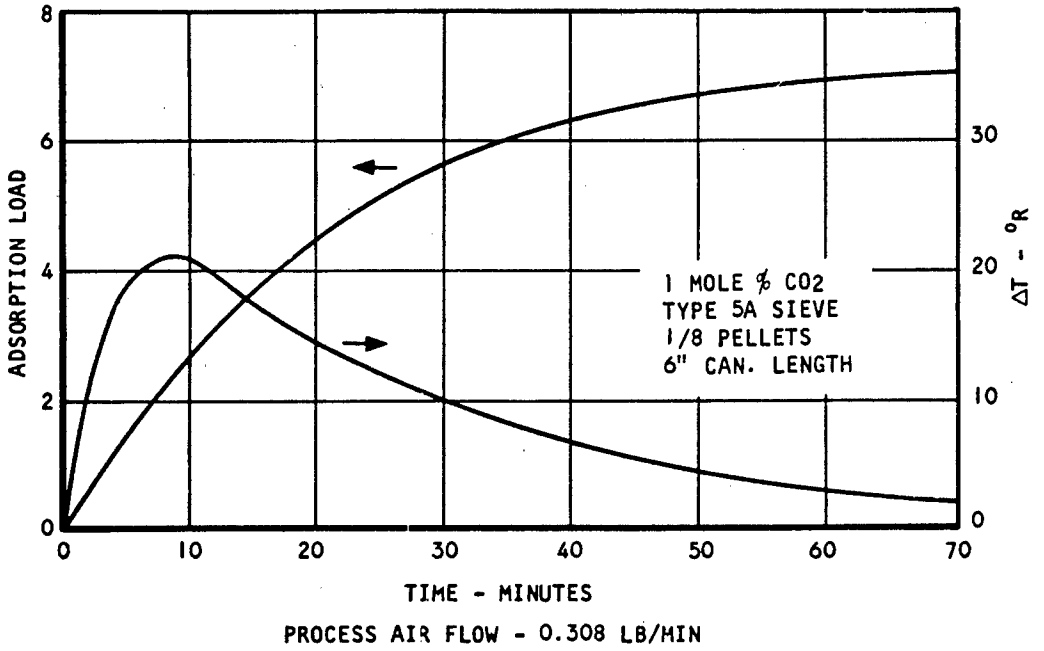


Figure 26. Process Air Temperature Increase Through Canister



