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Additive manufacturing of plastics — Environment, health, and safety — Test method for determination of particle and chemical emission rates from desktop material extrusion 3D printer

*Fabrication additive de plastiques — Environnement, santé
et sécurité — Méthode d'essai pour la détermination des
taux d'émission de particules et de produits chimiques des
imprimantes 3D de bureau par extrusion de matériau*

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Foreword

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This document was prepared by Technical Committee ISO/TC 261, *Additive manufacturing*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 438, *Additive manufacturing*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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Introduction

Academic communities have been releasing several papers warning that a significant number of particles and chemical substances emitted from material extrusion (MEX) AM processes commonly used in schools, private homes and similar non-industrial environments would be hazardous to humans when inhaled and absorbed into the human body.

However, currently, there is no well-known test method to measure particle and chemical substances emitted from desktop MEX-TRB/P machines, commonly called "3D printers" installed in the office environment, classroom, and residential space.

Therefore, the goal of this document is to provide test procedures in line with specific operating conditions for measuring particle and chemical emission rates emitted from desktop MEX-TRB/P machine, also known as a 3D printer which is widely used in the national marketplace.

Manufacturers of desktop MEX-TRB/P machines, also known as 3D printers, will be able to take advantage of this document to develop and improve their products by minimizing particle and chemical emission rates, and the end-users also would purchase more safe and improved machines from the market.

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Additive manufacturing of plastics — Environment, health, and safety — Test method for determination of particle and chemical emission rates from desktop material extrusion 3D printer

1 Scope

This document specifies test methods to determine particle emissions (including ultrafine particles) and specified volatile organic compounds (including aldehydes) from desktop MEX-TRB/P processes often used in non-industrial environments such as school, homes and office spaces in an emission test chamber under specified test conditions. However, these tests do not necessarily accurately predict real-world results.

This document specifies a conditioning method using an emission test chamber with controlled temperature, humidity, air exchange rate, air velocity, and procedures for monitoring, storage, analysis, calculation, and reporting of emission rates.

This document is intended to cover desktop MEX-TRB/P machine which is typically sized for placement on a desktop, used in non-industrial places like school, home and office space. The primary purpose of this document is to quantify particle and chemical emission rates from desktop MEX-TRB/P machine.

However, not all possible emissions are covered by this method. Many feedstocks can release hazardous emissions that are not measured by the chemical detectors prescribed in this document. It is the responsibility of the user to understand the material being extruded and the potential chemical emissions. An example is Poly Vinyl Chloride feedstocks that can potentially emit chlorinated compounds, which cannot be measured by the method described in this document.

2 Normative references

[ISO/FDIS 27548](https://standards.iso.org/standards/catalog/standards/iso/e2ec411e-b646-4cfl-be70-10c383110d8d/iso-fdis-27548)

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The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 554, *Standard atmospheres for conditioning and/or testing — Specifications*

ISO 16000-3, *Indoor air — Part 3: Determination of formaldehyde and other carbonyl compounds in indoor and test chamber air — Active sampling method*

ISO 16000-6, *Indoor air — Part 6: Determination of organic compounds (VVOC, VOC, SVOC) in indoor and test chamber air by active sampling on sorbent tubes, 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 27891, *Aerosol particle number concentration — Calibration of condensation particle counters*

ISO/IEC 28360-1:2021, *Information technology — Determination of chemical emission rates from electronic equipment — Part 1: Using consumables*

ISO/ASTM 52900, *Additive manufacturing — General principles — Fundamentals and vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/ASTM 52900, ISO/IEC 28360-1 and the following are applied.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

loading factor

ratio of the device volume to the volume of the unloaded Emission Test Chamber

Note 1 to entry: For the purpose of this standard, the device subjected to the testing is typically a desktop MEX-TRB/P machine, also popularly called a 3D printer

[SOURCE: ISO/IEC 28360-1:2021, 4.18, modified — “EUT” replaced by “device” and Note 1 to entry added.]

3.2

emission test chamber

ETC

enclosure with controlled operational parameters for the determination of chemical compounds and amount of particles emitted during the process

Note 1 to entry: For determining the emissions from AM process, typical controlled parameters include, but are not limited to, temperature, humidity, air exchange rate, and others

[SOURCE: ISO 16000-9:2006, 3.6, modified — Terminological entry is changed considering AM process]

3.3

differential electrical mobility classifier

DEMC

classifier able to select aerosol particles according to their electrical mobility and pass them to its exit

Note 1 to entry: A DEMC classifies aerosol particles by balancing the electrical force on each particle with its aerodynamic drag force in an electrical field. Classified particles are in a narrow range of electrical mobility determined by the operating conditions and physical dimensions of the DEMC, while they can have different sizes due to difference in the number of charges that they have.

Note 2 to entry: Another common acronym for the DEMC is DMA.

[SOURCE: ISO 15900:2020, 3.11]

3.4

differential mobility analysing system

DMAS

system to measure the size distribution of submicrometre aerosol particles consisting of a charge conditioner, a DEMC, flow meters, a particle detector, interconnecting plumbing, a computer and suitable software

Note 1 to entry: Another common acronym for the DMAS is MPSS (mobility particle size spectrometer).

[SOURCE: ISO 15900:2020, 3.12]

3.5

light scattering airborne particle counter

LSAPC

instrument capable of counting and sizing single airborne particles and reporting size data in terms of equivalent optical diameter

Note 1 to entry: The specifications for the LSAPC are given in ISO 21501-4:2007.

