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ECBC-TR-052

RECIRCULATION FILTER UNIT FOR THE M28 SIMPLIFIED COLLECTIVE PROTECTION SYSTEMS: ADSORPTION EQUILIBRIA OF CANDIDATE NONWOVEN MICROPOROUS MEDIA

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Adsorption equilibria of nitrogen, hexane, and DMMP are provided on two nonwoven adsorbent materials recommended for use in the recirculation filter unit (RFU) for the M28 Collective Protection System. The objective of this report is to correlate adsorption equilibria of the three adsorbates and to provide a model to predict the adsorption capacity of the media for toxic chemical agents over an extended range of vapor concentrations. This information may be used to select the best adsorbent materials for the RFU as well as to establish production acceptance criteria consistent with field use requirements. Adsorption equilibria of nitrogen, hexane, and DMMP on POLYSORB (PS) and Purification Products (PP) adsorbent materials show that there is much less adsorption capacity per unit packing volume than typical granular bed activated carbons such as BPL. A Dubinin-Radushkevich (DR) potential plot of the combined nitrogen, hexane, and DMMP data shows that the total estimated micropore volume of the PS and PP adsorbent materials is 0.113 and 0.077 mL/g of material, respectively. On a volume basis, the PP material may provide about 2.8 times more adsorptive capacity than the PS material. The estimated adsorbed phase loading of HD mustard at 0.029 mg/m^3 at 25 °C is about 0.035 and 0.015 mL/g of material for the PS and PP materials, respectively.

14. SUBJECT TERMS

Adsorption equilibria	Recirculation filter unit	M28
DMMP	Activated carbon	Nitrogen
Hexane	Nonwoven microporous Adsorbents	

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PREFACE

The work described in this report was authorized under Sales Order No. 9CEFD4. This work was started in June 1998 and completed in May 1999.

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Acknowledgments

Joseph Rehrmann is greatly appreciated for providing the nitrogen adsorption equilibria.

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RECIRCULATION FILTER UNIT FOR THE M28 SIMPLIFIED COLLECTIVE PROTECTION SYSTEMS: ADSORPTION EQUILIBRIA OF NONWOVEN MICROPOROUS MEDIA

1. INTRODUCTION

There is a need to provide filtration of agent vapors that may be introduced during entry/exit operations within shelters that provide collective protection against chemical warfare agent (CWA) vapors.[1,2] Recirculation filter units (RFU) have been identified as an effective technology to reduce toxic chemical vapors to acceptable levels.[3] Unlike the robust performance features of single-pass NBC filters, an RFU provides a means to scavenge CWA vapors at a cost and complexity that is significantly lower than traditional NBC filter systems.

The proposed RFU contains adsorbent media that removes chemical vapors by physical adsorption. It functions by recirculating contaminated air through an adsorbent bed and discharging the filtered air back into the shelter. To reduce power consumption of such a unit, a low airflow-resistant medium is used. Candidate adsorbent materials are nonwoven polymers containing activated carbon. These materials typically contain less adsorbent per unit volume than granular packed bed filters. The reduced packing density produces a greater void volume, thus enabling the use of higher superficial airflow velocities at a reduced pressure drop. For an RFU to be effective in contaminant control, the system must remove an amount of chemical that is at least as great as the rate at which it is introduced into the shelter. Because of the very low agent concentrations that can exist in shelters, high recirculation flows are needed to remove the mass of agent typically released from personnel and equipment entering the shelter. One advantage of such a system is that it can operate at lower efficiencies than standard NBC filters and still maintain effective contaminant control.

In any adsorptive process, the removal of contaminant vapors is affected by the rate of mass transfer (external and internal) of chemical to the micropores of the adsorbent and the capacity of the adsorbent to retain a given amount of adsorbed chemical. A fundamental aspect of an adsorption process is to understand the capacity of the filter for a given chemical vapor over a range of anticipated vapor concentrations. Adsorption equilibria provide the necessary information to determine the ratio of adsorbed chemical per unit amount of adsorbent for a given vapor concentration and operating temperature. This information is critical to the initial selection of candidate adsorbents and for sizing of the filter system with respect to the amount of chemical to be removed from the air.

