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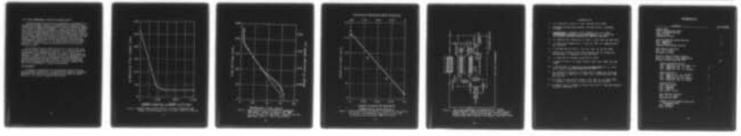

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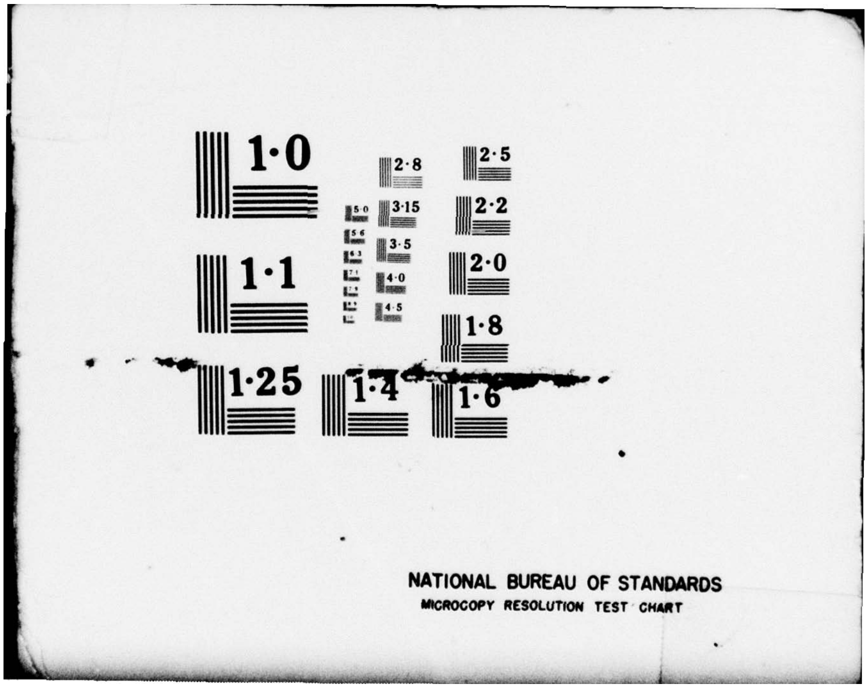
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ARCSL-CR-77043

RESIDUAL PROTECTIVE LIFE OF CARBON BEDS

Fourth Quarterly Progress Report

1 Dec. 76 - 28 Feb. 77

by

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Weakly sorbed probe gases, ethane and carbon dioxide, were used as a measure of the residual sorption capacity for dimethylmethyl phosphonate (DMMP) on ASC carbon. The reduced retention time of ethane decreased with increasing loading up to 0.1 gm DMMP/gm charcoal; above this level it was constant. In a dynamic sorption experiment, in which the bed loading was inhomogeneous lengthwise, ethane retention time decreased as the bed retained increasingly larger amounts of DMMP cont'd

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CONT

To follow the movement of zone of DMMP through BPL charcoal, partially loaded beds were prepared by coupling in series two sections; the inlet section presaturated with DMMP and the outlet-free of DMMP. The movement of DMMP was monitored by the change in weight of each section and in retention time of carbon dioxide through each section. The retention time decreased from 4.3 min for completely clean charcoal to 0.3 min for saturated charcoal.

Water also affected the retention time of ethane on an ASC carbon bed. Exposure to a feed stream at 18 mm Hg of water pressure resulted in decrease of the reduced retention time from 484 before the exposure to 31 at a loading of 0.24 gm H_2O/gm charcoal.

PREFACE

The work described in this report was authorized under Contract No. DAAA 15-76-C-0072, "Residual Protective Life of Carbon Beds". The work described covers the period of December 1, 1976 to February 28, 1977.

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RESIDUAL PROTECTIVE LIFE OF CARBON BEDS

A METHOD TO DETERMINE THE RESIDUAL CAPACITY OF AN ADSORBER

THE CHROMATOGRAPHIC RETENTION TIME OF A PROBE GAS MEASURES THE FRACTION OF UNUSED ADSORPTIVE CAPACITY.

