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Development and Testing of a Microwave Powered Regenerable Air Purification Technology Demonstrator

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ABSTRACT

Dielectric heating via microwave irradiation of contaminant laden sorbents offers distinct advantages in comparison to conventional thermal regeneration techniques. High temperatures may be achieved very rapidly because electromagnetic energy is absorbed directly by the sorbent material. A Technology Demonstrator, incorporating efficient rectangular waveguide based sorbent cartridge designs and effective microwave transmission systems was designed, fabricated and tested. Importantly, the performance of the Molecular Sieve 13X Waveguide Cartridge for the removal of water vapor, the Molecular Sieve 5A Waveguide Cartridge for the removal of CO_2, and the Activated Carbon Waveguide Cartridge for removal of volatile organics from air, were each validated by successive sorption/ microwave desorption cycles.

INTRODUCTION

The design, fabrication, and testing of a novel microwave powered thermally regenerable air purification device are described (1). Dielectric heating via microwave irradiation of contaminant laden sorbents offers distinct advantages in comparison to conventional thermal regeneration techniques. High temperatures may be achieved very rapidly because electromagnetic energy is absorbed directly by the sorbent material. Also, because the primary mode of heat transfer is through radiation, conductive and convective losses can be minimized, resulting in reduced power consumption. Significant results, which build upon previous work (2), include the identification of efficient rectangular waveguide based sorbent cartridge designs, establishment of effective microwave transmission systems, and demonstration of microwave powered thermal regeneration of a variety of sorbent/contaminant combinations. Performance of the integrated Technology Demonstrator was validated using a thermally regenerable Molecular Sieve 13X Waveguide Cartridge for the removal of water vapor, a thermally regenerable Molecular Sieve 5A Waveguide Cartridge for the removal of CO_2 from air, and a thermally regenerable Activated Carbon Waveguide Cartridge for removal of volatile organics, including oxygenates, aromatics, and chlorocarbons. The Technology Demonstrator will serve as a test-bed for future research in the areas of Advanced Life Support, EVA, and ground-based air purification and related environmental remediation applications. The integrated hardware will also serve as the basis for commercialization of the microwave powered regenerable air purification technology.

DEVELOPMENT SUMMARY

The development work for the microwave powered regenerable air purification technology followed four main paths. First, the characterization of the dielectric heating properties of a wide range of air contaminants and sorbent materials was performed using a Vector Network Analyzer based apparatus. This work has been reported elsewhere (3,4,5). Second, evaluations of the heating properties of candidate sorbent materials or sorbent/microwave susceptor composites were performed at the common 2.45 GHz microwave frequency. Third, microwave susceptible additives were identified and/or developed. Fourth, microwave transmission and waveguide cartridge designs were optimized. Only a brief summary of the sorbent evaluation study for CO_2 removal will be discussed here.

TEST APPARATUS

Initial microwave heating and thermal regeneration experiments were conducted using the bench-top rectangular waveguide based test system shown in Figure 1. The system utilizes a 2.45 GHz magnetron in conjunction with both WR430 and WR284 rectangular waveguide transmission elements. The essential features of the apparatus include: the microwave applicator test section, the three-stub tuner, and the variable power microwave generator. Autonomous data acquisition was achieved using a laptop computer.

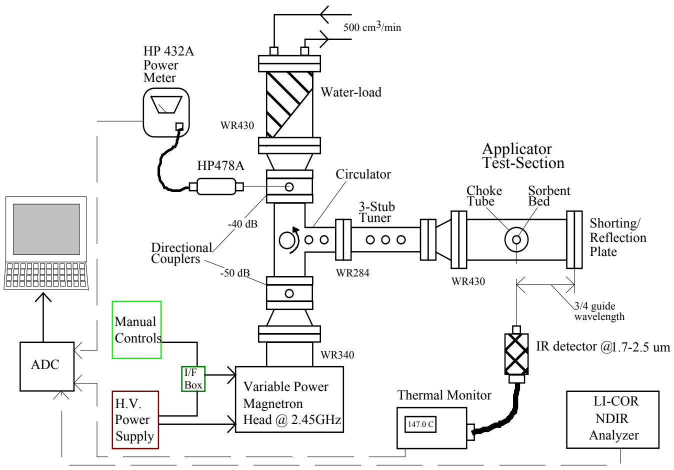

Figure 1. Rectangular Waveguide based Microwave Sorption-Desorption Test System.

Minimization of the power required for thermal regeneration of a loaded sorbent bed was an important goal of the development program. Each sorbent material has unique dielectric heating properties. To identify the minimum power needed to regenerate a given sorbent, the ability to vary the incident microwave power levels with precise control was essential. In previous work conducted in our laboratory, microwave powered thermal regenerations were performed using a rheostat (Variac) to control the voltage applied to the magnetron power supply. This method of variable power control proved to be relatively unstable and to have a lower control limit of $\approx$100 W. Substantial improvements have been made in the current test apparatus. Using, the UWHEAD2.0FAC-SM magnetron head and SM745 switch mode power supply, stable microwave power levels were continuously varied from 1 W to 1.9 kW. The power from the magnetron head was measured via a -50 dB directional coupler, connected to an HP478A thermistor based detector and HP432A power meter. A WFCC284-50/50-N circulator (Microwave Resources Inc.) diverted the power into a three-stub tuner. The circulator served to protect the magnetron head from potentially damaging reflections by redirecting reflected power into a water-load. This reflected power level was monitored via a -40 dB directional coupler. The Astex/Gerling model AX3041 three-stub tuner provided a means to attain maximal efficiency of power transmission by impedance matching between the source and load. By varying the insertion depths of the three metal tuning stubs into the waveguide, the reflection of power to the water-load was minimized and microwave power delivered to the sorbent bed maximized. This allowed maximum heating efficiencies at minimum input power levels.

