Nuclear Carbon Testing

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A. Proposed Standard Test Method For Nuclear Grade Gas Absorbers In Cartridge Containers (based on modifying ASTM D3803)

B. Auditing ASTM D3803 Testing – A Practical Guide

Nuclear Carbon Testing

C. Commentary to Current Practice

1. Regulatory Guide 1.52 Rev 3. Include Independent Qualification of Carbon.

2. ASTM D3803-98
    a. Section 4.1: Back up Beds are enough
    b. Section 6.2: Ambient Pressure Data- Consult US Weather Bureau.
    c. Section 8.2: Thyroid Counting- Delegate to Federal & State Agencies.
    d. Section 14.3: Reduce Needless Data Taking- Once Reaching Equilibrium.

A PROPOSED STANDARD TEST METHOD FOR NUCLEAR GRADE GAS ABSORBERS IN CARTRIDGE CONTAINERS

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INTRODUCTION

The ASTM-D-3803-1989 Standard and subsequent, nearly identical issues (1991-1998), was written for the purpose of evaluating the methyl iodide collection efficiency of bulk activated carbon (with a physical size distribution of about 8x16 mesh and impregnated with an additive; RDT M--16-1T 1976). This material is placed in a standard test bed geometry of 50mm diameter and 50mm depth . The standard flow defines the residence time of the gases within the beds. For new absorber materials the acceptance criteria limits methyl iodide penetration to a maximum of 2.5% (Reg Guide 1.52 Rev 3, 2001).

In many instances, nuclear industry users are not aware of the specific nature of this Standard as it applies to geometry, absorber type, mesh size, flow rate and pressure. Over the years since this Standard (and preceding Standards, 1979 & 1986) have been promulgated, nuclear industry participants have used this Standard both for activated carbon and other absorber materials. In addition, the industry has used a variety of absorber mesh sizes flow rates and test bed configurations. This Standard has been used, in essence, merely as a way of applying its rigorous tolerances and methodology for the quality assurance testing needs of the users. For over 20 years at our Laboratory, the overwhelming majority of absorber materials tested include only 2: activated carbon and silver zeolite. Similarly, the geometric configuration of choice, other than that described in the Standard is a prepackaged cartridge whose geometry is approximately 50mm in diameter and 25mm or less in depth.

We believe that it is prudent to recognize the widespread use of cartridges containing absorbers for collecting iodine forms in the nuclear environment as an Addendum to the D-3803 Standard or as a stand alone Standard. This will provide more comparable results from differing laboratory facilities. For this purpose the following draft version is submitted for consideration by the industry and D-28 Standards Committee.

The format is presented so as to easily identify sections of the ASTM Standard that remain applicable, as written, in contrast to the revised sections presented in expanded margin.

1. Scope

1.1 This test method is a very stringent procedure for establishing the capability of new and used activated carbon to remove radio-labeled methyl iodide from air and gas streams. The single test method described is for application to both new and used carbons, and should give test results comparable to those obtained from similar tests required and performed throughout the world. The conditions employed were selected to approximate operating or accident conditions of a nuclear reactor which would severely reduce the performance of activated carbons. Increasing the temperature at which this test is performed generally increases the removal efficiency of the carbon by increasing the rate of chemical and physical absorption and isotopic exchange, that is, increasing the kinetics of the radioiodine removal mechanisms. Decreasing the relative humidity of the test generally increases the efficiency of methyl iodide removal by activated carbon. The water vapor competes with the methyl iodide for adsorption sites on the carbon, and as the amount of water vapor decreases with lower specified relative humidities, the easier it is for the methyl iodide to be adsorbed. Therefore, this test method is a very stringent test of nuclear-grade activated carbon because of the low temperature and high relative humidity specified. This test method is recommended for the qualification of new carbons and the quantification of the degradation of used carbons.

1.1.1 Guidance for testing new and used carbons using conditions different from this test method is offered in Annex A1.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 2652 Terminology Relating to Activated Carbon[3]

D 2854 Test Method for Apparent Density of Activated Carbon[3]

E 300 Practice for Sampling Industrial Chemicals[4]

E 691 Practice for Conducting an Interlaboratory Test Study to Determine Precision of a Test Method[5]

2.2 *Code of Federal Regulations:*

CFR Title 49, Section 173.34, "Qualification, Maintenance, and Use of Cylinders"[6]

CFR Title 49, Part 178, Subpart C, "Specifications for Cylinders"[6]

2.3 *Military Standards:*

MIL-F-51068D Filter, Particulate High Efficiency, Fire Resistant[7]

MIL-F-51079A Filter, Medium Fire Resistant, High Efficiency[7]

MIL-STD-45662 Calibration Systems Requirements[7]

2.4 *Other Standards:*

ANSI/ASME N45.2.6 Qualifications of Inspection, Examination, and Testing Personnel for Nuclear Power Plants[8]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *counter efficiency (CE)*—the fraction of the actual number of disintegrations of a radioactive sample that is recorded by a nuclear counter.

3.1.2 *efficiency (E)*—the percentage of the contaminant removed from a gas stream by an adsorption bed; expressed mathematically as $E = 100 - P$, where E and P are given in percent.

3.1.3 *penetration (P)*—the percentage of the contaminant (CH_3I) which passes through the equilibrated test bed of standard depth, and is collected on the backup beds during the feed and elution periods under specified conditions.

3.1.4 *relative humidity (RH)*—for the purpose of this test method, relative humidity is defined as the ratio of the partial pressure of water in the gas to the saturation vapor pressure of water at the gas temperature and pressure. At temperatures below 100°C, this is the normal definition and relative humidity can range from 0 to 100 %.

3.1.5 "test bed" is interpreted as cartridge, when appropriate.

3.2 *Definitions*—for additional terms relating to this stan-rd, see Terminology D 2652.

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[1] This test method is under the jurisdiction of ASTM Committee D-28 on Activated Carbon and is the direct responsibility of Subcommittee D28.04 on Gas Phase Evaluation Tests.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 15.01.

[4] *Annual Book of ASTM Standards*, Vol 15.05.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] Published by the General Service Administration, 18th and "F" St., N. W., shington, DC 20405.

[7] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 bins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

[8] Available from American National Standards Institute, 11 W. 42nd St., 13th or, New York, NY 10036.

