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Validating Emissions Testing Laboratories: A Standard for Assessing Laboratories that Conduct Chemical Emissions Testing

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LABORATORY PROFICIENCY

Critical product purchasing decisions are being made daily based on chemical emissions data and analyses provided by commercial test laboratories. For these purchasers to make meaningful decisions, data must be based on accurate, comparable and reproducible test methods. This standard will establish a validation program that covers test methods and procedures for environmental chamber operation and analytical proficiency for emissions test laboratories.

This standard contains:

- Requirements for enforcement of a rigorous quality management system;
- Requirements for round robin and proficiency testing to confirm test chamber and analytical capabilities;
- Requirements for environmental test chamber performance;
- Explicit acceptable laboratory testing procedures and methods, using various established testing standards;
- Validation program criteria in conjunction with the above requirements; and
- A program for acceptable ongoing compliance.

Specifically, laboratories must comply with the quality management requirements as defined in this document, including external audits. It is expected that each laboratory will enter into a round robin testing program among all participating laboratories and proficiency testing for measured analytes. Laboratories that comply with the elements of this program must be ISO 17025 accredited to the applicable chamber and analytical test methods and standards.

2.0 GENERAL OVERVIEW

As an overview, the Laboratory Validation process requires laboratories to meet the following minimum qualifications.

2.1 Chambers (small and large) constructed and verified based on ASTM Standards D 5116 and D 6670.

2.2 Enforcement of a stringent ISO 17025 management system for quality, administrative and technical operations. Approved testing laboratories must maintain a quality control (QC) program which encompasses all facets of the measurement program from sample receipt to final review and issuance of reports; where product control, testing, data handling, and reporting protocols and procedures are standardized and controlled.

Measures to be routinely implemented in a product's evaluation program include but are not limited to:

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- Appropriate record keeping of sample identifications and tracking throughout the study
- Calibration of all instrumentation and equipment used in the collection and analysis of samples
- Validation and tracking of all chamber parameters including air purification, environmental controls, air change rate, chamber mixing, and sample recovery
- Analysis of spiked samples for accuracy determinations
- Duplicate analyses of samples evaluated and analyzed
- Replicate product sample testing
- Multi-point calibration and linear regression of all standards
- Analysis of controls including chamber backgrounds, sampling media, and instrumental systems.

2.3 Independent, third party status.

2.4 Qualified personnel and scientific staff.

2.5 Completion of round robin and proficiency testing to confirm chamber and analytical capabilities.

2.6 External audit of the laboratory.

3.0 ENVIRONMENTAL TEST CHAMBER REQUIREMENTS

3.1 Small chamber construction and operation must follow the guidance of ASTM D 5116 “Standard Guide for Small-Scale Environmental Chamber Determinations of Organic Emissions from Indoor Materials/Products”.

3.2 Large chamber construction and operation must follow the ASTM D 6670 “Standard Practice for Full-Scale Chamber Determination of Volatile Organic Emissions from Indoor Materials/Products”.

3.3 Size and Construction:

3.3.1 Small chambers are by definition less than 5 m^3 in volume.

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3.3.1.1 Small chambers for material testing are required to have a volume between 0.02 and 2 m^3.

3.3.1.2 Office equipment requires chambers with a minimum volume of 1 m^3.

3.3.1.3 Intermediate sized components such as seat backs and cushions, are typically tested in 0.5 - 2 m^3 sized chambers

3.3.2 Large chambers are by definition greater than 5 m^3 in volume.

3.3.2.1 Testing of chairs and other individual freestanding office furniture products typically requires chambers between 5 and 6 m^3.

3.3.2.2 Large furniture and workstation testing typically requires chambers with an interior volume between 25and 35 m^3 that can accommodate a full workstation in its entirety when assembled according to manufacturers' specifications.

3.3.3 All chambers must have controlled environmental operational parameters used for the purpose of providing accurate and reproducible emission measurements from sources of indoor air pollutants.