Sorbent beds were inserted vertically through the center of a modified WR430 waveguide section as illustrated in Figure 2. Inside the rectangular waveguide, the 0.75 wavelength separation between the sorbent bed center and the shorting plate, placed the sorbent bed center at a standing wave maximum. This ensured that the electric field component of the TE_{01} wave reflected by the shorting plate added constructively to the incident wave at the sorbent bed location. In this way, power transfered to the sorbent during thermal regeneration was maximized.

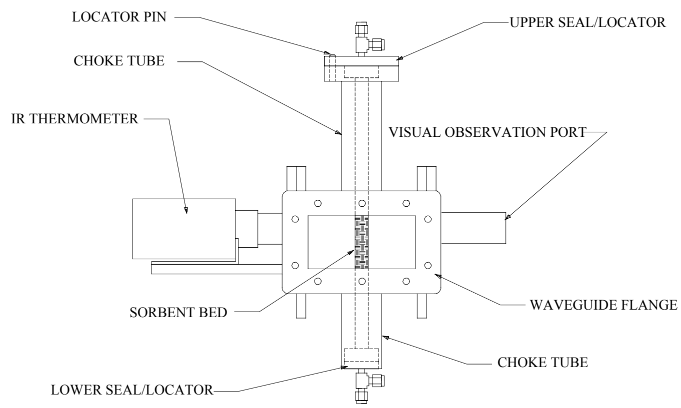

Figure 2. Cross Section View of Sorbent Bed Mounted in the Microwave Applicator Section of the Rectangular Waveguide Test System. The direction of Microwave Propagation is orthogonal to the paper. Gas is fed through the fittings located at the bottom and exits at the top.

EXPERIMENTAL

Carbon dioxide sorption and regeneration experiments were performed using Molecular Sieve 5A. A packed bed containing ~5.5 cm^3 of sorbent was confined within rectangular glass tubing and mounted in the applicator section of the microwave irradiation test system. Dry compressed air containing a nominal 350 ppm pCO_2 was fed to the sorbent at a flow rate of 320 cm^3/min. Effluent CO_2 levels were monitored in real-time using the LI-COR on-line NDIR detector. Once breakthrough reached influent levels, thermal regeneration was triggered under a dry nitrogen flow, also at 320 cm^3/min. Effluent CO_2 and bed temperature were monitored continuously. Microwave power was increased incrementally. Successive CO_2 sorption experiments, where breakthrough occurs at the same throughput demonstrates the adequacy of the intervening microwave powered thermal regenerations.

RESULTS

Two successive sorption experiments following regeneration are illustrated in Figures 3 and 4. For each of these, breakthrough began at approximately 6 L of throughput. A typical thermal desorption is illustrated in Figure 5. The bulk of the carbon dioxide was removed by 4 L of throughput, with relatively low input power levels (40 W), and bed temperatures $\leq$ 100 °C.

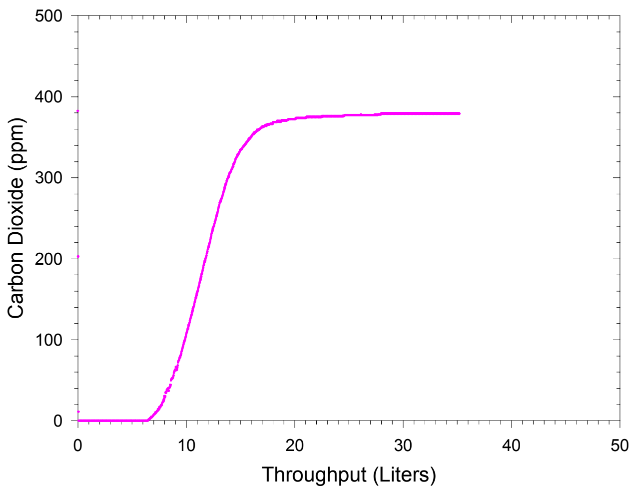

Figure 3. Second Sorption of Carbon Dioxide (dry) on Molecular Sieve 5A.

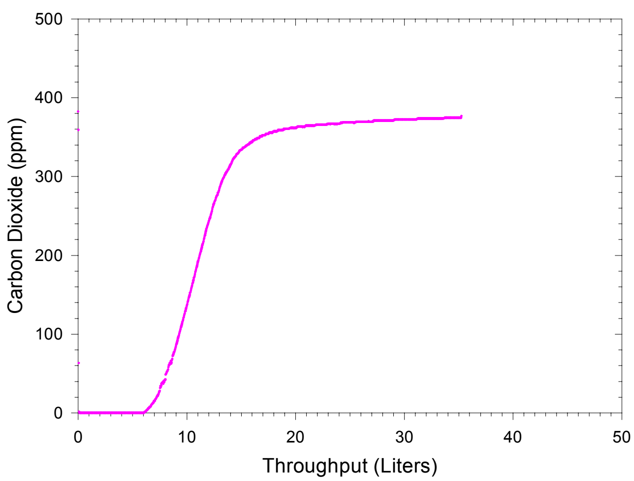

Figure 4. Third Sorption of Carbon Dioxide (dry) on Molecular Sieve 5A.

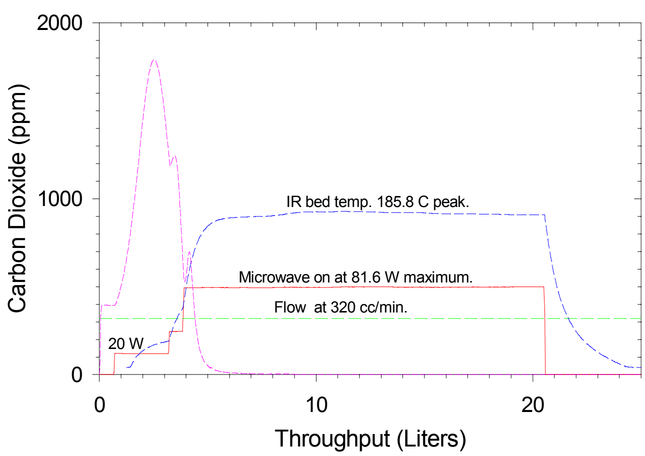

Figure 5. Microwave Powered Thermal Regeneration of Carbon Dioxide Loaded Molecular Sieve 5A.