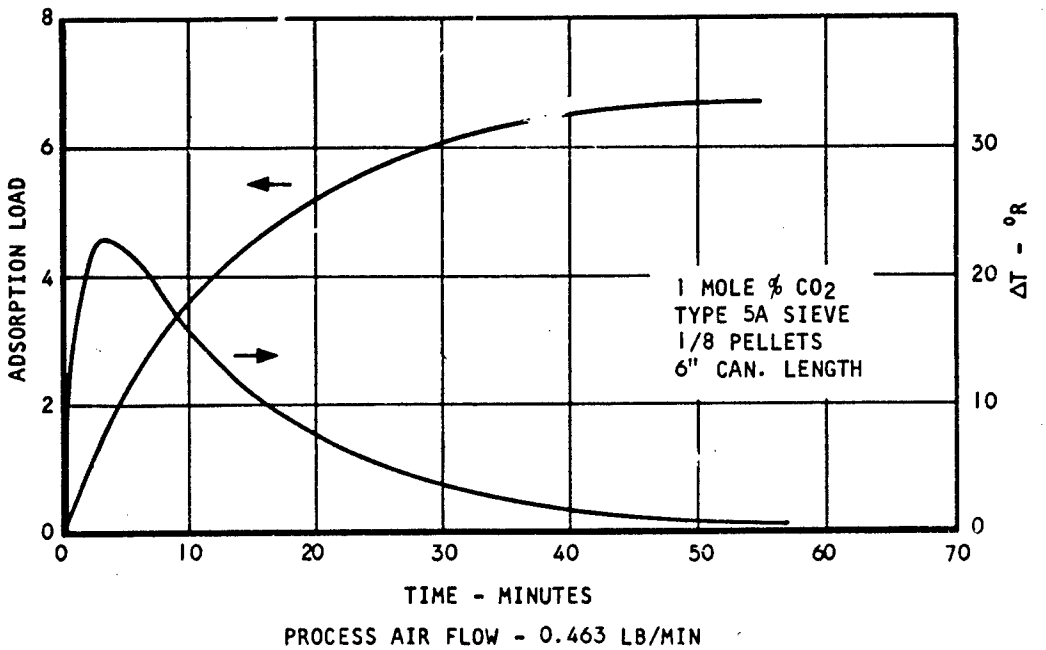
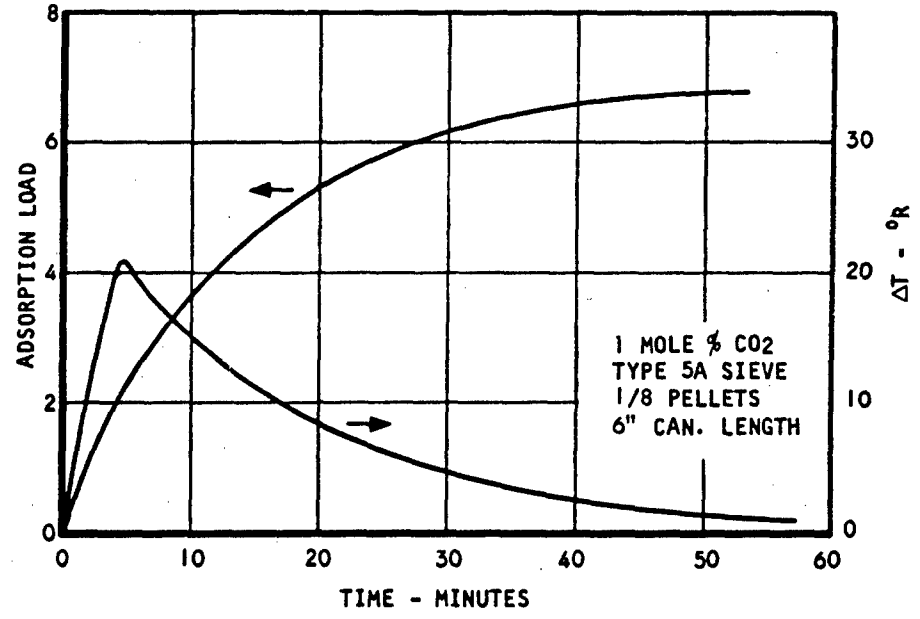
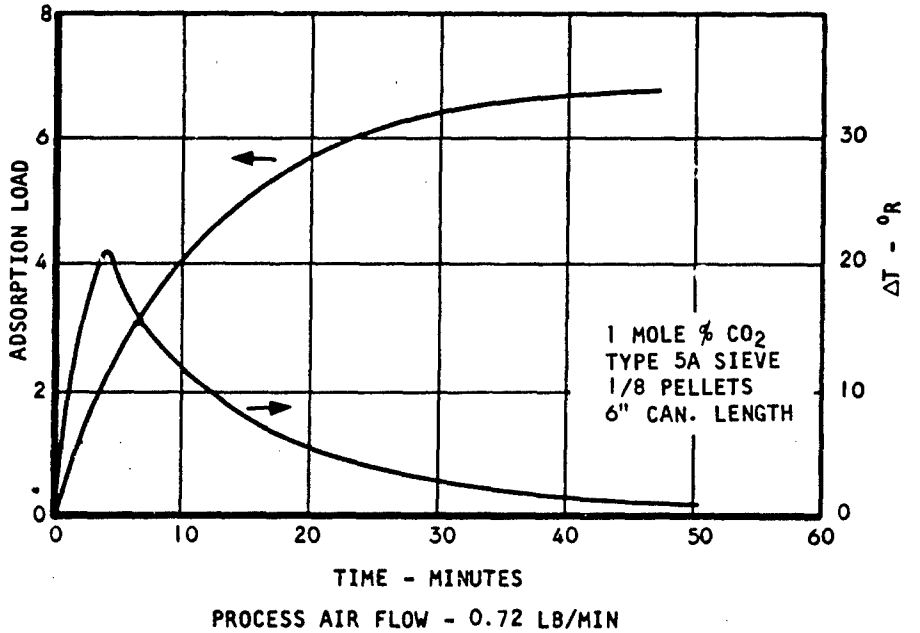