3.4 Material:

3.4.1 Environmental test chambers shall be constructed of inert, smooth surfaces such as stainless steel. Glass is inappropriate because of adsorption effects.

3.4.2 All joints and openings shall be sealed. All seals shall be made of non-VOC emitting and non-VOC adsorbing/absorbing materials.

3.4.3 The air within the chamber shall be free of any obstructions or contamination such as humidifiers or refrigeration coils. Internally or externally mounted fans may be used to keep the chamber air well mixed if it can be demonstrated through the use of quality control samples that the fans do not contaminate the chamber air samples or irreversibly absorb/adsorb formaldehyde or representative VOCs (toluene and n-decane). The internal chamber air shall only come in contact with inert materials.

3.4.4 The surfaces and seals of the chamber shall be sufficiently chemically inert such that formaldehyde at the level of 0.005 ppm and representative VOCs at the level of 10 $\mu g/m^3$ are not irreversibly retained on the interior surfaces.

3.4.4.1 Quality control testing must demonstrate recoveries between 80% and 120% for formaldehyde, toluene, and n-decane spiked at these levels. Recoveries of these compounds must show consistency. Other chemical recoveries may be required based on testing/certification standards.

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3.5 Air Exchange Rate:

3.5.1 Air shall be supplied to the chamber using a single pass system. The air that flows through the chamber shall be maintained at a constant flow rate that provides 1.0 ± 0.05 ACH and will depend on the chamber size. If a chamber with an interior volume of 1 m^3 is used, then the airflow shall be 1 m^3/h. Likewise, if a chamber with an interior volume of 29 m^3 is used then the airflow shall be 29 m^3/h. Note: Operation of excessively large chambers to meet this requirement may dilute the emissions and not provide accurate results.

3.5.1.1 Instrumentation must be available to control the air exchange rate with adequate accuracy, precision, and sensitivity and to monitor this parameter to document that the emission test is conducted within the control limits stated above.

3.5.2 At a minimum, the air exchange rate shall be monitored immediately before the product is placed in the chamber (at the same time background contamination checks are made) by accurately measuring the air flow into the chamber. ACH (h^{-1}) is then calculated as air flow (m^3/h) divided by chamber volume (m^3). The accuracy of this air exchange rate must be confirmed (with ± 5% accuracy) using procedures similar to those presented in ASTM Method E741 for tracer gas application. Alternatively, ASTM Method E741 may be used as the primary method for determining the air exchange rate.

3.6 Mixing:

3.6.1 The air within the chamber shall be well mixed.

3.6.1.1 The mixing of air within the chamber shall be evaluated against a theoretical model, and considered well-mixed if the result is within 5% of the theoretical well-mixed model.

3.6.1.2 Mixing shall be evaluated in an empty chamber.

3.6.2 Mixing may be achieved with or without the use of fans.

3.6.2.1 Ideally, mixing will be achieved by the introduction of air into the chamber via engineered manifolds with a matching exhaust manifold. Use of internally or externally mounted fans must demonstrate that they do not contaminate the chamber air or irreversibly adsorb/absorb VOCs.

3.6.3 Mixing Confirmation: The air within the chamber shall be well mixed and comply within 5% of the theoretical well-mixed model. Mixing shall be evaluated in an empty chamber. It shall be evaluated within 6 months prior to testing. Several procedures, as described below, may be used to evaluate chamber air mixing. If CO is used as the tracer gas, the laboratory should follow precautions, as it is hazardous.

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3.6.3.1 The methods referenced in ASTM standards D 5116 and D 6670 may be used for this evaluation for small and large chambers, respectively.

