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ERDEC-TR-067

LABORATORY-SCALE PRESSURE SWING ADSORPTION SYSTEM

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Fort Washington, MD 20744

May 1993

U.S. ARMY
CHEMICAL
AND BIOLOGICAL
DEFENSE AGENCY

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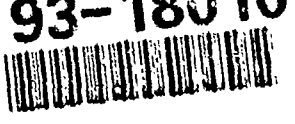

Aberdeen Proving Ground, Maryland 21010-5423

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	1993 May	Final, 90 Apr - 92 Sep

4. TITLE AND SUBTITLE
Laboratory-Scale Pressure Swing Adsorption System

5. FUNDING NUMBERS
PR-1C162622A553

6. AUTHOR(S)
Mahle, John J.; Buettner, Leonard C. (ERDEC); Friday, David K.; and Brady, Amanda B. (GEO-CENTERS, INC.)

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
DIR, ERDEC,* ATTN: SCBRD-RTE, APG, MD 21010-5423

GEO-CENTERS, INC., Fort Washington, MD 20744

8. PERFORMING ORGANIZATION REPORT NUMBER
ERDEC-TR-067

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES
*When this work was performed, ERDEC was known as the U.S. Army Chemical Research, Development and Engineering Center, and the ERDEC authors were assigned to the Research Directorate.

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

The design of a laboratory-scale pressure swing adsorption (PSA) system for air purification applications is described. Specific attention is provided to bed design, flow control, and feed generation systems. This apparatus is capable of automated, in-bed concentration, pressure and temperature measurements using computer control. Results are presented for some sample runs based on 150 SLPM of feed at three to four atmospheres of feed pressure.

14. SUBJECT TERMS
Pressure profiles
Experimental system
Laboratory scale
Pressure swing adsorption
In-bed profiles
Concentration profiles
(Continued on page 2)

15. NUMBER OF PAGES
25

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UL

14. SUBJECT TERMS (Continued)

Armored vehicle application
Regenerative air purification
Temperature profiles

PREFACE

The work described in this report was authorized under Project No. 1C162622A553, CB Defense/General Investigation. This work was started in April 1990 and completed in September 1992.

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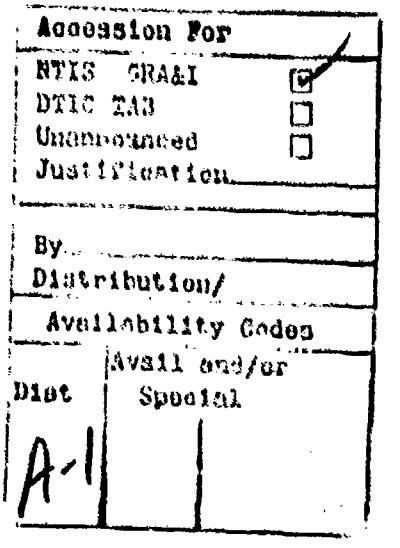

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1. INTRODUCTION

The Army is currently conducting programs to improve existing air purification systems using advanced technologies. One of the technologies being considered is pressure swing adsorption (PSA){1}. The advantage of this system is that the adsorption beds can be cycled between feed and purge steps to provide indefinite service life. PSA is a well accepted commercial process for air purification applications, the most important being air dryers. Typical specifications for such a system would reduce the water vapor concentration from 30,000 to 3 ppm a four order of magnitude reduction{2}. NBC applications can require five or even six orders of magnitude in purification depending on the chemical threat. PSA is more complex both mechanically and from an integration standpoint than current Army single pass, charcoal-based adsorption filters{3}. New methods must be developed to evaluate and qualify PSA for military applications, because there are a wide range of potential challenge vapors and operating conditions with no obvious design limiting scenario.

Recently a specific application, the next generation armored vehicle, has focused an extensive development effort on PSA for NBC protection. Size constraints are such that the NBC system would have to be designed to a fraction of the volume of adsorbent relative to a commercial air dryer unit to achieve this concentration reduction. Also there are a wide range of known, suspected and future chemical threats for which protection must be provided. Selecting the operating conditions, system geometry and adsorbent/adsorbents to provide this protection requires a combination of experimental and process modeling work.

Fundamental data required to design the system is best measured with small laboratory-scale apparatuses. The units can be fully instrumented to provide process design information. These pilot systems are at least an order of magnitude smaller than the full-scale systems they are intended to model. Results from the laboratory scale system can used to evaluate the design model from a fundamental standpoint. When questions arise in the operation of the full-scale unit, they can be answered by a combination of design model predictions and laboratory scale results. The small scale system provides an ability to reduce utilities and fabrication costs, chemical usage, and toxic chemical release.

It is of interest to characterize enough of the important parameters of the PSA process that the resulting data can be used for validation of a design model. These parameters include the size, flowrate, and transient behavior in concentration, temperature and pressure. One of the few studies of contaminant filtration at the lab-scale appears in a contractor's final report{4}, by Research Triangle Institute for

CRDEC, describing two laboratory PSA units. Some experimental work is reported using this system. The results are indicative of general trends expected in PSA performance but for a number of reasons this study does not provide the data required to evaluate a design for an armored vehicle application. For example, the range of superficial velocities considered are much lower than would be feasible, the cycle time too long and choice of vapors and adsorbents was too limited. In order to conduct extensive and conclusive testing of PSA a new design was initiated.

The present study describes a laboratory pressure swing adsorption system fabricated in support of armored systems collective protection development efforts. This unit was designed to provide data for fundamental studies of the PSA process in the NBC environment specifically for incorporation of the system into an armored vehicle application.