I INTRODUCTION

The determination of the residual capacity of an adsorber, such as an air purification filter, is an often encountered problem. This problem is particularly difficult when the feed to the adsorber contains a variable or unknown concentration of adsorbable material. Described below is a nondestructive, in situ method in which the flow of the adsorber feed is not interrupted.

The method treats an adsorber as a gas chromatography. A pulse of a probe gas is injected into the feed stream at the inlet end of the adsorber bed and the time for the pulse to traverse the length of the bed is measured. At the outlet end, the emergence of the probe-gas pulse can be detected either by changes in thermal conductivity of the gas stream or by other more sensitive detectors commonly used in gas chromatography (1). The most convenient measure of the probe gas transit time is the chromatographic retention time, the time from injection of the pulse to the time of the maximum concentration of the emergent peak. By proper selection of the probe gas, its retention time will vary with the bed loading. One method of selection, the one we used, was to choose probe gases which have a large value of the retention time per unit volume of adsorbent that is free of adsorbate relative to the gas retention time on adsorbent saturated with adsorbate. The retention time of such a probe gas will be primarily its retention time on the portion of the bed that is free of adsorbate. Thus, retention time becomes a measure of the residual capacity of the adsorber.

II EXPERIMENTAL APPARATUS AND MATERIALS

Commercial chromatographs (F and M models 720 and 810, Avondale, PA and Barber-Colman model 5340, Rockford, IL) with dual columns and thermal conductivity detectors were used in an isothermal mode. Gas-tight hypodermic syringes (inexpensive plastic from Becton, Dickinson and Company, Rutherford, NJ, and Teflon^R-sealed glass from Hamilton Co., Reno, NV) were used for injection of 0.01 to 1cc samples of probe gas. The columns of charcoal, ranging from 1 to 15 inches in length, were contained in either 1/4 inch copper (i.d. 0.170 in.) or stainless steel (i.d. 0.220 in.) tubing with glass wool plugs. The columns were operated at atmospheric pressure and had less than a 10 torr pressure drop. The charcoals were obtained from Pittsburgh Activated Carbon, Division of Calgon Corporation, Merck & Co., Inc., Pittsburgh, PA 15230, and were of two types (2): BPL, lot 7542, an impregnation-free material with a surface area of 1100 m^2/g and pores mainly between 1.5 and 2.0 nm and ASC, lot 3-339, a material made by impregnation of BPL carbon with various metal salts, resulting in a surface area of 850 m^2/g. Both charcoals were composed of irregularly shaped, approximately one mm particles with a size distribution between 12 and 30, U.S. Sieve Series. Dimethyl methylphosphonate (DMMP) Fyrrol^R grade, was obtained from Stauffer Chemical Co., Westport, CT, and was used without purification. According to the manufacturer it has a purity above 97%; chemical ionization mass spectroscopy and gas chromatography did not reveal any large impurity. Various grades of gases from technical to reagent were used without purification.

III EXPERIMENTAL RESULTS

Retention time is reported both directly and as a reduced dimensionless parameter, Θ, retention time divided by space time. The reduced retention time Θ is also equal to the retention volume divided by the volume of the charcoal bed. (The retention volume is the product of retention time and volumetric flow rate; the space time is the volume of the bed divided by the volumetric flow rate.) The use of Θ is based on "equilibrium" chromatographic theory (3) which shows this parameter to be independent of carrier flow rate and bed size.

Figure 1 shows the reduced retention time of 0.05cc ethane for a series of ASC charcoal columns with different levels of DMMP loading. The columns were homogeneously loaded by passing a feed stock of 5μg DMMP/cc He into the column at an appropriate constant temperature until the column came to constant weight. The reduced retention time decreased with increasing loading up to 0.1g DMMP/g charcoal; above this loading level it was constant.

Figure 2 describes the results of a typical dynamic sorption experiment in which the bed loading was inhomogeneous, lengthwise. The retention time of 0.4cc ethane is shown as a function of time for an 0.5g ASC carbon bed, initially free of DMMP, with a constant feed of 5μg DMMP/cc He. The lower abscissa shows the estimated loading based on complete sorption of the feed DMMP; saturation loading occurred at about 0.2g DMMP. The ethane retention time decreases as the column retains increasingly larger amounts of DMMP.

