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Standard Guide for Using Reference Material to Characterize Measurement Bias Associated with Volatile Organic Compound Emission Chamber Test[1]

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1. Scope

1.1 This guide provides procedures for using a reference material with a known emission rate of a volatile organic compound (VOC) to estimate the bias associated with a VOC emission chamber test.

1.2 This guide may be used to assess measurements of VOC emissions conducted in a variety of environmental chambers, such as small-scale chambers, full-scale chambers, emission cells, and micro-scale chambers.

1.3 This guide may be used to assess measurements of VOC emissions from a variety of sources including "dry" materials (for example, carpet, floor tile and particleboard) and "wet" materials (for example, paint and cleaning products).

1.4 This guide can be used to support quality control efforts by emissions testing laboratories, third party accreditation of testing laboratories participating in emissions testing programs, and quality control efforts by manufacturers of building and other materials.

1.5 This guide may be used to support the determination of precision and bias of other commonly used VOC emission standards including Guide D5116, Test Method D6007, ISO 16000-9, ANSI/BIFMA M7.1, and CDPH/EHLB/Standard Method ~~V1.1.~~V1.2.

1.6 This guide also describes the attributes of a suitable emission reference material and the different methods available to independently determine the reference material's VOC emission rate.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 *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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres
D5116 Guide for Small-Scale Environmental Chamber Determinations of Organic Emissions from Indoor Materials/Products
D5197 Test Method for Determination of Formaldehyde and Other Carbonyl Compounds in Air (Active Sampler Methodology)
D5466 Test Method for Determination of Volatile Organic Compounds in Atmospheres (Canister Sampling Methodology)
D6007 Test Method for Determining Formaldehyde Concentrations in Air from Wood Products Using a Small-Scale Chamber
D6196 Practice for Choosing Sorbents, Sampling Parameters and Thermal Desorption Analytical Conditions for Monitoring Volatile Organic Chemicals in Air
D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

[1] This guide is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.05 on Indoor Air.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the ~~standard's~~standard's Document Summary page on the ASTM website.

D6330 Practice for Determination of Volatile Organic Compounds (Excluding Formaldehyde) Emissions from Wood-Based Panels Using Small Environmental Chambers Under Defined Test Conditions
D6617 Practice for Laboratory Bias Detection Using Single Test Result from Standard Material
D6670 Practice for Full-Scale Chamber Determination of Volatile Organic Emissions from Indoor Materials/Products
D6803 Practice for Testing and Sampling of Volatile Organic Compounds (Including Carbonyl Compounds) Emitted from Architectural Coatings Using Small-Scale Environmental Chambers
D7143 Practice for Emission Cells for the Determination of Volatile Organic Emissions from Indoor Materials/Products
D7339 Test Method for Determination of Volatile Organic Compounds Emitted from Carpet using a Specific Sorbent Tube and Thermal Desorption / Gas Chromatography
D7440 Practice for Characterizing Uncertainty in Air Quality Measurements
D7706 Practice for Rapid Screening of VOC Emissions from Products Using Micro-Scale Chambers
D8141 Guide for Selecting Volatile Organic Compounds (VOCs) and Semi-Volatile Organic Compounds (SVOCs) Emission Testing Methods to Determine Emission Parameters for Modeling of Indoor Environments
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E741 Test Method for Determining Air Change in a Single Zone by Means of a Tracer Gas Dilution
E1333 Test Method for Determining Formaldehyde Concentrations in Air and Emission Rates from Wood Products Using a Large Chamber

2.2 *ISO Standards:*[3]

