Adsorbents and Carbon Technology for Air Sampling & Thermal Desorption

PEFTEC
November, 2015

Klaus Buckendahl, Jamie Brown

sigma-aldrich.com/analytical
Agenda

• Introduction
• Type of Adsorbents
• Selection Tool
What is Thermal Desorption?

A Sample Preparation technique for Air & Gas Analysis and GC.

- The gas /air sample is collected onto an adsorbent packed glass or stainless steel tube. *The sample is concentrated on the adsorbents.*

- The packed tube is heated (Thermal) and the compounds are released into the carrier gas (Desorption) transferred onto GC column for Analysis

![Diagram of Thermal Desorption setup](image)

- Carrier Gas
- Thermal Desorber (~300 °C)
- GC Detector (~300 °C)
- Gas Chromatograph
- Transfer Line
Adsorbent Characteristics for Thermal Desorption

- Able to retain & release the compounds of interest
- Able to withstand high temperatures ~ 300°C
- Low background levels
- Low metal content
- Hydrophobic

Desirable
- Consistent mesh/particle size
- Consistent density
- Low shrinkage
- Low amount of fines
Terms Defined

Surface Area:

- provides a general idea of the adsorbent strength, but it doesn't provide the whole picture.
- Other characteristics such as pore size, pore shape, and porosity can also play a role in the adsorbent’s ability to retain and release different compounds.

- General Rule:
  - The higher the surface area value, the stronger the adsorbent.
  - For surface area >800 m²/g, the size and shape of the pore becomes more important.
Terms Defined (cont.)

Pore Size:

- Macropores: > 50 nm diameter
- Mesopores: 2 - 50 nm diameter
- Micropores: < 2 nm diameter

Molecules cannot access pores smaller than their size
Will the strongest adsorbent work for everything?

Desorption of 43-compounds from Carbosieve-SIII

Desorption of 43-compounds from a Carbotrap-300

Carbotrap-300 = Carbotrap-C, Carbotrap-B, and Carbosieve SIII
Thermal Desorption Tube Adsorbent Beds

Single-Bed Tube
(e.g Tenax® TA)

Multi-Bed Tube
Carbotrap® 300
Adsorbent Types used in Thermal Desorption
Typical Adsorbents for Thermal Desorption

• Polymers
  • Tenax® TA (2,6-diphenyl-p-phenylene oxide)

• Graphitized Carbon Blacks (GCB)
  • Non porous
  • Names: Carbopack™, Carbotrap™
  • Various types available

• Carbon Molecular Sieves (CMS)
  • Porous
  • Names: Carboxen™, Carbosieve™
  • Various types available

• Glass beads
  • Used to retain large molecular weight volatiles

Key Expertise of Sigma-Aldrich / Supelco !!

Materials used on the NASA missions
Galileo (Jupiter) & Cassini-Huygens (Saturn-Titan)
Carbon Adsorbents
History in Supelco

>20 year Experience in preparing Carbon Adsorbents

Carbosieves
- First Family of Carbon molecular sieves (S-I, S-II, S-III)
- Carbosieve S-III one of strongest adsorbens
  - Microporous only
  - First CMS in air monitoring

Carboxenes (Introduced 1987)
- Carboxene-1000 highly efficient adsorbent
- Not as strong as S-III but far better kinetics
- Materials on NASA Missions
  - (e.g. Galileo, Cassini/Huygens to Titan)
Porous Polymers

Tenax®-TA

- The most popular adsorbent used in thermal desorption
- Maximum temperature: 350 °C
- Recommended desorption temp: 300 °C
- Recommended conditioning temp: 320 °C
- Methanol not retained (Good for spiking tubes with liquid calibration standards)

Typical Characteristics

- Granular - Tan in color
- Surface area: 35 m²/g
- Hydrophobic
Porous Polymers (cont.)