DMMP is adsorbed on charcoal rapidly. In contrast, desorption is slow. The adsorption experiment described in Figure 2 took less than four hours using a total of 48 liters of feed gas to achieve 0.4g DMMP/g charcoal; to remove enough DMMP to reach 0.3g DMMP/g charcoal took 1740 liters of DMMP-free He feed over a period of 140 hours.

Figure 3 shows the retention time of 0.25cc carbon dioxide for BPL charcoal beds of constant length as a function of the fraction of the length of the bed presaturated with DMMP. Each partially loaded bed was prepared by coupling together in series two sections, the inlet section presaturated with DMMP and the outlet section free of DMMP. The retention time decreases from 4.3 min for unit value of the fraction residual capacity (completely clean charcoal) to 0.3 min for zero value (completely saturated charcoal).

Within the range of experimental variables of superficial linear gas velocity and cross-sectional areas of the beds, the parameter Θ was essentially invariant for a probe gas at a given temperature. On an ASC bed at 24°C with ethane for six measurements ranging from 5 to 27 cm/sec, the range of Θ was 312 to 329. For four measurements with propane at 44°C from 2.4 to 21 cm/sec, the range was 286 to 328. For ethane at 44°C, with a bed of 0.16 cm^2 cross-section and superficial linear velocity of 9.5 cm/sec and a bed of 0.22 cm^2 and 9.0 cm/sec the values of Θ were 484 and 490 respectively.

The reduced retention time, Θ, is significantly affected by temperature. The temperature dependence can be expressed in terms of an energy Q obtained by plotting ln Θ vs. 1/T, to fit a Van't Hoff type of relationship $d \ln\Theta/dT = Q/RT^2$, where R is the gas constant and T the absolute temperature. On DMMP-free ASC charcoal the value of Q for propane was 11 kcal/mole; and on DMMP-free BPL charcoal 5 for methane, 8 for ethylene, 7 for ethane and 9 for acetylene.

At a fixed temperature, the ratio of the retention times on DMMP-free to DMMP-saturated charcoal can be very large. For example, at room temperature on 2.0g BPL beds at a superficial velocity of 2.3 cm/sec, the ratio of the actual retention times (min/min) were found to be 1.2/0.4 for air, 1.2/0.4 for methane, 10.8/0.8 for CO_2, and >15/2.2 for acetylene. The retention times on the saturated columns were determined by injecting the probe gas into a helium feed saturated in DMMP.

Water, the other polar adsorbate besides DMMP which was investigated, also affects the retention time. On an ASC bed with ethane as the probe gas, for as-received charcoal (manufacturer lists 2% water as typical) Θ was 484. On drying for 1 hour at 150°C with a 4 wt % loss, Θ increased to 570. Exposure to a feed stream containing 18 torr of water until constant retention time gave a Θ value of 31 at a loading of 0.24g H_2O/g charcoal.

IV DISCUSSION OF EXPERIMENTAL RESULTS

That the retention time of a pulse of a probe gas is a useful measure of the residual capacity of an adsorber is shown by Figures 1-3. Figure 1 shows that the retention time decreases as the ratio of adsorbate to adsorbent increases for a homogeneously loaded bed. The asymptotic behavior of the retention time at high loading suggests that less than saturation amounts of DMMP effectively block all the charcoal sites used by the probe gas. Complete blocking may be expected below DMMP saturation (0.4g/g charcoal) since saturation, for many adsorbates (4) below the critical temperature, occurs by filling of capillaries (80% lie between 1.5 and 2.0nm) with liquid-like adsorbate to the extent of about 0.3 to 0.4cc/g charcoal.

Figure 2 describes in situ results for a typical situation during the removal of an impurity (0.1 mole per cent) from a flowing feed stream by adsorption. From the sharp "breakthrough" curves (5) (concentration in the effluent as a function time for a bed with a constant feed) it is known that a sharp concentration profile moves down the bed, i.e., the length of the bed saturated with adsorbate from the inlet end increases with time.

Figure 3 shows that the retention time decreases for increasingly longer sections of a bed pre-saturated with DMMP. There is a linear relationship between residual capacity and retention time.

The flow rate and bed diameter studies show that factors other than adsorption equilibrium, such as diffusion to or within pores of the charcoal, do not dominate the retention time. Such a result is to be expected since chromatographic theory (6) suggests that the center of gravity of the chromatographic peak, the "first absolute moment", is only dependent on equilibrium adsorption properties. For a symmetrical chromatographic peak, this moment is equal to the chromatographic retention time. For our system with "tailings", the first-moment time is somewhat longer. For calibration purposes as a function of flow rate and bed size the dimensionless parameter, the reduced time, Θ is constant to good approximation.