2. SYSTEM DESIGN

2.1 System Schematic

Figure 1 presents a schematic of the PSA system. Regulated high pressure air is provided from an oil-free reciprocating compressor. A mass flow controller is used to regulate the feed flowrate. The chemical feed, as either a gas or liquid, is mixed with the air stream in a mixing column. Variations in feed pressure and concentration are buffered by the presence of a surge tank in the feed line upstream of the PSA beds. Alternately, one of the two beds is challenged while the other bed is purging with a portion of clean product. Bed switching is achieved by programable control of three-way valves. A multi-port sampling system is used to measure the vapor phase concentration in the bed and in the feed, product and purge streams. A mass flow controller is used to set the product flowrate and a control valve is set to provide the required system pressure. The purge gas is vented through a second surge tank.

2.2 Flowrate and Bed Diameter

In all laboratory type studies it is important to be concerned with the effects of scale-up. Phenomena that appear important at the laboratory scale may not be significant at full-scale and vice versa. For adsorption systems, a constant superficial velocity is generally used as an appropriate scale-up criteria. Another possible criteria could be to equate the Reynolds' Number based on particle diameter but for the present system the final particle diameter or even the adsorbent type was an unknown. It is established that for an armored vehicle such as the M1, approximately 100 SCFM of breathable air is required to maintain overpressure.

Preliminary estimates of the available space claim for a PSA system suggest that for a two-bed system the bed diameter would be approximately 12 inches. The available

pressure for the PSA system is likely to be between 30 and 60 psig. Pressures greater than 60 psig can not be economically provided while those less than 30 were thought to provide inadequate separation. Therefore 45 psig is chosen as the base-case condition pressure. At this pressure 200 SCFM feed (assuming 0.5 purge to feed ratio and 100 SCFM product) and a 12 inch bed diameter corresponded to a superficial velocity of approximately 30 cm/s. One choice of scaled conditions based on this superficial velocity is 150 SLPM (5 SCFM) feed to a 2 inch diameter bed. Based on this analysis a series of adsorption beds can be fabricated to bracket these conditions using one length and several diameters. The laboratory system is designed to operate with flowrates between 30-250 SLPM (1-9 SCFM) at pressure of 60 psig.

3. DESCRIPTION OF APPARATUS

3.1 Feed Generation

The feed air is supplied by a oil-free air compressor at a pressure of 95-110 psig in a receiver tank. The air is then conditioned using a combination of refrigerated and desiccant dryers providing a variable feed dew point of between -5 to -50 °C at atmospheric conditions. Higher dewpoints can be achieved by injection of liquid water to the feed stream.

Two types of feed generation procedures are employed to accommodate chemicals with a range of vapor pressures and boiling points. The ambient temperature in the room is maintained at 25±3 °C. For all chemicals with boiling points near ambient temperature, the chemical is kept liquified under pressure and dispensed using an HPLC pump (Waters Inc. Model No. 6000A). The chemical is metered into a gas-liquid contactor consisting of a 3 inch diameter, 16 inch long cylinder filled with high surface area nickel plated packing material. Liquid droplets cascade countercurrent to the flow. Additional evaporation efficiency can be obtained by heating the vessel. Chemicals which would not condense under the temperature and pressure of the PSA experiment are delivered by using a mass flow controller to meter the vapor directly. The challenge stream is then sent to a 38 liter surge tank used to maintain a constant pressure source and dampen challenge concentration fluctuations.

3.2 Flow Control and Valving

Flow switching is performed using four, three-way air actuated 3/8 inch trunion valves. Air actuation is chosen to provide rapid response of the valves. The trunion valves (Whitey no. SS-83XTS6-51DC) are found to provide long life (100K cycles) with minimal leakage. The sequencing of the 3-way valves is provided by solid state relays, controlled through a bus interface card on a PC.

It is quite difficult to characterize the instantaneous flowrates in the course of a

cycle. During pressurization and depressurization steps, flow rates, pressures and temperatures can change rapidly. For example pressurization and blowdown between 1 atm to 4 atm can occur in two seconds. Unfortunately, most flow measurement and control devices respond on a slower time scale. Therefore, there is always uncertainty in the absolute flowrate although the average may be fairly well represented. For the present system, flow control was accomplished using mass flow controllers on the feed and product streams and a valve to set the pressure in the feed bed. The feed mass flow controller (Tylan General Inc., FC-262) was upstream of the feed surge tank. During a typical thirty second half-cycle run the product flowrate reading changed approximately 10% from the setpoint and required about three seconds to return to the setpoint.

The following explanation may describe the operation of the controller during the cycle changeover. At the chosen flowrates and bed volumes enough gas is provided to pressurize the beds in less than three seconds. For a 2 inch diameter, 20 inches long bed the ratio of the volume of the 38 liter surge tank to the volume of the bed is approximately 40:1. Because of this large volume, the bed pressurizes as if the feed pressure is constant i.e. usually less than 2 psi change in the feed surge tank. The product mass flow controller response is sluggish so it probably does not move the valve stem appreciably during this interval. Tylan claims a typical response time of 30 sec to within 2% of full scale. This suggests that the mass flow controllers may be acting very similar to fixed-position valves.

Surge tanks on the purge and feed lines allow an integrated flowrate to be measured using a calibrated dry test meter. These values compared well with the readings taken from the mass flow controller. Typically these values agreed to within ±3%. The flowrates recorded for the experiment are those measured using the test meter. These values are recorded prior to and at the conclusion of the experiment.

The system pressure is controlled with a valve on the purge side of the purge-product split. Assuming an upstream pressure of 45 psig, a downstream pressure of 0.5 psig a globe valve with linear positioner is chosen having a Cv of 0.8 and turn down ratio of 40:1 (Research Control Inc., Globe Valve Type 807). This would allow the purge flowrate range of 20 to 800 SLPM.

