SESSION 9

ADSORPTION

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J. L. Kovach

RESIDENCE TIME DETERMINATION FOR ADSORBENT BEDS OF DIFFERENT CONFIGURATIONS
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E. M. Banks, W. P. Freeman, B. J. Kovach, J. L. Kovach, R. R. Sommer
RESIDENCE TIME DETERMINATION FOR ADSORBENT BEDS OF DIFFERENT CONFIGURATIONS

J.E. Otermat
W.O. Wikoff
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Abstract:

The residence time calculations of ASME AG-1 Code, Section FC, currently specify a screen surface area method, that is technically incorrect. Test data has been obtained on Type II adsorber trays of different configurations to establish residence time in the adsorber trays. These data indicate that the air volume/carbon volume ratio or the average screen area are more appropriate for the calculation of the residence time calculation than the currently used, smallest screen area basis.

Introduction:

The original adsorber residence time calculations of ASME/ANSI N-509 Standard were based on total screen area. The subsequently issued AG-1, Section FC, Mandatory Appendix I-1000, "Residence Time" calculations added new wording, changing from total screen area to an arbitrary value of the "gross screen area of all screens on inlet side or outlet side, whichever is smaller". This new definition, introduces a bias that favors adsorbers which are strictly parallel bed designs. Adsorbent beds, which do not conform to the parallel bed design, such as wrap around designs are penalized by the wording used. The easiest way to visualize the difference in residence time calculation is, by analogy with concentric adsorber beds, where the internal bed diameter is always smaller than the outer diameter. The current, major quantity military application adsorber, as an example, uses such configuration. The correct method of calculating residence time, for adsorbers with different inlet and outlet screen areas, is to use the average bed cross section and not the "smallest" screen area based flow path. The same thing is true for the wrap-around adsorber bed designs, discussed in Section FC of AG-1. In adsorption type unit operations, it is important to use strictly geometric average cross section for calculation, because the rate controlling steps of the adsorption process are also velocity dependent.

Test data has been generated to evaluate several Type II adsorber designs to establish actual residence time and to compare the adsorption based behavior with geometric configurations.

Experimental Methods.

Standard Type II adsorber cells of ASME/ANSI 509 cross section requirements (identical to the AG-1 Section FC cross section dimensions), but of different lengths and internal construction were filled with the same Lot/Batch adsorbent and tested in a wind tunnel at 333 SCFM. The pressure drop and the penetration of R-11 vapor were measured. The tests were conducted at ambient temperature and pressure in a wind tunnel where relative humidity and temperature were adjusted to result in uniform flow. The R-11 challenge vapor was introduced at a 1000 vppm inlet concentration and the outlet concentration was continuously monitored. The test was terminated when the adsorber tray outlet concentration reached 250 vppm R-11. The inlet and outlet concentrations were monitored by open diode type ionization detectors. The carbon fillable volume of the adsorbent beds was calculated both based on the physical measurements of the adsorber trays and from the adsorbent density and weight used to fill the adsorbent beds. All flow and R-11 penetration measurements were performed at 333 SCFM.

The adsorbent Lot/Batch used was in a moist condition, 19,0 wt. % water content, to increase the transport rate of the challenge gas through the adsorbent trays.
After filling, the adsorber trays were exposed to the ambient atmosphere for 30 minutes, then placed in the wind tunnel, where the 333 SCFM clean air was passed through the trays for 30 minutes prior to the initiation of R-11 challenge gas injection.

The breakthrough curves are shown on Figure No 1.

Test Results. Table 1.

