

Recommended Practice for Filter Weighing of Particulate Matter Emissions

RATIONALE

Since the introduction of the first particulate mass emissions standard for heavy-duty diesel engines in 1988, engine and vehicle manufacturers have substantially reduced particulate matter (PM) mass emissions from modern internal combustion engines. PM emissions standard levels have decreased from 0.6 g/bhp-h to the current 0.01 g/bhp-h over the U.S. Heavy Duty Federal Test Procedure (FTP). This reduction in PM emissions resulted mainly from improvements in fuel-air combustion, the introduction of high-pressure fuel injection systems, fuel property improvements, and exhaust aftertreatment.

PM emissions from an engine or vehicle are measured using particulate matter sample filters. The low PM emissions limits currently required for many applications, and the widespread use of exhaust particulate filters to achieve them, have resulted in the need to measure very low PM sample filter masses.

This document describes particulate filter weigh-room recommended practices to provide guidance in the procedures and processes associated with gravimetric filter weighing of engine or vehicle particulate matter emissions. This recommended practice provides proper procedures to build and maintain a particulate filter weigh room or enclosure to meet the requirements of EPA's 40 CFR Part 1065 and ISO 14065 for obtaining low measurement uncertainty.

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Figure 1

Example of particulate filter weigh room.....

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Figure 2

Schematic of single operator weigh room.....

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Figure 3

Picture of a sampling media (filter) pack (Source: California Air Resources Board, SOP #155)

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Table 1

Microbalance specifications

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1. SCOPE

This document describes particulate filter weigh-room recommended practices to provide guidance in the procedures and processes associated with gravimetric filter weighing of engine or vehicle particulate matter (PM) emissions. This recommended practice provides proper procedures to build and maintain a particulate filter weigh room or enclosure to meet the requirements of EPA's 40 CFR Part 1065 and ISO 14065 for obtaining low measurement uncertainty. The content of this recommended practice addresses the following areas:

- a. Particulate Filter Weigh Room
- b. Microbalance Requirements
- c. Filter Conditioning and Handling Requirements
- d. Weighing Process
- e. Equipment Calibration and Maintenance

1.1 Purpose

Individual particulate sample filters are weighed on an electronic micro-balance before and after sampling. Particulate matter is collected from the engine or vehicle exhaust on one or more of these filters. The net difference between pre- and post-sampling filter weights is used to calculate the exhaust particulate mass emissions.

The primary difficulty in measuring PM filter masses is that the mass of PM material collected on the sample filter can be very small.

Although the filter mass at the standard may be around 100 micro-grams in many cases, using current DPF technology the sample filter mass may be as low as a few micrograms (millionths of a gram), which makes accurate, repeatable measurements difficult. A very sensitive microbalance is required and the effect of static electricity, humidity, temperature variation, or any contamination can have a very large effect especially as the sample mass is the difference of two weighings.

This document seeks to guide the reader in the equipment, procedures and techniques necessary to make accurate and repeatable measurements for both engine certification and engine development.

2. REFERENCES

2.1 Applicable Documents

The following publications form a part of this specification to the extent specified herein. Unless otherwise indicated, the latest issue of SAE publications shall apply.

2.1.1 EPA Publications

EPA Title 40: Protection of Environment, Chapter 1, Subchapter U, 40 CFR Part 1065: Engine Testing Procedures

- a. 1065.190 PM-stabilization and weighing environments for gravimetric analysis
- b. 1065.290 PM gravimetric balance
- c. 1065.390 PM balance verifications and weighing process verification
- d. 1065.590 PM sampling media preconditioning and tare weighing
- e. 1065.595 PM sample post-conditioning and total weighing
- f. 1065.690 Buoyancy correction for PM sample media
- g. 1065.790 Mass standards

EPA Quality Assurance Handbook for Air Pollution Measurement Systems: "Volume II: Ambient Air Quality Monitoring Program", EPA-454/B-17-001, January 2017

California Air Resources Board, Procedure for the determination of particulate matter (PM) mass collected on filters SOP NO. MLD 145 Version 5.1, April, 2011, <https://ww2.arb.ca.gov/>

2.1.2 EU Publications

EU-PEMS PM Evaluation Program, doi: 10.2788/2264, <http://publications.jrc.ec.europa.eu/repository/handle/JRC60064>, accessed June 2020

2.1.3 NIST Publications

Available from NIST, 100 Bureau Drive, Stop 1070, Gaithersburg, MD 20899-1070, Tel: 301-975-6478, www.nist.gov.

