



AEROSPACE INFORMATION REPORT

AIR6037™**REV. A**Issued 2010-03
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Superseding AIR6037

Aircraft Exhaust Nonvolatile Particle Matter Measurement Method Development

RATIONALE

AIR6037 addresses aircraft exhaust nonvolatile particulate matter (nvPM) measurement method development. It continues the documentation of the state-of-the-art in sampling and measurement technologies, initially captured in AIR5892, issued in 2004. Many of the instruments, techniques, and methods described in AIR6037 had matured through application in aircraft engine field tests, generally occurring in the 2003 to 2008 time frame.

AIR6037 consists of technical appendices describing the technical advancements in particle mass, number and size measurement, particle sampling, and calculation of particle number and mass emission indices.

Research and test campaigns have continued since AIR6037 was issued, in 2010, driving further development of the sampling system and some of the measurement instruments. These developments are described in a subsequent aerospace information report, AIR6241, issued in 2013.

Because further improvements to both sampling system and measurement instrument components are contained in AIR6241, SAE E-31 Committee members have recommended stabilizing AIR6037 in order to maintain it as useful technical documentation in the progression of AIR5892, AIR6037, and AIR6241 toward the development of ARP6320.

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1. SCOPE

This report provides current practice measurement methods for quantifying nonvolatile particle matter at the exit plane of aircraft gas turbine engines. This document contains detailed information for many instruments and techniques, described in AIR5892A, that have been applied in aircraft engine field tests since AIR5892A was first issued in April 2003. There are four sections, identified as Technical Appendices (TA), presenting measurement techniques, sampling, and quantification of nonvolatile particles. The sections are written in the format of Aerospace Recommended Practice (ARP) documents and intended to progress to recommended practices upon overcoming existing technical challenges. Many important technical advances have been accomplished that comprise the Aircraft Engine Exhaust Nonvolatile Particle Matter Measurement Method Development techniques described in TA A: Particle Mass, TA B: particle Number and Size, TA C: Particle Sampling, and TA D: Calculation of Particle Number and particle Mass Emission Indices.

Various measurement methodologies and operability and compatibility issues are described within the TAs. The TAs briefly discuss degrees of sensitivity, accuracy, repeatability, and test operations acceptability for each measurement discipline. They reflect that many important technical advances have been accomplished for measurement techniques of nonvolatile particles. Additional research is required to transition the TAs to Aerospace Recommended Practices.

2. REFERENCES, DEFINITIONS, NOMENCLATURE

Each TA has an independent section identifying the references, definitions, and nomenclature appropriate to the information presented. Attempts were made to maintain consistency when information overlaps between TAs.

3. INTRODUCTION

3.1 Background

This AIR is the result of several years of particle measurement field tests performed on aircraft gas turbine engines. Researchers from government, industry, and academia participated in many test campaigns and collectively collaborated in various venues—most regularly within the forum of the SAE E-31 Committee, which is charged with developing appropriate total particle matter (nonvolatile + volatile) characterization techniques for routine certification of aircraft gas turbine engines. AIR5892A provides initial background information about E-31's role, introductory information on general particulate matter emissions, and distinguishes aircraft engine generated particulate matter as volatile and nonvolatile particle matter emissions.

3.2 Particle Matter Measurement Methods

Nonvolatile particle measurement methods are broadly divided into two general approaches: (1) measurement of the particle mass; and (2) measurement of size and number density of particles.

3.3 Mass Measurement

Regulatory interest in stationary source particulate matter emissions is directed toward total (nonvolatile + volatile) mass measurements. Only nonvolatile particle matter exists at the engine exit plane. Current practice sampling systems capture only the precursors of the volatile particle component that evolves downstream.

Most mass measurement systems were developed for ambient air and stationary source particulate matter quantification. TA A lists systems that can be applied to measure gas turbine engine generated black carbon, organic carbon, and carbonaceous mass. These measurement systems, referred to as analyzers in the text, are being used in current aircraft engine particle matter research. The researchers have developed common methods to measure nonvolatile particle matter mass emitted from aircraft engines using some of the more established analyzers. The analyzer routines, procedures, and general practices for these are described in TA A.

3.4 Particle Size and Number Density Measurement

Particle matter formed from aircraft engine exhaust is also distinguishable by size and number density of particles, as well as individual and total particle mass. The techniques for measuring particulate matter number and size distribution have been used extensively in atmospheric research and refined for measuring aircraft engine exhaust particle matter. TA B describes the methods researchers have developed to measure aircraft engine generated nonvolatile particle matter size distribution and number density. Measurement of these parameters can be used, with knowledge of particle density and morphology, to estimate nonvolatile mass.

