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IN-BED MULTIPORT SAMPLING SYSTEM

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Knowledge of the transient behavior in fixed-bed separation processes is required to design advanced air purification systems. Measuring transient behavior experimentally can be difficult because of rapid changes in pressure and temperature in the bed. An apparatus has been developed which simultaneously isolates multiple vapor phase concentration samples at selected axial positions within the bed for subsequent analysis. Results are presented for the calibration and operation of this sampling apparatus using a laboratory pressure swing adsorption system.

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PREFACE

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1. INTRODUCTION

Advanced air purification technologies that are being considered for military applications include pressure swing adsorption (PSA), temperature swing adsorption (TSA), and catalytic oxidation. For each of these processes, it is important to measure data that provides the most design information at the lowest cost. This is especially true when the tests take a long time to complete or are costly in terms of material or labor. In order to validate that a system provides protection over the appropriate range of conditions a combination of validation testing and mathematical modeling must be performed. Some of the important operating parameters to be considered include: feed concentration, flowrate, temperature, bed depth, and pressure. The following paper describes an in-bed sampling technique which eliminates the need to vary the bed depth parameter.

As an adsorption bed or catalytic reactor is challenged with a chemical vapor a concentration front is established. It is the shape of this front that provides the best information about the protection provided by the system. Through the appropriate placement of sample probes along the axial length of the reactor or adsorption bed, the shape of this front and the resulting concentration gradient can be determined in a single experiment. The placement of temperature probes in a PSA adsorption bed is described by Matz el al.(1) However, in-bed concentration measurements are seldom reported. For systems with fast transients such as PSA it is important that in-bed samples be gathered simultaneously in order to provide a profile of the in-bed concentration wave. Cycle times on the order of 5-60 seconds are commonly reported for PSA. In addition it is important that only small samples be used for the analysis in order that the overall flowpath of the bed not be disturbed.

In-bed concentration profiles are important when evaluating an air purification system for its ability to provide protection in a toxic environment. To gather the same information without an in-bed sampling system, duplicate experiments at different bed depths would have to be conducted. However, on large-scale systems it becomes increasingly difficult to hold all conditions constant from experiment to experiment especially temperature, relative humidity, and the packing of the adsorption bed. A second factor to consider is that the rate of sampling must be appropriate for the dynamics of the process. If the measured variables are changing every second and the sampling is limited to hours, then the results are not particularly informative.

The experimental system described here is designed to gather

several gas-phase samples with known volumes for subsequent analysis. A gas chromatograph is chosen as the analytical measuring device. Automation is another important feature of this system. Using computer code and appropriate hardware, in-bed concentrations and temperatures are measured over a period of days and weeks without operator intervention. Also, this sampling system can easily be used on larger size processes.

2. MATERIALS AND METHODS

A schematic of the multi-port sampling system is presented in Figure 1. The present application has a requirement for six sample ports but other configurations are possible. Using this system, six vapor streams can be sampled simultaneously from different locations, then held for sequential analysis using standard chromatographic techniques. There are two principle steps for the sampling system (1) feed, and (2) purge/analyze, which are controlled by the action of the two 12-port valves, SP1 and SP2 (Valco, model E12UWP). Four ports are required for each sample stream to complete the sample collection sequence (sample-in, sample-out, purge, and vent). Two 12-port valves are chosen as opposed to six 4-port valves because of the associated reduction in size and cost of the actuators.

During the sample collection, the 12-port valves direct the flow of the six sample probes within the bed to the sample loop on the corresponding 6-port valve, V1-V6 (Valco, model E6UWP). Using a flow-through stream selector valve, SSV1 (Valco ECSF6P), an unobstructed flow path is established for each sample line to the vent. Samples are collected by opening the sample solenoid valves, S1-S6, and allowing the vapor stream to flow through the sample loop. After an appropriate sampling time these solenoids are closed and the vapor contained within the sample loop is equilibrated to atmospheric pressure. Finally, the samples are isolated in the loops by rotating the 6-port valves, V1-V6. These steps insure that each gas sample is at a known pressure, temperature and volume.

After completing the feed step, each of the 12-port valves is rotated into the purge/analysis position. The choice of the sample to be analyzed is made by the synchronous positioning of the two flow-through stream selector valves, SSV1 and SSV2. A purge and analysis cycle is then conducted sequentially on the selected sample. The selection of either the purge or analysis step is performed using the 6-port valve, V7 (Valco model E6UWP). In the purge step, all transfer lines are capable of being purged clean with carrier gas. This important feature helps provide for a more accurate analysis of the vapor stream concentration by preventing contamination from previous samples. The carrier gas is split into two streams, one for the purge flow and the other for the carrier gas flow to the gas chromatograph. In the purge state, solenoid valve S7 is opened allowing the purge gas to

sweep any entrapped chemical remaining in the analysis flow path. Solenoid S7 is then closed to allow the pressure in the transfer lines to vent to the atmosphere. The clean flow path is flushed with the carrier gas and connected to the column inlet of the GC by rotating of valve V7. Once the detector signal has been stabilized, the contents of the sample loop are injected into this flow stream (by rotating valves V1-V6 back to the sample position) and analyzed.