<table>
<thead>
<tr>
<th></th>
<th>26 inch tray</th>
<th></th>
<th>30 inch tray</th>
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<tbody>
<tr>
<td></td>
<td>wrap around</td>
<td>parallel</td>
<td>wrap around</td>
<td>parallel</td>
</tr>
<tr>
<td>Adsorbent wt. lbs.</td>
<td>72.4</td>
<td>65.6</td>
<td>81.5</td>
<td>70.1</td>
</tr>
<tr>
<td>Pressure drop wg.</td>
<td>1.22</td>
<td>0.98</td>
<td>1.03</td>
<td>0.85</td>
</tr>
<tr>
<td>Cell volume cu.in.</td>
<td>3102</td>
<td>2770</td>
<td>3496</td>
<td>3019</td>
</tr>
<tr>
<td>Carbon volume cu.in.</td>
<td>3119</td>
<td>2826</td>
<td>3511</td>
<td>3020</td>
</tr>
<tr>
<td>Carbon based on ASTM AD</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Time to 100 vppm outlet conc. min.</td>
<td>5.75</td>
<td>5.00</td>
<td>7.18</td>
<td>6.77</td>
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<tr>
<td>Time to 250 vppm outlet conc. min.</td>
<td>12.52</td>
<td>11.43</td>
<td>15.75</td>
<td>14.43</td>
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</tbody>
</table>

Evaluation Results. Table 2

<table>
<thead>
<tr>
<th></th>
<th>26 inch tray</th>
<th></th>
<th>30 inch tray</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>wrap around</td>
<td>parallel</td>
<td>wrap around</td>
<td>parallel</td>
</tr>
<tr>
<td>Residence time based on AG-1, sec.</td>
<td>0.20</td>
<td>0.22</td>
<td>0.23</td>
<td>0.25</td>
</tr>
<tr>
<td>Residence time based on air/carbon volume, sec.</td>
<td>0.32</td>
<td>0.29</td>
<td>0.37</td>
<td>0.31</td>
</tr>
<tr>
<td>Residence time based on avg. screen area, sec.</td>
<td>0.24</td>
<td>0.22</td>
<td>0.27</td>
<td>0.25</td>
</tr>
<tr>
<td>Carbon weight/AG-1 residence time</td>
<td>362.0</td>
<td>298.2</td>
<td>354.3</td>
<td>280.4</td>
</tr>
<tr>
<td>Carbon weight/Air/Carbon Volume res.time</td>
<td>226.3</td>
<td>226.2</td>
<td>220.3</td>
<td>226.1</td>
</tr>
</tbody>
</table>

309
Evaluation Results. Table 2 (Continued)

<table>
<thead>
<tr>
<th></th>
<th>26 inch tray-wrap around</th>
<th>26 inch tray-parallel</th>
<th>30 inch tray-wrap around</th>
<th>30 inch tray-parallel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon weight/Avg. Screen Area res. time</td>
<td>301.7</td>
<td>298.2</td>
<td>301.9</td>
<td>280.4</td>
</tr>
<tr>
<td>Ratio of time to 250 vppm/carb wt.</td>
<td>0.173</td>
<td>0.174</td>
<td>0.193</td>
<td>0.205</td>
</tr>
<tr>
<td>Ratio of time to 250 vppm/AG-1 calc. residence time</td>
<td>62.6</td>
<td>52.0</td>
<td>68.5</td>
<td>57.7</td>
</tr>
<tr>
<td>Ratio of time to 250 vppm/Avg Screen area res. time</td>
<td>53.1</td>
<td>52.0</td>
<td>58.3</td>
<td>57.7</td>
</tr>
<tr>
<td>Ratio of time to 250 vppm/carbon vol. residence time</td>
<td>39.1</td>
<td>39.4</td>
<td>42.6</td>
<td>46.5</td>
</tr>
</tbody>
</table>

The data indicate that the residence time as derived from AG-1 Section FC Mandatory Appendix gives a misleading nominal residence time value which is biased toward parallel beds design. Either of the other two methods, i.e. the air to carbon volume or the average screen area (less baffles, etc.) gives a more correct actual residence time for any adsorber design, than the formula stated in AG-1, Section FC, Appendix I-1000.

**Conclusions:**

It is critical that formulae included in Code sections be based on sound technical information, and not favor any particular design style, as long as the other relevant parameters of the Code are met. It is equally important that changes and revisions of the Code sections are evaluated according to existing technical justification and supporting data and not on an arbitrary basis.

**DISCUSSION**

WREN: Did you look at the leakage rate as a function of flow rate? What kind of dependence do you expect?

KOVACH, L.: Leak rates were looked at under identical flow conditions. Our interest was mainly to determine whether different test agents, under the same conditions, gave an identical leak. Therefore, we used the standard conditions that are prescribed for this, i.e., 40 fpm through the carbon bed.