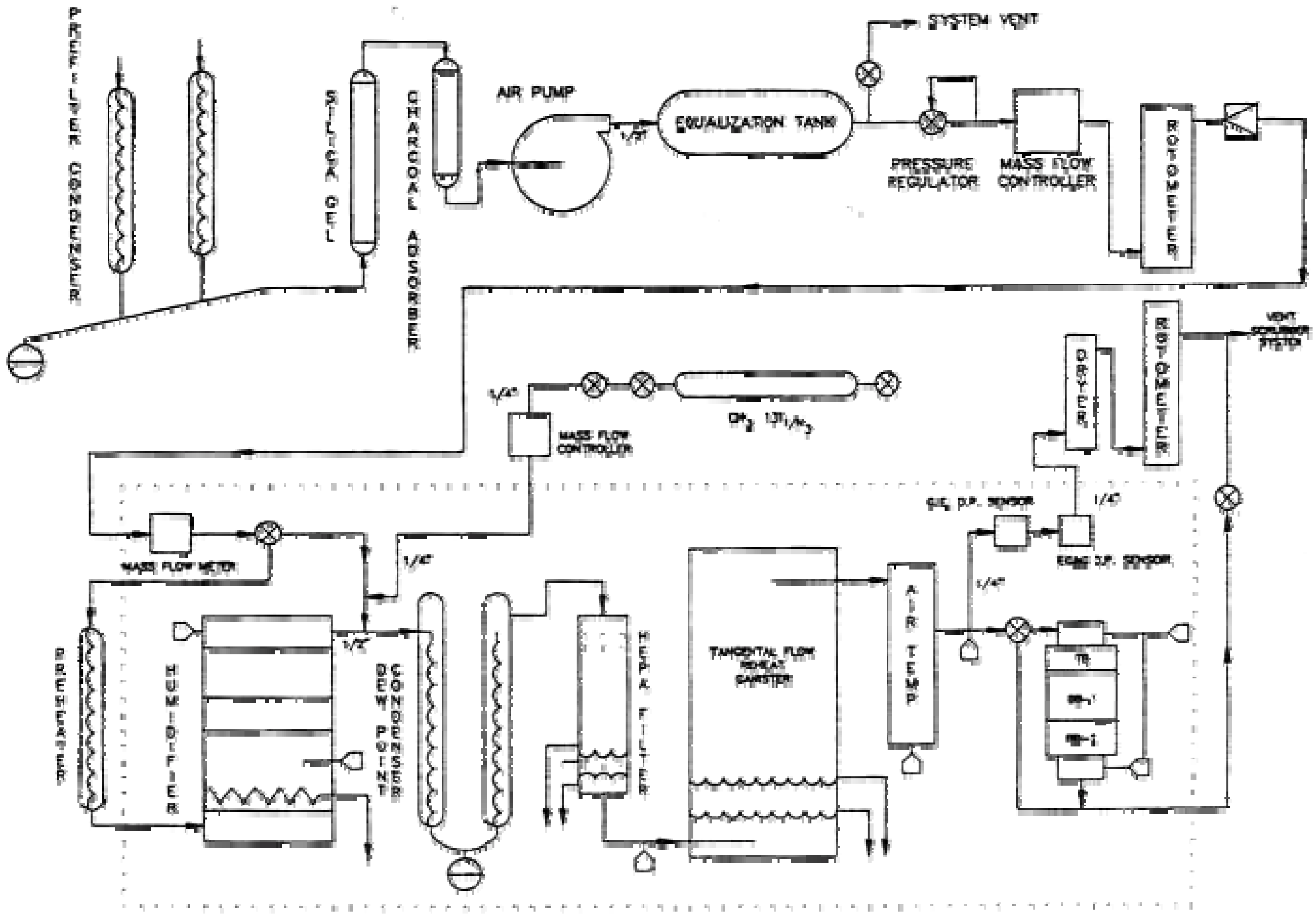


FIG. 2 Schematic of Activated Carbon Test System

TABLE 1 Parameter Specifications

NOTE 1—Temperature, relative humidity, pressure, and gas velocity are to remain constant within the specified maximum variations throughout the entire test, that is, for each test period. Parameter excursions outside the limits specified in this table will invalidate the test results. If results based on a test containing such variations must be reported, then these variations must be noted in the comments section of the external report forms and flagged in the parameter monitoring portion of the internal report.

Parameter	Pre-Equilibration (First 16 h)	Equilibration, Challenge, and Elution (Final 4 h)
Temperature, °C	30.0 ± 0.4	30.0 ± 0.2
Range	29.6 to 30.4	29.8 to 30.2
Relative humidity, %	91.0 to 96.0	93.0 to 96.0
Adsorbate concentration, mg/m³	. . .	1.75 ± 1.75
Test durations:		
Pre-equilibration, h	16.0 ± 0.1	. . .
Equilibration, min	. . .	120 ± 1
Challenge, min	. . .	60 ± 1
Elution, min	. . .	60 ± 1