In this report, adsorption equilibria of nitrogen, hexane, and dimethyl methylphosphonate (DMMP) are provided on two nonwoven adsorbent media recommended for use in the RFU. The objective is to correlate the adsorption equilibria of the three adsorbates and to provide a model to predict the adsorption capacity of the media for toxic chemical agents over an extended range of vapor concentrations. This information may be used to select the best adsorbent materials that meet the chemical capacity requirements of the RFU.

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[1] Blewett, W. K., Testing of Entry/Exit Procedures for the XM20 Simplified Collective Protection Equipment (SCPE), CRDC-TR-85008, U.S. Army Chemical Research and Development Center, Aberdeen Proving Ground, MD, August 1985, UNCLASSIFIED Report (AD-B095 495).

[2] Smith, J.M., and Moyer, R.H., Adsorption/Desorption of Agent and Simulant Vapors by Clothing, CRDEC-CR-88046, U.S. Army Chemical Research, Development and Engineering Center, Aberdeen Proving Ground, MD, March 1988, UNCLASSIFIED Report (AD-B121 911).

[3] Rok, C.A., Recirculation Filters for Collective Protection Systems, CRDEC-TR-019, U.S. Army Chemical Research, Development and Engineering Center, Aberdeen Proving Ground, MD, December 1988, UNCLASSIFIED Report (AD-B129 690).

These adsorption equilibria are important in establishing production acceptance criteria consistent with field use requirements. Performance of production acceptance at field use conditions is not practical because of the extremely long service life that would exist at field use concentrations (0.029 mg/m^3 for HD). The approach being taken is to conduct production acceptance testing at a relatively high challenge concentration that would result in a reasonable breakthrough time. The isotherm data would then be used to relate the filter capacity requirement under field use to the filter capacity at the production acceptance conditions. Further evaluations involving breakthrough testing are needed to assess the performance of the adsorbent media in actual operation. The latter effort would include information on mass transfer and adsorption capacity of selected adsorbents.

2. EXPERIMENTAL PROCEDURES

2.1 Adsorbent Material.

Two adsorbent materials were evaluated in this study. The first, referred to as PS, is a grade of POLYSORB material produced by Columbus Industries, Incorporated, Ashville, OH. The adsorbent product contains 25% by weight activated carbon supported on a nonwoven polyester fiber, which is coated with an acrylic latex polymer. This grade of material has an overall thickness of 1.58 cm. The second material evaluated, referred to as PP, is produced by Purification Products, Limited, Shipley, West Yorkshire, England. This grade of material also contains a nonwoven support of approximately 0.25 cm thickness. The Table reports the effective amount of activated carbon of PS and PP with a comparison to BPL granular carbon.

Table. Adsorbent Material Loading Properties

Adsorbent	Area Density mg-material/cm^2	Volume Density mg-material/mL	Carbon Loading mg/mg-material	Bulk Density mg-carbon/mL
P-S	53.5	33.8	0.25	8.4
P-P	35.3	138.9	unknown	----
BPL*	------		----	495.0
*Granular carbon, 12x30 mesh				

2.2 Adsorbates.

One design criteria of the RFU is to maintain a vapor concentration in a shelter below 0.029 mg/m^3 for chemicals such as HD mustard (bis-2-chloroethylsulfide). Therefore, it is necessary to determine the adsorbed phase loading that would produce this vapor concentration at normal ambient temperatures. The loading information can be used to determine the total capacity of adsorbent material needed to remove a given amount of vapor over the operational life of the filter.

In this investigation, nitrogen, hexane, and DMMP were used as reference adsorbates for the initial characterization of the PS and PP materials. Measured data were converted to relative pressures for comparing adsorbates with different vapor pressures and molecular weights. Nitrogen, the smallest molecule of three adsorbates, was used as a reference for comparing standard grade activated carbon adsorbents. Nitrogen is often used as a probe to estimate the surface area and pore-size distribution of adsorbents.