NIST/NVLAP Handbooks 150: Procedures and General Requirements, and 150-2: Calibration Laboratories Technical Guide, June 1996

NIST Handbooks 145: Handbook for the Quality Assurance of Metrological Measurements, Standard Operating Procedure 4 (SOP 4), May 2019

2.1.4 National Conference of Standards Laboratories (NCSL) Publications

Available from NCSL International, 2995 Wilderness Place, Suite 107, Boulder, CO 80301, Tel: 303-440-3339, www.ncsli.org.

Recommended Practice for Laboratory Design, RP-7, National Conference of Standards Laboratories, July 25, 1993.

2.1.5 ISO Publications

Copies of these documents are available online at <http://webstore.ansi.org/>.

ISO 14065:2013 Greenhouse gases — Requirements for greenhouse gas validation and verification bodies for use in accreditation or other forms of recognition

ISO 14644-1:2015 Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness by particle concentration

ISO 9000:2015 Quality management systems — Fundamentals and vocabulary

2.2 Related Publications

The following publications are provided for information purposes only and are not a required part of this SAE Technical Report.

2.2.1 SAE Publications

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, Tel: 877-606-7323 (inside USA and Canada) or +1 724-776-4970 (outside USA), www.sae.org

Chase, R., Schamp, H., "Measuring the Electrostatic Charge on a Filter," SAE Technical Paper 2007-01-0323, 2007, <https://doi.org/10.4271/2007-01-0323>.

2.2.2 Technical Papers

Davidson, S., Perkin, M., Division of Engineering and Process Control, National Physical Laboratory, Mike Buckley, South Yorkshire Trading Standards Unit (2004). The Measurement of Mass and Weight, ISSN 1368-6550, June 2004.

Troemner Mass Standards Handbook, Good Measurement Practices, Troemner, 201 Wolf Drive, Thorofare, NJ 08086, www.troemner.com.

3. DEFINITIONS

3.1 ²¹⁰Po ANTISTATIC STRIPS

²¹⁰Po is the most common isotope of the radioactive element polonium used to neutralize static electricity.

3.2 AS-BUILT

Condition where the installation is complete with all services connected and functioning but no production equipment, materials, or personnel present as per ISO 14644-1.

3.3 ATMOSPHERIC OVERPRESSURE

The action of keeping the pressure of an area higher than the atmospheric pressure.

3.4 AIR LOCK

A smaller room, like a waiting room, that is situated just before the entrance of the weighing room.

3.5 AUTOMATIC/AUTOMATED FILTER WEIGHING

A computerized system that will take filters loaded inside storage containers (petri dishes) and weigh them. This process tares the balance, corrects for drift, and prevents human error.

3.6 BUOYANCY CORRECTION

The calculations required to account for the buoyancy of air and its impact on mass, and correct the mass reading to reflect this impact.

3.7 CASSETTE

Filter holder used during PM sampling per 40 CFR Part 1065.170 requirements.

3.8 CASSETTE SEPARATOR

Device used to disassemble filter holder.

3.9 CHARGE MEASURING DEVICE

Otherwise known as an electrometer, used to measure electric charge.

3.10 CFR

Code of Federal Regulations is the formal process (codification) of permanent rules and regulations for the United States Government to be published in the Federal Register.

3.11 CORNER LOADING TESTS

Tests conducted during calibration to verify that any error produced by placing the filter off center meets the manufacturer specification.

3.12 CURIE (Ci)

A unit used to express the decay rate of radioactive material. The curie is equal to that quantity of radioactive material in which the number of atoms decaying per second is equal to 37 billion (3.7×10^{10}). It was based on the rate of decay of atoms within one gram of radium. It is named for Marie and Pierre Curie, who discovered radium in 1898.

3.13 PICOCURIE

One trillionth (10^{-12}) of a curie.

3.14 ELECTRONIC MICROBALANCE

An instrument that uses microchips/transistors for the capability of very precise mass measurement, e.g., micrograms (μg).

3.15 ELECTROSTATIC CHARGE

The electric charge on the surface of an insulated body when at rest.

3.16 EMISSIONS LIMIT/STANDARD

The limit on emissions from engines or vehicles as established by the respective governmental agencies. These limits/standards can be legally enforced.

3.17 ESD

Abbreviation for Electrostatic Discharge.

3.18 GLOVE BOX ENVIRONMENTAL CHAMBER

A smaller enclosure that contains all of the parameters to conduct PM testing in a controlled environment without the need to establish an entire separate clean room.

3.19 GROUNDING (ANTISTATIC) STRAP

A braided or otherwise insulated strap that prevents the buildup and discharge of static electricity from a person (microbalance operator).