ISO 12219-3 Interior Air of Road Vehicles—Part 3: Screening Method for the Determination of the Emissions of Volatile Organic Compounds from Vehicle Interior Parts and ~~Materials—Micro-scale~~Materials—Micro-Scale Chamber Method
ISO 16000-3 Indoor Air—Part 3: Determination of Formaldehyde and Other Carbonyl Compounds in Indoor Air and Test Change Air—Active Sampling Method
ISO 16000-6 Indoor Air—Part 6: Determination of Volatile Organic Compounds in Indoor and Test Chamber Air by Active Sampling on Tenax TA Sorbent, Thermal Desorption and Gas Chromatography Using MS or MS-FID
ISO 16000-9 Indoor Air—Part 9: Determination of the Emission of Volatile Organic Compounds from Building Products and Furnishing—Emission Test Chamber Method
ISO 16000-10 Indoor Air—Part 10: Determination of the Emission of Volatile Organic Compounds from Building Products and Furnishing—Emission Test Cell Method
ISO 16000-11 Indoor Air—Part 11: Determination of the Emission of Volatile Organic Compounds from Building Products and Furnishing—Sampling, Storage of Samples and Preparation of Test Specimens
ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories
ISO/IEC ~~17043~~17034 ~~Conformity Assessment—General Requirements for Proficiency Testing~~General Requirements for the Competence of Reference Material Producers
ISO/IEC Guide 98 Guide to the Expression of Uncertainty in Measurement (GUM)
ISO Guide 30 Terms and Definitions Used in Connection with Reference Materials
ISO Guide 33 Uses of Certified Reference Materials
~~ISO Guide 34 General Requirements for the Competence of Reference Material Producers~~
ISO Guide 35 Reference Materials—General and Statistical Principles for Certification

2.3 *Other Standards:*

ANSI/BIFMA M7.1 2011 Test Method for Determining VOC Emissions from Office Furniture Systems, Components, and Seating[4]
CDPH/EHLB/Standard Method ~~V1.1 2010~~ V1.2 Standard Method for the Testing and Evaluation of Volatile Organic Chemical Emissions from Indoor Sources Using Environmental Chambers, Version ~~1.1~~1.2, 2017[5]
Method TO-17~~–1999~~ "Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes," Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition ~~(EPA/625/R-96/010b)~~(EPA/625/R-96/010b), 1999[6]

3. Terminology

3.1 *Definitions*—For definitions and terms commonly used in Commitee D22 standards, refer to Terminology D1356. For definitions and terms commonly used when testing materials and products for VOC emissions, refer to Guide D5116. For definitions and terms commonly used to describe reference materials, refer to ISO Guide 30.

3.2 *Definitions of Terms Specific to This Standard:*

[3] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, http://www.iso.org.
[4] Available from Business + Institutional Furniture Manufacturers Association (BIFMA), 678 Front Ave. NW, Ste. 150, Grand Rapids, Michigan 49504-5368, https://www.bifma.org.
[5] Available from California Department of Public Health (CDPH), PO Box 997377, MS 0500, Sacramento, CA 95899-7377, http://www.cdph.ca.gov.
[6] Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC ~~20004,~~20460, http://www.epa.gov.

3.2.1 *constant emission source, n*—an emission source where the concentration of the chemical at the material surface does not change with time.

3.2.2 *diffusion-controlled source, n*—an emission source that is limited by the movement of contaminants within the material boundaries.

3.2.2.1 *Discussion*—

The rate of diffusion depends on the diffusivity of the VOC, the temperature, and the structure of the material.

3.2.3 *dynamic emission source, n*—an emission source where the concentration of the chemical at the material surface changes with time.

3.2.3.1 *Discussion*—

A dynamic source can still generate emissions in a predictable and consistent manner.

3.2.4 *evaporative-controlled source, n*—an emission source that is limited by the ability of a contaminant to transfer from the material surface through a boundary layer to the surrounding air.

3.2.4.1 *Discussion*—

The rate of mass transfer at the surface of an evaporative-controlled source is dependent on VOC volatility, air velocity, and turbulence near the material surface.

3.2.5 *primary reference measurement procedure, n*—"Reference measurement procedure used to obtain a measurement result without relation to a measurement standard for a quantity of the same kind" (**1**).[7]

3.2.6 *reference emission rate value, n*—the independently-determined mass of VOC emitted per unit of time from a reference emission source at specified conditions.