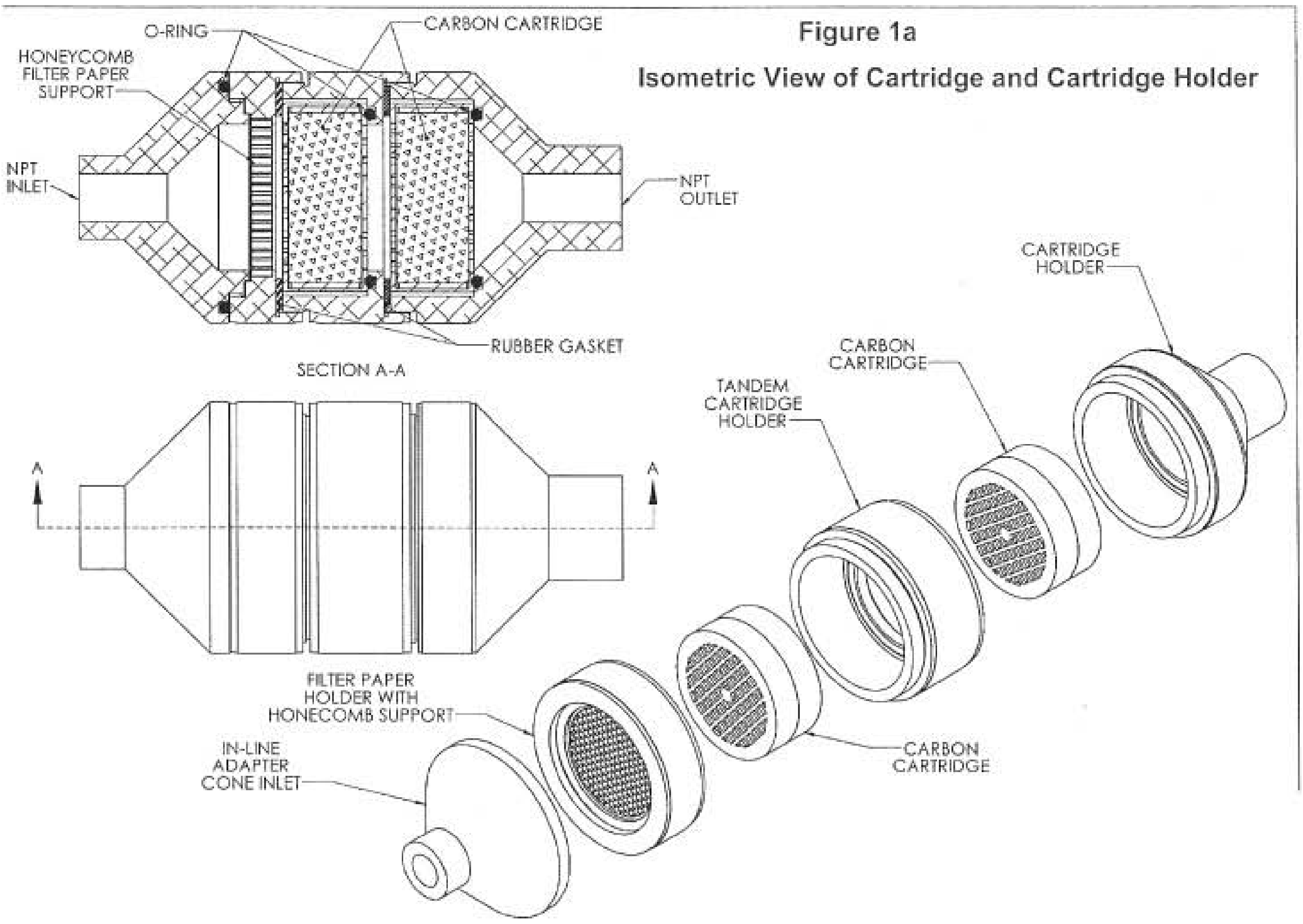


Figure 1a

Isometric View of Cartridge and Cartridge Holder

Drawing Courtesy HI-Q Environmental Products Inc
San Diego, CA

Figure 1b
External View of Typical Cartridge Holder

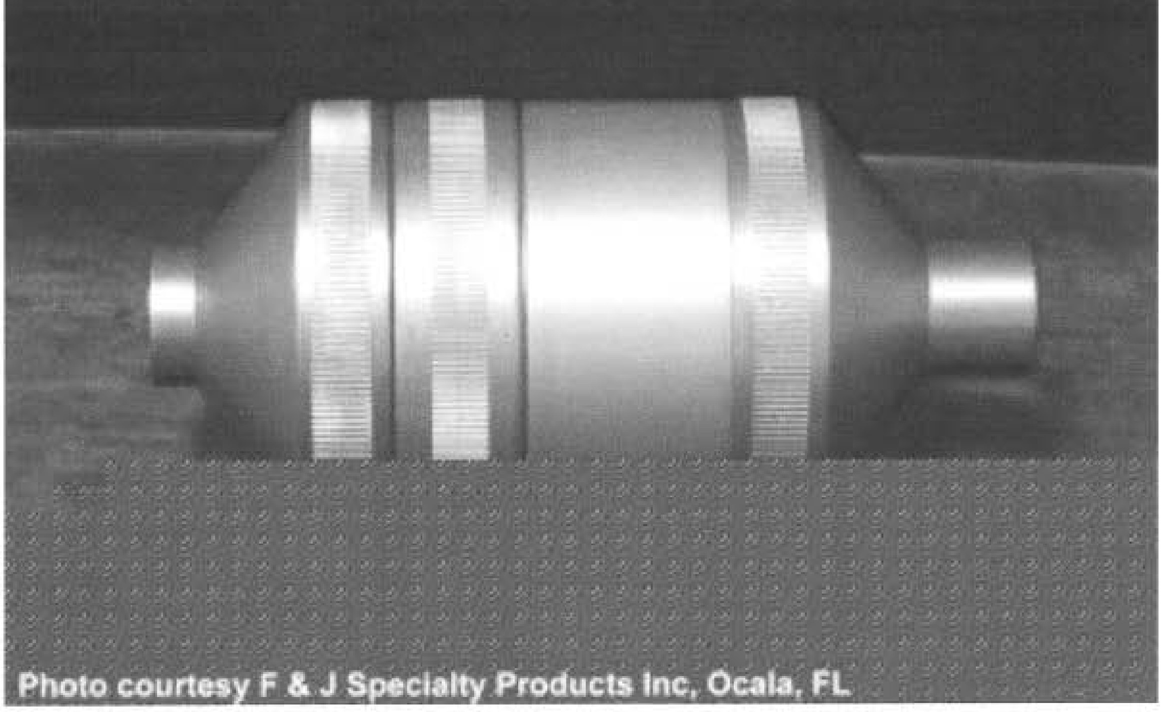


Photo courtesy F & J Specialty Products Inc, Ocala, FL

4. Summary of Test Method

4.1 Both new and used carbons are first exposed to humid air (pressure, approximately 1 atm; temperature, 30.0°C; relative humidity, 95 %) for a *pre-equilibration* period of 16 h. During this pre-equilibration period, the test system may be run unattended with the required parameter monitoring and adequate control devices. Following pre-equilibration, the air flow is continued for a two-hour *equilibration* period, during which the acceptable variability of all parameters is reduced. The test system must be closely monitored and controlled during the final four hours of the test. Qualification of personnel to perform this testing must meet or exceed ANSI/ASME N 45.2.6—1978, Level II, which requires a combination of education and actual test system operation experience. During the *challenge* or feed period, radio-labeled methyl iodide at a mass concentration of 1.75 mg/m ^{3} of humid air flow is passed through the beds for a period of 60 min. Following the feed period, humid air flow without test adsorbate is continued at the same conditions for a 60-min elution period. Throughout the entire test, the effluent from the sample bed passes through two backup beds containing carbon having a known high efficiency for methyl iodide. The two backup beds trap essentially all the radio-labeled methyl iodide that passes the test bed and provide a differential indication of their efficiency. At the end of the elution period, the gamma activity of ^{131}I in the test and backup beds is measured by a gamma counter, and the percent of adsorbate penetrating the test bed is determined.

5. Significance and Use

5.1 The results of this test method give a conservative estimate of the performance of nuclear-grade activated carbon used in all nuclear power plant HVAC systems for the removal of radioiodine.

6. APPARATUS

6.1 Cartridge and Backup Bed Assemblies:

6.1.1 The cartridge being tested is typically 50mm in diameter and 20-25mm in depth. A typical arrangement for testing adsorber materials in cartridges and their cartridge holders is shown in Fig. 1a and Fig. 1b. The only requirements for these assemblies are that they provide a smooth sealing face, uniform alignment of cartridge and sufficient clamping force so that the leak test in 10.2 can be met.

6.1.2 Sufficient backup beds must be used to provide evidence that essentially all the I-131 radioactivity was collected in each test. This may be up to 6 backup beds (50mm diameter X 25mm depth) depending upon the actual flow rate requirements of individual tests and the known collection efficiency of the backup material.

6.2 A schematic of a generalized test system is shown in Fig. 2. This system is designed to operate at approximately 30 C and 95% relative humidity, with a gas flow of 24.7 L/min at atmospheric pressure. Cartridges, however, are typically challenged at higher gas flows and may exibit considerable differential pressures when operating at these high gas flows. The design shown in Fig. 2 may require small modifications to accomodate these higher flows. In addition, small humidity corrections may be necessary due to the increased pressure developed upstream of the cartridges (particularly when small mesh materials are tested at high flow rates).

6.3 Deliberately left blank

6.4 *Saturator System*—This system may be a controlled temperature saturator (bubbler) or spray chamber (environmental condition generator), or any other device of sufficient stability and capacity to supply the required mass flow of water vapor at test conditions.

6.5 Flow Generator. This system should preferably be a combination of several air compressors upstream of the test system. A dryer, carbon adsorber and HEPA (high efficiency particulate air) filter are required to condition the inlet air. Flow measurement and control should be accurate and stable to within + 2% of the specified flow rate. System capacity shall meet or exceed the volumetric flow requirements as calculated from the specified flow rate. A surge tank and pressure control valve should be employed to help in stablizing flow. (typical flow rates for testing cartridges are 1-7 SCFM). To assure proper stability and therefore control, it is preferred that the multiple compressors, generally needed for high flows, are not operating intermittantly . They should be either on or off during the duration of the stabilization and load period (hrs. 16 & 17 of the 18 hr test). This may necessitate adjustment of controlling relief valves in the system preceeding the test facility.

6.6 *Moisture Separator*—A moisture separator should be used to protect the HEPA filter by removing large quantities of entrained particulate water, if present, after humidification. A HEPA filter (or equivalent) is required to function as a final droplet trap to remove small amounts of fine particulate water from the carrier gas ahead of the test bed.