The temperature dependence of the retention time, which is associated with the heat of sorption of the probe gas and typically 3 to 5% per °C, is an important factor in any calibration. The size of the probe gas injected is not a critical variable; typically, a five fold increase in size, decreased the retention time about 20%. The elution order in some cases changes with temperature, two elution sequences reported (7) on Carbosieve-B, a chromatographic grade charcoal of $1000m^2/g$ surface area with 1.2 to 1.5nm diameter pores, were the same as we found on the Pittsburgh charcoals, i.e., air<methane<carbon dioxide<ethane and methane<acetylene<ethylene<ethane.

The two polar adsorbates we studied are quite different. DMMP obeys a Dubin-Polanyi isotherm (8); even at a partial pressure of 10^{-11} times the vapor pressure of the liquid, the amount adsorbed is 50% of the saturation capacity of the adsorbent. In contrast, water follows an "S" shaped isotherm (*) with the steeply rising portion at about 50% relative humidity.

V SELECTION OF PROBE GASES FOR OTHER SYSTEMS

The substance used as the probe gas should be reversibly adsorbed and should be of a molecular size to have access to the same size pores as the adsorbate in the feed stream.

The selection of appropriate probe gases may be guided either by the extensive literature on gas chromatography (GC) for chemical analysis purposes or by adsorption isotherm data, since the retention time increases as dw/dp increases where w is the amount adsorbed at a partial pressure p. Chemical separation by GC is based on differences in retention times for

* L.A. Jonas, private communication.

chemical substances on one adsorbent; our method is based on the change in retention time of one substance (probe gas) as an adsorbent changes its properties with loading by an adsorbate from a feed stream. Loading has been widely used in GC to obtain desired differences in retention times among substances. "Activation" by removal of volatile material, e.g., H_2O, is widely practised in gas solid chromatography (GSC) (9). Additions of traces to eliminate dissymmetry in the shape of effluent peaks with tailings in GSC has been discussed (10). Gas liquid chromatography (GLC) is based on an inert solid covered by an active liquid phase in which the probe gas is soluble, and the amount of liquid determines the retention time. Modified GSC exploits low loading levels of a liquid phase to adjust the properties of the solid adsorbent (11).

Experimental screening of probe gases is easily carried out using clean and saturated adsorbents as columns in a GC. Scaling to other size adsorbers can be based on the invariance of the reduced time, Θ.

VI LARGE SCALE ADSORBERS

The determination of the residual capacity of large adsorbers by the retention time method may be carried out in two ways. Direct determination of the retention time of a probe gas may be done on the large-scale adsorber itself by injecting a probe gas pulse into the feed stream and monitoring the effluent stream for the emergence of the probe gas. An alternative method for the determination of the retention time involves the use of an off-the-shelf dual column gas chromatograph in parallel with the large-scale adsorber. As shown in Figure 4, a small slip-stream of the main feed stream is split equally through identical columns A and B of the chromatograph. Columns A and B, packed with the adsorbent, are operated at the same space time as the large-scale adsorber for the purpose of duplicating the fractional residual capacity of the large-scale adsorber. The lengths of the columns can be made the same as the adsorber for simulation of the linear gas velocity. The differential detector balances the signals from columns A and B if their outputs are identical, indicates a signal if they are not. Thus, the retention time of a probe gas, measured by injection into one of the columns, will be a determination of the fractional residual capacity of the large-scale adsorber.

While a chromatograph consisting of a single column with a detector can be used for the retention time, the differential-signal configuration, shown in Figure 4 with the matched-response detectors for each column, has the advantage of eliminating signals other than that from the probe gas. If the probe gas has an inconvenient retention time at the space time of the large-scale adsorber, the flow rate of the slip stream feeding the chromatograph can be appropriately adjusted for the length of time needed for measurement of the retention time. Selection of the appropriate flow rate can be based on the invariance of the product of the retention time and flow rate.