3.2.6.1 *Discussion*—

If the reference emission rate value is "certified," the reference value and associated uncertainty will be available on the accompanying certificate for the reference material.

3.2.7 *reference material, n*—a material that is "sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process" (ISO Guide 30).

3.2.7.1 *Discussion*—

A certified reference materials provides traceability assurance through ISO/IEC 17034. A reference material can also be produced that is not directly traceable through ISO/IEC 17034 but rather is fit for purpose.

4. Summary of Guide

4.1 This guide describes procedures for using a reference material to evaluate the measurement bias associated with tests of VOC emissions from materials and products. The reference materials described in this guide have independently-determined emission rates that can be measured in an environmental test chamber according to commonly used VOC emission and analytical standards and by following the instructions in the reference material's accompanying documentation. ~~Example instructions associated with published uses of reference materials are provided in Appendix X1.~~

4.2 In general, reference materials used in laboratory emission tests can provide traceability for test results determined at different times and in different laboratories using the same environmental conditions (for example, chamber airflow rate, temperature, and relative humidity). Chamber air samples are collected at specific times and analyzed according to referenced standards. If no analytical standard is prescribed, samples are analyzed according to a laboratory's standard operating procedures. Chamber concentrations and source emission rates are calculated. The measured emission rates, at defined time periods, are then compared with the known value for the reference material to estimate the measurement bias of the value obtained in the emission chamber. If applicable, the value of the measurement bias can be compared with acceptance criteria for the emission testing program of interest to the user.

[7] The boldface numbers in parentheses refer to the list of references at the end of this standard.

4.3 This guide also describes the qualities of an emission reference material, the different methods available to independently determine the VOC emission rate of a reference material, and the chamber operating parameters that potentially influence a reference material's emission results.

5. Significance and Use

5.1 Chamber testing is a globally-accepted method for measuring the emissions of VOCs from building materials and products. Chamber emission test data have a variety of uses including identification and labeling of products as low-VOC emitting for improved indoor air quality, manufacturing quality control, and development of new and improved products for reduced VOC emissions.

5.2 Currently, an inter-laboratory study (ILS) is the most frequently used method for assessing the bias of a laboratory's VOC emission test results. An ILS typically relies on a VOC source with an uncharacterized emission rate. Consequently, a large number of participants (Practice E691 recommends 30, with a minimum requirement of six) are needed to produce the data required to calculate a laboratory's performance relative to the central tendency and distribution of the results for all participants. Due to the participant size requirement and other logistical issues, an ILS involves significant planning and coordination to achieve useful results.

5.3 Inter-laboratory studies have often shown significant variations in measured VOC emission rates among participating laboratories for a given source. Variability in the emission rate from the source often is suspected to be a contributing factor, but it is difficult to be certain of the cause. Thus, better characterized sources are needed for evaluating the ability of laboratories to generate VOC emission test results with acceptable bias as discussed in 8.6.

5.4 Proficiency tests (PT) for VOC emission testing typically focus on a laboratory's analytical capabilities. For example, an analytical PT relies on a certified standard prepared by an accredited vendor as a reference. A laboratory analyzes the PT sample without knowledge of its concentration value. Acceptance of the results is judged by the deviation from the known value. Use of reference materials can expand analytical PT schemes to also include the impacts of test sample handling, test specimen preparation, chamber operation, and chamber air sampling.

5.5 Laboratories accredited under ISO/IEC 17025 are required to derive uncertainty estimates for their test results. Typically, this is done by developing an uncertainty budget and estimating an expanded uncertainty (ISO/IEC Guide 98, Practice D7440). Reference materials not accredited under ISO/IEC 17025 should still be delivered with documented uncertainty budgets. An uncertainty budget for a VOC emission test combines relevant sources of measurement uncertainty for all steps in the testing process from test specimen preparation through air sample analysis. A more efficient approach to determining the overall bias and precision for a VOC emission test is with repeated testing of a reference material (see ISO/IEC Guide 98, ISO Guide 33). This guide addresses the estimation of bias through comparison of the measured value to the reference material value. The precision is determined through repeated testing of multiple reference materials, ideally from the same production batch (see Practices D6299 and E691).