6.7 *Adsorbate Supply*—This system shall consist of a stainless steel cylinder, pressure gage, pressure regulator, and a flow regulator capable of providing a steady flow of the challenge gas, that is, radio-labeled methyl iodide in dry nitrogen, for the duration of the test feed period. The point of injection into the main gas flow of the system must be such that the cross-sectional distribution of the adsorbate at the face of the test bed can be ensured to be homogeneous. A mixing chamber, baffles, glass beads, etc. should be used to achieve adequate mixing.

6.8 Constant Temperature Cabinet: An enclosure and associated thermoregulatory system must be used that is capable of maintaining the inlet gas stream temperatutre from the point of humidity control to the cartridge at 30+ except during the first several hours of pre-equilibration , during which the adsorption of water by the adsorber material being tested may increase these temperatures slightly. The test assembly, including the cartridge and cartridge holder as well as much of the equipment associated with temperature, humidity and differential pressure ports should be located within the Cabinet enclosure. Ancilliary tubing and other necessary equipment that are affected by room temprature changes should be insulated. It is also recommended that the dew point sensor, critical to humidity measuement, be within the main air stream to assure accuracy (that is, in contrast to drawing a small portion of the flow gases to an external location). This is made possible by protecting the sensor from the bulk of the gas flow in a chamber just preceeding the test canister (Air flows beyond those specified by the dew point manufacurers will destabilize this instrument). Experimental experience is needed to determine the amount of this sensor protection for each facility. A bypass line must be included around the cartridge holder to avoid problems with startup, test termination and restart of the next cartridge to be tested.

6.9 *Flow Measurement and Control*—Mass flow controllers, control valve and orifice meter, rotameter or any other device with adequate stability and demonstrated measurement system accuracy of ± 2 % of specified flow rate at the test conditions. All flow measuring devices must use correction factors for interpretation and application to actual test conditions. These factors must be carefully predetermined and documented. No flow measuring device should be located directly downstream of the test bed such that it is subject to variable temperature and humidity conditions during a test as a result of water absorption by the carbon.

6.10 *Interconnecting Tubing*—Tubing must be non-reactive with methyl iodide, such as stainless steel, glass, etc., with a minimum of ⅜-in. outside diameter, and kept as short as possible to reduce the system pressure drop.

6.11 *Temperature Measurement Devices*— Platinum resistance thermometers (RTDs) with certified accuracy and measurement system calibration to ± 0.2°C are required for the measurement of test bed inlet air temperature and dew point. The placement of the air temperature RTD must be such that it is not subject to radiative heating from the test bed. It is critical to the exact measurement of relative humidity that the chilled mirror RTD and the inlet air temperature RTD be matched exactly (± 0.1°C) or that differences are exactly corrected for in relative humidity calculations.

6.12 Pressure Measurement Devices: Absolute pressure measuring devices must be accurate to within +1% of the reading at standard atmospheric pressure. It is recommended that a mercury manometer be installed for absolute and differential pressure measurement (this is the recognized standard for calibration of pressure). A 3-way valve will allow measurement of both from the same manometer.

6.13 *Humidity Measurement*—A humidity measuring device with demonstrated accuracy and calibration to ± 0.2°C at 30°C and 95 % relative humidity is required for measurement of relative humidity of the gas stream immediately upstream of the test bed. Note that for these test conditions only an optical dew point hygrometer currently meets these specifications. A secondary check on this measurement device is required to ensure that calibration offset has not occurred. This secondary device may be another optical dew point hygrometer, wet bulb/dry bulb, or any other device with a demonstrated accuracy of ± 3 % relative humidity. For this application, absolute accuracy is less important than reliability and reproducibility.

6.14 Data Recording: Temperature and relative humidity recording devices are needed to document these parameter (in intervals of no more than 5 min.) during the last 4 hours of each test.

6.15 Gamma Detection System: Any reliable and efficient detection system for gamma rays of 365 keV energy is permissible, provided actual count of gamma photons and not analog rate output is utilized. The system must provide adequate elimination of any interferences that might be present. Systems equipped with internal computers that make calculation or correction for such things as dead time, counting efficiency decay rates itc. are also permissible, provided they give accuracy equal to that required in this standard. In many cases, either thallium activated sodium iodide well crystals or single or multi-channel gamma spectrometers that use thallium activated sodium iodide, lithium drifted germanium, or intrinsic germanium detectors can be used with appropriate professional guidance. A reasonable arrangement includes one of these detectors and appropriate shielding as well as a multichannel spectrometer that focuses on the spectrum of the I-131 photopeak. This will provide an acceptable level of sensitivity (Efficiency/ background). This also minimizes the amount of radioactivity needed and consequently reduces the health hazard to personnel.

7. Materials

7.1 *Air*—Compressor, used for pressure systems, should be of the oil-free type to minimize injection of hydrocarbons into the system. Line filters shall consist of a dryer, activated carbon, and HEPA filters and shall be adequately sized and maintained.

7.2 *Water*—Specification D 1193 Type III reagent water, deionized or distilled, or both, must be used for water-vapor generation.

7.3 *Radio-Labeled Methyl Iodide*—Methyl iodide solution should be stored in the dark below 0°C to slow its decomposition to I_2. The activity of ^{131}I should be such that the total activity incident upon the detector in the entire spectrum from the test bed is between 10^3 and 5×10^5 counts/min.

7.4 *Backup Bed Carbon*, with a penetration of no more than 3 % when tested by this test method. The calculation of the efficiency of the first backup bed is required for each test.

7.5 *HEPA Filter Media*—In accordance with MIL-F-51079A. If a pleated filter is used in place of a flat sheet, it shall be constructed in accordance with MIL-F-51068D.

8. Hazards

8.1 **Warning:** *Overpressure*—The contaminant feed system makes use of dry nitrogen from standard high-pressure gas cylinders, a contaminant feed cylinder which is pressurized, and associated regulators and tubing for transport of the contaminant gas. This system must be designed with adequate safety factors. Standards for the fabrication of such pressure vessels and associated fittings are contained in 49 CFR 173.34. Elastomeric seals must be replaced on a regular basis or if damaged to ensure system integrity.

8.2 Warning: Radioactivity: The system must be adequately vented through a filter system capable of handling the maximum possible contaminant release. The radiotoxicity of I-131 is well documented,. The species used in this test is very volatile and easily inhaled. Rigorous health physics procedures must be folllowed whenever handling the radioisotope. State and Federal regulatory bodies are responsible for oversight of this issue. Personnel access to the facility should be limited and workers trained in health physics procedures.

9. SAMPLING: Guidance in sampling granular materials is given in Practice E 300.

10. PREPARATION OF APPARATUS

10.1 Leak testing of the system should be performed on a routine basis, and is recommended when increasing gas flow testing is needed. This test should be a pressure decay test for pressure induced flow systems. First the system should be pressurized to approximately 400kPa. The system should then be isolated, that is sealed at all atmospheric connections, and the pressure change with time noted. The maximum leak rate should not exceed 10kPa in 30 min to ensure the accuracy of flow measurement.

10. To ensure the accuracy of relative humidity measurement, a check of the differential pressure between the test bed and the sensor of the optical dew point hygrometer should be performed initially and whenever the system is modified, or semi-annually. This check should be performed with the test and backup canisters filled with carbon and with the system operating at the standard conditions specified, that is, temperature, flow, relative humidity, pressure, etc. This differential pressure should not exceed 1 kPa or must be corrected for either in the calculation of relative humidity, or preferably, by modification of the test system to reduce the pressure difference.