3.6.3.2 An inert tracer gas (SF_6 or CO) is introduced into the chamber (preferably with the inlet air) at a known concentration and constant flow. The chamber air concentration of the tracer gas is then measured over time at the same location as the sampling ports. The experimental curve (concentration vs. time) is compared to the theoretical curve for the same variables, assuming complete mixing, by estimating the relative standard deviation (RSD) of the mean of the deviation of the difference between the observed and theoretical air concentrations at selected time points. An estimate of the variance (S^2) is:

$$s^2 = \frac{\sum (o - t)^2}{n - 1} \qquad \text{(B-1)}$$

where,

o = observed value
t = theoretical value
n = number of observations

The mean of the differences would be:

$$m = \frac{\sum (o - t)}{n} \qquad \text{(B-2)}$$

The RSD is then:

$$RSD = s / m. \qquad \text{(B-3)}$$

Using this approach, the chamber air is considered well mixed if the RSD is less than 5%.

3.6.3.3 An inert tracer gas (SF_6 or CO) is introduced into the chamber (preferably with the inlet air) at a constant known concentration and flow. The chamber air concentration of the tracer gas is then measured over time at the same location as the sampling ports. The chamber concentration vs. time plot is compared to the theoretical curve for a completely mixed chamber:

$$C = C_o (1 - e^{-Nt}) \qquad \text{(B-4)}$$

where,

C = chamber concentration,
C_o = inlet concentration
t = time
N = air exchange rate, N = Q/V, where

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Q = flow rate through chamber
V = chamber volume

To evaluate mixing, the chamber volume (V) then is estimated by fitting the air concentration data for the trace gas to the theoretical curve. The chamber air is considered well mixed if the actual chamber volume and the chamber volume estimated from the tracer gas concentration agree within 5%.

3.6.6.4 An inert tracer gas (SF_6 or CO) is introduced into the chamber (preferably with the inlet air) over a short period of time (1 to 10 minutes). The chamber air concentration of the tracer gas is then measured over time in at least two and preferably three locations within the chamber. One location must be at the sampling ports; other locations should be toward the front one-third and/or rear one-third of the chamber. Chamber air exchange rates (air changes per hour) are calculated from tracer gas decay measurements as:

$$ACH = 1 / (t_o - t_i) \ln (C_i / C_o) \qquad \text{(B-5)}$$

where,

ACH = air changes per hour
t_o = final time (elapsed time in hours)
t_i = initial time
C_i = initial tracer gas concentration in ppm
C_o = final tracer gas concentration in ppm

3.7 Air Supply:

3.7.1 Oil-less piston type compressors shall be used to supply filtered air passed thru an air scrubbing system sufficient to meet required background air levels.

3.7.2 The purified air shall be supplied to the chambers via stainless steel lines.

3.7.3 Background air supply levels of contaminants shall be monitored weekly.

3.7.3.1 Upper limits for background levels are 2 µg/m^3 for formaldehyde, 10 µg/m^3 for total VOCs, 2 µg/m^3 for any individual VOC or aldehyde, and 10 µg/m^3 for respirable (<10µm) particles.

3.8 Temperature and Humidity:

3.8.1 The inlet air supply to the chamber must be monitored continuously, and maintained at a constant temperature and humidity of 23 ± 1° C and 50 ± 5% RH.

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3.8.1.1 Instrumentation must be available to control the temperature and humidity with adequate accuracy, precision, and sensitivity and to monitor these parameters to document that the emission test is conducted within the control limits stated above.

3.9 Air Tightness:

3.9.1 The chamber shall be operated at a slight positive pressure of less than 2 inches of water.

3.9.2 The chamber shall be airtight with an air-leakage rate of less than 0.03 ACH (sealed chamber at atmospheric pressure).

3.9.2.1 The tracer gas decay method referenced in ASTM E 741 “Standard Test Method for Determining Air Change in a Single Zone by Means of a Tracer Gas Dilution” is used for this validation.

3.9.2.2 Air tightness must be evaluated prior to the initiation of testing.

3.10 Sampling Ports:

3.10.1 Sampling ports for collecting chamber air samples may be integrated into the exhaust manifold exit or may be affixed to the chamber walls.