Enhancements are possible for this system in order to accomplish more complex steps. Product purity can be increased by pressurization with product rather than feed. This could be done with a shut-off valve on the purge line. By stopping the purge flow the purge bed come up to pressure with clean product air. Similarly more than two beds could be studied in order to implement a sequence of steps which increase the length of time a bed is purged.

3.3 Bed Design

A schematic of the adsorption bed is shown in Figure 2. The vessel is machined from 304 stainless steel stock to a length of 40 cm with 1/8 inch wall thickness. The top and bottom end caps are 5.0 cm long. Three beds at each of four diameters (2.54, 3.81, 4.08 and 7.62 cm or 1, 1.5, 2 and 3 inches) have been fabricated. A 60 U.S. sieve mesh screen reinforced with a perforated plate is used to contain the adsorbent. This screen is fitted to the bottom cap and the screen is held in place with the spring from the top. Sample ports, 1/8 inch NPT, were drilled at 5 cm intervals along the length of the bed to permit in-bed sampling.

There two aerosol/particulate filters placed in the lab scale PSA system. One is between the feed surge tank and the bed inlet (Matheson Inc., Model No. 6124-P12FF). This is used to trap any rust particles from the surge tank which was untreated steel. The second filter (Matheson Inc., Model No. 6134-P8FF) is used to prevent dust from entering the product flowmeter.

3.4 Concentration Measurement

Concentration is measured at several points in the system in order to provide a basis for completing the material balance, and evaluating system performance. A particularly unique aspect of this sampling is the ability to measure in-bed concentration profiles. Details of the multi-port sampling system are presented elsewhere{5}. That system is capable of collecting six samples simultaneously. These are usually chosen as feed, purge and four other ports depending on how far the adsorption wave had travelled in the bed.

Chemical analysis is conducted using an Hewlett Packard 5890 gas chromatograph equipped with a flame ionization detector and an Hewlett Packard 3336 integrator. Vapor samples may be taken from the feed, product and purge streams and in-bed ports. As many as six samples can be collected simultaneously. The vapor samples collected in the samples loops are sequentially passed to the GC for analysis. Due to the dynamic nature of the PSA process, bed exit concentrations vary over the course of the feed and purge steps. These must be averaged in order to complete a material balance. Ballast volumes of 38 liters are used to average the concentration for the purge and product streams. The residence time in this tank is approximately that of one half-cycle.

Two control algorithms are written in Pascal (Borland Inc., TURBOPASCAL) and executed on two IBM compatible PC's. One computer controls the timing of the 3-way flow direction valves while the second computer controls the valve sequencing of the in-bed gas sampling system and GC data acquisition. Contact closures on a relay board (Metrabyte Inc., Model No. ERB-24) provide the signal required of solenoids for

the pnuematic actuators for the flow direction valves and the electric actuators for the gas sampling valves. Care had to be taken to shield the EMF of the valve coils from the computer. Several runs had to be prematurely aborted due to interference from the coil with the cables between the computer and the relays.

3.5 Temperature Measurement

The in-bed temperature analysis is accomplished using sample ports at the same locations as the concentration sampling. The thermocouple probes, type T, 1/32 inch, ungrounded(Omega Inc No. TMQSS-032U-6) are placed in the center of an empty bed at fixed positions using Swagelok connections and Teflon ferrules. These thermocouples have a reported time constant of 1.8 seconds. Temperatures are recorded using an Apple Macintosh PC and A/D converter with cold junction compensation (National Instruments Inc., Model No. NB-MIO-16X).

3.6 Pressure Measurement

Transient in-bed pressure profiles could also be measured on the PSA system. Pressure transducers are connected to the system at the various port locations. These transducers (Validyne Inc., Model No. DP45-18) measure the absolute pressure over the 0-80 psid full scale. The transducers zero and span were set using a calibrated reference pressure gauge. Results were recorded by sampling at a rate of 100 Hz for each channel.

3.7 Humidity Measurement

Feed and product humidity can be monitored be during the course of the experiment. Feed air is analyzed at pressure by taking a slip stream of 1 SLPM from the feed surge tank using a dew point hygrometer(EG&G Inc., Model No. 911) capable of -25 C. The product dew point is always much lower than the feed for either adsorbent studied BPL or 13X. Product dew point readings as low as -70 C are recorded using a second hydrometer (EG&G Inc., Model No. 300). An external cooling system(5 °C water bath) is required to achieve low dew point measurements with these hygrometers. Results are recorded on a strip chart recorder.

3.8 Spring Sizing

One important aspect of PSA design is the appropriate sizing of the bed springs. If the spring provides too little force, then during each blowdown step where high velocities occur the resulting large pressure drop will fluidize the bed momentarily. Repeated cycling under such conditions can lead to bed attrition. Similarly if the spring force is too large then this could crush the particles without any cycling at all. For the present study the springs are sized so that with compression the pressure drop would not

displace the spring. Calculation of the bed pressure drop suggests that the bed depressurizes in approximately 1 second. This transient response is described using a exponential fit for pressure in psig and time in seconds.

$$P = 45e^{-8.4t} + 14.7 \tag{1}$$

The maximum velocity is calculated by integrating the equation of continuity with the assumption of negligible pressure drop through the bed. The velocity is expressed using only the length of the vessel and standard conditions.

$$v = L\left(\frac{1}{P_{ref}}\right)\frac{dP}{dt} \tag{2}$$

An estimate of the maximum bed pressure drop can then be obtained by using the Ergun equation.

$$-\frac{dP}{dz} = f_1 v + f_2 v^2$$
$$f_1 = \frac{150\mu(1-\epsilon)^2}{D_p^2\epsilon^3} \tag{3}$$
$$f_2 = \frac{1.75\rho(1-\epsilon)^2}{D_p\epsilon^3}$$

The calculated values for bed pressure and bed pressure drop are shown in Figure 3. Taking the maximum pressure exerted on the spring to be 10 psi the spring constant is calculated to give this pressure for a one inch displacement for the various bed diameters. This is much less than the crush strength of the adsorbents activated carbon and 13X molecular sieve which is on the order of 10 pounds per pellet.