5.6 Other uses of an emissions reference material include verifying quality control emission measurements of manufactured product batches and providing traceability for third party certification.

6. Reference Material Attributes

6.1 According to ISO Guide 30, a reference material with an "assigned quantity value" can be used to improve the accuracy of test methods and analysis, to evaluate laboratory equipment performance characteristics, and to establish metrological traceability. For material VOC emission testing, the assigned quantity value for the reference material is its emission rate of a specific VOC (see Guide D5116 for a discussion of material emission rates).

6.2 To meet ISO Guide 30 criteria, a reference material for material emission testing should consist of a known amount of VOC that transfers to the surrounding air at a predictable rate. In addition to meeting this "homogeneous and stable" criterion, reference materials for material emission testing should have the following characteristics.

6.2.1 An ideal reference material has a similar chemical matrix to a typical indoor material (for example, building product, furniture component, paint or cleaning product) and emits VOCs in a similar manner. For the purpose of this standard, the test item is referred to as a "material" regardless of its actual composition or description. In general, mass transfer rates of VOCs from materials are controlled by evaporative mass transfer from the surface, desorption of adsorbed compounds, or diffusion within the material, or a combination thereof (Guide D5116).

6.2.1.1 The dominating mass transfer process depends upon the age and type of material. For example, VOC emission rates from so-called "dry" materials (carpets, floor tiles, and particleboard) are initially affected by evaporation, but are ultimately controlled by internal diffusion processes. The VOC emission rates from so-called "wet" materials (for example, paints and cleaning products) tend to be more dynamic than dry materials with a high initial emission rate. The initial emission rate is primarily controlled by evaporation. Once most of the solvent has evaporated from a material, the emission rate is controlled by internal diffusion processes as described for dry materials.

6.2.1.2 The reference material's VOC emission rate should respond to laboratory chamber conditions similar to the way typical test materials respond. Therefore, the emission rate should be sensitive to environmental conditions that affect mass transfer processes (for example, temperature, relative humidity, airflow rate and air velocity). Additionally, the reference material's response to these different environmental factors should be well understood.

6.2.2 For commercial distribution, the reference material should be produced in batch quantities (typically on the order of hundreds of units) that are determined to be consistent (**2**). Stratified random sampling and a null-hypothesis statistical approach is used to verify the "homogeneity" of the batch (ISO Guide 35). Additional requirements for reference material producers are discussed in ~~ISO Guide 34.~~ISO/IEC 17034.

6.2.3 The reference material should emit VOCs that are measurable by the same sampling and analytical techniques used in VOC emission testing (for example, Test Method D5197, Test Method D7339, and ISO 16000-9). Although canisters can be used to sample VOCs, they are not typically employed for chamber emission testing. Chemical emissions should result in chamber concentrations appropriate to the level for which the measurement process is intended. Thus, reference materials of different sizes or numbers may be required for different types and sizes of test chambers.

6.2.4 The reference material should be packaged and shipped to prevent physical damage and minimize loss or degradation of the material. Short-term stability tests are described in ISO Guide 35.

6.2.5 The reference material shelf life should be known. ISO Guide 35 provides further information regarding long-term stability tests. Ideally, the shelf life should be several months or longer.

6.3 The temporal emission characteristics should be known for both constant and dynamic emission sources.

6.3.1 A reference material with a constant emission rate of a VOC allows test chamber air to reach a steady-state concentration. The time to reach near steady-state conditions is roughly three times the inverse of the air change rate. Thus, sampling should not occur until at least 3 h after the start of a test operating with a 1 ~~h~~1/h^{-1} air change rate. An example of a constant emission source is a filled liquid inner-tube diffusion film emission source described in Wei et al. (**3**). One advantage of a constant emission source is that chamber samples can be collected after only a few hours, allowing results to be obtained quickly.

