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A Testing Protocol for Organic Vapor Respirator Canisters and Cartridges

GERRY O. WOOD[A] and MARK W. ACKLEY[B]

[A]Los Alamos National Laboratory, Industrial Hygiene Group, Mail Stop K-486, Los Alamos, NM 87545; [B]Western New York Technology Development Center, 1576 Sweet Home Road, Amherst, NY 14221

A generic test plan has been developed for laboratory measurements of the performance of a selected organic vapor canister or cartridge against a selected vapor. It assumes that the test system is set up, the canisters or cartridges are prepared, and the capability for interpreting the resulting data exists. Portions of breakthrough curves are determined for a reference set of conditions and for ranges of bed depths, airflow rates, vapor concentrations, relative humidities, and temperatures. The minimum number of tests required to quantify effects of all the above parameters has been identified as 25. Objectives in choosing this protocol have been to (1) optimize the number of tests, (2) optimize the amount of information obtainable, (3) have the results in a form which can be correlated by any one of a variety of mathematical models, (4) define data reproducibility, (5) allow scaling of results to other bed geometries for evaluation or design purposes, and (6) provide a visual representation of results for field use.

Introduction

Performance data are required for initial design, improvements, and evaluations of organic vapor canisters, cartridges, and other activated carbon beds and for making decisions on using them in the field. Experiences in government and industrial testing laboratories have indicated a need for additional guidelines for testing canisters, cartridges, and sorbents. Factors affecting the performance of organic vapor cartridges have been reviewed elsewhere.[1-3]

The variety of potential use conditions (breathing rate, prior canister history, *etc.*) and environmental conditions (relative humidity, temperature, *etc.*) is one factor to consider in choosing laboratory testing conditions. In field applications these seldom are easily controlled. Canister designs and the carbons they contain also vary with manufacturer and even with time of manufacture. The types of vapors against which protection is required are diverse because of the scope of chemicals used in industry.

The resources available for testing always are limited. Testing equipment is complex and subject to frequent maintenance and calibration. Trained personnel are not always readily available, and testing is costly.

Considering the complexity of this situation, the best hope for defining the overall capability of a given canister, cartridge, or pair of cartridges is to select testing conditions which will define the fundamental parameters of the carbon/vapor interactions and the influences of environmental conditions on them.

Scope

In this paper the focus is on a testing protocol, including suggested conditions and ranges of variables, and the bases for selecting these. The authors assume as prerequisites that (1) a testing system is set up and ready for use, (2) the test canisters, cartridges, or beds are prepared for testing, and (3) a data analysis capability exists for extracting fundamental parameters and presenting data in a useful format.

Although the protocol description is presented for canisters and cartridges, it also can be scaled to apply to other packed carbon beds. Cartridges used in pairs on an air-purifying respirator should be tested in parallel in pairs, or the airflow rates should be halved for testing them singly.

The protocol is limited to a single gas or vapor, except for the ever-present covapor, water. Additional vapors can be added for multivapor testing once the removal of each single vapor has been defined. Filtration of particulates is a completely separate matter and is not considered here.

Protocol Objectives

The objectives in developing this protocol have been to

(1) Optimize the number of tests required to define performance for a particular vapor removal application
(2) Optimize the amount of information from testing, so that effects of environmental and use conditions on performance can be characterized
(3) Provide testing results which can be correlated by any one of a variety of mathematical models which have been or may be proposed
(4) Include enough test repetitions for at least one set of conditions so that a measure of the test data reproducibility can be defined
(5) Allow for scaling of the results to other bed size, geometry, packing density, sorbent particle size, *etc.*
(6) Provide a visual representation of results for field use.

Preconditioning

Canisters should be prepared for testing to match the condition they are expected to be in before actual use. For example, if the canisters are hermetically sealed until immediately before use, they should be tested in the laboratory "as-received." On the other hand, they may be expected to be used at or exposed to ambient temperature and humidity conditions for long periods prior to challenge by the vapor of concern. If so, the expected water (or other contaminant) loading should be determined and the canisters preconditioned to match.