Chemical analysis is conducted using an Hewlett-Packard, model HP5890 gas chromatograph equipped with a flame ionization detector and 3396A integrator. Computer control is used to automate the in-bed sampling system, GC data acquisition, and the relay board logic (Metrabyte Inc., model ERB-24) required for the proper sequencing of the valve actuators. A custom program is used with an IBM compatible PC using TurboPascal (Borland Inc.). This program controls the event timing, valve operation, GC operation and data logging. The mounting bracket for the sampling valves is constructed from 1/8 inch aluminum sheet. A hinge can be used to mount the sampling system in close proximity to the laboratory PSA, TSA or other filtration system.

3. RESULTS

Care is taken to determine the exact volume of the gas sample loops. The manufacturer of the sample loops provides a nominal volume for each gas sample loop. This value however is not exact. It is of interest to use the gas sampling system such that each of the sample ports indicates the same concentration when sampling a common stream. The sample loop volume determination is accomplished by using a gas calibration standard where the feed, purge and analysis steps are conducted sequentially for the two loop volumes of each sample valve. The resulting area counts are averaged for several sets of runs and normalized against the median value of the large loop.

Using a calibrated gas standard the relative volume of the gas sample loops are determined. Table I lists the area counts, and average for several samples using the six small sample loops 1a-6a and Table II for the large loops 1b-6b. Relative rankings are then established by assigned the loop with the largest area counts, 2b, to be a nominal 5.00 ml. All other volumes are assigned relative to this basis.

Typically it takes 30 minutes to process the six samples collected using this system. The first minute is used to purge the lines and collect the samples. The rest of the time is used to process the samples through the chromatograph.

Figure 2, gives an example of the in-bed sampling system used to monitor in-bed vapor concentration profiles of a high pressure adsorption system. This example represents the constant

challenge of R-113 to a two-bed PSA system with BPL activated carbon. The bed diameter is 5.1 cm and bed length 24 cm. The laboratory system used to conduct this run is described elsewhere(2). The concentration is plotted as a function of time at several locations in the bed. This provides dramatic evidence of the progression of the concentration front through the bed over the course of the experiment. The symbols represent discrete samples processed by the chromatograph. There is an inherent limit of detection of the chromatographic detector such that concentrations below this limit do not result in any response being recorded at the downstream ports until that detection limit is reached.

4. CONCLUSIONS

Details of an automated, analytical system to monitor the vapor concentration at six in-bed sampling ports for a dynamic regenerative filtration system are reported.

This system provides reproducible results with the sampling rate determined primarily by the chromatography.

Table 1.
Loop Volume Determination Small Loops, FID Response (Area)

	Loop 1a	Loop 2a	Loop 3a	Loop 4a	Loop 5a	Loop 6a
Trial 1	1,362,614	1,346,444	1,378,034	1,374,273	1,362,518	1,360,644
Trial 2	1,368,718	1,364,468	1,390,660	1,375,100	1,372,903	1,364,671
Trial 3	1,391,679	1,370,490	1,390,484	1,376,018	1,369,368	1,366,652
Average	1,374,337	1,360,467	1,386,393	1,375,130	1,368,263	1,363,989
Volume Ratio (ml)	0.492	0.487	0.496	0.492	0.490	0.488

Table 2.
Loop Volume Determination Large Loops, FID Response (Area)

	Loop 1b	Loop 2b	Loop 3b	Loop 4b	Loop 5b	Loop 6b
Trial 1	13,857,016	13,997,537	13,861,008	13,976,496	13,966,672	13,998,496
Trial 2	13,896,232	13,981,288	13,840,872	13,967,504	13,962,968	13,996,744
Trial 3	13,910,656	13,975,632	13,857,536	13,974,176	13,984,488	13,983,536
Average	13,887,968	13,984,819	13,853,139	13,972,725	13,971,376	13,992,925
Volume Ratio (ml)	4.97	5.00	4.96	5.00	5.00	5.01

Table 3.