10. Correction factors for flow measurement devices, especially rotameters, must be predetermined by the comparison of accurate pressure (± 1.0 kPa) and temperature (± 0.2°C) measurements made at the device and at the test bed under normal operating conditions. Correction of the measured flow to the actual flow at test bed for temperature, pressure, and water vapor can be made using Eq 2:

$$Q_A = \frac{(Q_M)(T_A)(P_M)}{(T_M)(P_A)} \left(1 + \frac{P_{H2O}}{P_A}\right) \tag{2}$$

where:

Q_A = actual gas flow at the test bed, L/min,
Q_M = flow of gas at the flow measurement device, L/min,
T_A = actual gas temperature at the test bed, °K,
T_M = gas temperature at flow measurement device, °K,
P_A = actual gas pressure at the test bed, kPa,
P_M = gas pressure at flow measurement device, kPa, and
P_{H2O} = partial pressure of water vapor at test bed, kPa.

10. 1 No flow measuring device should be located directly downstream of the test bed such that it is subject to variable temperature and humidity conditions during a test as a result of water adsorption by the carbon.

11. Calibration

11.1 The RTDs used to measure the test bed inlet gas temperature and the chilled mirror temperature of the dew point hygrometer must be calibrated together every six months by the National Institute of Standards and Technology (NIST) or a third party capable of certification to NIST standards. Check the hygrometer accuracy at the same time. In addition, the primary flow measuring device should also be calibrated every six months by NIST or a third party capable of certification to NIST standards. Other temperature, flow and pressure measuring devices, balances, radiation survey meters, and gamma detection systems shall be part of an established laboratory calibration program as specified in MIL-STD-45662, with initial calibration intervals of one month and periodic calibration intervals determined on the basis of instrument stability, purpose, and degree of usage. It is important to note that the measurement systems, that is, sensors, associated electronics, displays, etc., must be calibrated individually and together to ensure that the particular parameter monitoring system meets the accuracy and precision requirements.

12. Procedure

12.1 Stabilization Period: Install the cartridge into the system. Perform the leak test described in 9.1. Bring the system up to operating condition in the bypass lines prior to start of pre-equilibration . The duration of this stabilization period is recommended to be 2 h. during which the cartridge comes to thermal equilibrium at the specified test temperature.

12.2 Pre Equilibration Period (for new and used materials): Pass air with 95% relative humidity (range 90-92%) at a temperature of 30 + 0.4 C through the cartridge for 16 hr. There will usually be a sudden rise in the downstream temperature of the gas flow as the result of water adsorption by the material within the cartridge. (Thi change is dependant on the original humidity of the adsorber material). The extent of this temperature rise cannot controlled. The conditions at the cartridge inlet must be controlled at the specified conditions (see Table 1).

12.3 Equilibration Period (for new and used materials): Continue to pass air with 95% relative humidity (range, 93-96.%) at a temperature of 30 + O.2 C through the cartridge for 120 min. During this time interval, the backup beds are positioned at the end of the test facility and gas flow rate is slowly adjusted to the desired flow rate. This process can take 30 min or more, depending on the experience of the test operator and the amount of change from 24.7 L/min. It is important to anticipate and minimize any changes in temperature and humidity. When the system is stabilized at the operating conditions the pressure and differential pressure is observed and documented.

12.4 *Challenge Period (Feed)*—Humid air flow is already at the prescribed conditions (see Table 1) at the start of the feed period. Maintain flow at 30.0 ± 0.2°C at 95 % relative humidity (range, 93.0 to 96.5%) for 60 ± 1 min with 1.75 ± 0.25 mg/m^3 of radio-labeled CH_3I in the total system gas flow provided by the addition of a small and continuous flow of the challenge gas during the feed period.

12.5 Elution Period: To evaluate the ability of the adsorber material to hold the adsorbate once it is captured, continue flow at the end of the feed period without change of the test parameter for a period of 60 min (see Table 1)

12.6 Monitor and record gas stream temperatures upstream and downstream of the test bed. A decrease in the downstream temperature is indicative of bed flooding, where free water condenses in the sample bed; in this case, the test should be aborted.

12.7 At the end of the elution period, switch the system to bypass mode and shut down the system. Remove and disassemble the cartridge holder and backup beds. Transfer the adsorber material from the cartridge and backup beds to identical plastic containers of suitable geometry for the counting system. Homogenize the adsorber material thoroughly for about 1 min.

12.8 Counting Conditions: Because penetration (or efficiency) is simply a ratio of counting rates, absolute counting efficiencies are not necessary unless an independent determination of the total quantity of raioactive iodine is desired. If the geometry of the cartridge material is different from that of the backup beds utilized in the test, then a correction for the differences need to be applied to accurately determine penetration. These type of correction are generally predetermined for each cartridge variety. Count the radioactivity for a period of time necessary to obtain the desired sensitivity and precision. Calculate the results and propagate the statistical uncertainties as described in 11.9-11.14.

12.9 Gamma Count Corrections: If the test material and backup carbon are the same type material and are homogenized and counted under identically the same condition of height and geometry in identical counting bottles, no corrections are necessary. However, a self adsorption factor must be applied for materials being tested that are not similar in density to the carbon in the backup beds. For example, when testing cartridges containing silver zeolites, it is typically necessary to multiply the counting rate of this material by a factor of about 1.1 before calculating the penetration. These self adsorpion factors are predetermined by standard radiochemical methodollogy and applied, as necessary.

12.10 *Counting Efficiency*—Determination of the counting efficiency is unnecessary as far as the measurement of penetration is concerned, and is undesirable because of the extra time and the standard ^{131}I solution that are required. However, if a separate determination of the quantity of ^{131}I used is desired, the counting efficiency can be determined rather simply. Fill a standard counting bottle with carbon to the standard height used in the test procedure. Determine the volume of water required to fill the interstitial voids just to the top of the carbon. Count this sample under the standard counting conditions to determine the blank. Measure an exact volume of a standard solution of ^{131}I of such activity that dead time effects are kept below about 1 %. Dilute with water in a non-wetting plastic beaker to the volume determined previously to fill the carbon voids. Repack another counting bottle with carbon to the standard height and add the diluted iodine solution. Count under the identical conditions being used for the test samples, and as were used for the blank. The slight difference in attenuation of the gamma rays due to the water added will certainly be much less than the errors due to non-homogeneous absorption of small volumes of tracer in the carbon without water present. The counting efficiency is given by Eq 3:

$$CE = (R_s - R_b)(\exp 0.003592\ t)/A_s \qquad (3)$$

where:

CE = counting efficiency, net counts-per-minute/disintegrations-per-minute of ^{131}I at the same time,

R_s = counting rate of ^{131}I standard, counts/min,

R_b = counting rate of background, counts/min,

A_s = activity of ^{131}I standard taken, as of time of standardization of original solution, disintegrations/min,
t = length of time between standardization of original solution and counting, and
0.003592 = disintegration rate/h for 8.041-day ^{131}I.

12.11 *Decay Correction*—If the carbon from different canisters from a given test are counted at significantly different times, they must each be corrected for decay to some common base time in order that the counting rates obtained be comparable. Although other times can be used for zero time, it is convenient to correct all counts back to midnight of the first day in which counting for a particular test was done. Using the 24-h clock, times can be read directly from a watch to the nearest quarter hour, and the various beds can be counted in any order. For ^{131}I compounds, the correction is given in Eq 4:

$$R_0 = R_t \exp(0.003592\ t) \tag{4}$$

where:
R_0 = equivalent counting rate at time zero (midnight),
R_t = counting rate at time t, and
t = elapsed time between zero time and counting time, h.