WREN: It is my understanding that *in situ* tests do not necessarily use the same flow rate that is expected during an accident.
KOVACH, L.: I agree with that, because a leak is basically an orifice. Therefore, if you use a different flow rate, you will have a different pressure drop, and that would mean a different leak quantity.

WREN: Do you want to speculate about what kind of dependence you would expect?

KOVACH, L.: No, but I think it would not be difficult, based on the standard orifice equation, to predict what the pressure differential would be based on an equivalent leak rate.

WREN: In your methyl iodide removal efficiency, what fraction was not trapped due to leakage, and what fraction due to penetration?

KOVACH, L.: The methyl iodine test was performed in a laboratory test rig, so there was no leakage. Leaks occur only in the full-sized leak equivalency test.

PEARSON: In your paper, you described the method of detection for the leak test, and you mentioned a gas chromatograph equipped with an electron capture detector. You stated that the gas chromatographic column’s purpose is to separate oxygen from the tracer gas. The real purpose of a chromatographic column is to separate the tracer gas from other interferences, such as Refrigerants-11, -112, -114, and -13B1, which are commonly found in varying amounts, hour to hour, minute to minute, in nuclear power plants. You also described an open diode detector. My question to you is, what method does the open diode detector use to separate the interfering substances commonly found during filter leak testing?

KOVACH, L.: We are using the installed carbon bed in the same manner that the chromatographic column is used.

PEARSON: When Refrigerant-11 is adsorbed, it is very likely that you will desorb Refrigerant-12 or -22, which can be mistaken for a leak, when, in fact, the material has nothing to do with a leak.

KOVACH, L.: I have not seen a case like that in any industrial application when using either instrument. There has been no problem determining whether there was a leak or not. I think tests that show that you get identical results when running tests, one after another, using different test agents are a good indication of that.

PEARSON: Equipment manufacturers have suggested that anyone using HCFC-123 in air conditioning equipment should install detection monitors. When running leak tests using HCFC-123, would you need detection equipment in the area to make sure that the recommended TLV is not exceeded? How would you go about assuring compliance?

KOVACH, L.: The Federal Register describes very clearly what is required and under what conditions. I can tell you that the answer is, no, you do not need to install any other detectors than the one you are using for the leak test with HCFC-123.

PEARSON: Even when you are filling your generator, or getting your test equipment ready?
KOVACH, L.: That is correct.

BELLAMY: A lot of chemical names appeared on your slide. What compound are you using today for in-place testing?

KOVACH, L.: R-11.

BELLAMY: How long do you expect R-11 to be available?

KOVACH, L.: The reason we are using R-11 currently is because you cannot use a substitute according to the code. Until there is a change in the code, you will have to use R-11.

BELLAMY: And the cost is basically immaterial, it just has to get passed along to somebody.

KOVACH, L.: At the present time, I do not think anybody has changed the price for testing based on the increased price of R-11.

BELLAMY: If I can take the liberty, I would like to ask an NCS representative what compound they are using today?

PEARSON: R-11

BELLAMY: So, everybody is still using R-11 compound simply because it is a regulatory requirement and that is what we will have to use until the code case is approved?

KOVACH, L.: That is correct. One of the questions that may not have come up directly is why we are still looking at other test agents. It is because EPA's regulations relating to the use of some of the alternative compounds is also changing. EPA is publishing, under the Stratospheric Protection Division's Questions and Answers on Ozone Depleting Refrigerants and Their Substitutes, almost all of the various CFC applications. Concerning one of the presumed inert materials, perfluorocarbons, the EPA statement is as follows: "CFCs may only be used where no other agent is technically feasible, due to the performance or safety requirements. The user must conduct an evaluation of the other alternatives and must determine that they either will not perform properly, or that they will pose a risk to human health. In addition, CFC use must meet conditions discussed in..." and they refer to other sections. EPA's requirement is to look at test agents based on perfluorocarbons and show that you tested everything and found nothing suitable before they will agree to using some other product. The published data on perfluorocarbon reactions with carbon, that decomposition of the perfluorocarbon occurs, made it not worthwhile to look at perfluorocarbons as potential alternatives. At some point we may look at them again, if we have to show that we tested everything else. We would rather start looking at those that may be suitable, rather than proving there are none. This is the reason we are not conducting work on perfluorocarbons at the present time.
HALIDE TEST AGENT REPLACEMENT STUDY

E.M. Banks
W.P. Freeman
B.J. Kovach
J.L. Kovach
R.R. Sommer

NUCON International Inc.