Hexane (Aldrich, 99+%) and DMMP (Aldrich, 97%) were used to provide a range of physical properties accounting for steric and polar effects. Hexane is a nonpolar, straight-chain hydrocarbon that is more weakly adsorbed on activated carbon than DMMP. The DMMP is a branched molecule that is highly polar and is often used as a reference simulant for GB and other strongly adsorbed chemical agents (e.g., HD mustard).

2.3 Apparatus.

Three different adsorption equilibrium apparatuses were used to characterize the ratio of vapor concentration to adsorbed phase loading. Nitrogen adsorption equilibria at 77 K were measured on a volumetric/manometric apparatus (Autosorb-1C) produced by Quantachrome Corporation (Boynton Beach, FL). The system is capable of measuring adsorption isotherms for high vapor pressure chemicals at relative pressures from 1E-6 to 1. Measured data were applied to several standard models to estimate the surface area and pore size distribution of PS and PP.

The DMMP and hexane adsorption equilibria were measured on gravimetric and volumetric systems designed at this laboratory.* Each system provides a detection sensitivity suitable for a particular concentration range. The gravimetric system (modified Cahn 1000) was used for concentration measurements of about 0.1 g/g loading and higher, while the volumetric (Ballast/Dynatherm) was used for lower concentration measurements.

3. RESULTS AND DISCUSSION

Adsorption equilibria of nitrogen, hexane, and DMMP on the PS and PP adsorbent materials are presented in Figures 1 and 2. Analysis of the nitrogen data shows that the PS and PP adsorbent materials have a micropore volume (DR method) of 0.153 and 0.062 mL/g of material, respectively. These values equate to a surface area (BET method) of 325 m^2/g of material for the PS, and 130 m^2/g of material for the PP materials. Additional information on properties estimated from the nitrogen adsorption equilibria are provided in the Appendix. When corrected for the weight-fraction of actual carbon present (25% by weight carbon on PS), a micropore volume of about 0.6 mL/g of carbon and a surface area of 1300 m^2/g of carbon are obtained. By comparison, the micropore volume of granular activated carbon is about 0.4 to 0.7 mL/g of carbon, indicating that the carbon contained on the PS and PP materials is typical of highly activated carbons. The PS and PP surface area and micropore volume data indicate that the effective amount of carbon per bed volume is significantly less than a bed containing granular activated carbon (e.g., BPL activated carbon). This is determined by comparing a unit volume of the nonwoven materials to an equivalent volume of BPL granular carbon. The PS material provides about 8.4 mg of carbon/milliliter of material in comparison to 495 mg of carbon/millimeter of BPL as bulk density (see the table). Based on nitrogen micropore volume data, it appears that the carbon loading of the PP material is about 40% of the PS material (0.062-PP/0.153-PS mL/g of material). However, because the volume density of the PP material is about four times greater than that of the PS material (see the table), the total micropore volume based on nitrogen is about 66% higher. This is

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*Buettner, L.C., Mahle J.J., Buchanan J. H., and Friday D. K., <u>Adsorption Equilibria of Strongly Adsorbed Vapors Measured Using an Automated Gravimetric Apparatus</u>, U. S. Army Edgewood Research, Development and Engineering Center, Aberdeen Proving Ground, MD, unpublished data, March 1999.