12.11.1 Generally, the counting interval will be small compared to the decay time so that the beginning of the count can be used to calculate the elapsed time. However, the midpoint of the counting interval gives better accuracy and is just as convenient to use. It should be emphasized that the decay correction should be applied to the net counting rate after correction for background; that is, obviously the background does not decay with the half-life of ^{131}I.

12.12 *Radioactivity and Counting Times*— Corrections for dead-time losses of counting rate due to overloading the counting system by using too much activity can never be made as accurately or conveniently as avoiding such losses from the beginning. Such losses are particularly undesirable when the penetration is low and very large errors are incurred for the test bed with virtually no error from this source for the backup beds. Locating the test bed counting bottle some distance from the detector and counting only a small fraction of the total flux emitted to bring it within the proper range is neither desirable nor prudent. Consequently, the activity of the ^{131}I used in each test should be such that the test bed will not contain more than about 5×10^5 counts/min of total activity incident upon the detector and associated electronics to avoid the increased uncertainties of making large corrections for dead-time effects. When gamma spectrometry is used, this applies to the total events being processed by the analog-to-digital converter (ADC) for the entire spectrum, not just those of interest in the 365 keV photopeak. On the other extreme, the activity used should be kept sufficiently high to give 10^3 to 10^5 counts/min in the test bed to keep the sensitivity and precision of the measurement high without requiring prolonged counting times, particularly when using just the photopeak in gamma spectrometry. Thus, the activity on the test bed can be measured with a relative standard deviation of a few tenths percent with counting times of a very few minutes. For carbon backgrounds and backup beds containing low activity, the counting times should be 30 min to 1 h with gross counters or 1 to 2 h with spectrometers using just the iodine photopeak. This will permit the iodine activity in the backup beds to be detected above the carbon background and the Compton continuum with reasonable statistical certainty.

12.13 *Determination of Contaminant Mass*— The efficiency factor can provide an independent means for determining the mass of the contaminant. The equation is:

$$M = [\Sigma(R_t - R_b)]/(2.22 \times 10^6\ E \times A_s) \qquad (5)$$

where:

M = mass fed during test, g,

R_t = count rate for carbon bed, corrected to base time, counts/min,

R_b = background count corresponding to R_t, counts/min,

E = efficiency factor for gamma counter, and

A_s = contaminant specific activity at base time, µCi/g.

12.14 Contaminated Samples: Occasionally, samples are received for testing that have already been containated with various gamma emitting radionuclides such a Cs-137, Co-60, Ik-131 etc, during use in a reactor environment. No significant radioactive contamination is expected in testing cartridges. However, in testing the efficiency of used cartridges an estimate of potential contaminants should be made and subtracted as part of the background. A variety of experimental approaches are possible to quantify this added background, depending on the volatility of the contamination during the test.

12.14.1 A gamma spectrometer might be required to obtain sufficient resolution to separate the ^{131}I peak from contaminants having peaks in the same energy region.

12.14.2 A jig will be needed to hold the test sample reproducibly some distance from the detector to avoid overloading the system and causing unacceptable dead time effects. The distance will have to be sufficiently large that the contaminant activity will not cause more than a few percent dead time so that sufficient ^{131}I can be used in the test to give the precision desired at the increased distance without increasing the dead time prohibitively. The increase in total activity will also require additional health physics protection such as shielding of the detector and sample, and, possibly, ventilation. Unfortunately, the use of smaller samples or dilution with other carbon are not acceptable alternatives. Blanks and backup beds may be counted directly over the detector to obtain higher precision in shorter counting times provided the exact ratio of the counting rates between the two distances is determined and used in the calculation of penetration.

12.14.3 If one of the contaminants happens to be ^{131}I itself, it will have to be demonstrated that it will not elute during the test. Also. the activity of methyl iodide used in the test will have to be increased sufficiently over that already present that the net activity added can be measured with the precision desired. Consequently, the sample must be tested under the specified conditions without the addition of methyl iodide to determine the apparent penetration due to elution of iodine already on the sample. If the ^{131}I activity on the first backup bed is negligible, another sample may be tested with the methyl iodide challenge. The same sample used in the blank run should not be used for the test run because of uncertainties in how the blank run might have changed the distribution and elution characteristics of the iodine on the carbon. If ^{131}I activity eluted from the sample is relatively small compared to that to be obtained from the test, the activity eluted on the blank test can be subtracted from the test run as a correction with the understanding that the reliability of the results will decrease as the blank correction increases.

13. Calculation

13.1 *Penetration*—All counting must be corrected for the corresponding background counting rates before other corrections are applied. The net activities are then corrected for decay from counting time to some common time zero before calculation of penetration. The halflife and disintegration constant of ^{131}I are 8.041 days and 0.003592/hour, respectively. Because counting efficiencies are not required when counting conditions are kept the same for all fractions, calculate percent penetration using Eq 6:

$$P = 100\,(B + C)/(A + B + C) \qquad (6)$$

where:

P = penetration, %,
A = net counting rate of the ^{131}I activity collected in the test bed, counts/min,
B = net counting rate of the ^{131}I activity collected in the first backup bed, counts/min, and
C = net counting rate of the ^{131}I activity collected in the second backup bed, counts/min,

for beds of equal depth, counted under identical conditions, and corrected for decay.

Obviously, efficiency of the test bed can be given in Eq 7 as:

$$E = 100 - P = (100 \times A)/(A + B + C) \qquad (7)$$

where:

E = efficiency of test bed, %.

The efficiency with which the ^{131}I activity passing the test bed was retained by the first backup bed is, similarly:

$$E = (100 \times B)/(B + C) \qquad (8)$$

where:

E = efficiency of first backup bed, %.

The calculation given by Eq 8 is important in showing whether or not all the activity passing the test bed was collected, and whether or not the proper blank corrections are being made. When penetration is low and corrections for blanks or the Compton continuum, or both, are not made, C can be larger than B and the results will be grossly inaccurate. Specific equations are given in Annex A2 and Annex A3 for calculating both penetration of the test bed and efficiency of the first backup bed from the raw data obtained in a gross counter or in a gamma spectrometer, respectively.

13.2 *Error Propagation*—The uncertainty with which the measurement was made, expressed as one standard deviation, must be calculated for each measured value of penetration of the test bed and efficiency of the first backup bed. The uncertainty must include every statistical uncertainty incurred anywhere in the entire measurement process, all propagated to the final result by the well-known law of propagation of error. Thus, the standard deviation of percent penetration defined above is given by Eq 9:

$$S_P = 100\,\frac{\{(B + C)^2\,(S_A)^2 + (A)^2\,[(S_B)^2 + (S_C)^2]\}^{0.5}}{(A + B + C)^2} \qquad (9,$$

where:

S_P = standard deviation of percent penetration, and
S = estimate of the standard deviation of the net counting rates collected in this test.

It should also be noted that the standard deviation of efficiency by the test bed has the same absolute value as that of penetration of the test bed; that is, $S_E = S_P$. Similarly, the standard deviation for percent efficiency of the first backup bed is given by Eq 10:

$$S_{E,bu} = 100\,[C^2(S_B)^2 + B^2(S_C)^2]^{0.5}/(B + C)^2 \qquad (10)$$

Specific equations are also given in Annex A2 and Annex A3 for calculating the standard deviations of both penetration of the test bed and efficiency of the first backup bed from the raw data obtained in a gross counter and in a spectrometer, respectively.