WAVEGUIDE CARTRIDGE DESIGN AND FABRICATION

Based upon the experimental results derived using the rectangular waveguide based test system, a Microwave Powered Thermally Regenerable Air Purification Technology Demonstrator was designed and fabricated. The purpose of the device was to provide a flexible and convenient test-bed to facilitate future research and development efforts directed toward a variety of potential Advanced Life Support applications (including EVA), in addition to ground base air purification and related environmental remediation requirements. The hardware is also intended to serve as the basis for commercialization of the technology. The following goals were identified for the Technology Demonstration hardware:

- Demonstrate effective removal of CO_2, H_2O vapor , and trace organics at concentrations anticipated for the International Space Station (ISS) Portable Life Support System (PLSS).
- Demonstrate effective removal of typical airborne environmental pollutants including: halocarbons, aromatics, and oxygenates, as potential commercial applications.
- Demonstrate multiple regenerations of sorbents utilizing microwave energy.
- Demonstrate safe operation with no electrical hazards or microwave leaks.
- Demonstrate energy efficient operation during microwave powered thermal desorption.
- Demonstrate portability of sorption beds with quick and simple removal from the Technology Demonstrator test stand.

Sorbent cartridges employing MS 5A, MS 13X, and Activated Carbon were designed and fabricated to demonstrate the removal of airborne CO_2, water vapor, and organic contaminants, respectively. Design details are given for each of the waveguide sorbent beds in Figures 6, 7 and 8, respectively. A common configuration was employed for each cartridge. The primary differences are the lengths of the sorbent bed sections, stemming from variations in contaminant concentrations and sorption capacities. The horizontal dimensions for the MS 5A, MS 13X and activated carbon beds are 45.7 cm (18 inch), 22.9 cm (9 inch), and 15.2 cm (6 inch), respectively. The total length for each waveguide cartridge also includes additional waveguide sections, which provide gas connections, coaxial cable to waveguide transitions, and a silicon carbide microwave sorbent block termination.

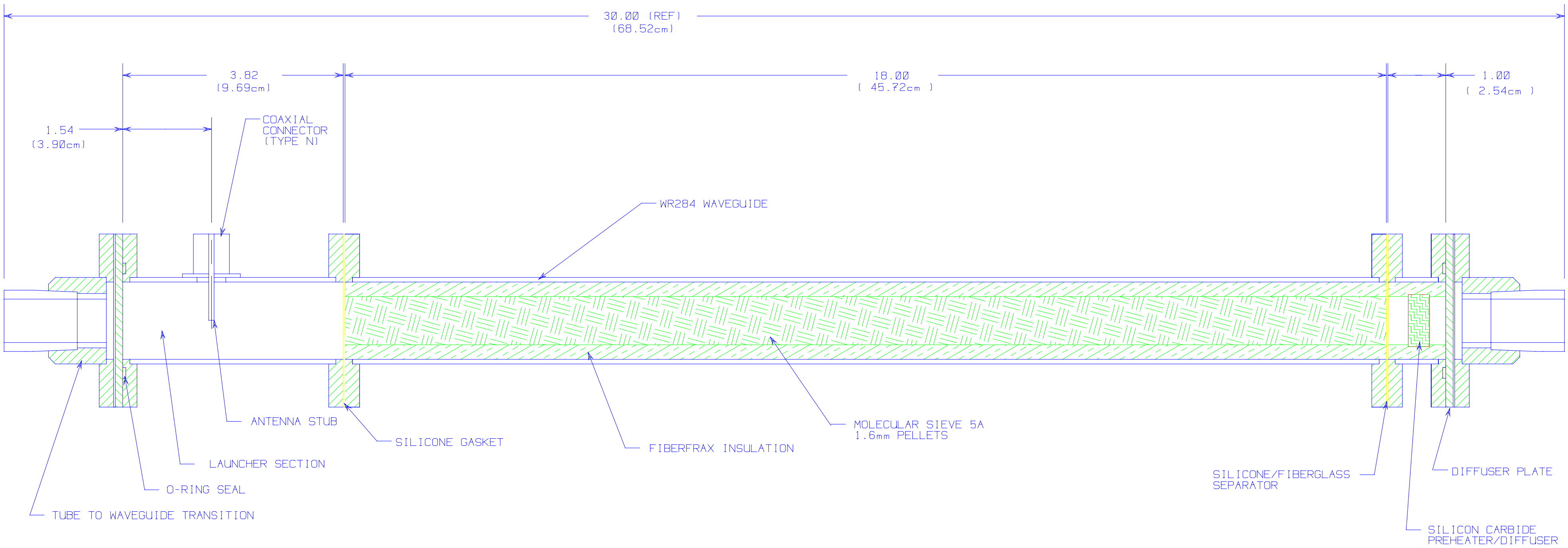

Figure 6. Molecular Sieve 5A Waveguide Cartridge.