Abstract

The intended phaseout of the chlorofluorocarbons (CFCs) from commercial use required the evaluation of substitute materials for the testing for leak paths through both individual adsorbers and installed adsorbent banks. The American Society of Mechanical Engineers (ASME) Committee on Nuclear Air and Gas Treatment (CONAGT) is in charge of maintaining the standards and codes specifying adsorbent leak test methods for the nuclear safety related air cleaning systems. The currently published standards and codes cite the use of R-11, R-12 and R-112 for leak path test agents. All of these compounds are CFCs. There are other agencies and organizations (USDOE, USDOD and USNRC) also specifying testing for leak paths or in some cases for special life tests using the above compounds. The CONAGT has recently developed criteria for the suitability evaluation of substitute test agents. On the basis of these criteria, several compounds were evaluated for their acceptability as adsorbent bed leak and life test agents.

The ASME CONAGT Test Agent Qualification Criteria.

The test agent qualification is based on the following parameters:

1) Similar retention times on activated carbons at the same concentration levels as one of the following: R-11, R-12, R-112 or R-112a.

2) Similar lower detection limit sensitivity and precision in the concentration range of use as R-11, R-12, R-112 and R-112a.

3) Gives the same in-place leak test results as R-11, R-12, R-112 or R-112a.

4) Chemical and radiological stability under the use conditions.

5) Causes no degradation of the carbon and its impregnant or of the other NATS components under the use conditions.

6) Is listed in the USEPA Toxic Substances Control Act (TSCA) inventory for commercial use.

Current Test Agent Use Information.

The utilization of CFCs has been pioneered by the USDOD at Edgewood Arsenal, to determine the presence of leak paths and bed packing uniformity for adsorbent beds intended for chemical agent protection. The development of radiological vapor protection adsorbers for the USAEC Savannah River site production reactors lead to the application of this methodology for nuclear safety related adsorbers. Initially these adsorbers operated at an approximately 75 FPM velocity through a one inch deep carbon bed. The evaluation of the various test agents is reported in regard to the suitability of the test agent under these conditions. (1)(2)(3)(4)(5)(6). On the basis of the original studies R-112 was found to be suitable for the leak testing of both the shop testing of individual adsorbers and the leak testing of the installed adsorber banks. R-112a was found to be an acceptable alternative agent to R-112. The other lower molecular weight and lower boiling point compounds were found to be unsuitable, mainly for the in-place tests, due to the fast penetration of the adsorbent bed at the high ambient temperature and humidity conditions of the site.
The identification of the presence of organic iodine compounds and the commensurate lower removal efficiency and capacity by the conventionally used adsorbents, led to the increase in adsorbent bed depth to a minimum of two inches and the operation at 40 FPM velocity through the adsorbent beds. Under these design and operating conditions, some of the smaller molecular weight and lower boiling point CFCs were suitable for the determination of the absence of the leak paths in both the individual adsorbers and the installed adsorber banks. The industrial practice became using R-12 for the leak testing of individual adsorbers, because when filled with new adsorbent and tested under controlled environmental conditions the adsorptive retention time was sufficiently long to enable the differentiation between a leak and the penetration of the adsorption wave front through the adsorbent bed. At the same time, it was relatively easy to purge off the R-12 used in the adsorber leak test and prevent potential interference with the subsequent in-place leak test. The R-12 was not suitable for the performance of the in-place leak test, because after the build-up of common organic compounds in the installed adsorber beds and their equilibration with the environmental moisture content, the penetration of the R-12 was too fast. Therefore a slightly larger molecular weight and higher boiling point compound, R-11, was selected as the in-place leak test agent. There was no specific development work performed anywhere for the selection of the R-12 and R-11 test agents. The selection was based on the available data from the original Savannah River site studies. However, the standard writing organizations included the specification for these two compounds on a "grandfathering" manner, without further evaluation. (7)(8)

When the perceived concern about the survival of the CFCs in the upper atmosphere became legislated, the continued use of the CFCs for leak testing of adsorbent beds became a concern of the users, standard writing organizations, testing agencies and test equipment manufacturers. At the present time, the USEPA policy is that the use of CFCs in the nuclear industry does not violate the prohibition of the intentional CFC release rules. However, the phase out of the manufacture of CFCs and the yearly increasing excise tax costs for the still manufactured CFCs, is an important driver for the evaluation of suitable replacement agents.