PoraPak™-N, Chromosorb®-106, HayeSep®-D

- Relatively low maximum temperatures: 225-290 °C
- Recommended desorption temp: 200 °C
- Recommended conditioning temp: 210 °C
- Typically has higher background levels than other adsorbents

Typical Characteristics

- Spherical - Light Yellow in color
- Surface area: 500 to 800 m²/g
- Hydrophobic
Other Adsorbents

**Glass Beads**  5 m²/g ("good pre-filter")

Rarely used for Thermal Desorption
- Petroleum Charcoal
- Coconut Charcoal
- Silica Gel
- Molecular Sieves (Zeolites)
Graphitized Carbon Blacks

Carbotrap® and Carbopack™

- Maximum temperature: 400 °C
- Recommended desorption temp: 330 °C
- Recommended conditioning temp: 350 °C
- Methanol not retained by most of them

Typical Characteristics

- Granular - Flat Grey/Black in color
- Surface area: 5 to 240 m²/g
- Designed to retain and release mid to large molecular weight compounds
- Hydrophobic
- High Purity - Low Background

Carbotrap = 20/40 mesh
Carbopack = 40/60, 60/80, 80/100, and 100/120 mesh
Water vapor retained by Carbopack(s) & Tenax-TA

Cumulative Water Uptake
Sampling at 75%RH (25°C)
## Adsorptive Strength of Graphitized Carbon Blacks

<table>
<thead>
<tr>
<th>Carbon Black</th>
<th>Adsorptive Strength (m²/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbopack X</td>
<td>240</td>
</tr>
<tr>
<td>Carbopack Z</td>
<td>220</td>
</tr>
<tr>
<td>Carbopack B</td>
<td>100</td>
</tr>
<tr>
<td>Carbopack Y</td>
<td>24</td>
</tr>
<tr>
<td>Carbopack C</td>
<td>10</td>
</tr>
<tr>
<td>Carbopack F</td>
<td>5</td>
</tr>
</tbody>
</table>

**Relative Adsorption Strength**

- Strongest: Carbopack X
- Weakest: Carbopack F
Graphitized Carbon Blacks

CALIBRATION STANDARD

CARBOPACK-X

CARBOPACK-B

CARBOPACK-Y

CARBOPACK-C

CARBOPACK-F

GLASSBEADS

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On September 29, 2015, the U.S. EPA issued a final rule - requiring all U.S. Petroleum Refineries to conduct passive air sampling along the perimeter of their properties. (40 CFR Parts 60 and 63) Coming Method will be EPA325A/B.

EPA Method 325 uses passive (diffusive) samplers to collect air samples at specific intervals along the fence line of the petroleum refineries property. The target compound is Benzene.

These passive air samplers are comprised of an inert-coated stainless steel thermal desorption tube packed with a graphitized carbon adsorbent. Carbopack™X is listed as primary adsorbent.

The Carbopack X is also validated by the EPA and listed in the Method.

*The I.D. of the stainless steel tube are coated with a protective layer (Supelcoat™), which masks any active sites that could be present on the inside of the tube.
EPA Method 325 (DRAFT) for Fenceline Monitoring (FLM)
Using Carbopack X in Specially Treated Thermal Desorption Tube

- Inert TD Tube with diffusion cap
- Carbopack X bed
  - retains wide range of analytes
    - recovery of key analytes, 1,3-butadiene, benzene, toluene, remain at 100% when sampling large volumes
- is hydrophobic
  - water will not be retained

Cat# 28686-U Stainless Steel w/SupelCoat Carbopack X (60/80) shown with diffusive sampling cap
Carbopack X was validated for the broadest compound portfolio due to its broad suitability, not just Benzene (or BTEX).