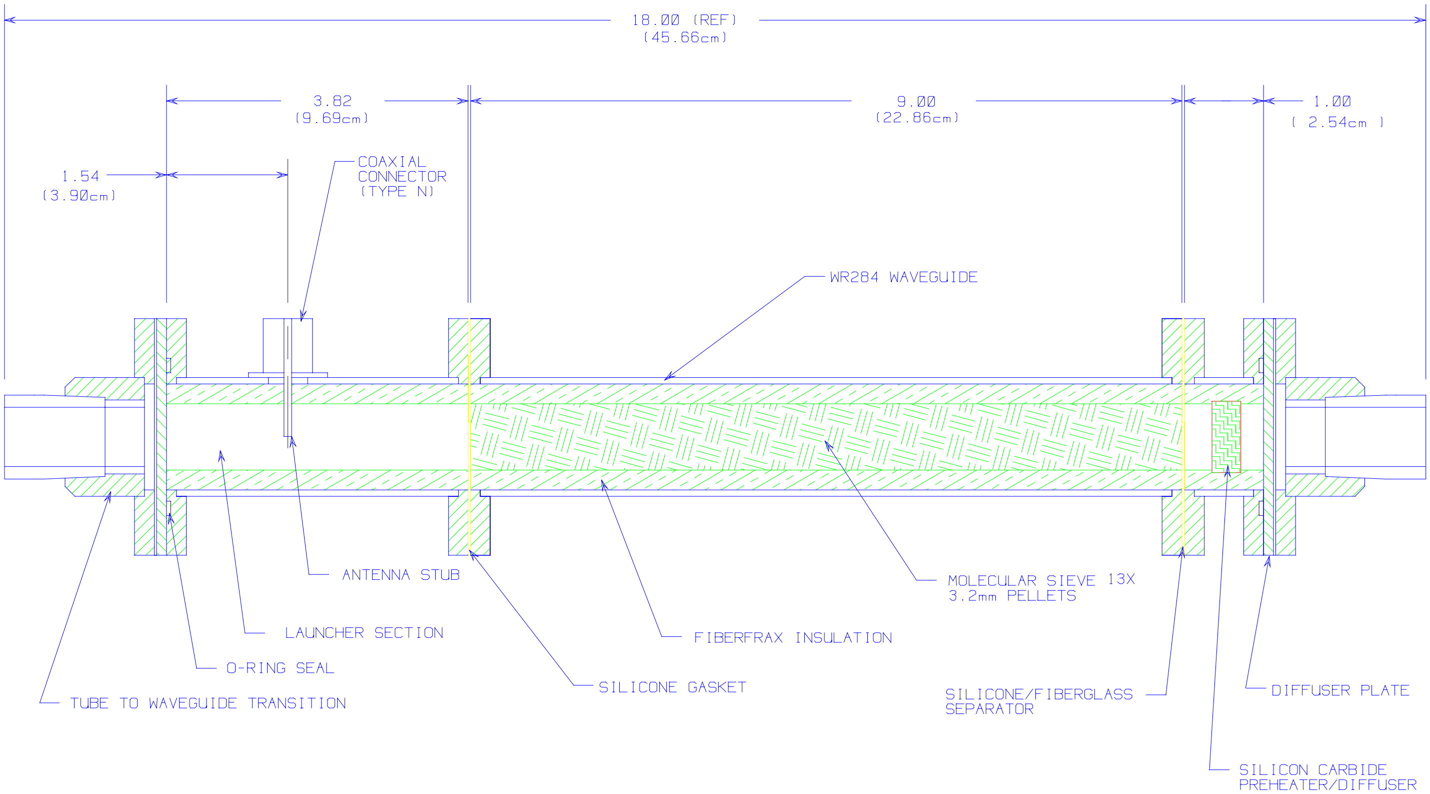

Figure 7. Molecular Sieve 13x Waveguide Cartridge.

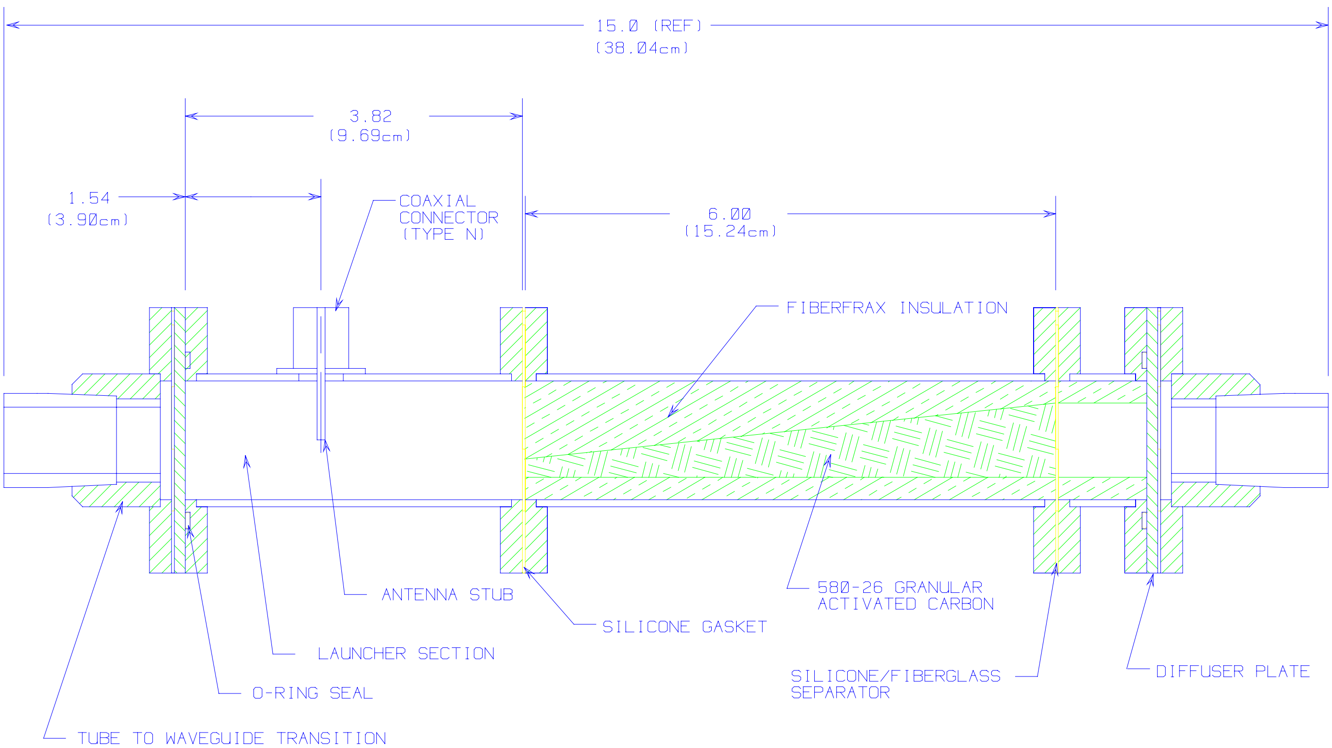

Figure 8. Activated Carbon Waveguide Cartridge.