Basically, the real technical criterion for a leak test agent is the detectability limit of the compound. The lower that limit, the less of the particular compound is used for leak testing. At the same time, it is important that the test agent is not strongly adsorbed on the particular adsorbent, otherwise the test becomes a destructive test. Because the current standards and codes specify a maximum leak rate of 0.05 % for the installed adsorbent banks, the required operating range for the test agent is in the 10,000 to 1 concentration range, thus the test instruments have to be able to detect the test agent in the same concentration range.

Currently, there are two typical detection systems in use. One is an electron capture detector utilizing radioactive ionization in the detector element and the separation of the oxygen peak from the test agent peak by the use of a chromatographic column. The test consist of extracting sub-milliliter quantities of sample from the air cleaning unit's air stream and injecting it into the column of the chromatograph. The other is an open diode detector, which is capable of detecting the presence of the test agent above the oxygen response on a continuous basis at sample flow rates of hundreds of ml per minute. The sensitivity of both detectors is in the ppb range for the conventional CFCs used as test agents and it is important that the same sensitivity range is maintained for the substitute test agents. There is also an economic reason for the utilization of the currently manufactured test equipment with the new test agents.

Alternative Test Agent Review.

In several countries, alternate test agents are being used even at the present time, because the US developed regulations and standards do not apply. The most commonly used in the non US nuclear safety related leak test application is non-radioactive methyl iodide or a mixture of radioactive and non-radioactive methyl iodide. While the detectability limit is satisfactory for methyl iodide by both commonly used detector systems and the penetration (retention) of the methyl iodide is not too different from that of the CFCs used in the past, the utilization of methyl iodide is complicated by the chemical hazards associated with it.

It is currently forbidden to air ship methyl iodide and it is a known carcinogen. The use of combination leak and efficiency test, using radioactive methyl iodide, is also dependent on the collection and detection limits of the upstream and downstream sampling cartridges, and the potential hazard of using radioactive compounds to determine the efficiency of the installed, but prior to testing, unknown efficiency air cleaning unit is questionable. The use of radioactive methyl iodide is also a health physics problem in US facilities due to current regulations.
Several NATO related agencies have selected 1-bromobutane as a test agent, after also evaluating chloroethane, iodomethane, chloroform, tert-butylchloride, chlorobenzene as candidate test agents and hexane-1-ol as a potential "poisoning" agent, (9) (10). The detectability limit for the 1-bromobutane is similar to that of the test agents used in the past. In the process of this test agent evaluation, an extensive study was also conducted determining the adsorptive and ambient environment effects on a parametric basis, (11).

NUCON has performed extensive testing with both HCFC-123 (a chlorofluorohydrocarbon) and 1-bromobutane as candidate materials. The behavior of the HCFC-123 is near identical in properties and leak test performance to the R-11. The 2-bromobutane is between the R-12 and the R-11 in chemical properties and the same as the R-11 and R-112 in leak test performance.

NCS Inc. has reported on the use of perfluorocompounds as potential test agents, (12). The perfluorocompounds are easily detected by electron capture detectors, but less easily by open diode detectors.

The NCS proposed optimum material, Dodecafluorodimethylcyclobutane (PDCB) was claimed as similar in leak test performance to that of R-11. (12)

**Future Regulation Considerations.**

The USEPA, while currently not prohibiting the use of CFCs for leak testing is discouraging its use for any application by the phase-out of manufacture and the high excise tax applied. It is also expected, that barring additional technical evidence relating to the behavior of HCFC materials, their use will also be restricted in future years. The USEPA ruled for many current applications of replacement for R-11, the use of HCFC-123 as "acceptable". (13) It is important to keep in mind, at least for US applications, that the USEPA ruled that "It is illegal to replace an ozone depleting chemical with a substitute listed by the Significant New Alternatives Policy (SNAP) as "unacceptable".