Sampling Event Table

Step No.	Step	Sample/Purge Valve (SP1, SP2)	GC/Purge Valve	Stream Selector Valve (SSV1, SSV2)	Purge Solenoid (S7)	Sample Loops (Hi/Low) 1-6	Sample Solenoid Valves 1-6
1	Power Off/Default	Purge	Purge	NS	OFF	NS	OFF
2	Presample Collection	Sample	-	-	-	UD	-
3	Sample Collection	-	-	-	-	-	ON
4	Depressurization	-	-	-	-	-	OFF
5	Sample Isolation	-	-	-	-	OP	-
6	Loop Analysis Selection	Purge	-	UD	-	-	-
7	Purge	-	-	-	ON	-	-
8	Depressurize	-	-	-	OFF	-	-
9	Connect GC	-	GC	-	-	-	-
10	Inject Analyze	-	-	-	-	OP	-
11	By pass GC	-	Purge	-	-	-	-

OP opposite state
NS non-specific
UD *user defined*
- unchanged from previous state
sequence runs from top to bottom.

1) for remaining sample to analyze, repeat steps 6 through 11
2) for new sample, repeat at step 2

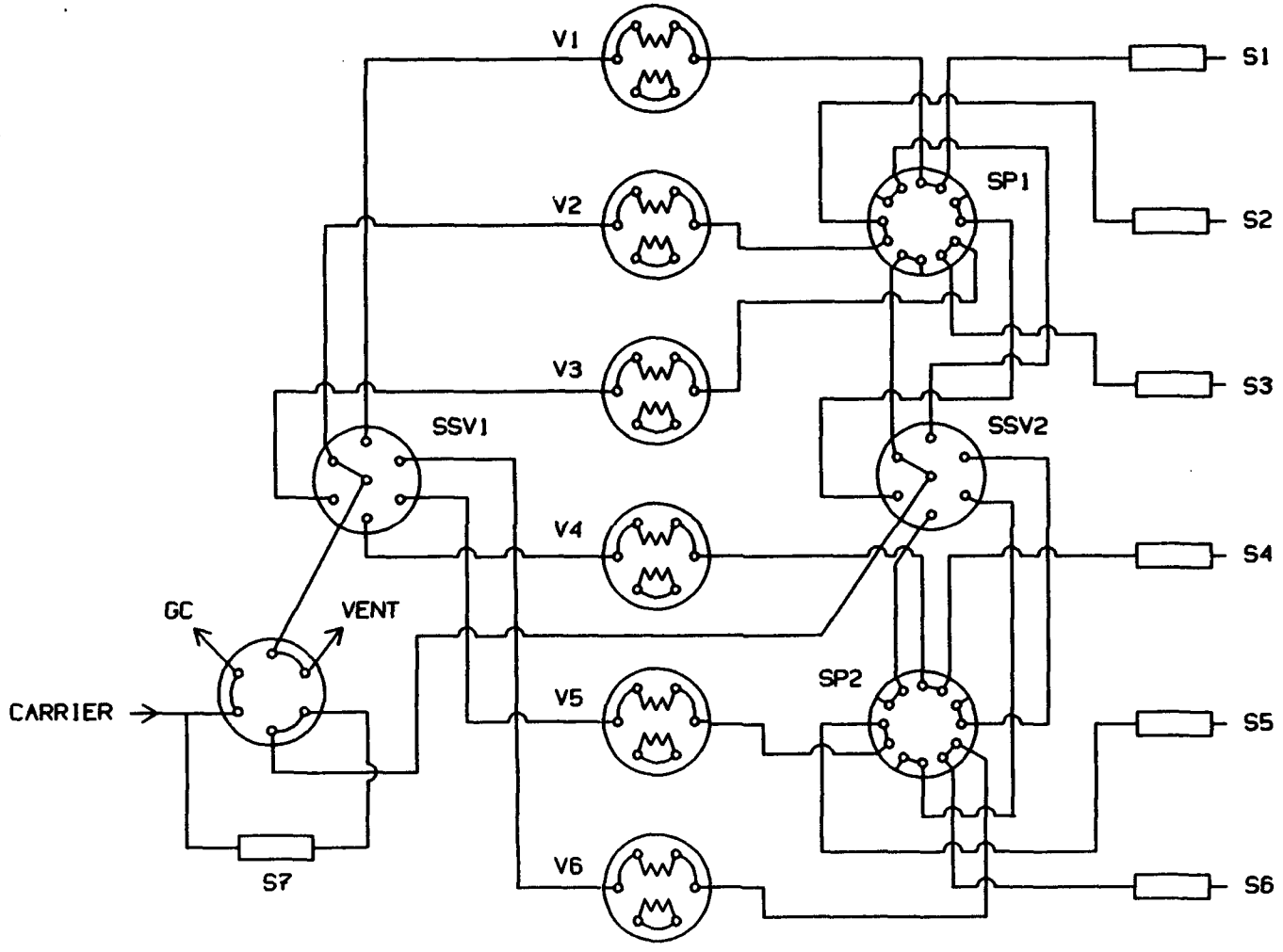

Figure 1. IN-BED SAMPLING SYSTEM

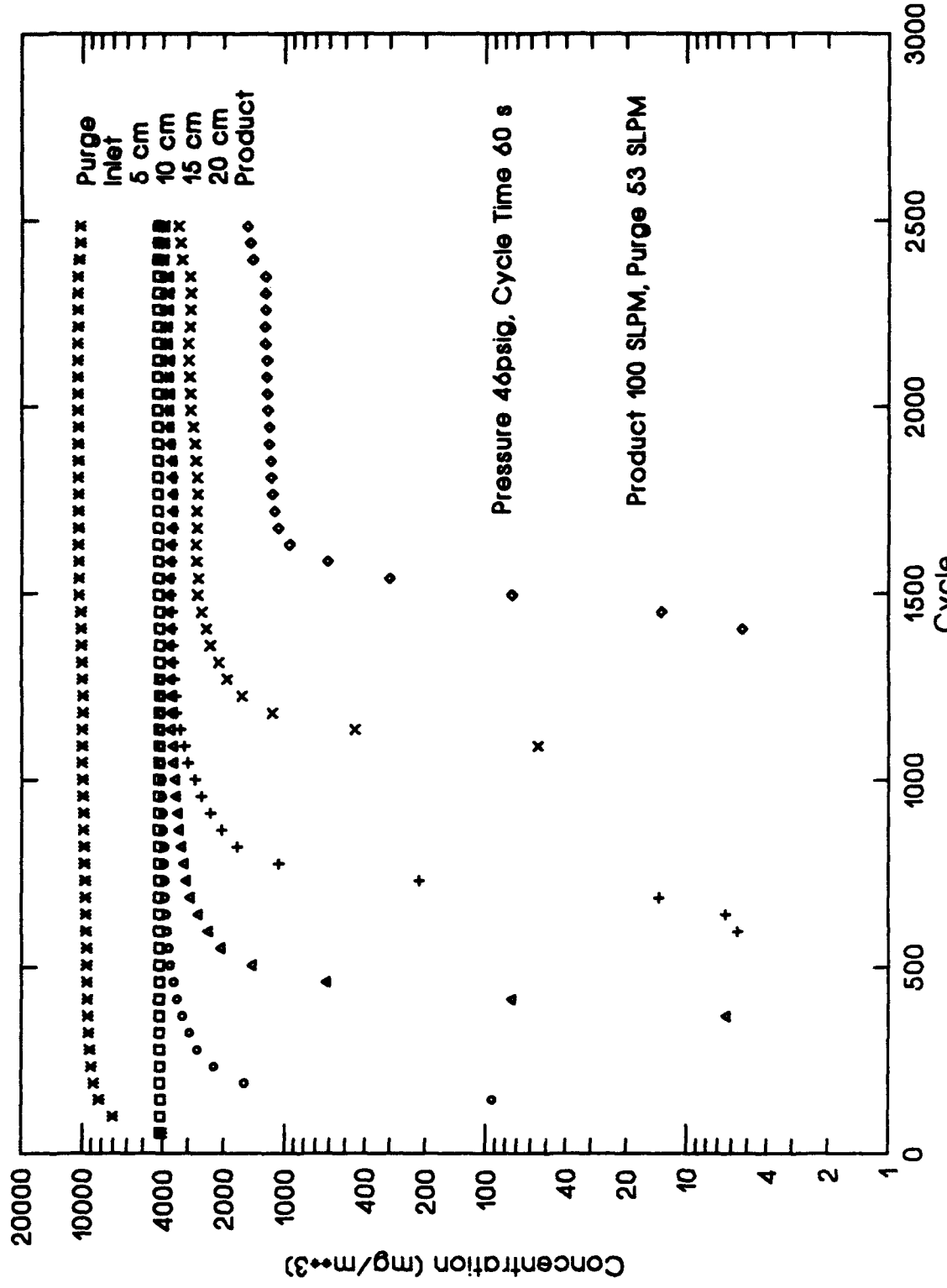

Figure 2. PSA Bed Profile for CFC113 on BPL - 3 Mar 92

5. LITERATURE CITED

1. Matz, M.J. and K.S. Knaebel, "Temperature Front Sensing for Feed Step Control in Pressure Swing Adsorption", I&EC Res., 26, 1638, 1987.

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