3.10.2 The sample ports should be designed to minimize the amount of tubing between the chamber and sampling media.

3.10.3 Sampling lines shall be made of nonabsorbent material, such as stainless steel.

3.10.4 Sampling ports and lines shall be located in a way that does not adversely affect the chamber airflow.

3.10.5 In large chambers, the sample lines shall be positioned to draw air at a height of 43 $\pm$ 6 inches, the midpoint of the breathing zone, as defined by ASHRAE Standard 55-2004.

4.0 QUALITY MANAGEMENT SYSTEM.

4.1 Quality Management

It is expected that laboratories meet the QA/QC requirements defined below and have an adequate quality system to manage the quantity of work performed. Documentation must be maintained and records management must be performed, and laboratories must also perform assessments and allow audits as specified by specific programs.

4.2 Quality Assurance and Quality Control

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A Quality Assurance Project Plan (QAPP) must be prepared and maintained. Elements of the plan must follow EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5. The QAPP will address all aspects of the measurement program from acquisition of test product to final review and data reporting. Required elements of the QAPP include:

4.2.1 Project Description

A brief description of the test program shall include objectives, identification of the product types to be tested, and how testing is to be conducted. The test conditions should be described, including the test temperature, air exchange rate, and material loading; sample collection schedule, procedures, equipment, and materials; analytical system procedures and equipment.

4.2.2 Project Organization and Description

A project organizational chart shall be provided that designates a Program Leader, a sample custodian, chamber testing supervisor, analysis supervisor, and a QA Officer. The QA Officer should be independent of the technical effort of the program to avoid real or perceived conflicts of interest. The responsibility of all individuals should be defined.

4.2.3 Data Quality Indicator Goals/Acceptance Criteria

The QA/QC Plan shall include data quality indicators and acceptance criteria. Data quality objectives may vary with the particular product and/or standard requirements. Data quality indicators shall be established for the following parameters prior to beginning the testing program:

4.2.3.1 Time and Environmental Conditions for Product Acquisition, Packaging, Shipping and Storage.

Specified limits for the elapsed time from sample packaging to testing under an acceptable range of specified environmental conditions. These will be defined by the specific product test method.

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Table 1 - General Data Quality Indicator Goals for Environmental Chamber Test Measurements

Parameter	Goal	Precision	Accuracy	Completeness[a]
Temperature	23º C	± 1º C	± 0.5ºC[b]	> 90%
Relative Humidity	50%	± 5% RH	± 5% RH[b]	> 90%
Air Flow Rate	1.0 ACH	± 5.0%	± 5.0%[b]	> 90%
Test Specimen Area	Variable	± 1%	± 10%	> 90%
Chamber Air Concentration ▪ Aldehydes ▪ TVOC ▪ IVOC	 < 90 ng < 180 ng < 36 ng	± 20 RSD[c]	± 20% [d]	> 90%

[a] Completeness characterizes the percentage of the planned measurements that are actually conducted.
[b] Accuracy certifications should be supplied by the manufacturers of the sensors who calibrate them against NIST-traceable primary sources. Precision measurements are obtained within the laboratory by continuous recording of the parameters. Non-compliance requires immediate correction and/or replacement of sensors. Calibrated replacements shall be kept in the laboratory. Experience indicates that routine calibration and tracking of precision prevents non-compliance.
[c] RSD = Relative standard deviation for duplicate chamber air samples based on a 10% measurement rate.
[d] Based on third party proficiency performance on quarterly basis.

4.2.3.2 Test Chamber Conditions and Test Results.

Precision, accuracy and completeness limits shall be met for each of the parameters listed in Table 1. Additional performance parameters will be required based on the specific test method and analytes measured. This will include, but not be limited to, analytical instrument and measurement performance criteria including calibration, detection limits, media blanks, range of detection, instrument blanks, and instrument response.