Recently, the EPA has advised of restrictions on the use of perfluorocompounds (PFC) for other than essential use. "PFCs may only be used where no other agent is technically feasible due to performance or safety requirements. The user must conduct an evaluation of the other alternatives and must determine that they either will not perform properly or that they will pose risk to human health". The restriction is based on the long term environmental survival of perfluorocarbons in the environment, (14). Thus, while multi-halogenated compounds are ideal from the detectability standpoint, the use of chlorofluoro, hydrogenchlorofluoro or perfluoro compounds does have some future use regulation uncertainty.

**Potential Test Agent Properties.**

<table>
<thead>
<tr>
<th>TEST AGENT</th>
<th>BP</th>
<th>MW</th>
<th>TSCA</th>
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<tbody>
<tr>
<td>Chloromethane****</td>
<td>-24.2</td>
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<td>R-12***</td>
<td>-29.8</td>
<td>120.92</td>
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<td>Chloroethane*</td>
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<td>R-11***</td>
<td>23.7</td>
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<td>HCFC-123</td>
<td>27.6</td>
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<tr>
<td>Iodomethane*</td>
<td>42.5</td>
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<td>PDCB**</td>
<td>45.0</td>
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<td>51.0</td>
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<td>Chloroform*</td>
<td>61.3</td>
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<td>Chlorobenzene*</td>
<td>132.0</td>
<td>112.56</td>
<td>yes</td>
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</table>
Adsorption Considerations for Test Agents.

There are different considerations for the leak test agent, when it is used on new adsorbent beds which have low moisture content and no "poisoning" contamination and for "in-place" leak testing agents, which need to differentiate between a mechanical leak and the penetration of the test agent through the adsorbent bed in installed systems, which may have unknown quantities of adsorbed moisture and other contaminants.

According to the work of Smith(11), the adsorbent moisture content interference effect becomes strong when the boiling point of the adsorbate is below 85 C. Thus, if a compound is selected for the determination of the mechanical leak, under most "as is" condition, installed adsorber banks, it is advantageous to use compounds with a high boiling point. In this case, there is a longer residence time of the test agent on the adsorber bed, however, the quantity of test agent used does not need to be at a level that would significantly influence the radiiodine control properties of the adsorbent, even after repeated testing. If the pulse mode leak test technique is applied, then even less test agent is used.

For the in-place test agent, it is also preferable, if the test agent is not identical to compounds commonly present in the plant environment, where the adsorbent banks are installed. This parameter was not considered originally, when the common refrigerant compounds were proposed and standardized into leak test methods. It would also be advantageous if the same test agent could be used for both nuclear safety system and military chemical agent protection adsorber banks, containing different adsorbents and various adsorbent bed depths, operated under varying face velocities.

The quality control testing on the freshly filled or refilled adsorbers, tested under controlled conditions, does not have the above discussed, high boiling point test agent, requirement.

For quality control testing of individual adsorbent beds, it is advantageous to use a test agent, that is easily stripped from the adsorbent, so no potential interference would exist when the installed adsorber bank is tested. However, it is possible to use the same test agent for both applications, if the quantity used is small (for example, as in pulse mode leak testing).

Equivalency of In-Place Leak Test Results.

A 6000 CFM full NATS installed at NUCON International Inc. has been used to evaluate the equivalency of the in-place leak test results using the different test agents on the full size systems. One of the sampling cartridges has been removed to simulate the leak. The total airflow, temperature and relative humidity were identical for the test series at 27 C, 55% RH.