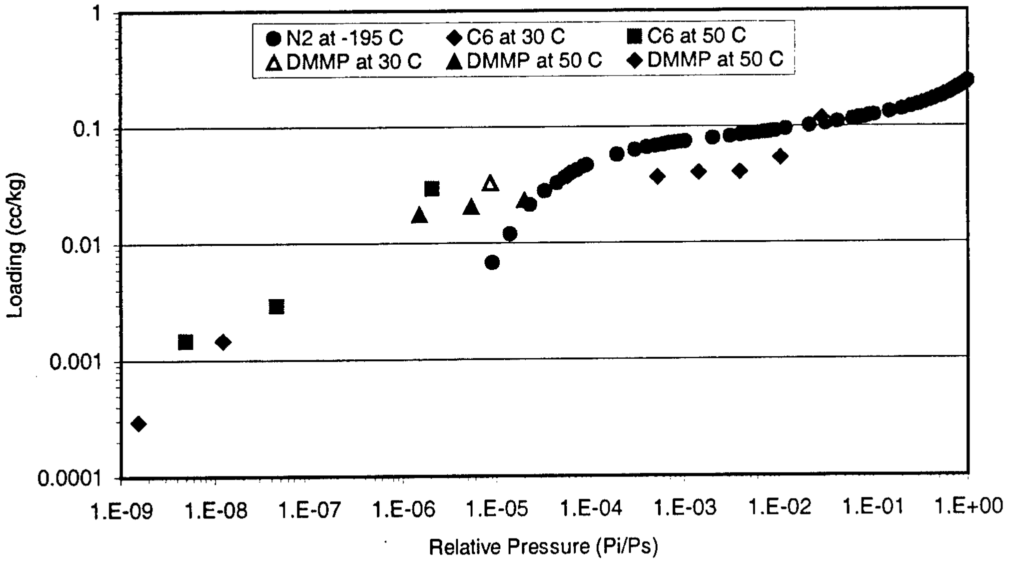

Figure 1. Adsorption Equilibria of Nitrogen, Hexane, and DMMP on PS Material

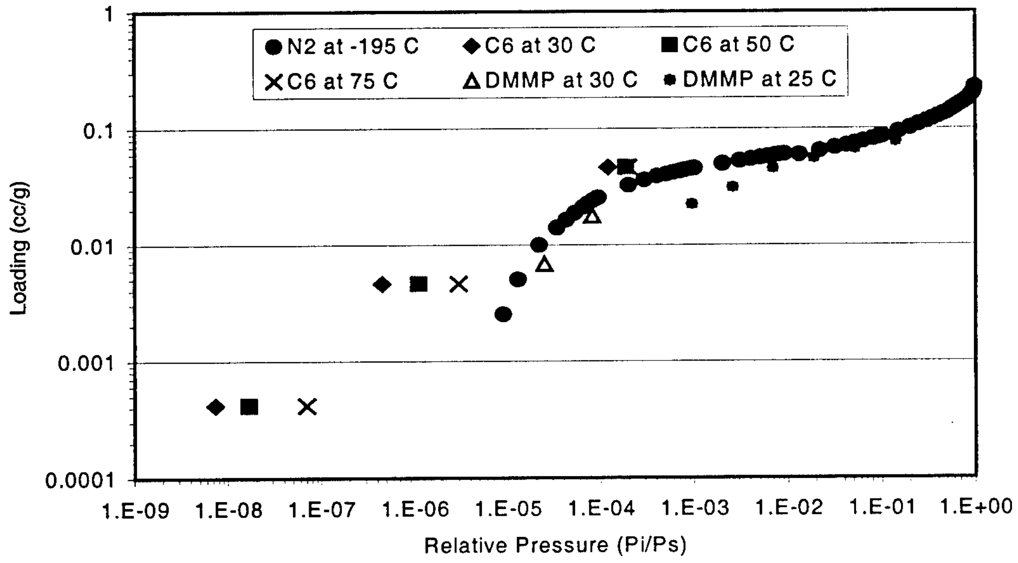

Figure 2. Adsorption Equilibria of Nitrogen, Hexane, and DMMP on PP Material

determined by multiplying the mass-per-volume density value shown in column three of the table by the corresponding micropore volume expressed as milliliters of N_2/g of material. For example,

PS material:

(0.038 g of material/milliliter of material) x (0.153 mL N_2 micropore /gram of material) (1)
= 0.00517 mL micropore/milliliter of material

and

PP material:

(0.1389 g of material/milliliter of material) x (0.062 mL micropore /gram of material) (2)
= 0.00861 mL micropore/milliliter of material