4. TESTING PROCEDURE

It is necessary to provide a procedure for consistent packing of the adsorption beds. First all required in-bed probes are connected: a 1/16 SS tube for concentration from one side and a 1/4 inch SS tube for a 1/32 inch thermocouple and 1/4 inch pressure transducer from the other side with both the sampling probes and thermocouple extending to the middle of the bed. Only one of the two beds is fitted for in-bed sampling. No preconditioning is performed when the adsorbent is activated carbon. It is

taken directly from the drum and weighed. BPL is loaded in the beds by pouring it through a drop tube of length four feet, diameter 2 inches. Overnight drying at 350 C under 3 lit/min nitrogen bleed is performed when the adsorbent is 13X molecular sieve. This is done using a tube furnace and quartz drying tube. Once the 13X is cooled the appropriate amount of adsorbent is weighed and poured directly in the bed without the droptube to minimize exposure to air. Approximately two inches of loose glass wool is added to the bed to capture any fines. A choice of spring lengths are available to accomodate a variety of bed depths and still keep the spring compression to 1 inch.

As a final step in bed preparation the beds are pressurized to 60 psig with clean, dry house air then suddenly depressurized. This is repeated thirty times when BPL is the adsorbent five times with 13X the latter to minimize the exposure to humid air. This process is effective in removing fines. Early results indicated that the presence of fines fouled the product mass flow controller, deposited in the product line, and provided adsorption sites in the gas sampling lines.

Prior to each run a calibration is performed of the sampling system and the gas chromatograph. A previously determined gas standard is used to purge out the multiport sample loops to determine the appropriate response factor. The flow controllers are also periodically calibrated against a NBS traceable standard flowmeter.

5. EXPERIMENTAL RESULTS

5.1 Concentration Profile

PSA experiments are usually run until a steady state condition is achieved. This is determined when the purge, product and in-bed concentrations remain constant. Often this required that the system operate several days unattended. Initially, two nontoxic chemicals were chosen for study in the PSA system. These have chemicals structures and vapor pressures similar to some compounds of military interest. The physical properties are listed in Table 1.

Results from a typical PSA experiment are presented in Figure 4. Concentrations are plotted versus cycle number for the run conditions listed in Table 2. The concentration is measured at six ports using the multiport sampling system: bed inlet, 5 cm, 10 cm, 15 cm, 20 cm from the top of the bed and the purge from the surge tank. Toward the end of the experiment the sample line at the 5 cm port is relocated to sample the product concentration.

For most experiments to this point the feed concentration is held at a constant value and remains there during the course of the experiment. The purge concentration rises quickly to a plateau value which is approximately twice the feed concentration. This result indicates that material is being purged off rather efficiently. If there were no

purge one would expect the two beds to saturate with chemical in 500 cycles based on a loading of 0.4 g R-113/g carbon.

During the course of a PSA experiment concentration profiles within the bed develops which eventually reaches a plateau concentration at or below the feed concentration. This result is characteristic of PSA and is referred to as the approach to periodic-state. This occurs when the amount of contaminant chemical entering in the feed is equal to the sum of the amount leaving in the product and purge streams. At this point it is possible to compute a material balance based on the feed, product and purge concentrations and flowrates. For the present example this difference is within 2 percent. The in-bed sampling profiles eliminate the need for bed depth studies to be run under exactly the same conditions. Due to slight variations in ambient temperature and bed packing, such identical conditions would be nearly impossible to achieve in practice.

5.2 Pressure Profile

A sample pressure profile measured in the laboratory PSA system is presented in Figure 5. Both steps of the two step process are shown. The pressure(psig) measured at three points in the bed are plotted versus time of the cycle. The data indicates that it takes approximately two seconds to pressurize and depressurize each bed. Also the measured pressure drop through the beds is negligible, a fraction of one psi. This suggests that the influence of the pressurization and blowdown steps will be most important for very short cycle times i.e. those less than ten seconds where the pressurization and blowdown times become a significant fraction of the total cycle time.

5.3 Temperature Profile

One interesting finding of the laboratory PSA experiments is the nature of the temperature behavior in the beds during a cycle. Temperature data is recorded with no chemical challenge. The conditions of this run are presented in Table 2. Figure 6 presents a plot of the recorded thermocouple responses. The temperature at each port is plotted versus time in the cycle. There is approximately a -1 °C temperature depression from ambient during the adsorption step and -13 °C change for the desorption step. This response is postulated to result from adsorption and desorption of oxygen and nitrogen.

6. CONCLUSIONS

- A laboratory pressure swing adsorption system has been developed for air purification studies.

- In-bed sampling systems have been developed to monitor concentration, temperature and pressure profiles.

- This system provides fundamental data for developing pressure swing adsorption design models.

Future reports will detail specific PSA experimental studies for the design implication of important parameters such as cycle time, pressure ratio, and purge to feed ratio.