Table 12.1: Validated Sorbents and Uptake Rates (mL/min) for Selected Clean Air Act Compounds

<table>
<thead>
<tr>
<th>Compound</th>
<th>Carbopack™ Xa</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,1-Dichloroethene</td>
<td>0.57±0.14</td>
</tr>
<tr>
<td>3-Chloropropene</td>
<td>0.51±0.3</td>
</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>0.57±0.1</td>
</tr>
<tr>
<td>1,2-Dichloroethane</td>
<td>0.57±0.08</td>
</tr>
<tr>
<td>1,1,1-Trichloroethane</td>
<td>0.51±0.1</td>
</tr>
<tr>
<td>Benzene</td>
<td>0.67±0.06</td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
<td>0.51±0.06</td>
</tr>
<tr>
<td>1,2-Dichloropropane</td>
<td>0.52±0.1</td>
</tr>
<tr>
<td>Trichloroethene</td>
<td>0.5±0.05</td>
</tr>
<tr>
<td>1,1,2-Trichloroethane</td>
<td>0.49±0.13</td>
</tr>
<tr>
<td>Toluene</td>
<td>0.52±0.14</td>
</tr>
<tr>
<td>Tetrachloroethene</td>
<td>0.48±0.05</td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>0.51±0.06</td>
</tr>
<tr>
<td>Ethylbenzene</td>
<td>0.46±0.07</td>
</tr>
<tr>
<td>m,p-Xylene</td>
<td>0.46±0.09</td>
</tr>
<tr>
<td>Styrene</td>
<td>0.5±0.14</td>
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<tr>
<td>o-Xylene</td>
<td>0.46±0.12</td>
</tr>
<tr>
<td>p-Dichlorobenzene</td>
<td>0.45±0.05</td>
</tr>
</tbody>
</table>

US EPA Method 325B Compliance Flyer (RNW)

Passive Fenceline Sampling Solution for Benzene and Other VOCs

sigma-aldrich.com/flm
Passive Sampling Options for Thermal Desorption Using Carbopack X

Axial Sampler

Radial Sampler
(RAD141 for 1,3 Butadiene)

Radial Sampler (radiello®) due to design
• Higher Sampling rates
• Higher sensitivity
Radiello (RAD141) Sampler for 1,3-Butadiene

After sampling, the RAD141 adsorbent cartridge is placed in an empty stainless steel thermal desorption tube for analysis.
Carbon Molecular Sieves

Carbosieve® and Carboxen®

- Maximum temperature: 400 °C
- Recommended desorption temp: 330 °C
- Recommended conditioning temp: 350 °C
- Methanol is retained

Typical Characteristics

- Spherical (Carbosieve-G is granular)
- Shiny/Dull Black in color
- High surface area 400 to 1500 m²/g
- Designed to retain and release small molecular weight compounds
Adsorptive Strength of Carbon Molecular Sieves

Relative Adsorption Strength

<table>
<thead>
<tr>
<th>Material</th>
<th>Adsorptive Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carboxen-1016</td>
<td>75 $m^2/g$</td>
</tr>
<tr>
<td><em>Carbosieve-G</em></td>
<td>1160 $m^2/g$</td>
</tr>
<tr>
<td>Carboxen-1012</td>
<td>1500 $m^2/g$</td>
</tr>
<tr>
<td>Carboxen-564</td>
<td>400 $m^2/g$</td>
</tr>
<tr>
<td>Carboxen-1000</td>
<td>1200 $m^2/g$</td>
</tr>
<tr>
<td>Carboxen-1001</td>
<td>500 $m^2/g$</td>
</tr>
<tr>
<td>Carboxen-569</td>
<td>485 $m^2/g$</td>
</tr>
<tr>
<td>Carboxen-1003</td>
<td>1000 $m^2/g$</td>
</tr>
<tr>
<td>Carboxen-1018</td>
<td>675 $m^2/g$</td>
</tr>
<tr>
<td><em>Carbosieve-SIII</em></td>
<td>975 $m^2/g$</td>
</tr>
<tr>
<td>Carboxen-1021</td>
<td>1160 $m^2/g$</td>
</tr>
</tbody>
</table>