Figure 27. Process Air Temperature Increase Through Canister



PROCESS AIR FLOW - 0.616 LB/MIN



PROCESS AIR FLOW - 0.72 LB/MIN

Figure 28. Process Air Temperature Increase Through Canister

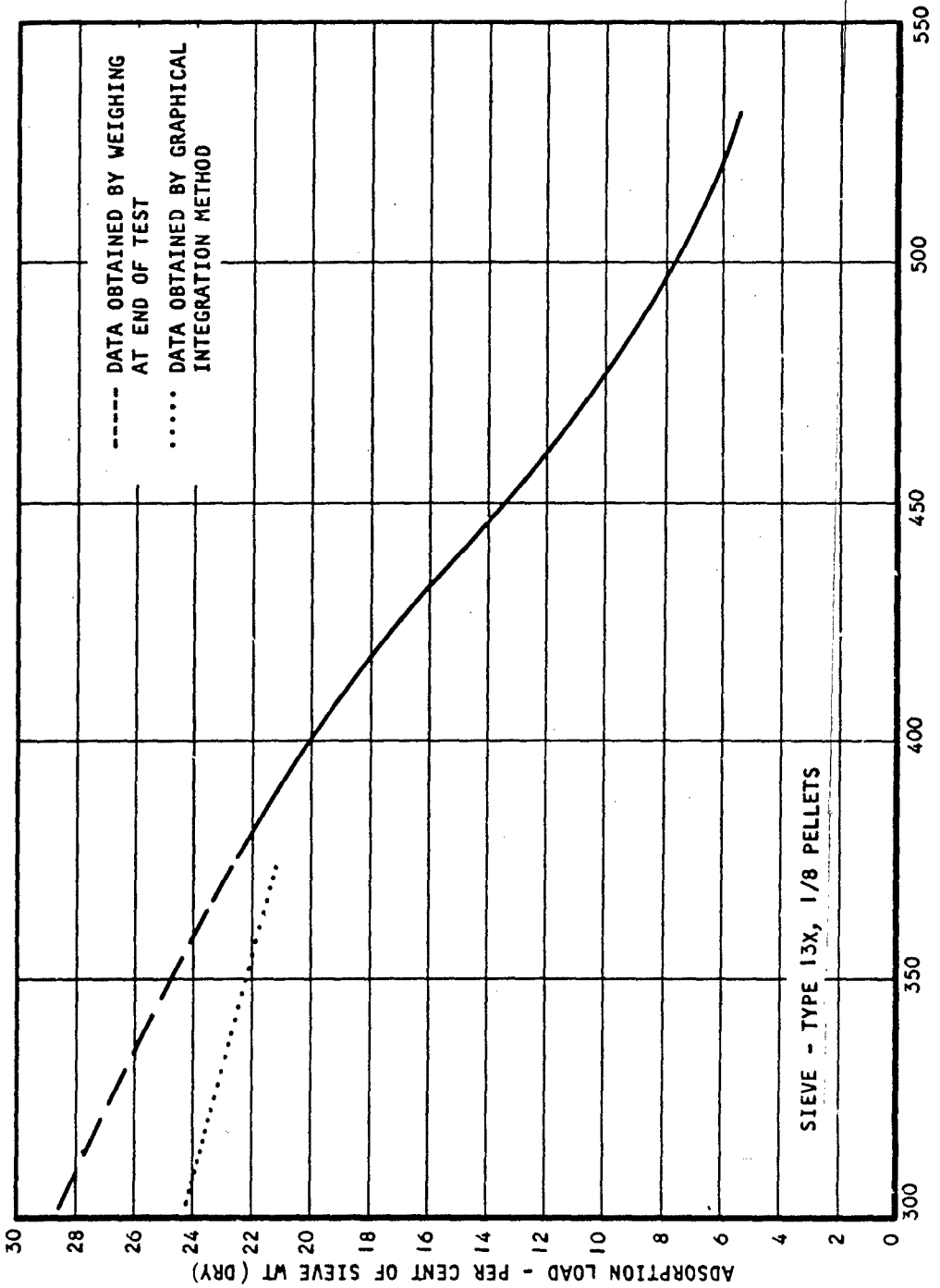


Figure 29. Effect of Process Air Temperature on Dynamic Adsorption Capacity, Type 13X Sieve

SECTION V

CONCLUSIONS

The results presented should be of value to scientists and engineers engaged in the study of carbon dioxide removal for space vehicle atmospheric control. Although the data is of primary interest to those engaged in the design of regenerable carbon dioxide removal systems, it should also be beneficial in establishing empirical relationships for the adsorption process.

The tests conducted indicated the variance of results for identical test conditions. Precise predictability of the adsorption process is impossible, and the results presented must be used with this in mind. However, the data was obtained with care and is representative of several tests conducted at each test condition.