VIII OTHER CHROMATOGRAPHIC METHODS FOR RESIDUAL CAPACITY

Although we have not carried out any experimental investigations, in principle, the residual capacity of an adsorber can be measured by other methods which are based on some property of a pulse of a probe gas changing as a function of loading of an adsorber. A minor variation of our method might consist of selecting a probe gas with appreciably greater retention time on the saturated adsorbent than on the bare adsorbent, the inverse of our selection. Another possibility is the use of a two-component probe gas pulse and measurement of the difference of retention times of the two components. A third possibility involves the fractional conversion of a heterogeneously catalyzed reaction; an exploitation of the Kokes-Emmett micro-reactor technique (12). In this method, the adsorbate is either a poison for a reaction catalyzed by the bare surface or is the seat of catalytic activity.

We are aware of only one report of the use of a pulse of gas to determine the residual capacity of an adsorber. It is based on the change in dispersion of a chromatographic peak with loading. Stamulis and Thompson (13) showed that the dispersion of a pulse of CO_2 decreased with loading of a charcoal filter. Retention time has less dependence on mass transport by diffusion and rates of adsorption and more dependence on equilibrium adsorption than does dispersion. The ease of experimental determination of retention time and the availability of retention time data in the chromatographic literature are advantages.

ACKNOWLEDGEMENT

D. Sartorelli, supported by an NSF Undergraduate Research Program, made experimental contributions. We appreciate the support of the Defense Department under Contract No. EC-CR 77031 and advice from the Project Monitor, Dr. L.A. Jonas of Edgewood Proving Ground.