On both ends of the cartridges, 1.91 cm (3/4") Swagelok™ to 1.91 cm (3/4") NPT adapters are threaded into custom fabricated air to WR284 waveguide adapters. These adapters were machined and corner welded to CPR - flat flanges. Silicone gaskets form air tight seals between the flanges and the 0.318 cm (1/8") aluminum diffuser plates. The coaxial cable-to-waveguide transition sections are 9.7 cm (3.82") from flange surface to flange surface. These pieces were assembled using commercially available flanges, waveguide tubing, coaxial panel mount connectors, and copper antennas. The 3.9 cm (1.54") separation distance between the antenna center and the diffuser plate was found to be ideal for maximum power transfer at 2.45 GHz from the coaxial cable to the WR284 waveguide. A special grooved flange seals against one side of each coaxial to waveguide transition, compressing a silicone rectangular O-ring trapped in the waveguide groove. The opposite end of each transition is sealed to the sorbent filled section of waveguide by a pair of silicone gaskets. Trapped between these gaskets is a piece of fiberglass fabric, which serves to confine the sorbent media in the waveguide section. Similar fiberglass fabric retainers are located on the opposite ends of the sorbent media sections. In the MS 13X cartridge, the coaxial cable-to-waveguide transition piece is internally insulated, to minimize condensation of water vapor within the waveguide.

The length for the MS 5A sorbent filled waveguide section was roughly determined by the sorption capacity and the CO_2 concentration representative of a typical life support air stream. The lengths of the MS 13X and activated carbon beds were driven by other considerations. The MS 13X bed was intentionally oversized to guarantee that in a sequential sorbent application, in which a contaminated air stream first passes through the MS 13X bed to remove water vapor, that no water vapor would ever enter a downstream MS 5A CO_2 removal bed and degrade performance. The activated carbon volume needed to remove typical trace levels of airborne organics is actually much smaller than that embodied in the cartridge design. It was established that in order to achieve uniform flow in the bed, a minimum waveguide size of 15.24 cm (6 inch) was necessary to achieve a Length/Diameter ratio > 2. (L/D > 2 is the minimum requirement from fluid dynamics for uniform flow through a channel).

The molecular sieve containing cartridges differ from the activated carbon cartridge in that silicon carbide blocks with holes drilled through to allow air passage, are placed on the ends opposite the microwave source in a 2.54 cm (1inch) section of waveguide. The silicon carbide blocks serve to distribute the air flow, and also to absorb any microwave radiation that passes through the molecular sieve sorbent material. Carrier gas flow during regeneration of these beds is countercurrent to the direction of applied microwave power. Thus, as the air enters the waveguide chamber, it initially comes in contact with the silicon carbide block, recovering heat generated in the block, and then passing into the sorbent bed, thereby enhancing the efficiency of regeneration.

The activated carbon containing cartridge differs from the others in that a wedge shaped sorbent bed geometry is employed, and a silicon carbide block is not needed. This cartridge was fabricated using a 15.24 cm (6 inch) length of Carborundum Fiberfrax® material with the same cross sectional dimensions as the interior of the WR284 waveguide (7.2 cm x 3.6 cm). A hollow wedge shape was carved into this block. The resulting monolith was placed inside a 15.24 cm (6 inch) length of WR284 waveguide, as measured from flange face to flange face. Barneby-Sutcliff 580-26 activated carbon was then used to fill the wedge shaped void in the Fiberfrax® monolith. A 2.45 cm (1 inch) waveguide spacer was placed on the side opposite the microwave source to allow air entering the bed during adsorption to be evenly distributed.

PERFORMANCE TESTING

Bench-top testing of each Waveguide Sorbent Cartridge designed for the Technology Demonstrator was performed using the apparatus shown in Figure 9. Microwave power was generated using a variable power 0 - 1.9 kW magnetron and transmitted to the waveguide cartridges through a coaxial cable connection. Applied and reflected power levels were measured via a solid state transducer attached to the directional coupler. Power levels, temperatures, and effluent contaminant concentrations were monitored in real-time and saved on disk for further analysis.

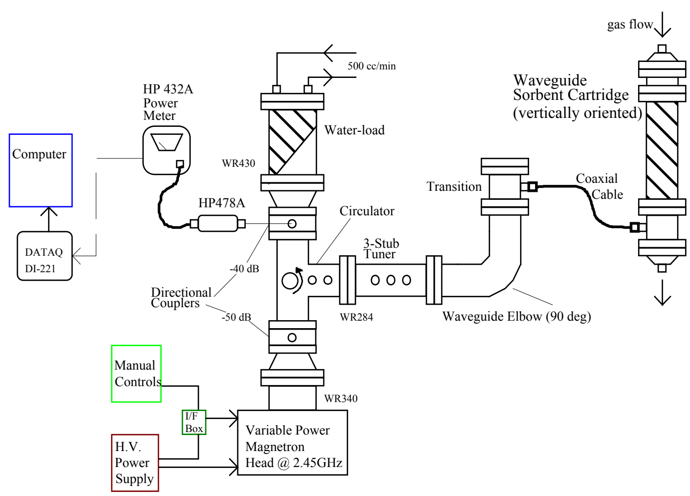

Figure 9. Technology Demonstrator Bench-top Testing Microwave Power Diagram.

The flow path varied with each waveguide cartridge. Variations included, the type of sensor(s) placed in the effluent air stream, the addition of oxygen to the effluent stream for VOC monitoring, use of a syringe pump to directly inject VOCs into the air stream at a controlled rate, a water trap placed in-line directly downstream of the MS 13X waveguide cartridge, and the type of the carrier or purge gas stream. During adsorptions, the contaminant effluent levels were allowed to rise to a point that was significantly below the influent level. Adsorption was then terminated. This was because, in most applications high levels of contaminant breakthrough are not acceptable, for either medical or regulatory reasons.

