Leak Testing Filters Containing Activated Carbon Media with Different Moisture Contents Using a New Halide Detector

by W. Peter Freeman
NUCON International, Inc.
Columbus, Ohio

Abstract

Leak testing filters that contain activated carbon media that are contaminated (either with a high moisture content or other contaminants)has always presented a challenge for the test technician because of the difficulty in discerning the breakthrough of the test agent from a mechanical leak. One of the most successful techniques for solving this problem has been the use of the pulse injection technique which takes advantage of the chromatographic effect of the carbon media to separate the leak peak from the breakthrough. However, when the moisture (or other contaminant) content of the carbon is high, the time difference between the leak and the breakthrough can be shorter than the response time of the detector and a leak can not be distinguished from a breakthrough. One solution to this problem is to use a test agent whose breakthrough time is not affected as much by the adsorbed contaminants on the activated carbon. Typical examples of these kinds of agents are bromobutane, HFC43-10 (decafluoropentane) and PDCB (perfluorodimethylcyclobutane). Reported here are results of leak tests performed on activated carbon media (both plain carbon and carbon impregnated for radioiodine removal) using a new fast response detector developed by NUCON called the BBD halide detector. Activated carbon samples with moisture contents up to nearly 50 % were leak tested using pulse injection to determine the effect of the moisture content on the breakthrough time for R-11(trichlorofluoromethane), HCFC-123(2,2-dichloro-1,1,1-trifluoroethane), HFC43-10, PDCB and Bromobutane. These times were then compared with the times for a peak to appear from a known mechanical leak. Some of the results were surprising. For example HFC 43-10, although expected to be strongly adsorbed by the activated carbon based on its physical properties, could not be used successfully as a leak test agent when the moisture content of the media was near 50%. Test agents that were found to be successful for leak testing activated carbon with high moisture contents were additionally tested for their effect on the radioiodine removal performance by loading a given amount of test agent on the carbon and performing a radioiodine removal test according to ASTM D3803-95.

Introduction

The pulse mode injection technique was developed to perform in-place leak testing of the adsorber section of air cleaning systems where the breakthrough of the test agent is too rapid to distinguish from a mechanical leak.[1] Causes for this rapid breakthrough include poisoning of the adsorbent(including high moisture content) and high flowrates and/or short residence times. One solution to this problem has been to use a more strongly adsorbed test agent like PDCB(perfluorodimethylcyclobutane) and gas chromatography.[2] For similar reasons the British have used bromobutane as a test agent and the pulse injection technique along with a rapid response halide detector developed by NUCON.(3) Additionally, the military has replaced the leak test agent with HFC43-10(decafluoropentane)in an effort to use more environmentally benign test agent and to also use a test agent whose hold up on the adsorbent is not affected by moisture as much as R-11. HCFC123(2,2-dichloro-1,1,1-trifluoroethane has also been suggested as a replacement for R-11 for ASME N-510 leak testing.(4) Leak tests using these agents were performed on both plain carbon and impregnated nuclear grade carbon with moisture contents to up to 50 percent using the pulse injection technique to determine the affect of moisture content on the breakthrough time. These times were then compared to the time for a peak to occur resulting from a known mechanical leak using the same leak test agents. Test agents that could be used to distinguish a leak from breakthrough for nuclear grade carbons with high moisture contents were additionally tested for their effect on the radioiodine removal performance of these carbons. This was accomplished by loading different amount of test agent on the carbon and performing a radioiodine removal test according to ASTM D3803-1995.

Description of Test Procedure

A 2 inch diameter x 2 inch deep glass bed was filled with carbon using the method of ASTM D2454. The plain carbon for the leak tests met the requirements for base carbon of ASME N509-1980. The same carbon impregnated with KI and reacted amine salts had an efficiency of 99.76 % when tested according to ASTM D3803-1995. Weighed amounts of water were placed on the carbon to give the different moisture contents.