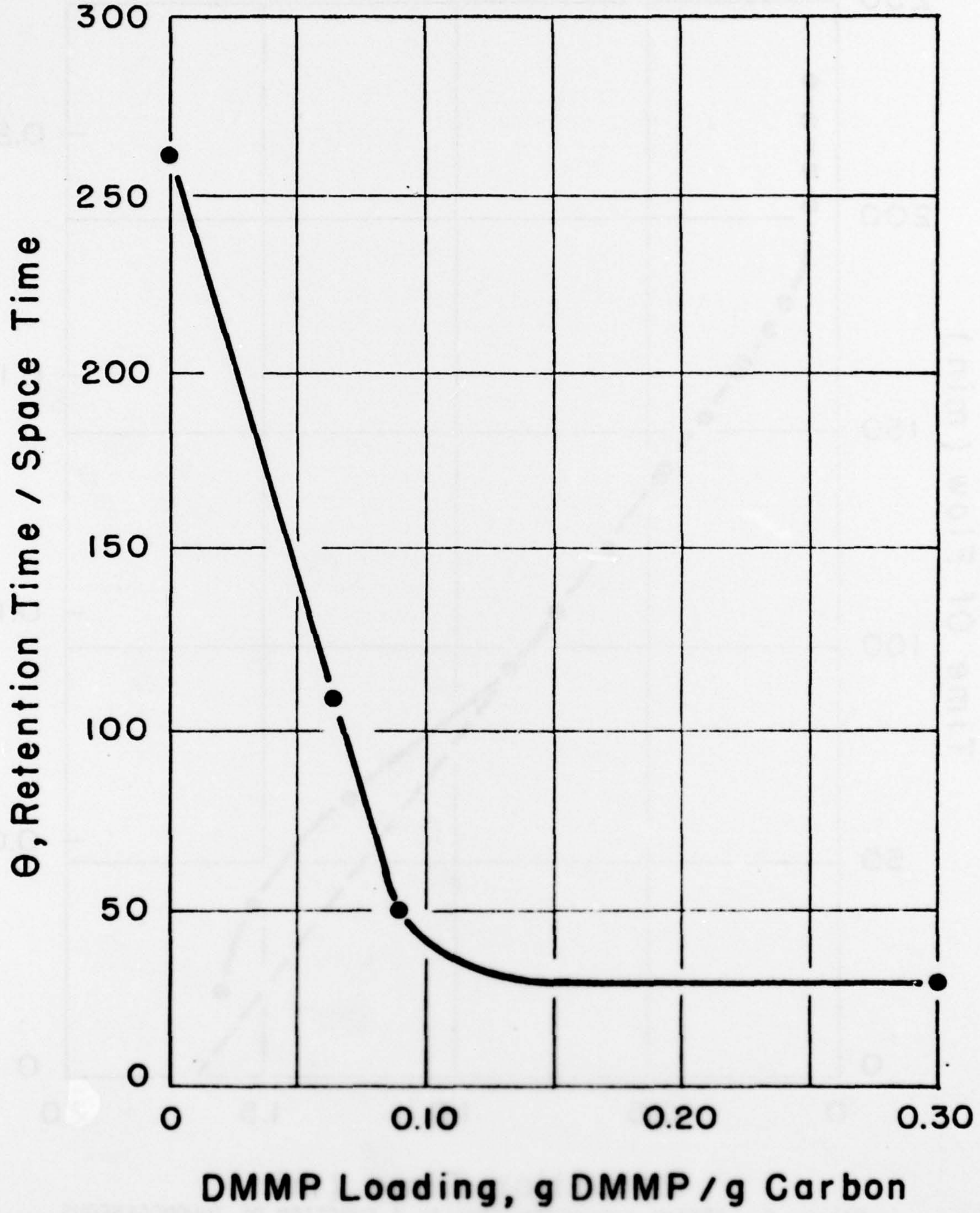
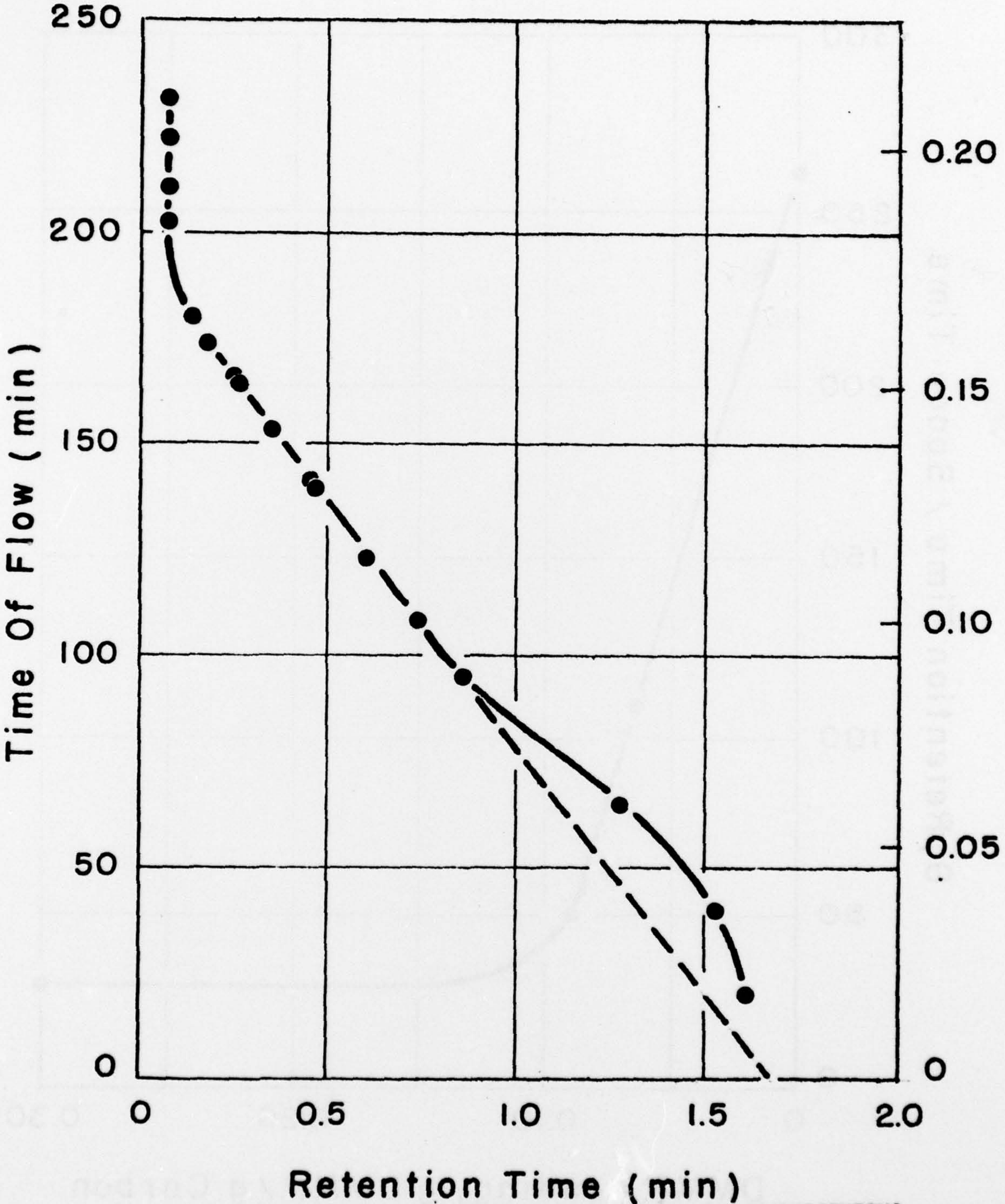