3.5 Sampling

Sampling and transport of aircraft exhaust nonvolatile particle matter from the engine exit plane to a mass, size, or number density measurement analyzer is a complex process that requires special treatment to ensure particle matter physical characteristics are maintained. Much research has been performed on particle sampling since AIR5892A was issued. However, several research issues remain before a fully developed sampling methodology can be defined appropriately as a recommended practice. TA C describes sampling approaches currently recommended by researchers, and suggests further research needed to provide flexibility in probe design, line size and conditioning, and dilution schemes. Guidance is based on the various research activities undertaken in the past six years. Some research needs are obvious, and the committee believes that the particle matter sampling methodology will likely mature once efforts are made to resolve the issues through focused research studies.

3.6 Quantification

TA D describes the calculation methodology to determine particle matter emission indices for mass and number. Specific instruction is presented for various sampling and measurement scenarios. Method derivation and examples are shown. This technique is a good candidate to become an Aerospace Recommend Practice (ARP) once comparative studies are undertaken with existing or new data and results supported by industry practitioners.

4. NOTES

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APPENDIX A (TECHNICAL) PARTICLE MASS

A.1 SCOPE

AIR6037 addresses procedures applicable to quantifying the emission of nonvolatile particulate matter at the exit plane of aircraft gas turbine engines. The distinction between nonvolatile and volatile particle types is a critical task in the measurement of particles in aircraft engine exhaust, since these particle types require different approaches for measurement. While both volatile and nonvolatile particulate matter (PM) are present in aircraft gas turbine exhaust, only nonvolatile particles exist at the exit plane. The methods used to measure nonvolatile particles are more advanced and are addressed in this document. This Technical Appendix describes the measurement of particulate mass of nonvolatile aerosol constituents in the exhaust of gas turbine engines.

A.2 REFERENCES

A.2.1 SAE Publications

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

A.2.1.1 AIR5892A Nonvolatile Exhaust Particle Measurement Techniques

A.2.1.2 ARP1179C Aircraft Gas Turbine Engine Smoke Measurement Practice

A.2.2 Other References

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A.2.3 Definitions

AERODYNAMIC DIAMETER: The diameter of an equivalent unit density sphere with the same settling velocity as the particle in question, also often referred to as just "classical aerodynamic diameter."

AGGREGATE: Two or more black carbon primary particles fused together to form a single nonspherical larger particle.

AIRCRAFT GAS TURBINE ENGINE: Any gas turbine engine used for aircraft propulsion or power generation, including those commonly called turbojet, turbofan, turboprop, or turboshaft type engines.

BLACK CARBON: All light-absorbing (visible spectral range) carbon-based particle products produced by the incomplete combustion in an aircraft gas turbine engine.

CLASSICAL AERODYNAMIC DIAMETER: The diameter of an equivalent unit density sphere with the same settling velocity as the particle in question, also often referred to as just “aerodynamic diameter.”

ELEMENTAL CARBON (PARTICULATE MATTER): The refractory carbon found in combustion-generated particulate matter. Also known as graphitic carbon.

EMISSION INDEX (MASS): The mass of emissions of a given constituent per thousand mass units of fuel burned (e.g., g/kg fuel).

ENGINE EXIT PLANE: Any point within the area of the engine exhaust nozzle at an axial distance within 0.5 diameters (or equivalent, if not circular) downstream from the outer edge of the nozzle.

NONVOLATILE PARTICLES: Particle emissions that exist at gas turbine engine exit plane temperature and pressure conditions, and that do not contain volatile particle contributions that condense at lower temperatures.

ORGANIC CARBON (PARTICULATE MATTER): All carbon-based particulate matter resulting from condensation and chemical reaction after the combustion process. Organic carbon contains volatile components of carbon combined with hydrogen and other elements.

PRIMARY PARTICLE: Spherical black carbon based particles formed during the combustion of hydrocarbon fuels

PM 2.5: Particulate matter <2.5 µm in classical aerodynamic diameter.

PM 10: Particulate matter <10 µm in classical aerodynamic diameter.

VOLATILE PARTICLES: Particles formed from condensable gases after the exhaust has been cooled to below engine exit conditions (e.g., sulfuric acid particles).