[SOURCE: ISO 14644-1:2015, 3.5.1]

**3.6
accumulated particle number concentration**

C_p
time-dependent number for the concentration of particles in a specified size range

**3.7
total particles**

number of particles as calculated based on the measured *accumulated particle number concentration* (3.6) in the sampled volume and the duration of the particle emission test

**3.8
particle emission rate**

PER
particles emitted from AM process per unit time (1/h) in a specified size range that is calculated from *accumulated particle number concentration* (3.6) divided by the build time in h

**3.9
particle emission yield**

Y_{particle}
number of particles emitted per mass of extruded material during the build cycle

**3.10
chemical emission yield**

Y_{chemical}
mass of chemical compounds emitted per mass of extruded material during the build cycle

**3.11
chemical emission rate**

average mass of organic compounds emitted from an AM process per unit of time

**3.12
toluene response factor**

toluene equivalents used to quantify the unidentified substances detected with a flame ionization detector (GC-FID) or mass spectrometric detector (GC-MS)

**3.13
total volatile organic compounds**

TVOC
sum of the concentrations of identified and unidentified volatile organic compounds eluting between and including n-hexane and n-hexadecane.

Note 1 to entry: For a MEX-TRB/P-process, the total volatile organic compounds are typically measured using a non-polar capillary GC column and the concentrations of the converted areas of unidentified peaks using the toluene response factor

[SOURCE: ISO 16000-9:—, 3.14, modified — Note 1 to entry rewritten and Note 2 to entry deleted.]

4 Abbreviated terms and symbols

4.1 Abbreviated terms

ABS	Acrylonitrile butadiene styrene
CPC	Condensation particle counter
DNPH	Dinitrophenylhydrazine
FP	Fine particles
GC/MS	Gas chromatography/mass spectrometry
HPLC	High performance liquid chromatography
PLA	Poly lactic acid
RH	Relative humidity
RPD	Relative percentage difference
RSD	Relative standard deviation
TP	Total particles
UFP	Ultrafine particles

4.2 Symbols

β	particle loss-rate coefficient (h^{-1})
C_{av}	arithmetic average of $C_p(t)$ between t_{start} and t_{stop} (cm^{-3})
C	VOC concentration during the extrusion phase ($\mu\text{g}\cdot\text{cm}^{-3}$)
C_b	VOC concentration during the pre-extruding phase ($\mu\text{g}\cdot\text{cm}^{-3}$)
L	loading factor ($\text{m}^3\cdot\text{m}^{-3}$)
$P E R (t)$ $[r_{\text{pe}}(t)]$	time-dependent particle emission rate (s^{-1})
$P E R$ $(r_{\text{pe,h}})$	particle emission rate for an average hour (h^{-1})
Δt	time difference between two successive data points (s)
t_{start}	time when print command sent (s)
t_{stop}	time when extrusion ends (s)
r	air exchange rate (h^{-1})
P_m	final test specimen mass after extrusion completes (g)
V_c	emission test chamber volume (m^3)
V_s	sample volume during the extruding phase (m^3)

5 Method overview

This document specifies test methods to determine particle and chemical emission rates during the operation of the desktop MEX-TRB/P machine. Particle and chemical emissions are determined by the chamber concentration emitted from the operation of the desktop MEX-TRB/P machine inside an ETC where temperature, humidity, air exchange rate, and air velocity are controlled. Test procedures are divided into three phases: pre-extruding, extruding and post-extruding.

The observed chamber concentration during the extruding phase is converted to the particle emission rate per hour or used material mass by mathematical calculations. The procedures for the build conditions should be under the standard operating conditions (see [A.2](#)) of the desktop MEX-TRB/P machine. Chemical emissions (TVOC and aldehydes) are directly calculated from the chamber concentration as mass per hour.

There are various reasons for performing these measurements. For example, determining the maximum emissions for using machines or comparing emissions from different AM machines. The procedures used for the test can be tailored for the specific purpose of the test. In the case of determining maximum emission rates, the AM machine should be set at the conditions that result in maximum emissions, which are typically the fastest extruding speed, the thickest layer, and the highest nozzle temperature recommended by the manufacturer. For comparing emission rates from different AM machines, the process settings shall be referred to values that are outlined in [Annex A](#).

6 Requirements of the instrument for measurement

6.1 General

6.1.1 Emission test chamber (ETC)

The ETC shall be designed with stainless steel electropolished materials so that it does not emit or absorb substances that can affect measurements during background and AM process tests. During operation, the ETC shall be controlled for constant temperature, humidity, and air exchange rate (see [7.1](#)), and they shall be continuously monitored by using data logging instruments that are calibrated and traceable to primary standards.

General requirements for other materials comprising of an air supply system, mixing equipment and air tightness which are used to construct ETC shall be tested in the ETC to confirm that they do not contribute to the emission test chamber background concentration through emission or adsorption. The test setup of ETC shall not recirculate chamber air so as not to have the contaminated air put into the ETC again.

When flow changes are made to chamber air, a tracer gas test shall be performed to confirm the accuracy of the air exchange rate. The verification process for the test conditions of the ETC such as a tracer gas test procedure and a recovery test shall be performed in accordance with ISO 16000-9 or ASTM D6670.

6.1.2 Instruments for chemical analyses

VOC emitted from MEX operation inside the ETC shall be analysed by thermal desorption GC/MS with the use of a sorbent like Tenax TA^{®1)} or a multi-bed tube as the one consisting of Tenax[®] GR plus Carbopack^{™B2)}. The multi-bed tube Tenax/Carbopack consists of 30 mm Tenax[®] GR plus 25 mm of Carbopack^{™B} separated by 3 mm of preconditioned quartz wool, or one having in equal performance. These are commercially available prepacked and preconditioned if required.

An electron impact instrument (EI) of GC/MS shall be operated in the scanning mode over a mass range of at least m/z 35-350. The general analytical method for the emission of VOC from MEX AM machine using ETC shall be based on ISO 16000-6, EPA Method TO-17, and ASTM D6196.

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