6.3.2 A reference material with a dynamic emission rate is representative of a dry material whose emissions change with time as the source becomes depleted. To evaluate sampling at multiple concentrations, chamber air samples should be collected at a minimum of two elapsed times (for example, 24 and 72 h) in order to generate an emission profile. An example of a dynamic emission source is a polymer film loaded to equilibrium with a VOC as described in Cox et al. (**4**). A dynamic emission source also allows for the assessment of a laboratory's ability to measure analytes at different concentration levels.

6.4 According to NIST (**5**), there are several ways to determine a reference material's emission rate value.

6.4.1 A "certified" reference value is determined using at least two independent measurement methods. The optimal approach to determine a material's reference value is with a primary reference measurement procedure. For example, gravimetric determination is a primary method to measure the chemical content of a standard gas mixture.

6.4.2 Non-certified, reference values may be determined by one or multiple laboratories using a single method. For example, an ILS may be designed to determine an average reference value for a small subset of samples from a single batch. The resulting ILS value and uncertainty are then assigned to the remaining samples from that batch.

6.5 Temporal emission profiles may be determined by modeling. For example, the emission rates of VOCs from a relatively homogeneous polyvinyl chloride (PVC) flooring product have been well-characterized by fundamental mass transfer models (**4, ~~6 and 7~~6, 7**). Although not commonly used to assign reference values, such models may provide additional information regarding the performance of a test chamber. Cox et al. (**4**) developed a mass transfer model to predict the emission rate of toluene from a dynamic emission source. And, Wei et al. (**3**) developed a mass transfer model to predict the emission rate of toluene from a constant emission source.

6.6 When a new batch of reference materials is introduced, the associated reference emission rate value and its uncertainty should be re-established through accepted methods. An expanded uncertainty for a reference emission rate value may be determined using procedures in ISO/IEC Guide 98.

7. Apparatus and Operating Parameters

7.1 The state of the art for measuring VOC emissions from indoor materials is environmental chamber testing. An environmental chamber test exposes a material or representative portion of a material of known dimension (typically the material area, but results also may be normalized to material length or mass as appropriate) to a specified temperature, relative humidity and airflow rate. At these conditions, VOCs diffuse or evaporate from the material surface to chamber air. At a specified elapsed time, the resulting concentration of a VOC in chamber air is measured to determine the material's VOC emission rate or area-specific emission ~~rate, termed emission factor~~ rate (Guide D5116). Reference materials are designed to mimic material sources that are commonly evaluated for VOC emissions by chamber testing.

7.2 The emissions of VOCs during a test primarily depend upon the physicochemical properties and application of the material sample and the environmental exposure conditions. Chamber operating parameters have the potential to affect VOC emission rates. Thus, use of a reference material whose emission rate is also impacted by these parameters provides a check of chamber performance. Chamber parameters are addressed in the documentation accompanying a reference material. Detailed descriptions of the impacts of chamber parameters on VOC emission rates from materials can be found elsewhere (Guide D5116 and Practice D6670). A brief summary is provided here.

7.2.1 Temperature affects a VOC's volatility and diffusivity properties. As such, higher material and ambient air temperatures will result in faster emission rates. A commonly used temperature setpoint for chamber testing is 23°C with an associated precision and accuracy of ±0.5°C (Guide D5116). A relatively small deviation in chamber temperature can significantly affect a material's emission rate, and some reference materials can behave similarly. For example, a chamber study of environmental factors using a diffusion bottle source **(3)** estimated that the emission rate of toluene at 23.5°C was approximately 10 % higher than the emission rate for the same source at 22.5°C.

7.2.2 Relative humidity can impact the emission rates of water-soluble compounds such as formaldehyde. Relative humidity may also affect VOC emissions from materials that are hygroscopic since the adsorbed water may change the diffusion properties of the material and how the VOC desorbs from the surface. A commonly used relative humidity setpoint for chamber testing is 50 % with an associated precision of ±5 % and accuracy of ±10 % (Guide D5116). Some existing reference materials contain hydrophobic compounds and mimic sources that don't contain significant amounts of water.