The performances of the various contaminant sensors used in the Technology Demonstrator were evaluated against conventional laboratory instruments. The two Viasala sensors (for CO_2 and for water vapor) performed at least as well as the laboratory counterparts. The solid state VOC sensor, however, proved to be very non-linear and exhibited significant calibration drift. This sensor, while failing to monitor sorption breakthrough concentrations accurately, functioned adequately for the control of thermal regeneration. The solid state VOC sensor was used initially to monitor the MEK sorption/desorption experiments. However, when calibration drift was recognized, further testing with toluene and trichloroethane utilized a Beckman on-line Hydrocarbon Analyzer.

Carbon Dioxide. During adsorption, pure CO_2 was bled into a dry air stream to achieve a total concentration of 1% (10,000 ppm). An air flow rate of 8 L/min was used. During regeneration, the microwave power level was 300 W. Nitrogen was used as the purge gas to carry desorbed contaminants from the cartridge at a flow rate of 4 L/min. Nitrogen flow was in the opposite direction of the gas flow during adsorption. Seven complete adsorption/regeneration cycles were performed, with 40% breakthrough occurring at: 1220, 1282, 1205, 1264, 1228, 1302, and 1239 L of cumulative throughput, respectively. These successive adsorption and desorption cycles as functions of throughput for carbon dioxide loaded on the MS 5A Waveguide Cartridge are shown in Figures 10 and 11, respectively. The sorption capacities were very consistent from cycle to cycle, with less than 5% variation from the mean throughput of 1249 L observed over all seven cycles. In addition, no net reduction in the capacity was evident. Clearly, the waveguide cartridge is both fully regenerable, and capable of sustained operation for removal of airborne CO_2.

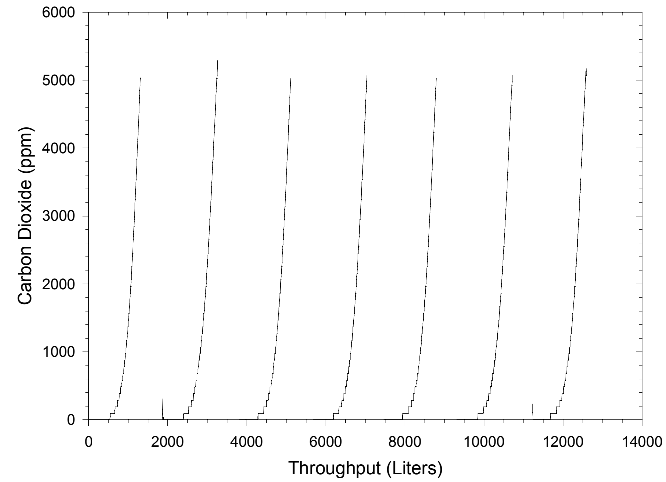

Figure 10. Multiple Carbon Dioxide Adsorptions for MS 5A Waveguide Cartridge.

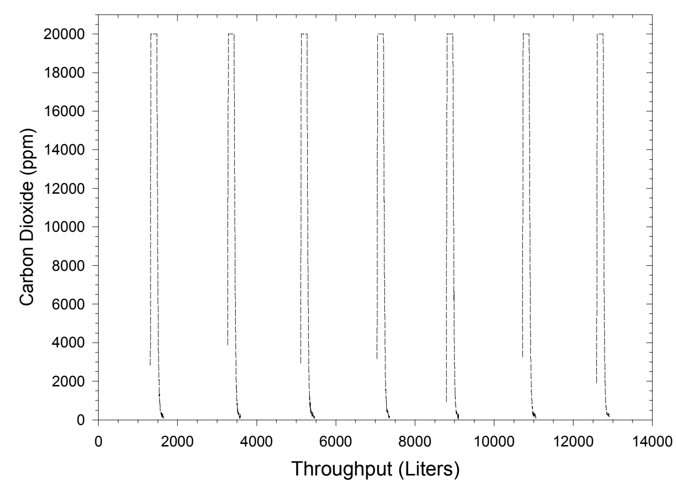

Figure 11. Multiple Carbon Dioxide Desorptions for MS 5A Waveguide Cartridge.

Water Vapor. As with the previous series of tests, the flow directions were reversed between adsorption and desorption to maximize heat transfer during regeneration. Compressed air was saturated with water vapor by sparging and then fed to the sorbent at a constant flow rate of 8 L/min. Thermal regenerations were conducted at 300 W incident microwave power, with a 4 L/min nitrogen carrier gas flow rate. Liquid phase water which condensed during regeneration was collected in water traps. When condensation ceased to occur, the water traps were bypassed for the remainder of the desorption cycle.

The test included five complete adsorption/desorption cycles. These are summarized in Figures 12 and 13. Throughputs of water vapor laden air to achieve 5% breakthrough were: 1511, 1497, 1505, 1656, and 1719 L, for the five cycles, respectively. No net decrease in sorption capacity was observed. The final two sorptions showed an increase in capacity, as compared to the earlier sorptions. Overall, less than 9% variation about the mean throughput of 1578 L was observed from the first cycle to the fifth. These results strongly support the validity of the MS 13X Waveguide Cartridge design.

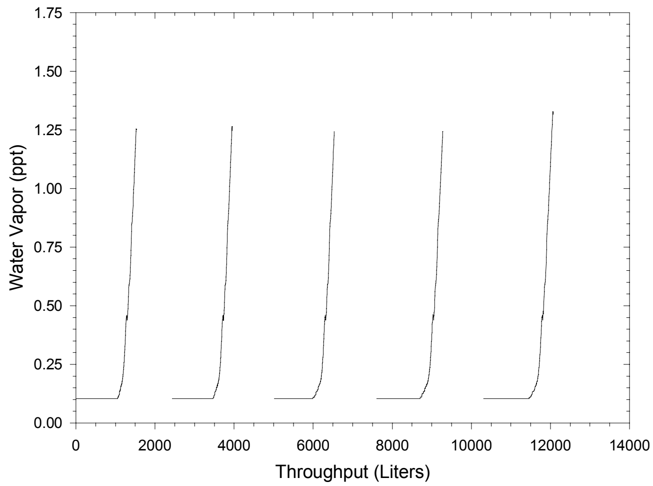

Figure 12. Multiple Water Vapor Adsorptions for MS 13X Waveguide Cartridge.