A.2.4 Symbols and Terminology

All units are SI. Other symbols are defined in the text as applicable to the mathematical expression at hand.

BC - black carbon

cfm – cubic ft per minute

cm - centimeter

CO₂ - carbon dioxide

EC - elemental carbon

g - gram

m - meter

mg - milligram

µg - microgram

µm - micrometer

nm - nanometer

OC - organic carbon

P - pressure, hPa

PM - particulate matter

V - volume

L - liter

slpm - standard liter per minute

SN - smoke number per ARP1179C

T - temperature, Kelvin

t - time, seconds, hours

TC - total carbon

A.3 INTRODUCTION

Aircraft gas turbine engines emit small particles ($<10\text{ }\mu\text{m}$) as a result of the combustion of hydrocarbon fuels. These are classified into two groups, nonvolatile and volatile. The nonvolatile particles are comprised mostly of carbonaceous matter from fuel combustion, particles ingested in the engine inlet, and metals from engine component erosion. By definition, only nonvolatile particles exist at the engine exit plane conditions. Volatile particles nucleate in the cooling exhaust gas from gaseous precursors. The emission of nonvolatile particles is related to the design and operating conditions of the investigated engine and to fuel properties. Current regulatory interest in stationary source particle emissions is directed toward total mass measurements. Total mass measurement techniques require measurements of volatile and nonvolatile PM, but only the measurement of nonvolatile particles is addressed in this technical appendix.

Similar to particles emitted from diesel engines (Ref. A.2.2.4, A.2.2.5), nonvolatile particles emitted from aircraft engines can continue to grow by adsorption and condensation of organic and water-soluble inorganic (e.g., sulfates) gaseous compounds. The organic compounds are reactive and may be volatile or semi-volatile, i.e., they oxidize at temperatures as low as 400 K. Elemental carbon (EC) is chemically almost inert at low and moderate temperatures and thermally stable up to temperatures of about 700 K. Thus, the EC fraction is the most stable fraction of combustion particulate matter in terms of particle transformation and temperature change during sampling. The temperature threshold separating volatile organics from nonvolatile matter is set to 350 °C (625 K). Engine exhaust gas temperature is close to or even higher than 600 K. Following the definition of nonvolatile particle matter, this PM fraction contains all material in the particle phase at the exhaust plane.

Downstream of the exit plane as the exhaust cools and volatile PM forms, the distribution between organic carbon (OC) and EC depends on the operating conditions of the engine. The respective fractions may vary from 10% EC and 90% OC at idle conditions to approximately 100% EC at take-off thrust conditions (Ref. A.2.2.7). The light absorbing material, termed black carbon (BC), is strongly correlated to the EC fraction of the aerosol. The most significant feature of the BC fraction is its graphite-like structure. These particles can absorb light in the visible spectral region very efficiently and appear as black. Light absorption by combustion particles is therefore closely linked to the key traceable compound of the nonvolatile fraction of the gas turbine exhaust aerosol.

Nonvolatile PM mass measurement methods can be divided into two general approaches: the measurement of total particulate mass and the measurement of the light-absorbing black carbon fraction. Optically-based methods which rely on the measurement of light absorption by particles measure the BC. Methods based on physical but non-optical methods measure total particulate mass. Optical methods may require extractive sampling and particle deposition on appropriate filter matrices or can be operated non-intrusively. The corresponding measurement techniques are summarized in Table A1A.

TABLE A1A - AVAILABLE METHODS FOR THE MEASUREMENT OF PARTICLE MASS CONCENTRATIONS IN AIRCRAFT ENGINE EXHAUST

Measurement Method	Measurement	Analysis
Gravimetric analysis	Total particulate mass	off line, filter samples
Tapered Element Oscillating Microbalance TEOM	Total particulate mass	on-line, extractive
Quartz Crystal Microbalance ^(a)	Total particulate mass	on-line, extractive
Combustion of filter samples and CO ₂ detection	Total carbonaceous mass, TC	off line, filter samples
Combustion analysis including OC/EC separation	Organic carbon (OC), elemental carbon (EC), OC + EC = TC	off line, filter samples
Optical absorption photometry ^(a)	Black carbon (BC), BC $\cong$ EC	on-line, extractive, filter samples, time resolution $\geq$ 1 min
Laser induced incandescence	Black carbon (BC)	on-line, real time, extractive
Laser induced incandescence	Black carbon (BC)	on-line, in situ, real time, nonintrusive
Transmission measurement	Opacity	on-line, in situ nonintrusive or extractive
Light scattering	Forward scattering	on-line, in situ nonintrusive or extractive

^(a) May require upstream dilution prior to measurement.