Leak test agents were injected upstream of the carbon bed into a heated manifold using a μL syringe. The concentration downstream of the carbon bed was monitored using a NUCON BBD halide detector calibrated for bromobutane. The downstream concentration for other leak test agents was converted to their corresponding concentrations using calibrations factors determined relative to bromobutane.(5) Air at 30°C was allowed to flow through the bed at 40 fpm. A mechanical leak through the bed was made by placing a plastic coffee stirrer vertically through the bed.

Presentation of Results

Table 1
Injection Leak Tests on Plain Carbon, Dry

Test Agent	Inlet Concentration, ppm	Outlet Concentration, ppb	Estimated Efficiency, %
R-11	10	0	≥99.99
Bromobutane	100	0	≥99.99
Bromobutane	300	0	≥99.99
HFC43-10	100	0	≥99.99

Table 1
Injection Leak Tests on Plain Carbon, Dry

HFC43-10	200	0	≥99.99

Table 2
Injection Leak Tests on Plain Carbon with 50% Moisture

Test Agent	Inlet Concentration, ppm	Outlet Concentration, ppb	Estimated Efficiency, %
R-11	10	1 @ 20 seconds	99.99
R-11	20	2@ 20 seconds	99.99
R-11	30	3@ 20 seconds	99.99
Bromobutane	100	1@ 400 seconds	≥99.99
Bromobutane	300	2@ 600 seconds	≥99.99
HFC43-10	100	32@ 9 seconds	99.97
HFC43-10	200	42@ 10 seconds	99.98

Table 3
Injection Leak Tests on Plain Carbon, Dry with Mechanical Leak

Test Agent	Inlet Concentration, ppm	Outlet Concentration, ppb	Estimated Efficiency.%
R-11	10	66@ 6 seconds	99.34
HFC43-10	100	413@ 6 seconds	99.59
Brombutane	100	72@ 12 seconds	99.93

Table 4
Injection Leak Test on Plain Carbon with 50% Moisture and Mechanical Leak

Test Agent	Inlet Concentration, ppm	Outlet Concentration, ppb	Estimated Efficiency, %
R-11	10	30@ 5 seconds	99.7
HFC43-10	100	198@ 8 seconds	99.8
Bromobutane	100	61@ 12 seconds	99.94

Table 5
Injection Leak Tests on Dry Nuclear Grade Carbon(7.6% Moisture)

Test Agent	Inlet Concentration, ppm	Outlet Concentration, ppb	Estimated Efficiency, %
R-123	100	5@ 120 seconds	>99.99
PDCB	100	3@ 20 minutes	>99.99

Table 6
Injection Leak Tests on Nuclear Grade Carbon with 20% Moisture

Test Agent	Inlet Concentration, ppm	Outlet Concentration, ppb	Estimated Efficiency, %
R-123	100	17@ 26 minutes(6 sec.)	99.98
PDCB	100	0	>99.99
HFC43-10	100	0	>99.99
Bromobutane	100	0	>99.99

Table 7
Injection Leak Tests on Nuclear Grade Carbon with 30% Moisture

Test Agent	Inlet Concentration, ppm	Outlet Concentration, ppb	Estimated Efficiency, %
R-123	100	>500 @ 8 seconds	<99.5
HFC43-10	100	18 @ 25 seconds	99.98
Bromobutane	100	0	>99.99
PDCB	100	0	>99.99

Table 8
Leak Tests on Nuclear Grade Carbon with 50% Moisture

Test Agent	Inlet Concentration, ppm	Outlet Concentration, ppb	Estimated Efficiency, %
R-123	100	>500 @ 4 seconds	<99.5
PDCB	100	26 @ 8 seconds	99.97
Bromobutane	100	0	>99.99

Table 9

Injection Leak Test on Nuclear grade Carbon with 50% Moisture and Mechanical Leak