In the absence of any guidance on preuse conditioning, the canisters can be preconditioned to a constant weight loading corresponding to 50% relative humidity (RH). This may take up to 48 hr with 50% RH airflow through the canister. If canisters are not preconditioned at the testing humidities, heating or cooling of air passing through them will occur because of water adsorption or desorption, respectively.[4] These latter situations are more difficult to treat theoretically. Canister equilibration at 50% is an intermediate water loading.[5]

In any case, the amount of water present as-received should be determined by carbon drying. The amount added or removed prior to testing should be determined by weight change upon humidity equilibration (if any) of the original bed. This information can be useful in characterizing the carbon and identifying possible improvements in manufacturing and storage.

Phase I: Initial Testing

A flow chart summarizing the specific recommendations for the initial phase of testing is shown in Figure 1. Phase I testing requires at least nine tests (three reference + two bed weight doubling + four flow rate varying) with 11 canisters or 22 cartridges tested in pairs. The extra units are for double-depth tests. Discussions of the choices for initial test conditions follow.

Reference Conditions and Reproducibility

The first step is to select a reference set of conditions for testing. A high volumetric flow rate, Q, of 64 L/min represents a high respiratory minute volume.[6] Steady, rather than cyclic, flow is recommended for simplicity, although cyclic flow may be used.[7] Ambient temperature, T, near 25°C is attained and maintained most easily. A 50% RH for testing represents an intermediate value for the possible range.

The initial challenge concentration, C_{oi}, of the chosen vapor should greatly exceed (preferably by at least 100 times) TL, the permissible exposure limit (threshold limit value [TLV®], permissible exposure limit [PEL], *etc.*), but also give practical testing times. The initial suggested limits (between 20 min and 4 hr) also were selected to give practical breakthrough times for subsequent testing.

A portion of the breakthrough curve should be measured—from the minimum detection limit, which should be no more than 1% of challenge concentration, to at least 10% of the challenge concentration. The maximum allowable breakthrough concentration, defined by the application, should be included. The data should span at least a factor of 10 (preferably at least 50) in breakthrough concentration to allow derivation of both capacity and kinetic parameters for each test.

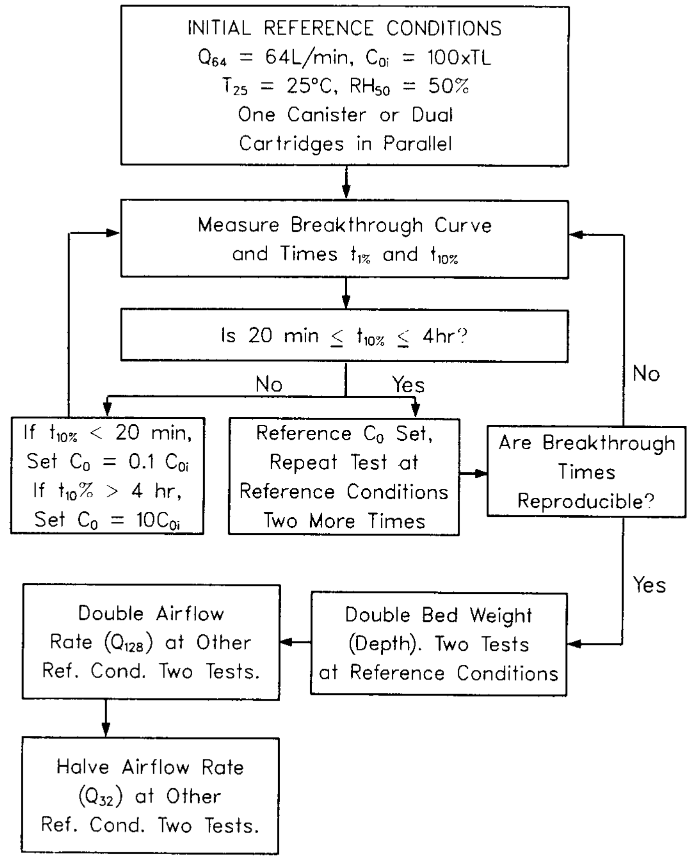

Figure 1—Phase I: Basic testing protocol to determine experimental reproducibility, bed depth effects, and airflow rate effects.

At least triplicate tests should be run at the selected set of reference conditions to define the combined reproducibility of the test and the integrity of the sorbent bed packing. Subsequent tests should be done at least in duplicate at each set of conditions. If the range of any breakthrough time (maximum value minus minimum value) exceeds 20% of the average (estimated relative standard deviation exceeds 12%),[8] then the following steps should be taken: (1) repeat measurements and eliminate proven outliers, (2) improve testing equipment and procedures, and/or (3) investigate the uniformity of canister packings.