Strongest: *Carbosieve-G* (1160 $m^2/g$)

Strongest: Carboxen-1021 (1160 $m^2/g$)

Weakest: Carboxen-569 (485 $m^2/g$)

Weakest: Carboxen-1001 (500 $m^2/g$)
Relative Hydrophobicity

Glass Beads
Graphitized Carbon Blacks
Porous Polymers

Carboxen-1016
Carboxen-569,1001,1003
Carboxen-563
Carboxen-564
Carboxen-1000
Carboxen-1012
Carboxen-1018,1021, Carbosieve G & SIII

Silica Gel, Mole Sieve 5x, 13x

Very Hydrophobic

Retains Less H₂O

Retains More H₂O

Carbon Molecular Sieves
**Information on Carbon Adsorbens**

**Wide Range of Properties**

How to choose?

<table>
<thead>
<tr>
<th>Relative Analyte Size</th>
<th>Adsorptive Strength</th>
<th>Recommended Adsorbents</th>
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</thead>
<tbody>
<tr>
<td>C20+</td>
<td>Weakest</td>
<td>Carbotrap F, Carbopack F</td>
</tr>
<tr>
<td>C12-C20</td>
<td></td>
<td>Carbotrap C, Carbopack C</td>
</tr>
<tr>
<td>C9-C14</td>
<td></td>
<td>Carbotrap Y, Carbopack Y</td>
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<tr>
<td>C5-C12</td>
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<td>Graphsphere 2027, Graphsphere 2029, Carbotrap B, Carbopack B, Graphsphere 2016, Graphsphere 2017</td>
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<tr>
<td>C3-C9</td>
<td></td>
<td>Carbotrap X, Carbopack X, Carbopack Z</td>
</tr>
<tr>
<td>C2-C5</td>
<td>Strongest</td>
<td>Carboxen 1012, Carboxen 1034, Carboxen 1000, Carboxen 1008, Carboxen 1026, Carbosieve G, Carboxen 1005, Carboxen 572, Carbosieve S-II, Carboxen 1003, Carbosieve S-III, Carboxen 1032, Carboxen 1030, Carboxen 1006, Carboxen 1018, Carboxen 1010, Carboxen 1021, Carboxen 563, Carboxen 1001, Carboxen 569, Carboxen 1033, Carboxen 564</td>
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</table>