4.2.3.3 Record Keeping and Logs.

Various logging requirements shall be implemented for all test parameters, including chamber and analytical performance. Many of these are identified in ASTM D5116. Additionally, personnel conducting each procedure shall be so noted. Records of the devices used, date and time of tests, and the test results shall be part of the QA/QC recording process. The completeness of records indicates the care and attention given the QC process. Logs that shall be maintained include:

- Sample tracking to record receipt, storage, and disposition of materials to be tested.

- GC standards preparation to document preparation of all standards.
- Calibration to contain environmental systems calibration data.

- Instrument maintenance to document repairs and maintenance on all equipment.

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- Work orders to record all pertinent information for each test, including sample details, sample identification number sampling requested, and sampling times.

- GC runs to record run number and test identification number

- Sample tube preparation logs.

4.2.3.4 Sample Management and Custody.

Products are to be logged into an automated data management system. Chamber air samples shall be tracked through the automated data management system via a defined numbering and work order system. Chain of custody forms shall be used to document the receipt and disposal of all products tested. A sample custodian shall be designated.

4.2.4 Quality Control Procedures.

All procedures shall be evaluated for acceptable performance at the point that the data are generated. This minimizes the need to repeat testing due to out of control situations. Project staff shall carry out Quality Control activities in a routine, consistent manner to provide necessary feedback in the operation of all measurement systems, including:
- Routine maintenance and calibration of systems

- Daily recording of GC calibration accuracy and precision.

- Collection and analysis of duplicate analytical samples, as well as material replicates.

- QC check of sorbent tubes.

- Periodic analysis of proficiency samples supplied by an independent source.

- All records (including temperature, relative humidity, air change, and background levels) shall be maintained and available for review.

- Internal auditing of the quality management system is to be performed annually, at a minimum, to evaluate compliance with established criteria. Internal audits shall be conducted in accordance with established procedures. All audits are to be documented in an audit report and made available for review.

4.2.5 Control Charts

QC charts will allow visual analysis of system performance and observation of anomalistic or unacceptable deviations. This may be done by use of the Shewart Chart (reference: Shewart, W.A., 1931, Economic Control of Quality of Manufactured Products, Bell Telephone

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Laboratories). (Cf. 'Manual on Presentation of Data and Control Chart Analysis', 6th ed., prepared by Committee E-11 on Quality and Statistics, ASTM, 1991.)

4.2.6 Internal Performance and System Audits

All major components of the test shall be audited yearly by the QA Officer and as required by ISO 9001 and/or 17025. These may include, but not be limited to, the preparation of samples, laboratory systems, analytical measurement systems, data entry and processing.

4.2.7 Corrective Action.

The need for corrective action may be identified through reviews, internal QC checks, audits or observations made during routine sampling and analysis activities. All corrective actions will be documented and root cause determined. No further work may be performed until the problem has been satisfactorily resolved, and the QA Officer has acknowledged approval.

4.2.9 Quality Assurance Reporting

All data reported on this project shall be accompanied by the applicable QA/QC data, including the results of internal QC checks, audit results, and any necessary corrective actions. The QA Officer will maintain current records of all QA/QC activities.

4.2.10 Document Control

Policies should be stated and procedures maintained to control all documents as part of the quality system including test methods, internal standard operating procedures (SOPs), software, equipment manuals, product documentation forms, data algorithms, and report formats.

4.2.11 Control of Records

Retention policies should be stated and procedures maintained to control all quality and technical records. Policies to identify, collect, maintain, access, file, store and dispose of quality and technical records must be documented. Computer files are satisfactory, provided copies can be obtained as needed and data edits are documented.

4.2.12 Sample Retention and Disposal

Policies shall include manner and duration of sample retention and disposal and must comply with test method specific requirements.