14. Reports

14.1 Two separate reports are to be written for each test of a sample of activated carbon. The first, or external, report is intended for clients and contains only information essential for their use. The second, or internal, report contains all parameter monitoring and radioactivity counting data and should be kept on file together with a copy of the external report as a cover page at the test laboratory for a period of no less than one year. These laboratory reports may be used for test validation if there are questions regarding results and may also be used for quality assurance (QA) audit purposes.

14.2 *Information Presented in Both Internal and External Reports*:

14.2.1 Name, address, and phone number of laboratory making the test.

14.2.2 Name and signature and experience at ANSI/ASME Level II of technician performing test, and name and signature and ANSI/ASME qualification level and experience of supervisor approving test.

14.2.3 Date of test.

14.2.4 Source of sample and sample identification.

14.2.5 The nominal test conditions; that is, the specified test period durations, temperature, relative humidity, flow, etc.

14.2.6 Overall time-weighted average and standard deviation for temperature, relative humidity, flow, and pressure.

14.2.7 Any notable deviations (see note in Table 1) from the specified conditions must be included in a comment section following the nominal test condition section.

14.2.8 The penetration of the test bed must be reported as a finite number to the proper number of significant figures as indicated by the value of the standard deviation, including negative signs if obtained. No subjective judgements are permitted, such as rounding negative results to zero or reporting results as less than some arbitrary figure. There must be a statement included after the penetration value which states that the standard deviation included is simply that associated with the precision of the radio-analytical result and that the overall accuracy of the penetration result must be estimated from the test method bias and precision data which indicates an accuracy of approximately ± 25 % at the 1 % penetration level, and ± 6 % at the 10 % penetration level for laboratories which rigorously follow the test protocol. These reporting requirements are illustrated in Annex A4.

14.3 *Information Required for Internal Report Only:*

14.3.1 Maximum, minimum, average, and standard deviation for gas temperature immediately upstream of the test bed for each of the test periods.

14.3.2 Maximum, minimum, time-weighted average, and time-weighted standard deviation for absolute pressure at the test bed for each of the test periods.

14.3.3 Maximum, minimum, time-weighted average, and time-weighted standard deviation for relative humidity as measured just prior to the test bed for each of the test periods.

14.3.4 Maximum, minimum, time-weighted average, and time-weighted standard deviation for the actual gas flow for each of the test periods.

14.3.5 The penetration of the test bed and the efficiency of the first backup bed will be accompanied by an estimate of the statistical uncertainty with which each measurement was made, reported as one standard deviation of all random uncertainties incurred in the entire measurement process, not merely the standard deviation of sample counts. All raw data obtained will also be reported along with the calculated result, including total counts, counting times and decay times of the test bed, all backup beds, carbon backgrounds, etc. (see Annex A4).

15. Precision and Bias

15.1 *Precision*—The values in these statements were determined using data from six laboratories which participated in the Second NRC/INEL Interlaboratory Comparison.[9] Using the method of analysis in Practice E 691 presented in Annex A5, the precision of this test method based on the interlaboratory test results is as follows:

15.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method exceed the following values only in one case in twenty:

Repeatability = 0.76 at 1 % Penetration
(95 % Confidence Interval: 0.32 to 1.85 % Penetration)
Repeatability = 1.77 at 10 % Penetration
(95 % Confidence Interval: 8.30 to 11.84 % Penetration)

15.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical material would, in the long run, exceed the following values only in one case in twenty:

Reproducibility = 0.77 at 1 % Penetration
(95 % Confidence Interval: 0.31 to 1.85 % Penetration)
Reproducibility = 1.77 at 10 % Penetration
(95 % Confidence Interval: 8.30 to 11.84 % Penetration)

15.2 *Bias*—Bias depends on exact conformance to the empirical conditions of the test. Interlaboratory comparisons have shown that results from laboratories which do not rigorously follow the specifications for test system design, operation, and calibration often exhibit a very significant bias. This bias cannot be corrected for because of the non-uniformity of the effects of variations of the specified parameters and procedures on different carbons.

A PRACTICAL GUIDE FOR AUDITING ASTM-D-3803 TESTING

J. D. LUDWICK

Considerable experience, as well as proper procedures are needed for implementing the test described in the D-3803 Standard. Each laboratory situation is different. There are a number of external factors that subtly influence the stability during this test and the experimenters ability to properly maintain control of the critical test parameters.

These include

1. **Variations in room temperature**
2. **Variations in outside air temperature, relative humidity and pressure during the test.**
3. **Intermittent operation of pumps that provide gas flow.**
4. **Other factors that must be evaluated at each laboratory**

For these and other reasons that will become apparent, it is recommended that an on-site audit of the test be conducted to provide sufficient assurance that accurate and reproducible results are obtained. Much of the extensive documentation of overnight parameters described in the Standard is of limited value.

WHAT IS IMPORTANT: The critical test parameters during the 60 min load period, particularly, humidity. Humidity is fundamentally defined in the Standard as the difference between dry-bulb and dew-point. The importance of this is clearly documented in ANNEX 1, Fig. A1.1 of the Standard. This ANNEX is provided as an addendum for convenience. The penetration of methyl iodide , through activated carbon, in the relative humidity region of interest (95%) is extremely sensitive to small changes in relative humidity. These small changes in relative humidity are caused by small changes between the dry-bulb and dew-point temperatures. These latter temperature changes are caused by the relative ability of instruments to carefully control the temperatures as well as the external factors listed above.

PROCEDURE AND RATIONAL

First verify, by inspection, the documentation containing the most recent records of temperature (and humidity) and flow calibrations and their trace-ability to NIST standards within the last 6 months. Then, make note of the dry-bulb calibration and differences, if any at 30 C vs NIST. This defines the actual temperature reading needed during the test.

Note the position of the dew point and dry-bulb temperature sensors within the test bed.

Verify that the sample of interest is properly loaded into the test beds.

Verify temperature, humidity and flow rate are within acceptable limits (ASTM D-3803) when instruments take over control as personnel end their work day.

Return with operating personnel and re-verify by observation all the above parameters

During this time of stable test conditions make note of the temperature of the dew-point sensor and its difference, if any, from the dry-bulb. This will provide an instant cross calibration of the dew-point sensor vs the dry-bulb (test) temperature. Use the differences, if any to calculate actual humidity, or simply reference Table 1 throughout the critical test time period.

American Society for Testing Materials "Standard Test Methods for Nuclear-Grade Activated Carbon" ASTM Standard D-3803-1991 (Re-approved 1998).