TABLE A1B – SELECTED METHODS FOR THE MEASUREMENT OF PARTICLE MASS CONCENTRATIONS IN AIRCRAFT ENGINE EXHAUST

Measurement Method	Measurement	Analysis
Gravimetric analysis	Total particulate mass	off line, filter samples
Tapered Element Oscillating Microbalance TEOM	Total particulate mass	on-line, extractive
Quartz Crystal Microbalance ^(a)	Total particulate mass	on-line, extractive
Optical absorption photometry ^(a)	Black carbon (BC), BC $\cong$ EC	on-line, extractive, filter samples, time resolution $\geq$ 1 min
Laser induced incandescence	Black carbon (BC)	on-line, real time, extractive
Laser induced incandescence	Black carbon (BC)	on-line, in situ, real time, nonintrusive

^(a) May require upstream dilution prior to measurement.

Based on an assessment of robustness, maturity, and applicability of available mass measurement techniques, a set of six methods was agreed upon as potential approaches for the measurement of nonvolatile PM mass emitted from gas turbine engines. The selected methods are either sensitive to PM mass (gravimetry, microbalance methods) or to light-absorbing BC (absorption photometry, laser induced incandescence). This subset of methods is compiled in Table A1B and described in detail in Sections A.4 to A.9.

For clean modern gas turbine engines, however, the detection limit of mass-based methods like gravimetry and microbalance methods becomes a serious limitation because the testing time has to be kept at a level acceptable for engine manufacturers as the main users of this Technical Appendix (TA). Absorption photometry and extractive laser-induced incandescence are sensitive down to 1.0 $\mu\text{g}/\text{m}^3$ (Ref. A.2.2.6, A.2.2.8, A.2.2.10), compared to a detection limit of the TEOM which is quantified as 200 $\mu\text{g}/\text{m}^3$ (Ref. A.2.2.3). A similar difference in detection limits holds for off-line gravimetry (detection limit approx. 3 μg) and optical absorption photometry (approx. 17 ng).

Particle sampling requirements prior to measurement are treated in TA C. TA C also covers the subject of sample dilution which is an essential sampling pre-requisite for some measurement methods (see Table A1B). Dilution, however, is always required for a simultaneous measurement of particle number as described in TA B.

The amount of particulate matter emitted from aircraft engines can be quantified in terms of mass of particles per volume of gas. Typical units are mg/m^3 or $\mu\text{g}/\text{m}^3$. Reference conditions for the gas volume have to be specified, e.g., $T = 273.15 \text{ K}$, $p = 1013.25 \text{ hPa}$ for standard temperature and pressure (STP) conditions or $T = 288.15 \text{ K}$, $p = 1013.25 \text{ hPa}$ for sea level Static (SLS) conditions. Unless stated otherwise, the particle mass concentration does not refer to a specific range of particle sizes.

A.4 GRAVIMETRY

Time-averaging methods are used to sample the emitted particles on appropriate fibrous filters or membrane filters (Ref. A.2.1.2, A.2.2.1, A.2.2.2). Since a considerable fraction of aircraft engine exhaust particles is smaller than $0.1 \mu\text{m}$ in diameter, filter materials of high sampling efficiency in this size range are required. Common practice is to use glass fiber, quartz fiber, or Teflon filters for exhaust particle sampling that show a filtration efficiency of $>99\%$ in the relevant size range. Cellulose fiber filters like Whatman No. 4, used for the current Smoke Number method (Ref. A.2.1.1), show significantly lower filtration efficiency for particles smaller than $0.1 \mu\text{m}$ in diameter (Ref. A.2.2.1) and are not recommended because of this and hygroscopic issues. To determine particulate matter mass, the technique used must specify both temperature and flow rate for the sampling system and filter efficiency. Applying gravimetry, the sampled particle matter is analyzed gravimetrically by weighing the filter before and after loading, which yields the total particle mass. The accumulated exhaust volume penetrating the filter is recorded for each sample in order to obtain a mass concentration measurement.