5.0 STANDARD OPERATING PROCEDURES

The laboratory shall prepare laboratory specific SOPs for all aspects of the analytical procedures. The SOPs shall be specific and be readily available to those involved in the

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analysis and testing. A copy of the method shall be retained in the laboratory. The SOPs shall cover the following areas:

- Sample handling, storage, unusual preparation

- Test sample preparation

- Environmental chamber operation and control

- Assembly, calibration, and operation of the sampling system

- Preparation, handling, and storage of the sorbent collection media

- Air sample collections

- Description and operation of the instrumentation systems, including the sampling device, sample introduction system, separation chemistry, and data system

- All aspects of data recording and processing and reporting

-QA/QC procedures to ensure data quality objectives are met for specific test method and analytes being measured

-Corrective Actions.

6.0 PROFICIENCY TESTING

As indicated in section 4,2,4 all analytical laboratories must participate in proficiency testing as part of their quality program and be found to be proficient. This establishes the bias and accuracy of test results. Regular participation in a proficiency-testing scheme provides independent verification of the analytical competence of a laboratory and shows a commitment to the maintenance and improvement of performance. It demonstrates to the public, customers, accreditation bodies, and regulators, that analytical procedures are under control and gives analysts confidence that the service which they provide will withstand scrutiny and meet the overall quality performance requirements.

The proficiency program operates by providing participating laboratories with sorbent samples containing known analytes at concentrations found in the emissions of products. Tenax tubes are conditioned by the respective labs as used for their sampling media. The tubes are then sent to GEI's contract laboratory where chemicals are loaded and then the tubes are returned to the respective laboratory for thermal desorption analysis and results are sent to GEI. The laboratory is then provided with a report showing how closely their results agree with the accepted value, and where necessary, can then take appropriate action to improve performance. Performance must be within 20% of the real value to be acceptable. New

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laboratories must show two back to back proficient rounds for acceptance on the quarterly proficiency program, including acceptable performance on the Euro Proficiency Testing program offered by GREENGUARD and Blue Angel, as globally recognized throughout Europe. The chemical spikes on the tubes will be in a concentration range of 0.1 - 1 µg/tube, which corresponds to a concentration range of 20 - 200 µg/m³. The analysis is to be conducted using gas chromatography with thermo desorption according to the guidance of ISO 16000 and appropriate GEI standards. Specific chemicals loaded on the tubes will be those representative of chemicals found in the emissions of indoor products. Some chemicals that may be included are demonstrated below:

Alcohols	2-butoxyethanol, 2-(2-butoxyethoxy)ethanol, 2-ethyl-1-hexanol, 2-phenoxyethanol, propylene glycol
Alkanes	Decane, dodecane, pentadecane
Aromatic hydrocarbons	styrene, toluene, trimethylbenzenes, o-xylene, 4-phenylcyclohexene
Esters	2-(2-butoxyethoxy)ethyl acetate, 2-butoxyethyl acetate, n-butyl acetate, texanol isobutyrate, texanol (2,2,4-trimethyl-1,3-pentandiol monoisobutyrate)
Ketones and Aldehydes	acetophenone, hexanal, octanal
Terpenes	3-carene, limonene, longifolene, α-pinene

Following demonstration of analytical proficiency, the laboratory will be expected to participate in an annual round robin testing of representative products being tested for the Greenguard Certification program. Acceptable performance within two standard deviations of the average across all laboratories is expected for specific target emissions of a product. Participation in this effort will follow the guidelines of the Euro Proficiency Testing Program.

7.0 SAFETY AND HEALTH

Laboratories are expected to follow applicable federal, state and local regulations regarding safety and health, for example, OSHA Standard 29 CFR 1910.1450, "Occupational Exposures to Hazardous Chemicals in Laboratories."

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Table of Revisions

Rev. No.	Rev. Date	Description of Revision
0	**3/1/06**	Original Release
1	**12/13/07**	General formatting changes, removed revision year of ASTM documents referenced.
2	**10/29/08**	Removed acceptance of ISO 9001 as a requirement.
3	**2/10/10**	Updated to current format, revised entire document; title change.

Document Author	Date	Approval for Adequacy	Date
Signature		Signature	

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