NOMENCLATURE

P	= pressure
t	= time
v	= superficial velocity
L	= bed length
z	= axial distance
f_1, f_2	= parameters defined in equation (3)
D_p	= particle diameter
ε	= void fraction
μ	= viscosity of the gas
ρ	= density of the gas

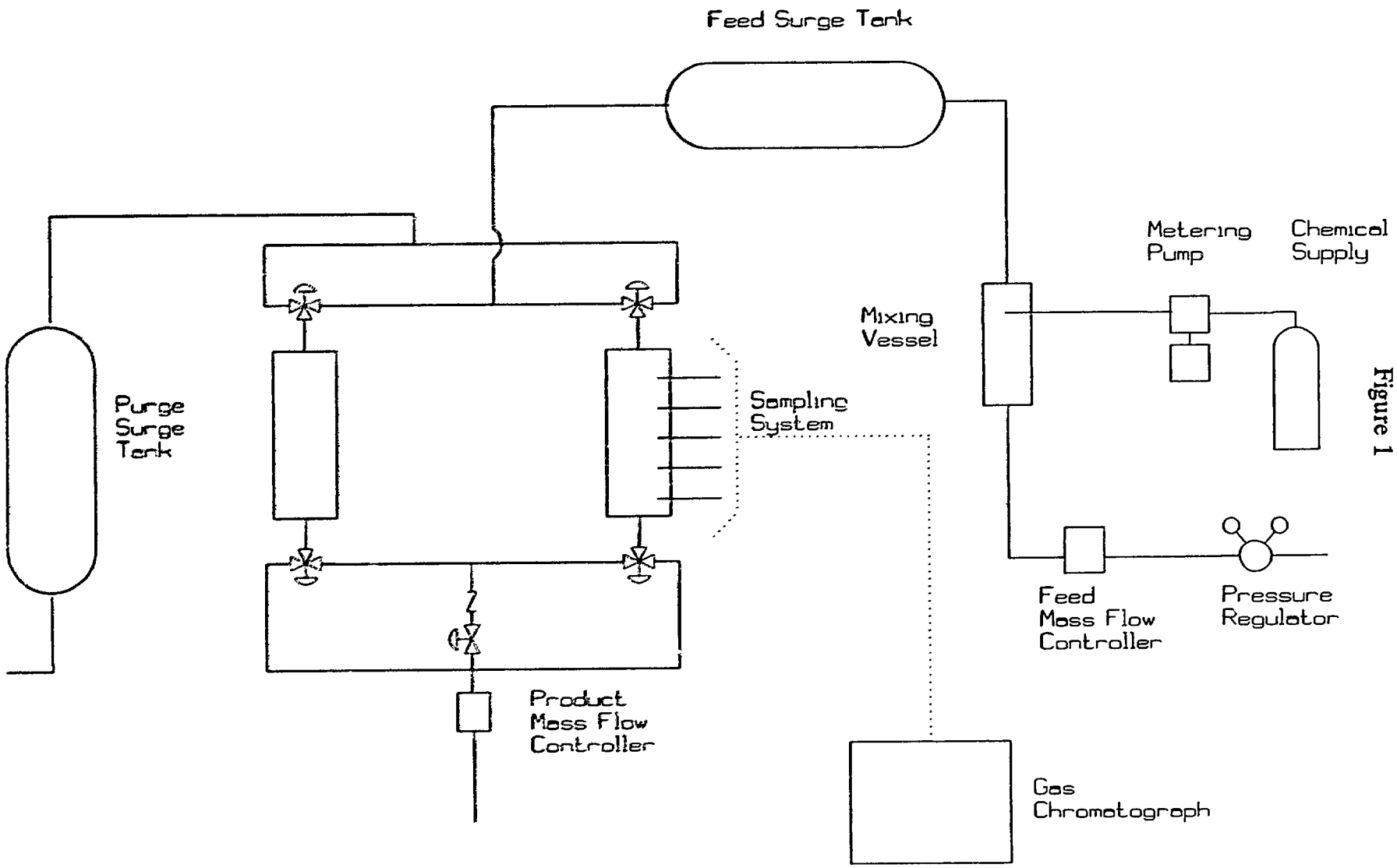

Laboratory PSA
System Schematic
Feed Surge Tank
Metering
Pump
Chemical
Supply
Mixing
Vessel
Purge
Surge
Tank
Sampling
System
Feed
Mass Flow
Controller
Pressure
Regulator
Product
Mass Flow
Controller
Gas
Chromatograph