A.4.1 Equipment

This method is based on a sound principle - a mass measurement is obtained by sampling directly from aircraft engine exhaust, collecting the soot on a quartz filter, and weighing the sample. The method can utilize an existing SAE ARP1179C type smoke analyzer and procedure, both slightly modified, to improve the process from a qualitative to quantitative basis. Each sample collected during a specified interval provides a unique mass measurement. The technique requires pre-treating the quartz filter, heating each to above 100°C to remove condensable matter. After obtaining a sample the filter is safeguarded from contamination, by placing it inside a preconditioned container. Pre-heated glass or Pyrex Petri dishes, free from exposure to humidity, are sufficient for storage, prior to analysis.

A.4.2 Analyzer Routines

A grab sample approach is used to collect soot. The analysis method consists of weighing the collected soot to obtain mass directly. Additional information can be obtained by analyzing the sample using carbon burn-off to determine elemental carbon and organic content. Both elemental carbon and organic carbon particle composition, by percentage, is obtained. However, carbon burn-off requires a carbon analyzer (considerable capital expensive) and is not required for the direct mass measurement. A schematic diagram of a representative flow path is shown in Figure A1. An untreated (raw exhaust) sample is collected at the filter holder. Control and solenoid valves are protected from soot build up by filters internal to the system and all components are heated to 339 K (150°F) to prevent condensation and water accumulation. A suitable undiluted probe, gaseous emissions type, is used for sample collection and connected to sampling lines meeting ARP1179C Section 5.3 criteria. These sampling lines are connected to the sample inlet identified in the flow schematic. The filter holder firmly clamps quartz filter paper (4 cm diameter) using sample holders having soft seals, which prevents paper tear and minimizes any system leakage. The filter holder design is based on ARP1179C Figure 2 criteria. In addition, valves, temperature control, sample pump, sample size measurement, and sample flow rate measurement are specified identical to ARP1179C Sections 5.5 to 5.9 criteria.