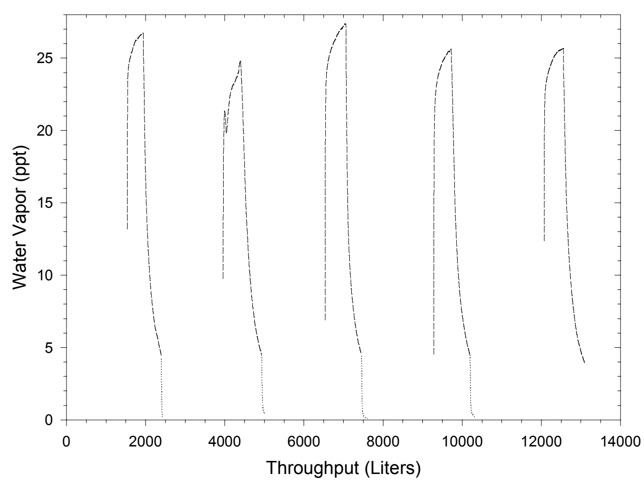

Figure 13. Multiple Water Vapor Desorptions for 13X Waveguide Cartridge.

Volatile Organic Chemicals (VOC). The Activated Carbon Waveguide Cartridge was designed for the removal of a broad variety of airborne VOCs. In separate tests, the cartridge was challenged with methylethylketone (MEK), toluene, and 1,1,1-trichloroethane each carried in a dry compressed air gas stream. While the presence of water vapor is known to suppress the adsorption capacity of VOCs on activated carbons, it has been shown (6) that significant capacity remains for VOC removal. The organic compounds, used in this study, represent oxygenates, aromatics, and halocarbons, respectively, three important classes of air pollutant. The volatility of each of these compounds has a two order of magnitude increase when the temperature is raised 200°C above room temperature. As activated carbon is heated in excess of 200 °C, the significant increase in equilibrium vapor pressure in the gas phase in contact with the adsorbed VOCs promotes the rapid desorption of the organics from the carbon surface. Due to time constraints, the potential thermal decomposition of these VOCs during microwave desorption was not investigated.

The Activated Carbon Waveguide Cartridge was first challenged with methyl ethyl ketone (MEK). The contaminant stream consisted of 1407 ppm MEK in dry compressed air. Flow rates of 4 L/min were used for both sorption and thermal regeneration via an inert nitrogen carrier gas. Thermal regenerations were conducted at a microwave power level of 34 W. The test was continued through nine complete adsorption/desorption cycles. The adsorptions and thermal regenerations are illustrated in Figures 14 and 15, respectively. The throughputs to achieve 8.6% breakthrough were 1233, 1089, 519, 503, 561, 671, 751, 697, and 693 L, for the sequence of nine cycles. A significant drop in the sorption capacity of this cartridge was observed following the second regeneration. The reason for this anomaly is unclear. However, the sorption capacity appeared to stabilize at the lower level.

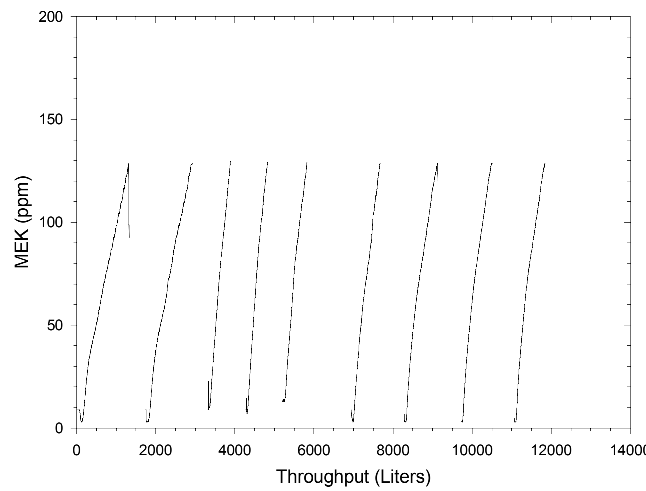

Figure 14. Multiple MEK Adsorptions for Activated Carbon Waveguide Cartridge.

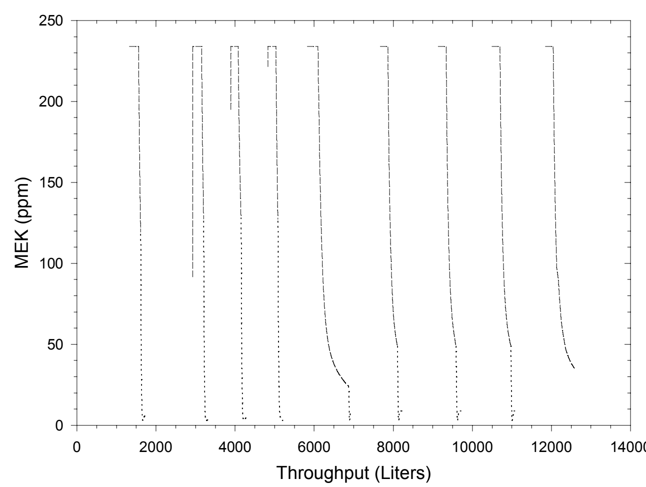

Figure 15. Multiple MEK Desorptions for Activated Carbon Waveguide Cartridge.

Similar experiments were conducted using a contaminant stream consisting of 1977 ppm toluene in dry compressed air. As illustrated in Figures 16 and 17, seven adsorption/desorption cycles were completed using this contaminant. For these experiments, the Beckman hydrocarbon analyzer was used as the real-time monitor. Significant calibration drift occurred. The data acquisition system also experience a failure during the second cycle. Cumulative flows required to achieve 10% breakthrough were: 1058, ------, 705, 852, 857, 689, and 623 L, for the sequence. Again, a significant decline of sorption capacity was observed following the second regeneration.