Figure 1

Figure 2

PSA Bed With Sample Probes

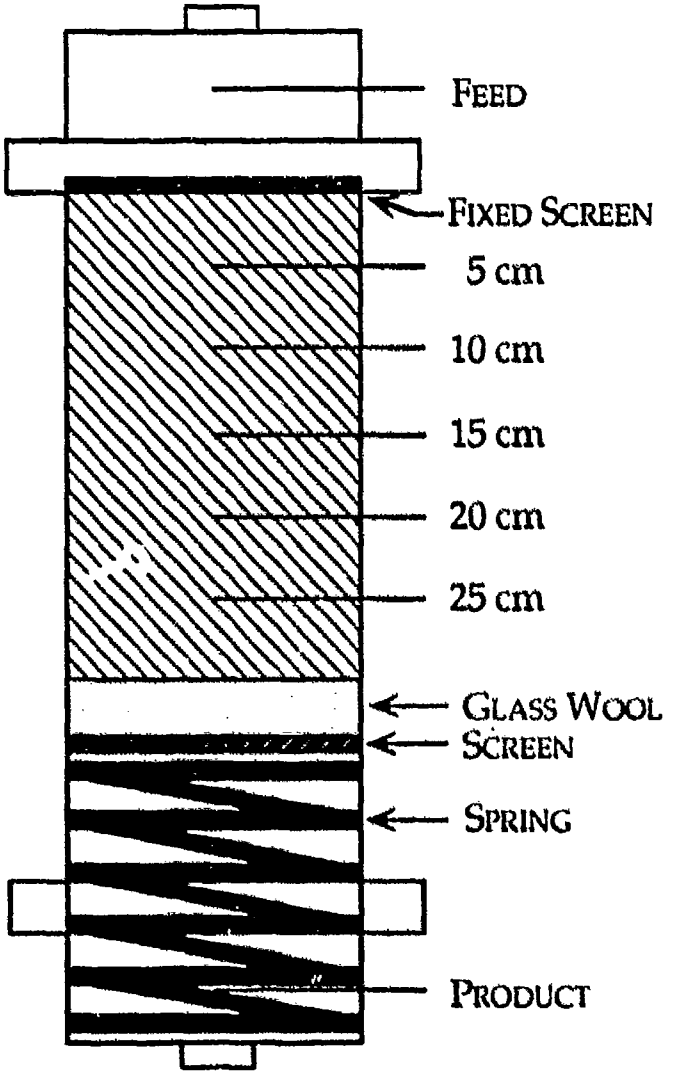


Figure 3

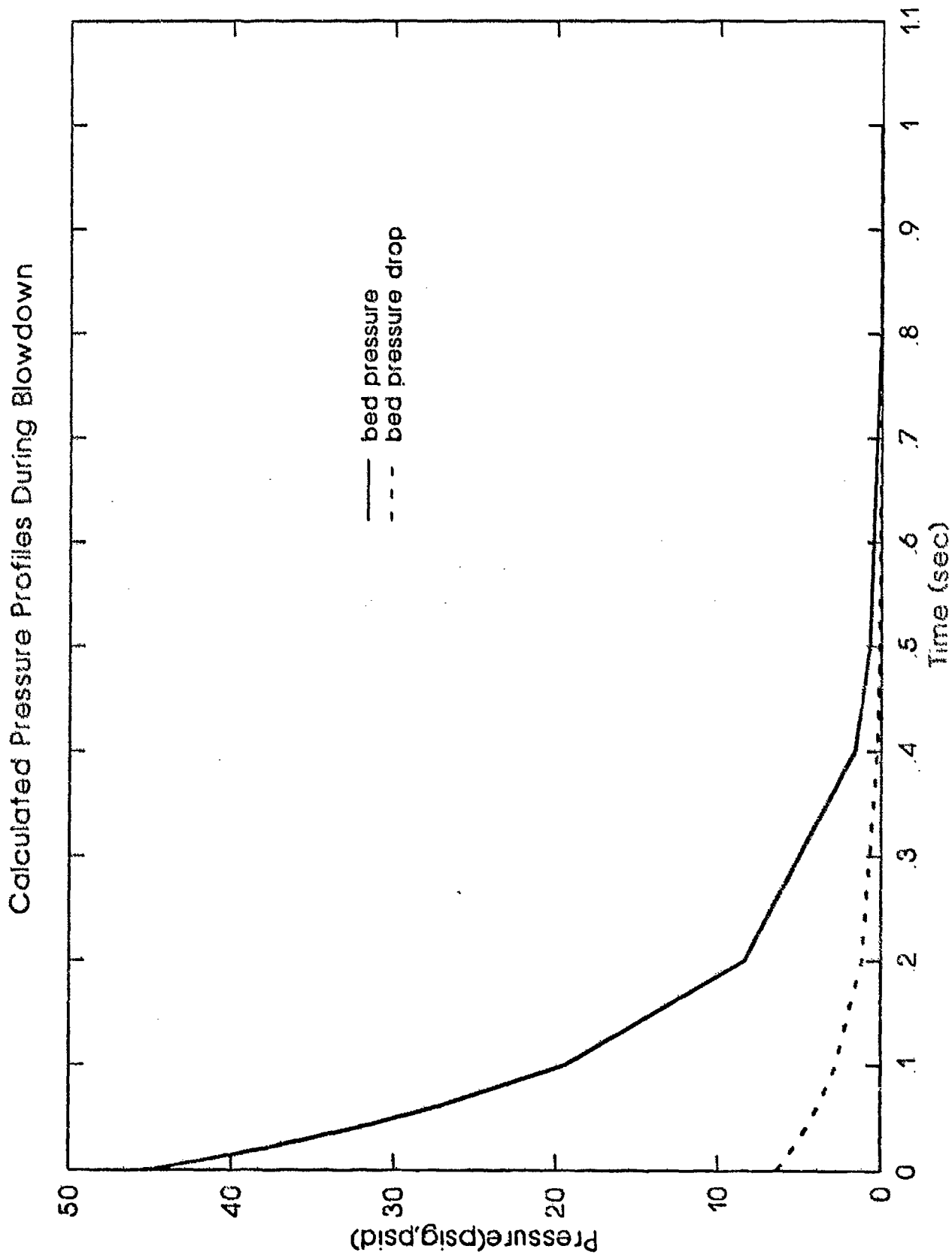

Calculated Pressure Profiles During Blowdown
bed pressure
bed pressure drop
Pressure(psig,psid)
Time (sec)
0
10
20
30
40
50
0
.1
.2
.3
.4
.5
.6
.7
.8
.9
1
1.1