Bed Depth and Airflow Velocity Effects

Constant pattern of the adsorption wave in a packed bed is defined as no additional spreading of the wave (and, therefore, the breakthrough curve) as it passes further through the sorbent.[9] This assumption is basic to most models of breakthrough behavior. It can be confirmed only by deepening the bed (*e.g.*, putting two canisters in series) and observing the relative shapes of the breakthrough curves. Mathematically, differences in times for two bed depths (D_B) should be nearly the same at each selected breakthrough fraction (*e.g.*, 0.01 and 0.1). If this is not found to be the case, no constant pattern has been formed in the first canister, and models based on this assumption do not apply. Testing following this suggested protocol still will provide useful data for estimating service lives, however.

Airflow rates (velocities) double and half the reference rate provide a wide enough range to determine effects on kinetic parameters and, perhaps, mass transfer rate limiting mechanisms.[9-11]

Phase II: Environmental Effects Testing

While bed size and airflow velocity can be controlled to some extent by canister design, environmental conditions at the point of application must be taken as they are. Therefore, the effects of environmental parameters (vapor concentration [C_o], relative humidity [RH], and temperature [T]) on canister service life (at a predefined breakthrough concentration [C]) must be known by the user. Phase II testing requires at least 16 tests (2 C_os × 2 RHs × 2 Ts) with 16 canisters or 32 cartridges used in pairs.

Experimental Design

The two-level factorial experimental design[12-14] is proposed for testing effects of these three environmental conditions on canister breakthrough times. The advantages of this approach are (1) it yields more and more precise information about the effects of the variables, (2) it provides a way of identifying suspect results, (3) it avoids the bias which may result from varying only one parameter at a time, and (4) it provides a graphic presentation of the parametric effects, which is useful for determining service life and, therefore, applicability in actual field situations.

Two levels of each parameter in all combinations form the corners of a cube when plotted on three axes of Cartesian coordinates (Figure 2). When these levels are the extremes of each parameter, the volume of the cube represents all possible conditions. Values of the dependent variables (breakthrough times) are determined experimentally at the corners (sets of challenge concentration, relative humidity, and temperature conditions) and repeated. Values at intermediate conditions can be estimated by looking at the three-dimensional representation. Tentative extrapolation beyond the test range also can be made by estimating or, better, by using proven mathematical models. This should be done with caution, however, and confirmed experimentally, when possible.

Parameter Range Selection

Challenge concentrations 10 times and 1/10 the selected reference concentration (see Phase I above) provide a wide enough range to include a variety of applications. Data at these points plus the intermediate (reference) point data can be used to define effects of RH and T on capacity parameters (*e.g.,* adsorption isotherms).

Testing should be done at high and low extremes of relative humidity. It is not easy to exceed 85% RH, however, without condensation on cold spots in the apparatus. It may be preferable to extrapolate results to higher relative humidities. Therefore, 85% RH has been selected as the upper level for this test protocol. The dry bed and dry air condition is of importance in defining the single-component capacity (adsorption isotherm); however, if completely dry air is not available, the lowest humidity practically attainable should be used. Adsorbing water vapor usually reduces a carbon's capacity for adsorbing other vapors. When the vapor removal mechanism is by reaction, however, water actually may enhance the process.[9] Comparisons of effects of high, low, and intermediate RHs can be used to identify the critical range of effects and to select additional testing relative humidities.

Temperature is one of the most difficult parameters to control in canister testing. Temperature effects on canister service life vary with the mechanism of vapor removal. Physical adsorption capacities are reduced, but diffusion, catalysis, and chemical reactions usually are enhanced at higher temperatures. Therefore, a range of 25 ± 10°C (*e.g.,* 15 and 35°C) is useful for preliminary identification of these effects. Again, additional testing temperatures (intermediate or more extreme) may be suggested by the observed effects on service lives. It is important to remember, however, that at extreme low or high temperatures, physiological conditions may become more limiting to respiratory protection than canister or cartridge performance.