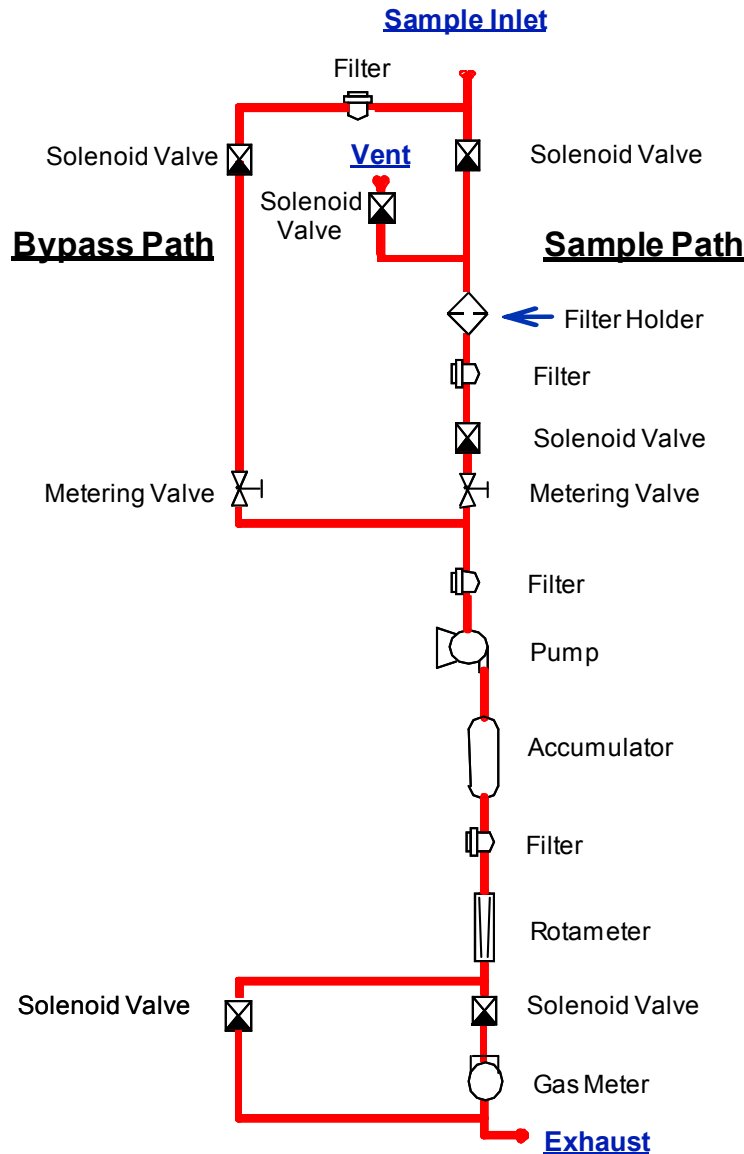


FIGURE A1 – SCHEMATIC OF THE GRAVIMETRIC SAMPLING STREAM FLOW PATH

A.4.3 Calibration Standards

A.4.3.1 Filter Media

Samples shall be acquired on quartz filters. Whatman type QMA filters have 99.95% particle retention at 0.3 μm , are often used for measuring total carbon mass deposited on the filter. However, their efficiency below 0.3 μm must be established before being considered for certification use. Because research suggests new aircraft engines commonly emit a majority of particles in the range of 10 to 300 nm, this technique may not be suitable to use in these new engines until the filter efficiency characteristics are quantified, or appropriate filters are manufactured, for the lower range of interest. Total mass is obtained using suitable scales, as defined in Section A.4.3.2.

A.4.3.2 Balance

A microbalance is required to accurately measure the sample mass obtained. The balance shall have a readability of 0.0001 mg (0.1 μg) and minimum capacity of 1000 mg.

Performance specifications shall be:

Repeatability $\leq 0.25 \mu\text{g}$

Linearity $0.5 - 1 \mu\text{g}$

Sensitivity 1 ppm/K

Stability $\leq 0.1 \text{ ppm}$

Follow manufacturer guidance to properly calibrate the balance before each use. It is recommended the balance is calibrated to ISO traceable standards annually.

A.4.3.3 Carbon Burn-off

A carbon analyzer is used to determine the total soot mass deposited on the quartz filter, by measuring the carbon dioxide produced while increasing the temperature of the filter in an oven, in the presence of excess oxygen. This technique is commonly referred to as carbon burn-off. It provides another quantitative and comparative approach to obtain particle mass, although it is a non-gravimetric technique. Typical carbon analyzers are the model RC-412 Multiphase Carbon/Hydrogen/ Moisture Determinator, manufactured by LECO and the Carbon Aerosol Analysis Lab Instrument, manufactured by Sunset. The manufacturer's procedures and standards shall be used for the equipment calibration. An example analysis signal for a quartz filter sample, shown in Figure A2, is integrated to obtain total carbon, which is proportional to the amount of carbon dioxide produced. The total carbon is weight corrected to obtain organic and elemental carbon concentrations.

A.4.4 System Layout

The system layout is composed of the components identified in the schematic of the gravimetric sampling stream flow path diagram (Figure A1) internal to the enclosed system. All sample path lines and components are insulated and maintained at 339 K (150°F). A representative gravimetric sampling system is shown in Figure A3. The legend provides identification of all control inputs and outputs, monitoring units and connections for external air and nitrogen sources.

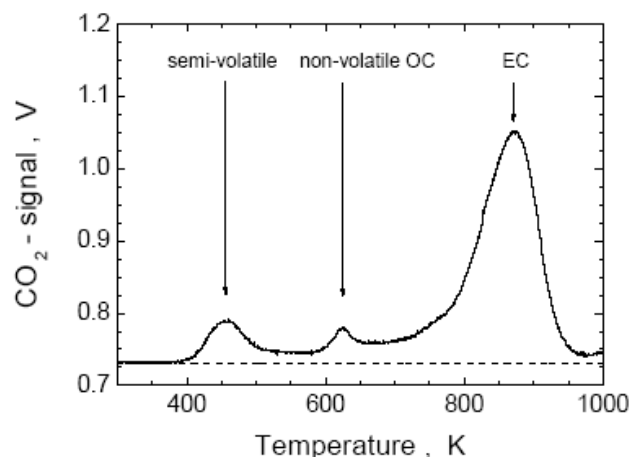
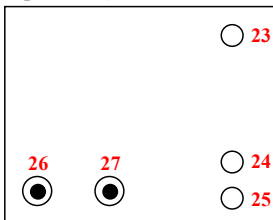


FIGURE A2 – CARBON MASS (TEMPERATURE PROGRAMMED OXIDATION)