The percent ratio is

$$PP/PS = [1-(0.00861/0.00517)] \times 100\% = 66\% \qquad (3)$$

In comparison, hexane and DMMP adsorption equilibria shown in Figures 3 and 4 are remarkably similar to the nitrogen. Although some scatter exists, the trend shows that each adsorbate occupies about the same pore volume for a given relative pressure. Deviations are effected primarily by differences in the temperature of the measured data and the molar volume of each adsorbate (34.6, 130.3, 108.2 mL/mole for nitrogen, hexane, and DMMP, respectively). These factors can be correlated on an adsorption potential plot (Dubinin-Radushkevich) as shown in Figures 3 and 4. Plotted are loading (milliliters/kilogram) versus potential energy of adsorption $(T/V_m)^2LN(P_s/P_i)^2$ of nitrogen, hexane, and DMMP, where T is temperature (K), V_m is the adsorbate molar volume, P_s is the adsorbate vapor pressure at T, and P_i is the partial pressure of the adsorbate in equilibrium with the adsorbed phase loading.

Figure 3 shows the adsorption potential plot for the PS material. As expected, the potential energy of adsorption increases with decreasing adsorbed-phase loading for a given temperature. With lower loadings, there is naturally a decrease in the vapor-phase concentration (shown as partial pressure, P_i) for each of the three chemicals with temperature held constant. A regression of the data produces an intercept of 0.1138 mL/g, which is the approximate micropore volume when all three adsorbates are considered. This value is lower than the derived nitrogen micropore volume of 0.153 mL/g, which is to be expected, since smaller pores are more accessible to nitrogen than they are to hexane and DMMP.

Similarly, Figure 4 shows the adsorption potential plot for the PS material. A regression of the data produces an intercept of 0.0777 mL/g, which is a little higher than the nitrogen derived micropore volume of 0.062 mL/g. One reason for the difference is that scattering of the data affects the slope. The second reason is that the slope of the potential plot for the PP material is lower than the PS plot, suggesting that there is a lower number of the smallest micropores available for adsorption. This is shown by the slope of the DR potential fit and the difference in the magnitude of the calculated energy terms. With reduced scatter in the data, it is likely that the nitrogen, hexane and DMMP data would produce equivalent micropore volumes.

A comparative analysis of both materials shows that on a weight basis, the micropore volume (combined nitrogen, hexane, and DMMP) of the PP material is about 68% of the PS material (0.0777/0.113 mL/g). However, because volume density is more meaningful in comparing filter beds of similar volumes, the effective amounts of carbon per bed volume are 0.00380 and 0.0107 mL micropore/milliliter of material for the PS and PP materials, respectively. This is determined in a similar combined isotherms shown in Figures 3 and 4. The PP material provides about 2.8 times more adsorptive capacity for the same volume of material than the PS material (0.0107/ 0.0038 mL micropore/millimeter of material).

The DR correlation of the combined nitrogen, hexane, and DMMP data can be used to estimate the adsorption equilibria of other chemicals (e.g., mustard). Estimates of the adsorbed phase concentrations of mustard on the PS and PP materials are made for a vapor concentration of 0.029 mg/m^3 at 25 °C. This vapor concentration is equivalent to a partial pressure of 0.000451 Pa. The saturation pressure of mustard at 25 °C is 14.08 Pa, and the molar volume is 125.2 mL/mole (159.08 g/mole/ 1.27 g/mL). These values were used to calculate the adsorption potential at about 530 $(T/V_m)^2LN(P_s/P_i)^2$ as shown in Figures 3 and 4. The estimated adsorbed phase loading under these conditions would be about 0.035 and 0.015 mL/g of material for the PS and PP materials, respectively.

4. CONCLUSIONS

The following conclusions are presented as a result of tests performed:

- Two nonwoven fabric adsorbent materials [POLYSORB (PS) and Purification Products (PP)] were evaluated based on adsorption equilibria of nitrogen, hexane, and DMMP vapors. Both materials provide significantly less adsorption capacity per unit packing volume than typical granular bed activated carbons (e.g., BPL).

- A DR potential plot of the combined nitrogen, hexane, and DMMP data shows that the total estimated micropore volume of the PS and PP materials is 0.113 and 0.077 mL/g of material, respectively. Based on a comparison of the actual bed volume of adsorbent material for nitrogen, hexane, and DMMP, the PP material may provide about 2.8 times more adsorptive capacity than the PS material.