The test results presented indicate the advantages gained by low temperature adsorption of carbon dioxide from simulated space vehicle atmospheric environments. Any decrease in the process air temperature proves beneficial to the adsorption of carbon dioxide by increasing the dynamic adsorption capacity of the sieve and raising the total weight of carbon dioxide removed from the process air for a combined adsorption-desorption cycle. Desorption of the sieve at the reduced temperature is not satisfactory. Warming of the sieve to ambient, or near ambient temperatures followed by vacuum desorption, gives an increased desorption rate and an increased overall (adsorption-desorption) carbon dioxide removal rate. This means that, for a given set of conditions, either the sieve weight can be reduced or the cyc'e time lengthened, the magnitude depending upon the minimum process air temperature that can be obtained.

The design of a regenerable system using synthetic zeolites must involve a design compromise of the adsorption and desorption requirements. When desorption is accomplished by vacuum, the desorption rate is controlling, because the adsorption rate can be varied by the process air flow rate, while the desorption rate (which is lower than the adsorption rate) is fixed by the canister design. If the carbon dioxide can be dumped to space, the vacuum of space eliminates the problem of the pumping speed of the vacuum source.

The results of the tests indicate that optimum adsorption occurs with a long canister length, low superficial velocity, small pellet size, and low process air temperature. Optimum desorption is obtained using a canister with a short flow length giving a large free surface area of zeolite.

Since the desorption cycle controls the amount of carbon dioxide that can be removed from the process air, a short canister or bed length would be desirable. The adsorption test results indicated that a short canister length reduces the amount of carbon dioxide adsorbed at the high dynamic adsorption efficiencies but increases the amount adsorbed at the intermediate efficiencies. Only a small decrease in the dynamic adsorption capacity results from the shorter canister length. The desorption tests indicated that the desorption rate decreased rapidly as the sieves desorbed, the maximum

desorption rate occurring when the bed was fully loaded (saturated). More carbon dioxide can be desorbed by not desorbing to the 100 per cent desorbed condition but rather cycling the canister faster and desorbing only in the range of high desorption rates. Therefore, so far as the overall carbon dioxide removal rate from the process air is concerned, a short canister length will not be detrimental since the amount of carbon dioxide adsorbed is not affected to any extent and the desorption is minimized. A short canister length will be advantageous from a systems standpoint because of the lower pressure drop and the resultant weight reduction by using a smaller compressor and the lower compressor power requirement.

A canister design will have a maximum superficial velocity for the process air. For velocities higher than the maximum, the carbon dioxide adsorption rate will increase very little and the increased compressor or fan power will be wasted. Adsorption at the high dynamic adsorption efficiencies is not important, remembering that for a cycling or regenerative canister, 100 per cent desorption by vacuum is not advantageous because of the low desorption rate and the long desorption time required. The effect of low superficial velocities was to increase the amount of adsorption at the high dynamic efficiency range. Therefore, superficial velocities approximating the maximum superficial velocity for the canister do not considerably affect the overall carbon dioxide removal rate. However, the higher pressure drop will be a penalty.

A canister design should incorporate a short bed length, perhaps of even a radial flow design. The process air should enter at as low a temperature as possible, which system limitations will dictate. The canister size and weight can be reduced by decreasing the cycle time of the canister, and the only limitation in this case will be the increased mechanical wear of the valves and the resulting reliability problem. Molecular sieve stability, as a function of adsorption-desorption cycling, is not considered but may also prove to be a factor.

Aeronautical Systems Division, Dir/Aeromechanics, Flight Accessories Lab, Wright-Patterson AFB, Ohio
Rpt Nr 62-560. LOW TEMPERATURE ADSORPTION OF CARBON DIOXIDE. Final report, Sep 62, 44p. incl illus, tables.

Unclassified report

A test program was conducted to study low temperature adsorption of carbon dioxide by synthetic zeolites for possible application to space vehicle environmental control systems. Type 5A molecular sieves and a 1 mole per cent carbon dioxide concentration were used for most of the tests. The range of the conditions included temperatures from

(over)

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1. Low temperature adsorption
2. Carbon dioxide removal system
3. Space capsule atmosphere
4. Regenerable adsorption system

I. AFSC Project 6146, Task 614609
II. Contract AF33(616)-8323
III. AiResearch Manufacturing Co., Los Angeles 9, Calif.
IV. G. Christensen
V. SS-715-R
VI. Not aval fr OTS
VII. In ASTIA collection

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