7.2.3 Air change rate determines the rate of contaminant removal from the chamber system. When describing the amount of chamber air dilution, the air change rate is often normalized by the material surface area (area specific airflow rate). By the process of dilution with clean air, VOC concentrations in the chamber air will be lower for higher air change rates. In addition, the test chamber air concentration provides resistance to the rate of VOC evaporation. Thus, the greater the difference in concentration between the material surface and the chamber air, the greater the evaporation rate. This effect will be more pronounced for materials where VOC mass transfer is more impacted by the evaporation rate rather than by the diffusion rate in the bulk material. Ideally, reference materials result in chamber air concentrations of a VOC that are similar to concentrations of interest in emission testing of indoor materials.

7.2.4 Surface air velocity and turbulence affect the mass transfer evaporation rate of a VOC by changing the resistance of the boundary layer between the material surface and the chamber air. At higher air velocities and greater turbulence, the boundary layer resistance is reduced resulting in greater emissions, especially for wet materials. This effect, however, does have a threshold such that air velocities greater than a certain value will result in similar mass transfer rates (Guide D5116). Air velocity at the surface of a material is difficult to measure and often varies substantially between chambers used in different laboratories **(8)**. Thus, this parameter may be an uncontrolled source of bias when comparing chamber measurements to reference material values.

7.2.5 In general, the emission profiles of indoor materials change with time, often following a first or second order decay rate. As a result, the elapsed time at which the material emission rate is determined will have an impact on the result. The impact of elapsed time is potentially greater for sources with a faster mass transfer rate (for example, evaporative "wet" sources). Some reference materials are dynamic sources. Exposure and sampling times must be controlled carefully when using dynamic sources.

7.3 Reference materials can be applied to a broad range of chamber designs and sizes including small chambers (Guide D5116, Test Method D6007, Practice D6330, Practice D6803, ISO 16000-9, ISO ~~16000-11~~16000-11, and CDPH/EHLB/Standard Method ~~V1.1),~~V1.2), full-scale chambers (Practice D6670, Test Method E1333, ANSI/BIFMA M7.1), emission cells (Practice D7143, ISO 16000-10) and micro-scale chambers (Practice D7706, ISO 12219-3). More general descriptions of environmental chamber systems are provided in Guide D5116 ~~and~~ , Practice D6670, and Guide D8141.

7.4 Descriptions of sampling and analysis of VOCs in material emission tests are found in several standards, for example, Test Method D5197, Test Method D5466, Practice D6196, Test Method D7339, ISO 16000-3, ISO ~~16000-6~~16000-6, and Method TO-17. Possible sources of error during the collection and chemical analysis of air samples that may impact material emissions and reference material test results include collection efficiency, recovery, contamination, storage losses, analytical equipment calibrations and data analysis. In general, a laboratory's analytical accuracy is assessed regularly through a PT program.

7.5 As described in Guide D5116, the chamber concentration at a specific time point for a reference material is converted to an ~~emission factor [EF~~area specific emission rate [$EA(t)$ in μg/(m^2h)] using Eq 1, with the assumption that this steady-state approximation is applied to constant sources and to dynamic sources with relatively low decay ~~rates.~~rates:

~~$$EF = C_t(N/L) \tag{1}$$~~

~~where:~~

~~C_t = chamber air concentration at time t (μg/m^3),~~
~~N = chamber air change rate (h^{-1}), and~~
~~L = sample loading factor (m^2/m^3).~~

$$EA(t) = C(t)\frac{N}{L} \tag{1}$$

where:

$C(t)$ = chamber air concentration at time t (μg/m^3),
N = chamber air change rate (1/h), and
L = sample loading factor (m^2/m^3).

The material emission rate ($ER(t)$ in μg/h) is equivalent to the product of the area specific emission rate ($EA(t)$) and the sample surface area (A).