Figure 4

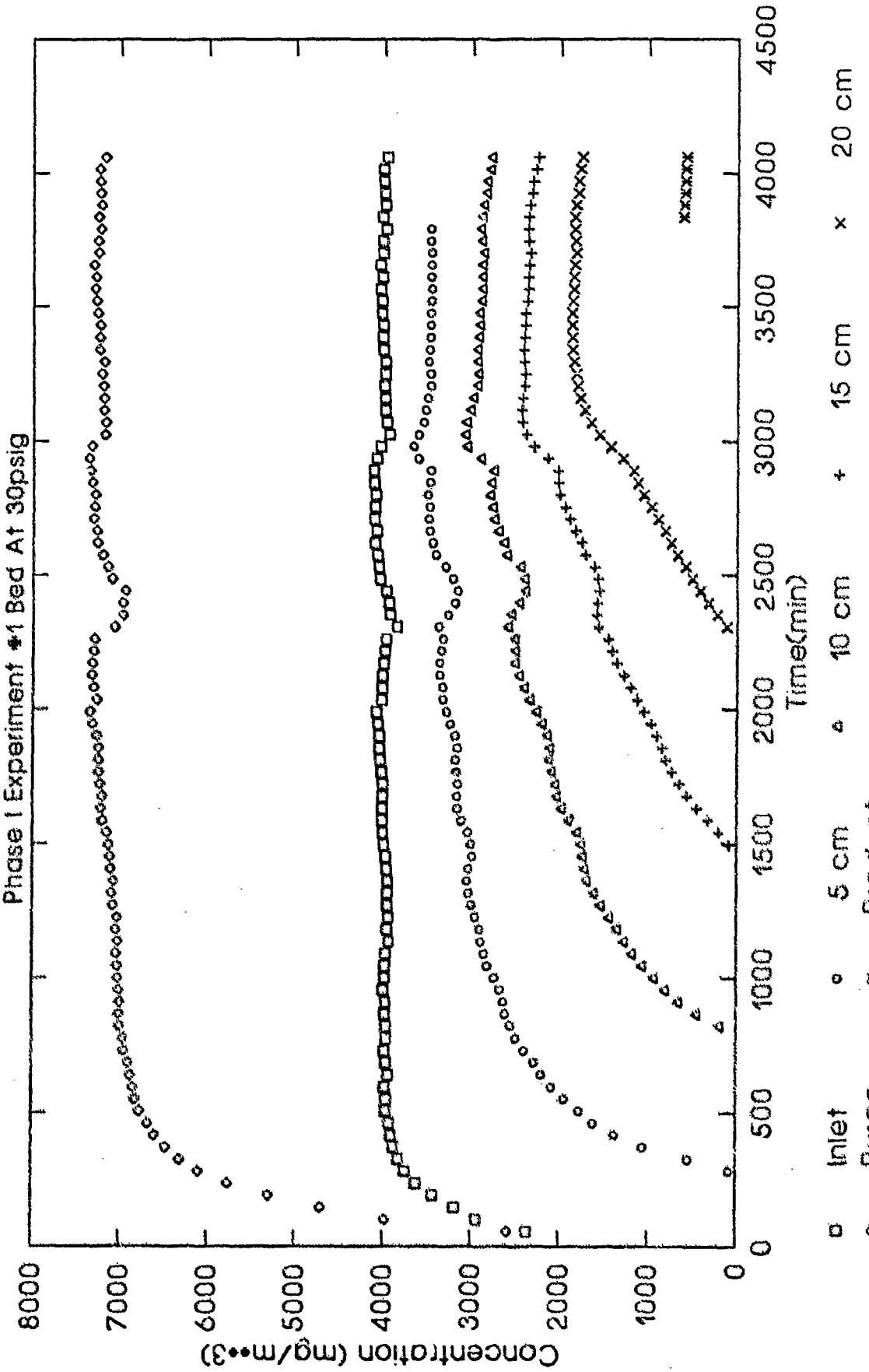
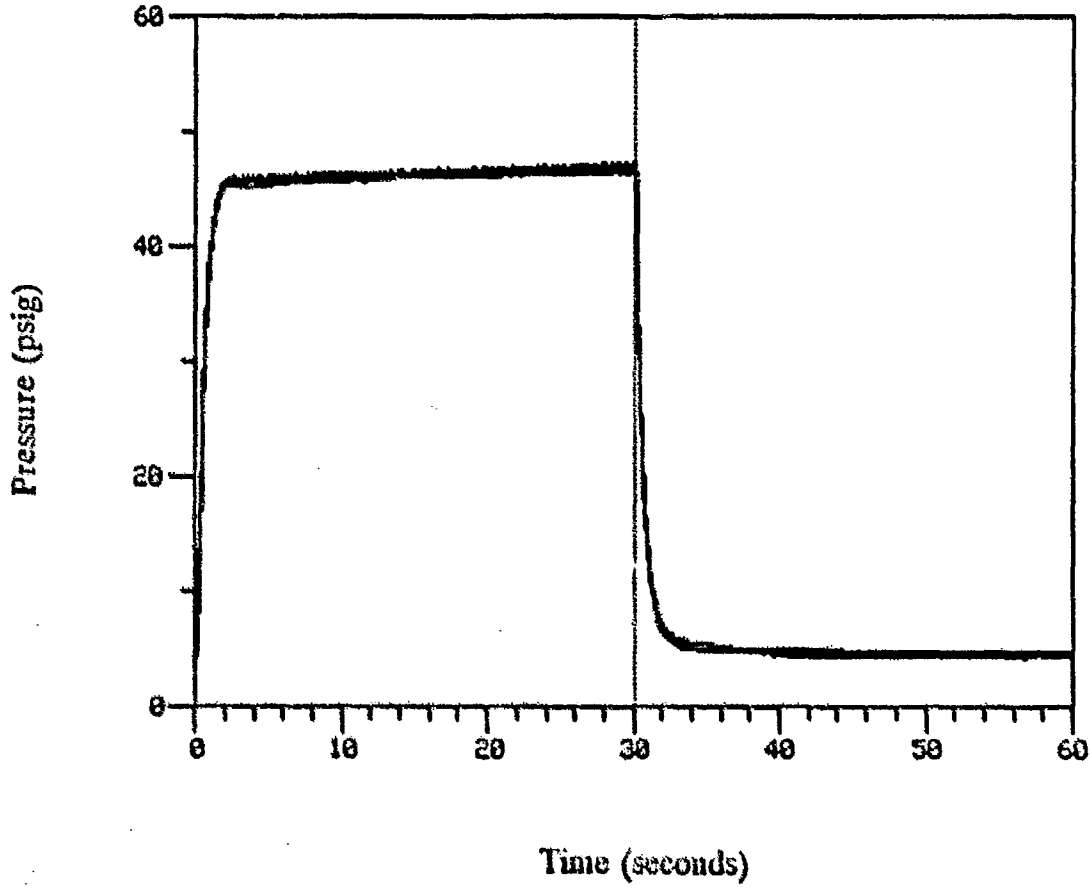