Test Agent	Inlet Concentration, ppm	Outlet Concentration, ppb	Estimated Efficiency, %
R-123	100	>500 @ 6 seconds	<99.5
PDCB	100	39@ 8 seconds	99.96
Bromobutane	100	72@ 12 seconds	99.93

Discussion of Results

The efficiencies shown in the tables were calculated using the peak concentrations downstream and the upstream concentrations based on the amount injected. The results in Table 1 show that all of the leak agents used provide satisfactory results on the dry plain carbon. However, with 50% moisture on the carbon, we find rapid breakthrough for R-11 and HFC-43-10. In Table 3 with dry carbon and a mechanical leak, R-11 and HFC43-10 over estimate the leak when the peak heights are used. The same situation is seen in Table 4 for the carbon with 50% moisture and a mechanical leak for R-11 and HFC43-10 while the bromobutane gives a comparable result for the leak with or without the moisture.

The results in Table 5 on the dry nuclear grade carbon show that the R-123 and the PDCB provide satisfactory results. In Table 6 where 20% moisture has been added to the carbon, the R-123 shows a breakthrough that stars in 6 seconds and over estimates the leak. The PDCB, HFC43-10 and bromobutane still show no breakthrough. This moisture content corresponds to a % RH of about 70%. In Table 7 with 30% moisture added to the carbon, R-123 goes off scale on the detector almost immediately while the HFC43-10 shows a breakthrough after 25 seconds. The bromobutane and PDCB are unaffected. This moisture content corresponds to about 95% RH. In Table 8 with 50% moisture added to the carbon, R-123 is again off scale, while now the PDCB shows an early breakthrough at 8 second with the bromobutane again showing no breakthrough. In Table 9 the test is repeated with 50% moisture on the carbon with a mechanical leak added. Comparing the results for the previous table with this table show that the leak cannot be distinguished from breakthrough for R-123 and PDCB while the bromobutane gives satisfactory results. The 50% moisture content of the carbon is not usually encountered in air cleaning systems although the weapon destruction facilities are an exception.

In conclusion, the results show that the leak test agent bromobutane can provide satisfactory results even if the carbon has a moisture content of 50%. The leak test agent PDCB does the same for carbons that have up to 30 % moisture. This would include most situations likely to be encountered in the field with the possible exception as mentioned before of the weapons destruction facilities. The rapid breakthrough for PDCB when the carbon has a high moisture content means that it has little effect on the radioiodine efficiency even at 5% loading while the bromobutane, because of its strong adsorption on the carbon, reduce the radioiodine efficiency from 99.76% to 98.5% for the nuclear grade carbon used here and when tested to ASTM D3803-1995. Also it should be mentioned that a more accurate method of determining the leak(compared to using peak heights) has been developed at NUCON that allows the leak to be calculated by simply knowing the flow rate of the air cleaning system, the quantity of test agent injected and the area under the leak peak.

References

1. Kovach, B. J., et al, " Field Test Results of In-Place Charcoal Adsorber Leak-Testing by Pulse Mode Halide Injection", Proceedings of the 20th DOE/NRC Air Cleaning Conference, 1988.

2. Pearson, J. R., et al, "Replacement Tracer Agents for the In-Place Testing of Adsorbers in NATS", Proceedings of the 22nd DOE/NRC Nuclear Air Cleaning Conference, 1992.

3. Jones, J. W. F., et al, "Instructions for the Use of the NUCON Detector for Shipboard Testing of NBC Air Filtration Units", PLSD, Defence Evaluation and Research Agency, CBD. Porton Down, Salisbury, Wiltshire, 1997.

4. Banks, E. M., et al, "Halide Test Agent Replacement Study", Proceedings of the 23rd DOE/NRC Air Cleaning Conference, 1994.

5. Kovach, B. J., "Report on the Sensitivity of the NUCON BBD Series Halide Detector to Various Candidate Vapor Phase Challenge Agents Used for Leak Testing of Charcoal(Carbon) Adsorbers", NUCON 331, May 1997.