- The estimated adsorbed phase loading of mustard at 0.029 mg/m^3 at 25 °C is about 0.035 and 0.015 mL/g of material for the PS and PP materials, respectively.

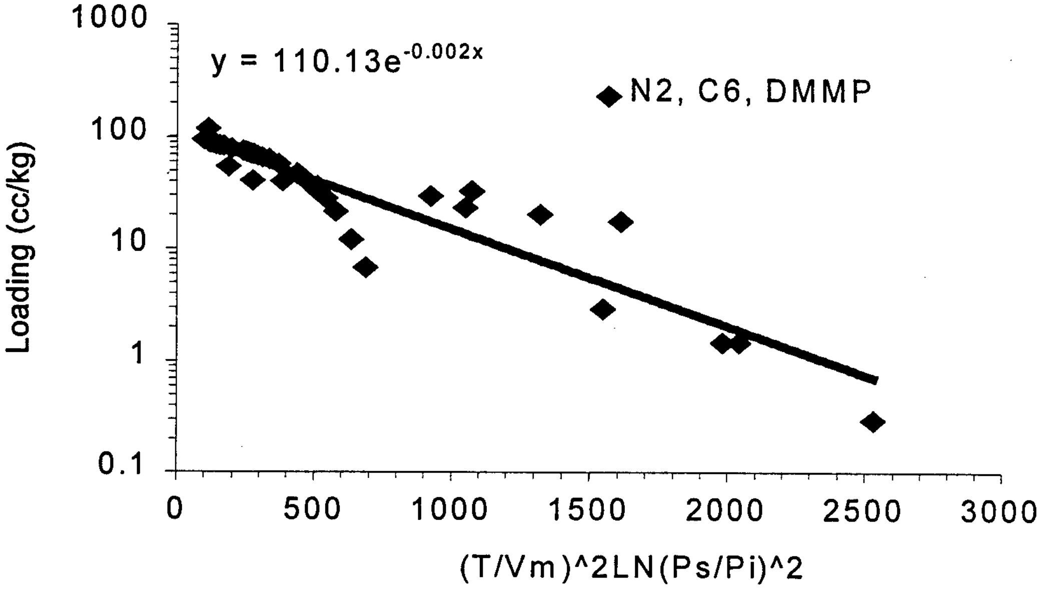

Figure 3. Adsorption Potential Plot of Nitrogen, Hexane, and DMMP on PS Material

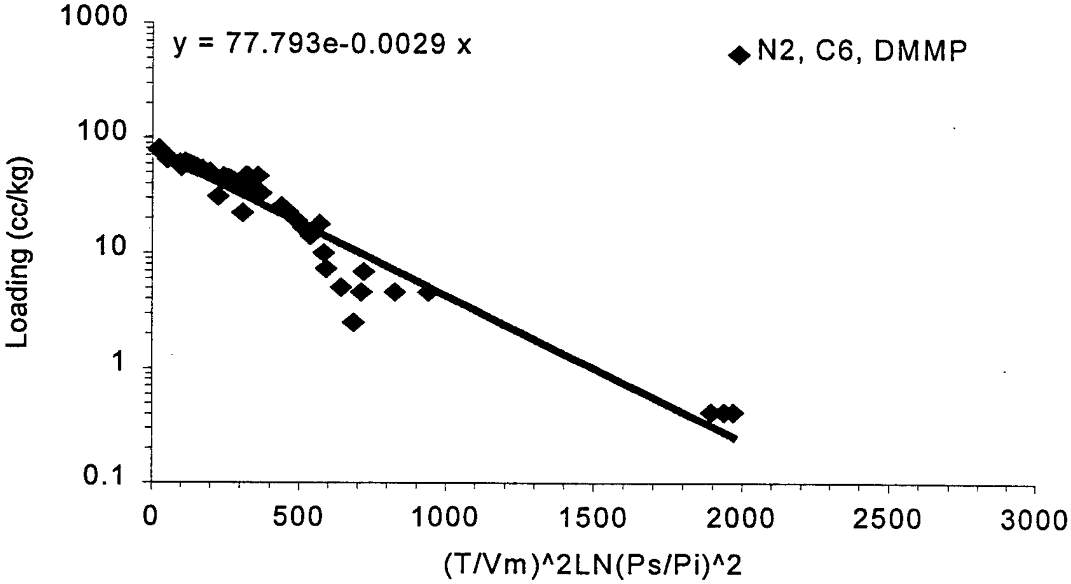

Figure 4. Adsorption Potential Plot of Nitrogen, Hexane, and DMMP on PP Material

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APPENDIX A

NITROGEN PORE SIZE ANALYSIS

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Date: 04/06/2000                                                      Page 1

                         Quantachrome Corporation
           Quantachrome Autosorb Automated Gas Sorption System Report
                    Autosorb for Windows®  Version 1.16

Sample ID         Thin Filter Material
Description       Recirculation Filter Material
Comments
Sample Weight     0.0362 g
Adsorbate         NITROGEN           Outgas Temp    150.0 °C   Operator        JR
Cross-Sec Area    16.2 Å²/molecule   Outgas Time    6.0 hrs    Analysis Time   945.0 min
NonIdeality       6.580E-05          P/Po Toler     0          End of Run      09/04/1998 03:21
Molecular Wt      28.0134 g/mol      Equil Time     5          File Name       AS89030M.RAW
Station #         1                  Bath Temp.     77.40


                      AREA-VOLUME-PORE SIZE SUMMARY

                            SURFACE AREA DATA

   Multipoint BET...............................................  1.305E+02  m²/g
   Single Point BET.............................................  1.200E+02  m²/g
   Langmuir Surface Area........................................  2.782E+02  m²/g
   BJH Method Cumulative Adsorption Surface Area................  4.130E+01  m²/g
   BJH Method Cumulative Desorption Surface Area................  4.550E+01  m²/g
   DH Method Cumulative Adsorption Surface Area.................  4.226E+01  m²/g