PSA Profiles for R113 on BPL - 10 Feb 92
Product 76SLPM Purge 77SLPM 2in 24cm 60s
Phase I Experiment #1 Bed At 30psig
Concentration (mg/m**3)
Time(min)
0
1000
2000
3000
4000
5000
6000
7000
8000
0
500
1000
1500
2000
2500
3000
3500
4000
4500
Inlet
Purge
5 cm
Product
10 cm
15 cm
20 cm

Figure 5

PSA Transient Pressure Response
At Six In-Bed Sample Ports
Feed Pressure 45 Psig



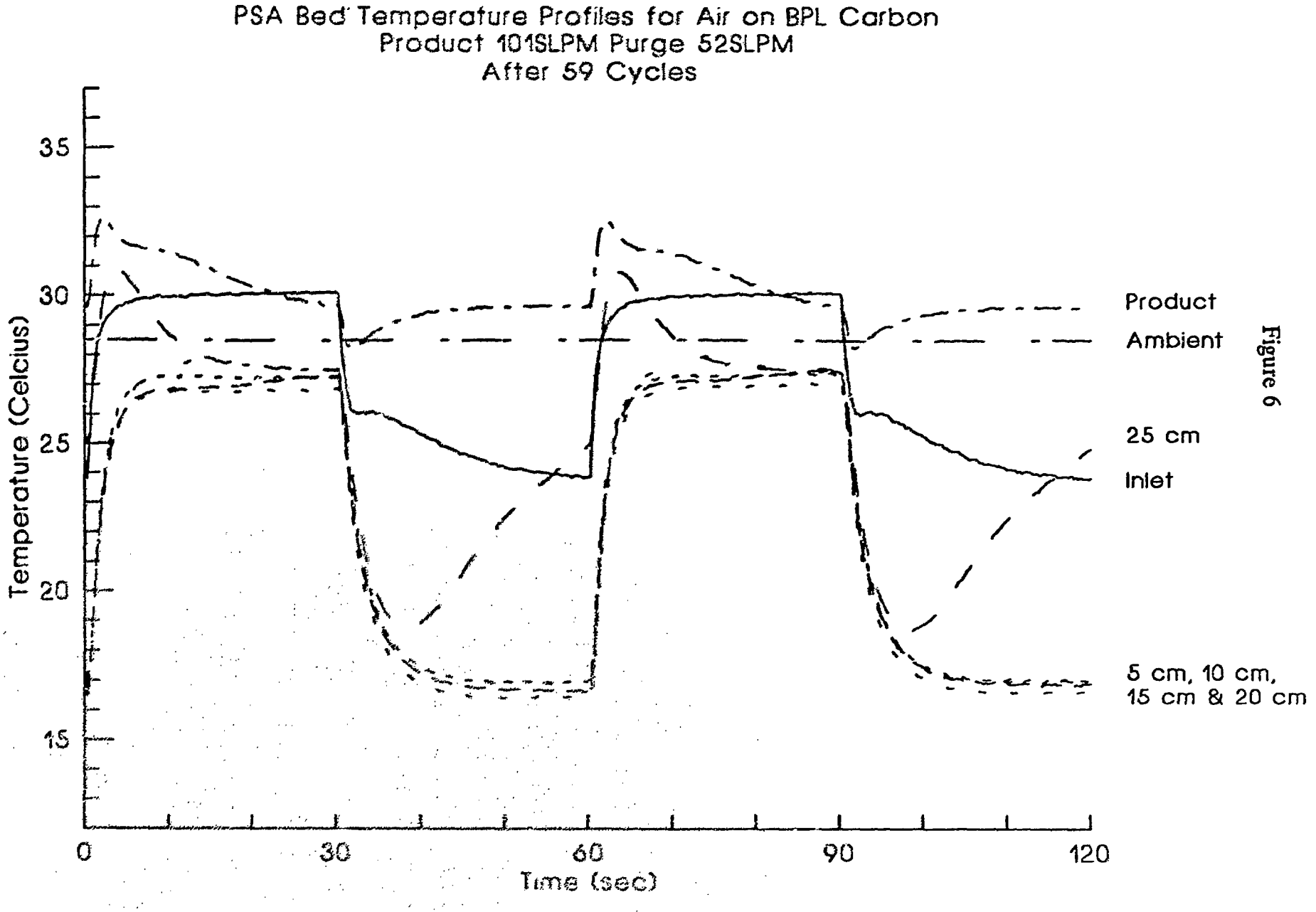

PSA Bed Temperature Profiles for Air on BPL Carbon
Product 101SLPM Purge 52SLPM
After 59 Cycles
Temperature (Celcius)
35
30
25
20
15
0
30
60
90
120
Time (sec)
Product
Ambient
25 cm
Inlet
5 cm, 10 cm,
15 cm & 20 cm

Figure 6

Table 1. Physical Properties of Chemicals

Chemical	Boiling Point	P_{sat} @ 25 °C	C_{sat} @ 25 °C
R-113	47.6 °C	332 torr	3.34×10^6 mg/m^3
R-318	-5.8	2327	2.50×10^7

Table 2. PSA Run Parameters

Run Date	Feed Chemical	Adsorbent	Particle Diameter	Bed Diameter	Bed Length	Feed Flowrate	Half-Cycle Time	Pressure Ratio	Purge/ Feed Velocity Ratio	Ambient Temperature	Feed DewPoint
2/2/92	R-113	BPL	1 mm	5.08 cm	24 cm	153 SLPM	30 sec	3.0	1.5	25 C	-26 C
4/29/92	Air	BPL	1 mm	5.08 cm	25 cm	153 SLPM	30 sec	4.0	1.3	28 C	-26 C

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