Figure 1: REDUCED ETHANE RETENTIME TIME AS A FUNCTION OF HOMOGENEOUS DMMP LOADING. 0.4g ASC charcoal, 28°C, 9.5 cm/sec superficial velocity.



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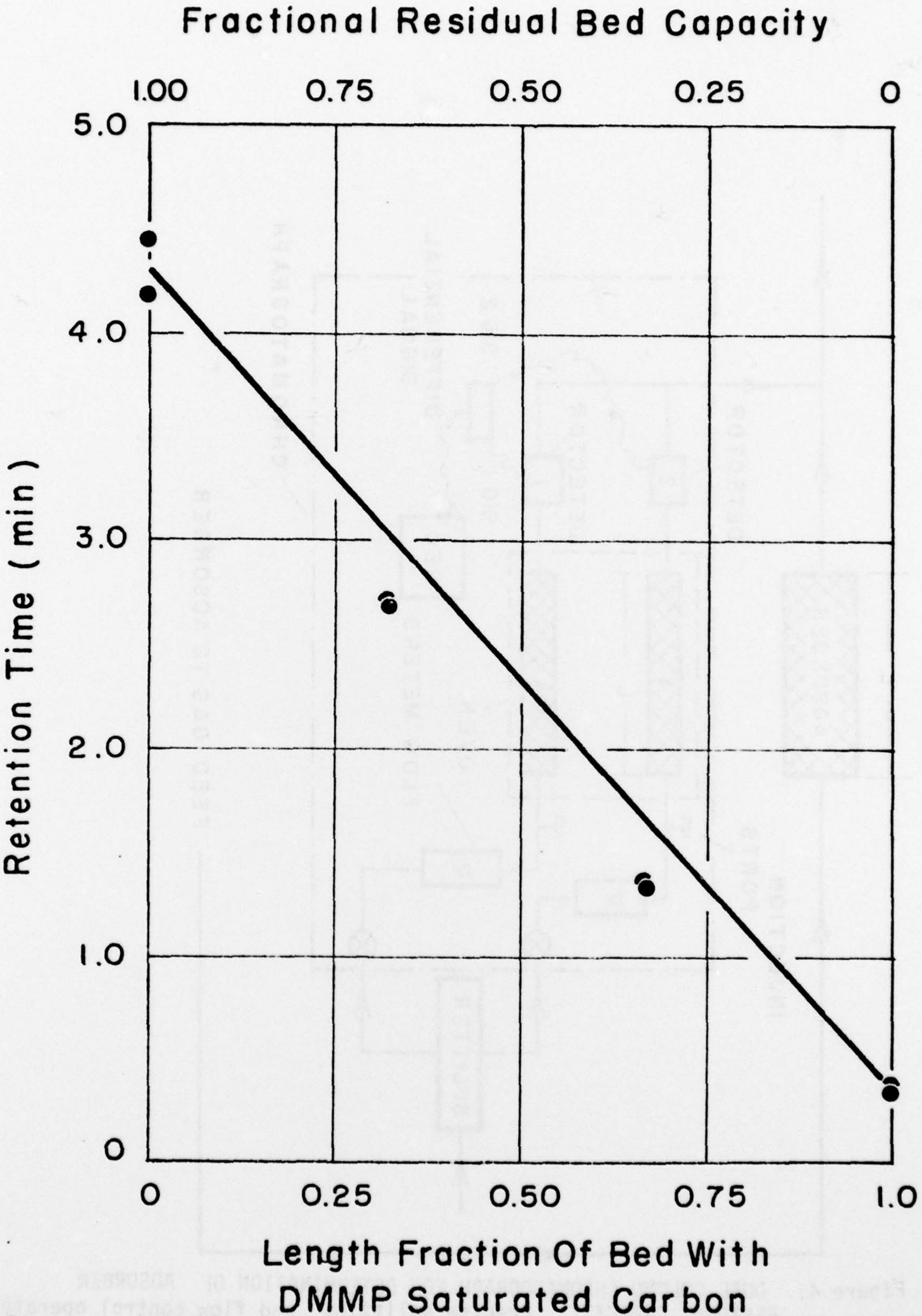