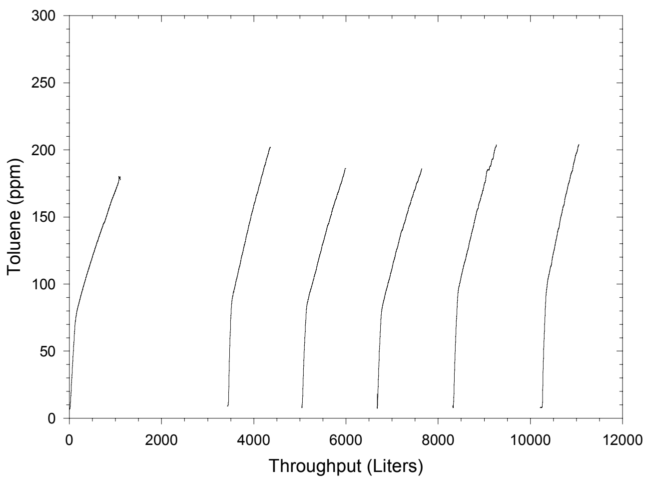

Figure 16. Multiple Toluene Adsorptions for Activated Carbon Waveguide Cartridge.

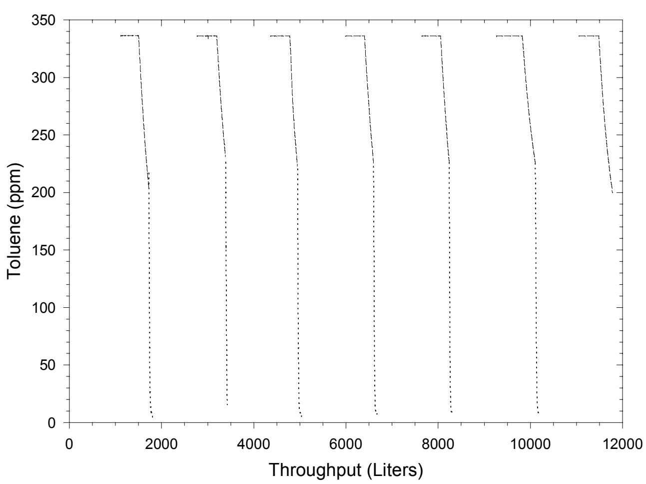

Figure 17. Multiple Toluene Desorptions for Activated Carbon Waveguide Cartridge.

The third VOC challenge of the Activated Carbon Waveguide Cartridge consisted of 1504 ppm of 1,1,1-trichloroethane (TCA) in dry compressed air. As with previous experiments, flow rates were 4 L/min for both sorptions and regenerations. Incident microwave power during thermal desorption was 38 W. Seven adsorption/desorption cycles were completed for this contaminant. The throughputs required to reach 6.7% breakthrough were: 732, 785, 521, 526, 493, 473, and 652 L, for the sequence. The experimental results for sorptions and desorptions are illustrated in Figures 18 and 19, respectively.

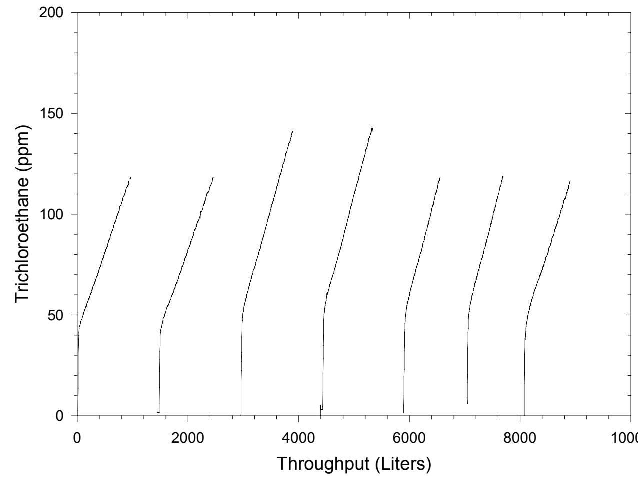

Figure 18. Multiple TCA Adsorptions for Activated Carbon Waveguide Cartridge.

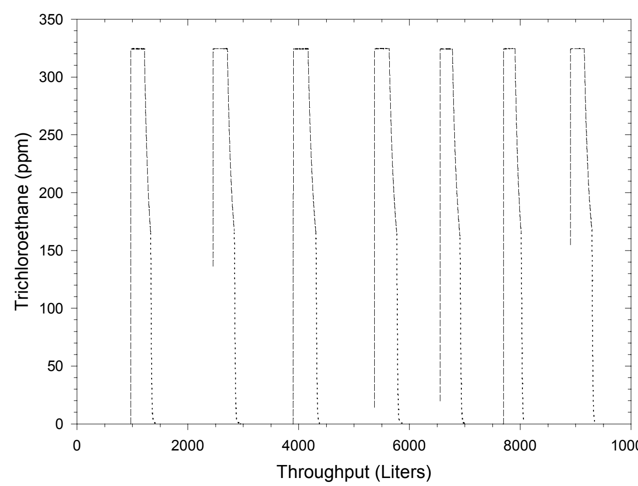

Figure 19. Multiple TCA Microwave Desorptions for Activated Carbon Waveguide Cartridge.

CONCLUSION

The Phase II research and development effort resulted in the design, fabrication, assembly, and testing of rectangular waveguide based thermally regenerable sorbent cartridges for the removal of airborne carbon dioxide, water vapor, and trace organic contaminants. The efficient microwave induced thermal desorption of loaded contaminant beds was demonstrated over numerous cycles of operation, with minimal input power levels. The Waveguide Sorbent Cartridges were then incorporated into the Microwave Regenerable Air Purification Technology Demonstrator, shown in Figure 20. This hardware will provide the capability for

Figure 20. Microwave Regenerable Air Purification Technology Demonstrator.

continued development of novel sorption based air purification methods for application in the fields of regenerative life support and ground-based environmental remediation.

ACKNOWLEDGMENTS

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