### Approx. Surface Area and Pore Properties

<table>
<thead>
<tr>
<th>Carbon</th>
<th>Approx. Surface Area (m²/g)</th>
<th>Micro</th>
<th>Mesop.</th>
<th>Macro</th>
<th>Approx. Pore Diam. (Å)</th>
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</thead>
<tbody>
<tr>
<td>Carbotrap F / Carbopack F</td>
<td>5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
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<tr>
<td>Carbotrap C / Carbopack C *</td>
<td>10</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Carbotrap Y / Carbopack Y *</td>
<td>24</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
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<tr>
<td>Carboxen 1017</td>
<td>61</td>
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<td>0.33</td>
<td>-</td>
<td>-</td>
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<tr>
<td>Graphitized carbon black</td>
<td>70</td>
<td>0.01</td>
<td>0.23</td>
<td>-</td>
<td>137</td>
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<td>Carboxen 1016</td>
<td>75</td>
<td>-</td>
<td>0.34</td>
<td>-</td>
<td>-</td>
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<tr>
<td>Carbotrap B / Carbopack B *</td>
<td>100</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Mesoporous carbon</td>
<td>203</td>
<td>-</td>
<td>0.49</td>
<td>-</td>
<td>96.3</td>
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<tr>
<td>Purified carbon black</td>
<td>214</td>
<td>0.06</td>
<td>0.28</td>
<td>-</td>
<td>63.9</td>
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<td>Carbopack Z *</td>
<td>220</td>
<td>-</td>
<td>1.73</td>
<td>-</td>
<td>255</td>
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<tr>
<td>Carbotrap X / Carbopack X *</td>
<td>240</td>
<td>-</td>
<td>0.62</td>
<td>-</td>
<td>100</td>
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<tr>
<td>Carboxen 564</td>
<td>400</td>
<td>0.24</td>
<td>0.13</td>
<td>0.14</td>
<td>6-9</td>
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<td>Carboxen 569</td>
<td>485</td>
<td>0.20</td>
<td>0.14</td>
<td>0.10</td>
<td>5-8</td>
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<td>Carboxen 1001</td>
<td>500</td>
<td>0.22</td>
<td>0.13</td>
<td>0.11</td>
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<td>Carboxen 563</td>
<td>510</td>
<td>0.24</td>
<td>0.15</td>
<td>0.24</td>
<td>7-10</td>
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<tr>
<td>Carboxen 1021</td>
<td>600</td>
<td>0.30</td>
<td>-</td>
<td>-</td>
<td>5-8</td>
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<tr>
<td>Carboxen 1010</td>
<td>675</td>
<td>0.35</td>
<td>-</td>
<td>-</td>
<td>6-8</td>
</tr>
<tr>
<td>Carboxen 1018</td>
<td>675</td>
<td>0.35</td>
<td>-</td>
<td>-</td>
<td>6-8</td>
</tr>
<tr>
<td>Carboxen 1006</td>
<td>715</td>
<td>0.29</td>
<td>0.26</td>
<td>0.23</td>
<td>7-10</td>
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<tr>
<td>Carbosieve S-III</td>
<td>975</td>
<td>0.35</td>
<td>0.04</td>
<td>-</td>
<td>4-11</td>
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<tr>
<td>Carboxen 1003</td>
<td>1000</td>
<td>0.38</td>
<td>0.26</td>
<td>0.28</td>
<td>5-8</td>
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<tr>
<td>Carbosieve S-II</td>
<td>1059</td>
<td>0.45</td>
<td>0.01</td>
<td>-</td>
<td>6-15</td>
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<tr>
<td>Carboxen 572</td>
<td>1100</td>
<td>0.41</td>
<td>0.19</td>
<td>0.24</td>
<td>10-12</td>
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<tr>
<td>Supelcarb</td>
<td>1150</td>
<td>0.47</td>
<td>0.26</td>
<td>0.28</td>
<td>5-8</td>
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<tr>
<td>Carbosieve G</td>
<td>1160</td>
<td>0.49</td>
<td>0.02</td>
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<td>6-15</td>
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<td>Carboxen 1000</td>
<td>1200</td>
<td>0.44</td>
<td>0.16</td>
<td>0.25</td>
<td>10-12</td>
</tr>
<tr>
<td>Carboxen 1012</td>
<td>1500</td>
<td>-</td>
<td>0.66</td>
<td>-</td>
<td>19-21</td>
</tr>
</tbody>
</table>
"A Tool for Selecting an Adsorbent for Thermal Desorption"

www.sigmaaldrich.com/air-monitoring
Spiking the Tubes with the Gas Mix

Adsorbent Tube Injector System (ATIS)
used for spiking tubes

- 20 mL of gas mix
  - 43 Compounds: 50 to 260 in molecular weight, -30 to 215°C in boiling point.
  - Concentration: 1 ppm of each compound
- Injected into a stream of N₂
- N₂ carried the compounds to the tube
  - Challenges volumes tested

sigma-aldrich.com/atis
ATIS - Principle

- Adsorbent Tube
- Glass Cuvett
- Heater Block
- Inert Gas Inlet
- Thermometer
### How to use the performance charts

#### Information about the adsorbent

The 6 volumes studied:

<table>
<thead>
<tr>
<th>Volume (Liters)</th>
<th>0.2</th>
<th>1</th>
<th>5</th>
<th>10</th>
<th>20</th>
<th>100</th>
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<tbody>
<tr>
<td>1</td>
<td></td>
<td></td>
<td></td>
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<td>100</td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