Smoke Sampling Machine Parts Legend

1. Thermocouple (5-channel) Indicator
2. Pressure Indicator
3. Ball Valve
 - Up-Position: pressure at flow meter
 - Down-Position: pressure at sample line
4. Clamping Pressure Indicator
 - Set to 414 KPa (60 psig)
5. Sample Holder Close Lamp (green)
6. Sample Holder Open Lamp (blue)
7. Flow Meter (L/min)
8. Bypass Metering Valve
9. Sample Metering Valve
10. Filter Holder Diffuser Unit
11. Timer Indicator & Control
12. Volume Indicator & Control
13. Sample Mode Switch (Open/Close)
14. Leak Check Switch & Lamp (clear)
15. Constant Sample Switch & Lamp (yellow)
16. Bypass Switch & Lamp (green)
17. Sample Switch & Lamp (red)
18. Reset Switch
19. Drain Switch
20. Pump "On" Switch
21. Master Power Switch
22. Pump "On" Lamp (red)
23. Sample Exhaust Line Connection
24. Nitrogen Connection
25. Air Connection
26. Sample Input Quick-Disconnect
27. Quick-Disconnect Plug (no connection)
Used to secure sample line without connecting to the machine

Top View (Line Connections)



1.5 m height, 110 Kg, 8.8 amps

FIGURE A3 - REPRESENTATIVE GRAVIMETRIC FILTER SAMPLING SYSTEM

A.4.5 Test Procedure

The following is a typical procedure used to perform system checks and sample the aircraft engine exhaust.

A.4.5.1 System Checkout Procedure

1. Check oil reservoir in pump. Oil reservoir should be at least half full.
2. Check air supply line. It should be installed at 690 kPa (100 psi).
3. Check Nitrogen purge line. This purges and creates a back pressure on probe.
4. Ensure vent line is installed and vents out of the test cell.
5. Insulated sample line should be attached, powered on, and set for 422 K (300 °F).
6. Ensure the equipment is powered by a 9 amp, 120 volt circuit.
7. Turn on master power and pump "on" switch.
8. Allow one hour for complete warm-up.
9. Flow meter should be set to 14 L/min (0.5 cfm) for both bypass and sample lines.

10. Clamping pressure should read 414 kPa (60 psi).
11. Sampling should occur to obtain a volumetric reading of 7 L/min (0.25 cfm) and typically takes about 30 seconds, if the flow meter is achieving the correct amount of flow.
12. Check sample line and flow meter pressures using the ball valve on front console. Sample must be engaged to check these pressures. If line pressure is below 55 kPa (8 psi) the sample line could be clogged.

A.4.5.2 Sampling Procedure

The sampling flow rate shall be maintained at 14 L/min $\pm$ 0.5 L/min (0.5 cfm $\pm$ 0.02 cfm). The sample shall be taken over a period of 30 s intervals, and time averaged, to obtain sufficient soot mass for gravimetric assessment. Three samples at each engine condition are recommended.

1. Ensure the filter holder diffuser unit is open, operated by using the sample mode open/close switch.
2. Place a conditioned quartz filter into the filter holder and set inside the diffuser unit.
3. Close the diffuser unit using sample mode open/close button.
4. Press the reset switch.
5. Press the sample switch.
6. After sample is complete open the diffuser unit, remove filter holder, and store it in a pre-treated glass, or Pyrex container.
7. Repeat as necessary to obtain more samples.
8. When not sampling, connect the sample line to nitrogen purge.

A.4.6 Data Flow

For each sample, the time-averaged result of total measured exhaust volume shall be assessed to obtain the mass/volume, after weighing the sample. The particle mass shall be reported in units mg/m³, after performing mass and volume conversions. Upon determining the gravimetric mass, the quartz filter sample shall be further assessed using the carbon burn-off method to obtain total carbon mass and relative organic and elemental and mass concentrations.

Data reported shall include sample exhaust flow rate, mass concentration averaged over 30 s time intervals, total carbon mass, percent organic carbon, and percent elemental carbon.

A.4.7 Calculation of Results

Fuel-specific PM EIs shall be calculated from the gravimetric data using the procedure outlined in TA D.

A.5 MICROBALANCE – TAPERED ELEMENT

Two on-line methods that have been used for the determination of total particulate matter (PM) mass emissions are the tapered element oscillating microbalance (TEOM) and the quartz crystal microbalance (QCM, see Section A.6). Both these methods have been used successfully in similar evaluations of automotive internal combustion engines, with the TEOM also recently introduced for measurements in the aerospace industry.

In the TEOM instrument, a hollow vibrating tube equipped with a small in-line filter is installed inside a heated chamber. The change in harmonic oscillation measured by a sensitive frequency counter over a prescribed averaging time is proportional to the additional PM mass collected on the filter. The TEOM was originally designed to monitor PM ambient air quality but has been adapted for use in automotive engine testing (Refs. A.2.2.2, A.2.2.3). The TEOM mass values must be corrected for pressure loss across the filter and total sample flow through the TEOM. In general, the TEOM has been shown to correlate reasonably well to the total filter method as determined over extended averaging periods, but it has high instrumental noise making shorter term measurements problematic.