Data Reporting

All the breakthrough curve data (breakthrough concentrations versus times) for each specified set of preconditioning and testing conditions should be tabulated and made acces-

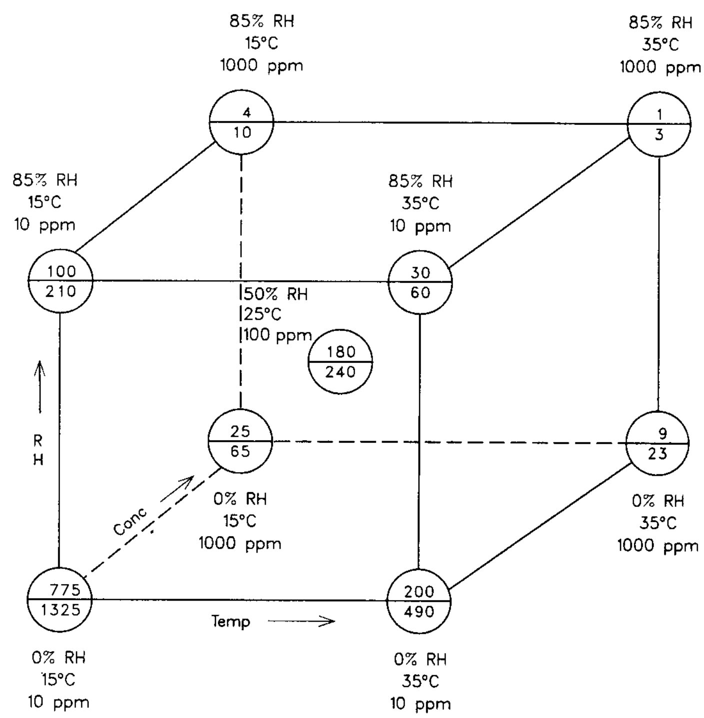

Figure 2—Sample two-level factorial analysis representation of service life data. The numbers in the circles represent average 1-ppm breakthrough times (min) for 50% RH preconditioned canisters (top half circle) and as-received canisters (bottom half circle) at some specified set of testing conditions.

TABLE I
Proposed Values of Parameters for Preliminary Canister Testing

Parameter	Low	Reference	High
C_o[A]	10 × TL[B]	100 × TL	1000 × TL
RH[C]	dry	50%	85%
Q[D]	32 L/min	64 L/min	128 L/min
T[E]	15°C	25°C	35°C
D_B[F]	—	1 canister	2 canisters in series

[A]C_o = vapor concentration
[B]TL = permissible exposure limit
[C]RH = relative humidity
[D]Q = volumetric flow rate
[E]T = ambient temperature
[F]D_B = bed depth

sible to others. Such data can be used in testing and applying empirical and theoretical correlations for predicting canister efficiencies and service lives. Graphical plots of breakthrough curves can be useful for showing service lives and environmental effects on them. The canister (test bed) specifications (sorbent weight, volume, and type) also must be reported in as much detail as is available.

Data Presentation

The two-level factorial method allows the summarization of service life test results for the proposed sets of testing conditions in the cube representation shown in Figure 2. The numbers given for service lives in the circles within the cube (reference conditions) and at the corners (combinations of two levels for three parameters) are for illustration only. In this example, the canister obviously is useless (< 5 min service life) near or above 85% RH, 35° C, and 1000 ppm; but it has a useful service life for more moderate conditions. A fresh canister (not preconditioned) tested at 85% RH and 35° C against 10 ppm of vapor gave an average service live of 60 min (upper, front, right corner). At lower humidity and/or temperature, a longer service life can be expected (moving down and/or left on the front face of the cube).

Two service life values are given in Figure 2 at each set of environmental conditions, one for each of two initial canister conditions, as-received and preconditioned. Such a representation could show data for two breakthrough levels, two different canisters, two work (airflow) rates, two separate vapors, *etc.*, just as well.

Conclusions

This canister, cartridge, or packed-bed testing protocol can be used to provide consistent and comparable data useful for

(1) Deciding on the applicability and service life of a given unit for removing a given vapor under a variety of environmental and use conditions
(2) Designing new or improved canisters, cartridges, and sorbents
(3) Testing and development of models for describing and predicting sorbent bed performance

Table I summarizes the proposed values of the testing parameters.

A minimum of 25 tests (9 in Phase I + 16 in Phase II) with 27 canisters or 54 cartridges used in pairs has been identified as necessary. The results may suggest additional tests at intermediate or more extreme vapor concentration, relative humidity, and/or temperature conditions.

Acknowledgment

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