#### Green = Recommend for use

Recoveries are greater than 80%

#### Yellow = Use caution

(Watch the trend)

Recoveries are 21 to 79%

#### Red = Not recommend

Recoveries are below 20%

### Carboxen-1000

- **Surface Area**: 1200 m²/g
- **Desorption Temperature**: 350 °C

### Performance Key

- **Green**: Recoveries are greater than 80%
- **Yellow**: Recoveries are 21 to 79%
- **Red**: Recoveries are below 20%

* indicates this analyte was strongly adsorbed.

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SIGMA-ALDRICH®
The outcome of this research ...
“Inlet” First adsorbent bed

### Carbopack B

(Graphitized Carbon Black)

<table>
<thead>
<tr>
<th>Surface Area: 100 $m^2/g$</th>
<th>Desorption Temperature: 330 °C</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>Challenge Volume (Liters)</th>
<th>0.2</th>
<th>1</th>
<th>5</th>
<th>10</th>
<th>20</th>
<th>100</th>
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</thead>
<tbody>
<tr>
<td>Vinyl chloride</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Methylene chloride</td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Toluene</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1,2,4-Trichlorobenzene</td>
<td>Retained</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Breaks through

Retained

Carryover was observed (Too Strongly Adsorbed)

Most likely irreversibly adsorbed

Back-up adsorbent bed

### Carboxen-1003

(Carbon Molecular Sieve)

<table>
<thead>
<tr>
<th>Surface Area: 1000 $m^2/g$</th>
<th>Desorption Temperature: 330 °C</th>
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</table>

<table>
<thead>
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<th>Challenge Volume (Liters)</th>
<th>0.2</th>
<th>1</th>
<th>5</th>
<th>10</th>
<th>20</th>
<th>100</th>
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<tr>
<td>Vinyl chloride</td>
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<td></td>
<td>Retained</td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>Retained</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Toluene</td>
<td>Carryover was observed (Too Strongly Adsorbed)</td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>1,2,4-Trichlorobenzene</td>
<td>Most likely irreversibly adsorbed</td>
<td></td>
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</tr>
</tbody>
</table>
Test different Carbon Adsorbents?

Supelco Specialty Carbon Adsorbents
High-Tech Materials Engineered for Applications of This World and Beyond!

Selection Guidelines
Physical Characteristics
Custom Capabilities
Carbon Adsorbent Sampler Kits
Carbon Molecular Sieves
Spherical Graphitized Petroleum Carbons
Graphitized Carbon Blacks

(MQR)

Custom Tubes possible!

Sampler Kits
Conclusion

• Thermal desorption covers a wide range of analytes
  • Available Adsorbents offer enables broad sampling scope
• Single bed tubes are often have limits for a wider analyte portfolio
  • Multibed tubes provide a wider range
• Synthetic carbon adsorbents (Carbopack, Carboxen) are most suitable due to stability, reproducibility & purity
  • Sigma-Aldrich / Supelco has long year experience and wide selection
• Adsorbent selection can be done
  • by literature research (official methods, journal articles, vendor information)
  • using „A Tool for Selecting an Adsorbent for Thermal Desorption”
• Custom selected adsorbents for a multibed tube allow to achieve optimal retention & release characteristics for desired application
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Kristen Schultz - Air Monitoring Product Manager

Bellefonte, Pennsylvania USA

Trademarks:
Carbotrap®, Carbopack™, Carboxen®, Carbosieve® – Sigma Aldrich, USA
Chromosorb® - Imerys Minerals California, Inc. USA
HayeSep® - Hayes Separations, USA
PoraPak™ - Waters Associates, USA
Tenax® - Buchem B.V., Netherlands
Thank you!

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sigma-aldrich.com/air-monitoring