3.20 HEPA

High-Efficiency Particulate Air (filter) being, using, or containing a filter usually designed to remove 99.97 percent of airborne particles measuring 0.3 micrometers or greater in diameter.

3.21 HIGH PRECISION MASS STANDARD

A mass standard that is measured accurately to a very small value, like 0.5 μg .

3.22 INERTIAL MASS

A measurement of the resistance of an object to acceleration in response to all types of force.

3.23 ION GENERATOR

A device that creates ions (ionized air) using high voltage.

3.24 ISO

International Organization for Standardization. An independent, non-governmental organization with international members from 162 countries that develop voluntary standards and promote the communication of standards between nations.

3.25 LABORATORY SUPERVISOR

The person who oversees the activities that involve the laboratory; including the collection and verification of data in the weigh room/chamber.

3.26 MASS METROLOGY

The science of measuring mass.

3.27 MICROBALANCE

A balance designed to measure very small weights with great precision.

3.28 MICROBALANCE OPERATOR

A person responsible for using and maintaining the microbalance in the weighing room. Including the measurement of test and reference filters.

3.29 MIRROR IMAGE CHARGE INTERFERENCE

Apparent opposite charge produced when charged object is placed near a conducting surface which can lead to error in charge measurement (SAE 2007-01-0323)

3.30 NIST

National Institute of Standards and Technology. This organization has the responsibility for both maintaining and supporting the development of commercial and scientific metrology. The accuracy and traceability of commercial/industrial measurements are not enforced by NIST.

Establishing use and enforcing the NIST measurements depends on governmental organizations, both state and federal, as well as professional groups (i.e., SAE).

3.31 NON-SERRATED FORCEPS

A pair of smooth tipped tweezers.

3.32 OHMMETER

An instrument used to measure electrical resistance.

3.33 OIML

Organisation Internationale de Métrologie Légale (International Organization of Legal Metrology). Legal metrology is the application of legal requirements to measurements and measurement devices.

3.34 OMNIDIRECTIONAL THERMAL ANEMOMETER

An all directional anemometer that measures and indicates air flow using the cooling effect of the air flow on heated wires.

3.35 PARTICLE NUMBER (PN)

Solid particles and liquid droplets that are found in the air; some are produced from vehicle/engine exhaust.

3.36 PTFE (Polytetrafluoroethylene) MEMBRANE FILTERS

Filters that are specifically designed to collect the particulate matter from engine or vehicle exhaust. The PTFE membrane minimizes absorption of water and gases on the filter and allow for more accurate mass measurements.

3.37 QUALITY CONTROL (QC)

A system established to maintain the standards of products and/or test results. This includes testing and documentation of the test results to verify compliance with the specifications.

3.38 REFERENCE FILTER

A filter that has been conditioned for a minimum of 24 hours and never leaves the clean room environment. The filter will be weighed before and after each weighing session to monitor system compliance. This filter will not be used to collect samples.

3.39 REVERSE OSMOSIS

A process whereby water passes through a semi permeable membrane backwards, with a higher pressure, to remove ions and particles from the water.

3.40 SUBSTITUTION WEIGHT

An NIST traceable weight, of similar mass to the sample filter, used for substitution weighing as described in 8.7.4.

3.41 STANDARD ERROR OF ESTIMATE (SEE)

Measure of accuracy of estimates made with a regression line created from known data.

3.42 STICKY MATS

A mat, which has a tacky (sticky) surface, placed at the clean room entrance. Its purpose is to remove dirt and other debris from shoe bottoms in order to prevent clean room contamination.

3.43 TARE

A deduction from the gross weight of a substance and its container made in allowance for the weight of the container.

3.44 THERMAL EQUILIBRIUM

A condition where two substances/items that are touching do not exchange any heat.

3.45 THERMOGRAPH & HYDRO THERMOGRAPH CHARTS

Charts that continuously record the temperature, dew point, and/or humidity for the filter weigh room.

3.46 ULPA

Ultra-Low Penetration Air (filters) removes at least 99.999% of airborne particles with size 0.1 micrometers or greater.

3.47 UNCERTAINTY

A non-negative value associated with the incomplete knowledge of the value being measured.

4. ABBREVIATIONS AND SYMBOLS

The abbreviations used in the document have the following meanings in both capital and lower case.

%	Percent
°C	Degree Celsius
CFR	Code of Federal Regulations
cm	Centimeter
DPF	Diesel Particulate Filter
EPA	Environmental Protection Agency