The leak percentages determined using the NUCON F-1000 test agent generators and detectors were as follows:

<table>
<thead>
<tr>
<th>Test Agent</th>
<th>Continuous Test</th>
<th>Pulse Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>R-11</td>
<td>0.26%</td>
<td>0.27%</td>
</tr>
<tr>
<td>HCFC-123</td>
<td>0.25%</td>
<td>0.26%</td>
</tr>
<tr>
<td>1-bromobutane</td>
<td>0.26%</td>
<td>0.27%</td>
</tr>
</tbody>
</table>

The inlet concentration, for each of the test series, was either a continuous ~ 15 vppm, or three successive 6.0 ml pulses. The downstream detector was always connected to the duct downstream of the blower, to assure good mixing. The injection point was verified according to ASME/ANSI N-510 requirements.
The lower detectability limits for the evaluated compounds using the F-1000 instruments were as follows:

- R-11: 0.75 vppb
- HCFC-123: 1.00 vppb
- 1-bromobutane: 1.00 vppb
- Bromochloromethane: 0.75 vppb

It is expected that other currently used test agent detector instruments have similar detection sensitivity levels.

The change in radioactive methyl iodide penetration of adsorbent beds (NUCON NUSORB KITEG II, Lot 55, Batch 54), when exposed to 0.5% by weight of each of the above test agents, which corresponds to approximately 500 in-place leak tests total test agent load, was insignificant. (Within the error limit of the tests.)

Unexposed: 99.66% efficiency at 30 C, 95% RH,
R-11 exposed: 99.60%
HCFC-123 exposed: 99.55%
1-bromobutane exposed: 99.30%

It is expected that longer than 2 hours of sweep time would displace more of the test agents and the co-loading effect would even be less if the NRC required air sweeps are performed on a monthly basis on the NATS. It is estimated that an adsorbent bed installed in a NATS may see approximately 50 tests in its life.

So the above test was conducted at 10 times higher loading than the expected normal test agent load.

In connection with the evaluation of organic compound poisoning of adsorbent beds, a detailed study was made of the quantity of organic compounds that would result in a significant effect on the radioactive methyl iodide removal efficiency of the adsorbent.

While, for that particular case, not the most stringent test conditions were used, the data indicated that higher than 1 wt. % loadings of organic compounds are required to show differences in the adsorbent performance. (15)

A technical evaluation of the potential effect of the proposed test agents on other NATS components was based on the effects of potential dissolution of HEPA filter potting and sealant compounds and the potential detrimental effect on the commonly used gasket materials, neoprene and silicone rubber. In the quantities used there were no measurable effects.

The retention on ASME/ANSI N-509, 1980 grade activated carbon for the test agents was performed under identical process conditions of 40 FPM, 30 C and preequilibrated at the appropriate %RH, on a two inch deep bed. The results of a 5 minute, 20 vppb test agent penetration test through the adsorbent bed were:

<table>
<thead>
<tr>
<th>Test Agent</th>
<th>Penetration time to 10% breakthrough on Preequilibrated Adsorbent</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Minutes At 95 %RH</td>
</tr>
<tr>
<td>R-11</td>
<td>1.3</td>
</tr>
<tr>
<td>HCFC-123</td>
<td>1.9</td>
</tr>
<tr>
<td>1-bromobutane</td>
<td>120 +</td>
</tr>
<tr>
<td>R-112</td>
<td>120 +</td>
</tr>
</tbody>
</table>
So far, only screening tests were performed with chloromethane, as a potential substitute for R-12, in the quality control tests on freshly filled adsorbers. The preliminary data indicates, that the substitution will be probably acceptable, however additional data needs to be generated.

Conclusions from the Test Program.

On the basis of the evaluation of the properties and the performance of actual in-place leak tests and of the other relevant parameters of utilization of the evaluated test agents, it was found that either HCFC-123 or 1-bromobutane are acceptable substitutes for the R-11 in-place leak test agent.

Based on the very extensive testing at Porton Down (11), the 1-bromobutane is not affected by the expected in situ moisture content of the adsorbent or the potentially existing "poisoning" compound levels in either nuclear or military applications.

None of the test agents evaluated so far, were found to adversely effect either the adsorbent performance or the downstream NATS components at the loading levels expected in NATs in-place leak testing.

It is expected from the screening tests performed with electron capture detectors, that other manufacturer’s test instruments would have similar detector sensitivity as that of the NUCON instruments.

Additional compounds are being tested to further optimize test agents under the CONAGT criteria. Of these bromochloromethane is currently in the evaluation stage for replacement of R-11 and chloromethane for the replacement of R-12.

References.