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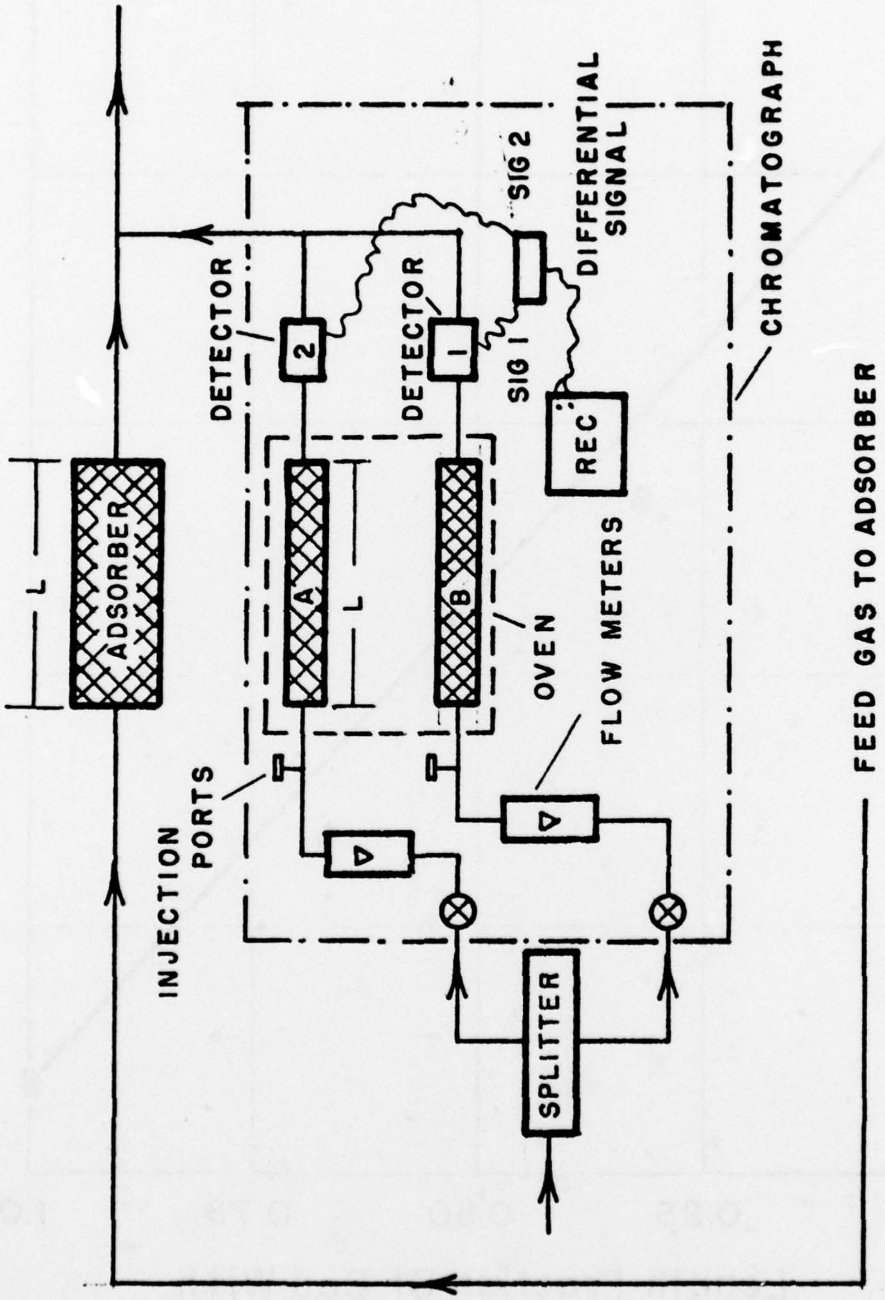

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