   DH Method Cumulative Desorption Surface Area.................  4.660E+01  m²/g
   t-Method External Surface Area...............................  3.180E+01  m²/g
   t-Method Micro Pore Surface Area.............................  9.870E+01  m²/g
   DR Method Micro Pore Area....................................  1.736E+02  m²/g

                            PORE VOLUME DATA

   Total Pore Volume for pores with Diameter
   less than 3731.4 Å at P/Po = 0.99484.........................  1.235E-01  cc/g
   BJH Method Cumulative Adsorption Pore Volume.................  7.610E-02  cc/g
   BJH Method Cumulative Desorption Pore Volume.................  7.549E-02  cc/g
   DH Method Cumulative Adsorption Pore Volume..................  3.242E-02  cc/g
   DH Method Cumulative Desorption Pore Volume..................  3.219E-02  cc/g
   t-Method Micro Pore Volume...................................  6.143E-02  cc/g
   DR Method Micro Pore Volume..................................  6.169E-02  cc/g
   HK Method Cumulative Pore Volume.............................  5.207E-02  cc/g
   SF Method Cumulative Pore Volume.............................  5.327E-02  cc/g

                             PORE SIZE DATA

   Average Pore Diameter........................................  3.785E+01  Å
   BJH Method Adsorption Pore Diameter (Mode)...................  3.063E+01  Å
   BJH Method Desorption Pore Diameter (Mode)...................  3.054E+01  Å
   DH  Method Adsorption Pore Diameter (Mode)...................  3.063E+01  Å
   DH  Method Desorption Pore Diameter (Mode)...................  3.824E+01  Å
   DR  Method Micro Pore Width..................................  3.537E+01  Å
   DA  Method Pore Diameter (Mode)..............................  1.800E+01  Å
   HK  Method Pore Width    (Mode)..............................  5.375E+00  Å
   SF  Method Pore Diameter (Mode)..............................  9.321E+00  Å
   DFT Method Pore Width    (Mode)..............................  1.429E+01  Å