TABLE 1

Relative Humidity at 30 Degrees Celcius as a Function of Dew-Point

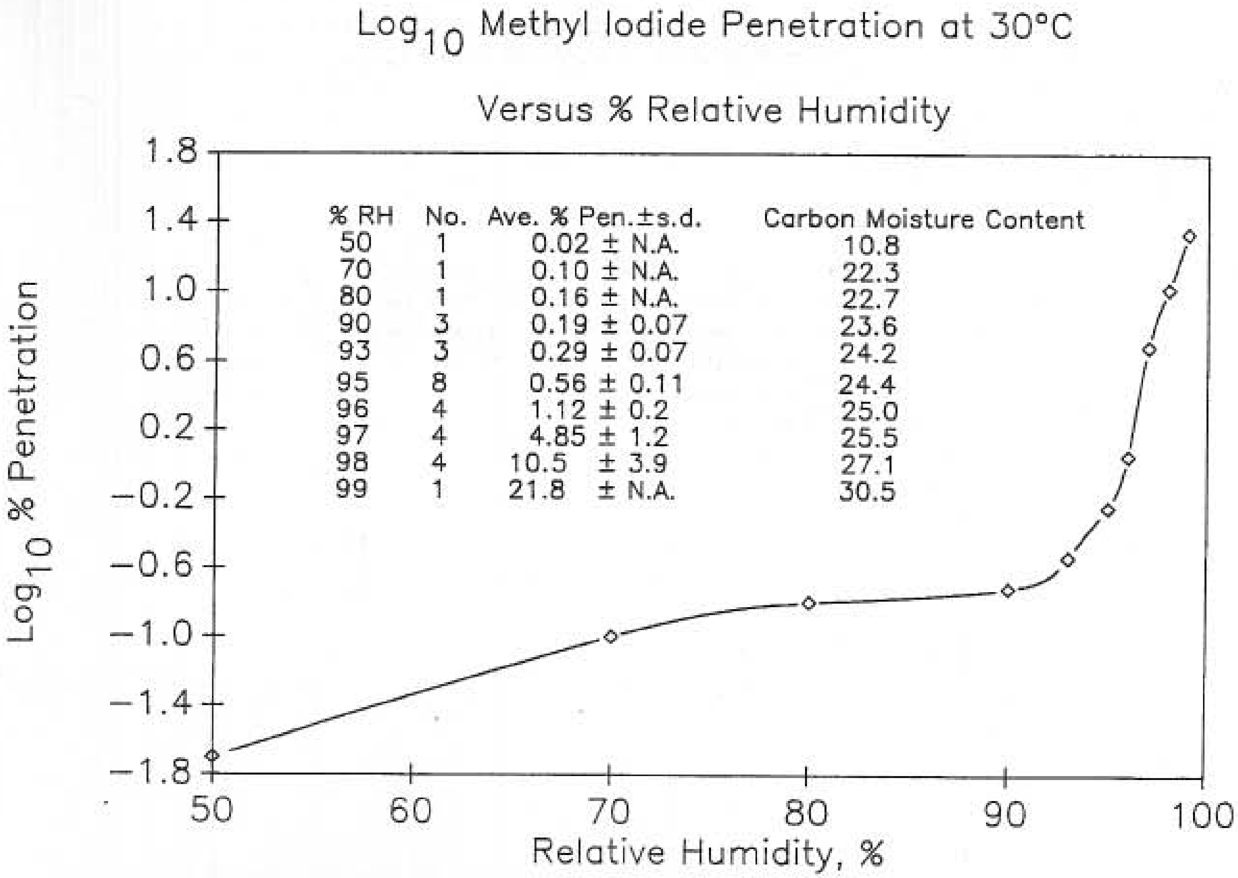
Temp. (C)	Dew-Point (C)	Temp (F)	Dew-Point (F)	Relative Humidity
30	28.8	86.0	83.84	93.34
30	28.9	86.0	84.02	93.88
30	29.0	86.0	84.20	94.42
30	29.1	86.0	84.38	94.97
30	29.2	86.0	84.56	95.52
30	29.3	86.0	84.74	96.07

When methyl iodide delivery is started, and for 5 min after the delivery is terminated, auditing personnel should maintain a vigil , taking particular note of the temperature of gases at the inlet of the test bed - this is the dry bulb sensor. Although the Standard allows for a variance of between 29.8-30.2 C, preferably, the variance should be no more than 29.9-30.1 C during this critical test period (hour 19 of the 20hr test). Also make particular note of the dew-point temperature that corresponds to the dry-bulb temperature. Use Table 1 to quickly estimate relative humidity. Any relative humidity outside of the Standard is not acceptable. In addition, routinely note the values expressed on the inlet and outlet flow measuring devices. These should not vary, at any time, beyond the stated Standard limits of + 2%.

This procedure works well if the dew-point sensor and dry-bulb are very close and essentially in the same air. Therefore, humidity device installations that require a small bypass flow to an external dew-point sensor location are not preferred.

Any indication of moisture condensation is also reason to terminate a test. For example, unusual cooling of a dry-bulb and heating of its corresponding wet bulb is positive evidence of this process. Since each test requires two humidity measuring devices a vigil on both should be maintained.

When the test is completed, observe the test bed disassembly and verify the identification of each test section. Then assure that the measurement for radioactivity properly follows this identification.



% RH	No.	Ave. % Pen. ± s.d.	Carbon Moisture Content
50	1	0.02 ± N.A.	10.8
70	1	0.10 ± N.A.	22.3
80	1	0.16 ± N.A.	22.7
90	3	0.19 ± 0.07	23.6
93	3	0.29 ± 0.07	24.2
95	8	0.56 ± 0.11	24.4
96	4	1.12 ± 0.2	25.0
97	4	4.85 ± 1.2	25.5
98	4	10.5 ± 3.9	27.1
99	1	21.8 ± N.A.	30.5

FIG. A1.1 Results of Sensitivity Testing of New, Co-impregnated, 2*N*Carbon Using the IC Protocol and Varying Only Relative Humidity

COMMENTS BY J. D. LUDWICK

1. Reg Guide 1.52 Section 4.11 should include a statement that requires qualification or batch tests be conducted by a Laboratory that is independent of the manufacturer.
Clearly, the intent of Appendix A, 10CFR 50 AND NQA-1 WOULD BE -- YOU DO NOT CERTIFY YOUR OWN MATERIAL.

PERTAINING TO ASTM-D-3803-98:

Section 4.1, Line 19 Change to read: Throughout the test, the effluent from the test bed passes through sufficient backup beds containing carbon having a known high efficiency for methyl iodide so as to demonstrate that essentially all of the radio-labeled methyl iodide has been collected.

Section 8.2 Do not require routine thyroid counting: Authority for protection of personnel working with radioactivity has been delegated to federal and state radiation protection agencies. We already have to jump through enough hoops.

Section 6.1.2 Pressure measuring devices that record? The US Weather Bureau has them . Consult them for the current barometric pressure. Table 1, NOTE: How do you control pressure when the atmospheric pressure changes during the test. This requirement may have come, historically, from the 1986 Standard. Why record such data. Our pressure data comes from a mercury manometer. Rotometer corrections are in order for changes in atmospheric pressure.

During 35 years of operation I have never seen useful information come from our differential pressure gage at test temperatures of 30 degrees C or less. Typical differential pressures across the test bed are in the range of 1" water (0.025 kPa). During a 30 degree C/ 95% RH test about 0.7 grams of water/min pass through the system. Small amounts of condensation, if present, are not able to block flow to produce significant changes in differential pressure. The 1986 Standard included the 130 degree C steam-air pressurized test. On rare occasions, some useful differential pressure data was observed. The amount of moisture passing through the system during this test was considerable, about 36 grams/min.

Section 14.3 Too burdensome: The only test period of particular interest is hours 17-19 of the test. Proper equilibration during or at the end of each test period can be assured by noting the lack of any heating changes. For this purpose, a Pt RTD device capable of distinguishing 0.05 degrees C (1 degree F), should be adequate. When no change is noted during a 20 min interval, equilibrium has been established. This suggestion is not meant to replace the different test periods; just to reduce the need for accumulating needless data (particularly during the pre-equilibration period.

Thanks for listening