                        DATA REDUCTION PARAMETERS

                       Thermal Transpiration : ON
                  Effective Molecule Diameter (D) 3.5400 Å
              Effective Cell Stem Inner Diameter (d) 4.0000 mm

                      BJH/DH Moving Average Size : 1

               Interaction Constant (K) 2.9600 nm^3 x kJ/mol
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Date: 04/06/2000                                                     Page 1

                              Quantachrome Corporation
                  Quantachrome Autosorb Automated Gas Sorption System Report
                         Autosorb for Windows®  Version 1.16

Sample ID        Thick Filter Sample
Description      Recirculation Filter Material
Comments
Sample Weight    0.0200 g
Adsorbate        NITROGEN           Outgas Temp    100.0 °C    Operator        JR
Cross-Sec Area   16.2 Å²/molecule   Outgas Time    54.0 hrs    Analysis Time   900.0 min
NonIdeality      6.580E-05          P/Po Toler     0           End of Run      09/11/1998 10:05
Molecular Wt     28.0134 g/mol      Equil Time     5           File Name       AS89110M.RAW
Station #        1                  Bath Temp.     77.40


                          AREA-VOLUME-PORE SIZE SUMMARY

                               SURFACE AREA DATA

   Multipoint BET............................................   3.256E+02  m²/g
   Single Point BET..........................................   3.112E+02  m²/g
   Langmuir Surface Area.....................................   6.036E+02  m²/g
   BJH Method Cumulative Adsorption Surface Area.............   8.051E+01  m²/g
   BJH Method Cumulative Desorption Surface Area.............   7.442E+01  m²/g
   DH Method Cumulative Adsorption Surface Area..............   8.245E+01  m²/g
   DH Method Cumulative Desorption Surface Area..............   7.625E+01  m²/g
   t-Method External Surface Area............................   3.562E+01  m²/g
   t-Method Micro Pore Surface Area..........................   2.900E+02  m²/g
   DR Method Micro Pore Area.................................   4.305E+02  m²/g

                               PORE VOLUME DATA

   Total Pore Volume for pores with Diameter
   less than 5049.2 Å at P/Po = 0.99620......................   2.417E-01  cc/g
   BJH Method Cumulative Adsorption Pore Volume..............   1.222E-01  cc/g
   BJH Method Cumulative Desorption Pore Volume..............   1.058E-01  cc/g
   DH Method Cumulative Adsorption Pore Volume...............   5.217E-02  cc/g
   DH Method Cumulative Desorption Pore Volume...............   4.522E-02  cc/g
   t-Method Micro Pore Volume................................   1.748E-01  cc/g
   DR Method Micro Pore Volume...............................   1.530E-01  cc/g
   HK Method Cumulative Pore Volume..........................   1.312E-01  cc/g
   SF Method Cumulative Pore Volume..........................   1.333E-01  cc/g

                                PORE SIZE DATA

   Average Pore Diameter.....................................   2.969E+01  Å
   BJH Method Adsorption Pore Diameter (Mode)................   3.415E+01  Å
   BJH Method Desorption Pore Diameter (Mode)................   3.048E+01  Å
   DH  Method Adsorption Pore Diameter (Mode)................   3.415E+01  Å
   DH  Method Desorption Pore Diameter (Mode)................   3.048E+01  Å
   DR  Method Micro Pore Width...............................   3.195E+01  Å
   DA  Method Pore Diameter (Mode)...........................   1.680E+01  Å
   HK  Method Pore Width    (Mode)...........................   5.325E+00  Å
   SF  Method Pore Diameter (Mode)...........................   9.228E+00  Å
   DFT Method Pore Width    (Mode)...........................   1.429E+01  Å

                           DATA REDUCTION PARAMETERS

                          Thermal Transpiration : ON
                    Effective Molecule Diameter (D) 3.5400 Å
                 Effective Cell Stem Inner Diameter (d) 4.0000 mm

                          BJH/DH Moving Average Size : 1

                     Interaction Constant (K) 2.9600 nm^3 x kJ/mol
```