A.5.1 Equipment

A tapered element oscillating microbalance (TEOM) shall be used for the determination of total PM mass concentration. An instrument such as the Thermo Scientific Series 1105a Diesel Particulate Mass Monitor (Figure A4), or equivalent analyzer, is acceptable for this purpose. The following procedures are based on the Series 1105a and its associated Operating Manual.



FIGURE A4 - THERMO SCIENTIFIC SERIES 1105A TEOM

A.5.2 Analyzer Routines

A.5.2.1 Leak Checks

Leak checks must be performed upon installation and periodically thereafter. If using a typically configured Monitor, only the system's sample flow path must be checked. If using the bypass/purge option, both the sample flow path and the bypass/purge flow path must be checked. Refer to the instrument operating manual for step-by-step instructions on performing sample flow path and bypass/purge flow path leak checks.

A.5.2.2 Flow Audits

A flow audit verifies that flow rates in the Monitor meet specifications. A flow audit must be performed upon installation and at least once per year thereafter. A leak check must be performed before the flow audit. Refer to the instrument operation manual for step-by-step instructions on performing a flow audit using a NIST-traceable flow standard.

A.5.2.3 Differential Pressure Verification/Calibration

The differential pressure compensation across the TEOM element shall be verified upon installation and at least once per year thereafter. The same procedure is used to audit and/or calibrate the differential pressure across the TEOM filter element. A needle valve and filter (e.g., Pressure Compensation Test Kit, Part No. 55-008602, or equivalent) shall be used for this purpose as outlined in the instrument operating manual. If the "New mDP" value is not between 0.002 and 0.006, the instrument shall be returned to the manufacturer for repair and recertification.

A.5.2.4 Differential Temperature Verification/Calibration

The differential temperature compensation across the TEOM element shall be verified upon installation and at least once per year thereafter. The same procedure is used to audit and/or calibrate the differential temperature across the TEOM filter element as described in the instrument operation manual. If the "New mTemp" value is not $4.3 \times 10^{-3} \pm 10\%$ the instrument shall be returned to the manufacturer for repair and re-certification.

A.5.3 Calibration Standards

The spring constant (K_0) of the mass transducer shall be verified upon installation and at least once per year thereafter. A filter element of known mass (e.g., Mass Calibration Verification Kit, Part No. 59-008298, or equivalent) shall be used for this purpose as described in the instrument operating manual. If the new K_0 determined by this procedure is not within $\pm 2.5\%$ of the original factory calibration, the mass transducer shall be returned to the manufacturer for repair and re-certification.

A.5.4 System Layout

The sampling system shall consist of a heated sample line connected to the "EXT. HEATER" port on the back of the 1105a and the Monitor itself as illustrated in the instrument operation manual. The heated sample line shall be set to a temperature equivalent to that of the TEOM mass transducer (nominally 50 °C). Additional information on set-up of the sampling system can be found in instrument operation manual.

A.5.5 Test Procedure

A.5.5.1 Starting a Sampling Run

Refer to the instrument operation manual for more specific instructions on setting up the software parameters.

1. Install a new TEOM filter.
2. Ensure the sensor unit is off. Always start the RP1105A software before turning the sensor on.
3. Start the RP1105A software program from the program icon located on the computer's desktop.
4. Select the data saving settings
 - a. Select a directory to which the data files are saved.
 - b. Select the types of data to save.
 - c. Change the filename.
 - d. Select the Auto Save function.
 - e. Change the text delimiter of the data files, if necessary (i.e., comma or tab).
 - f. Include settings, caption, and format information in the data files, if required.
 - g. Go to the Configuration screen and verify that all configuration settings are appropriate for the sampling run then select OK.
5. Turn on the sensor unit.
6. Go to the Main screen and set the Text Display, Graphic Control, and Graphing Area frames to the desired parameters (Refer to the instrument user manual for more detailed information on these options).
7. Select Instrument and then Start from the menu bar to begin collecting data. Selected data values will display in the Text Display and in the Graphing Area frames.

A.5.5.2 Data Retrieval

1. To stop data collection, from the Main menu select Instrument, and then Stop from the menu bar.
2. If the Auto Save option was selected for the sampling run (See Step 4a, above), go to Step 3, below. If the Auto Save function was not used:
 - a. On the Main screen, select File, Save Data >, and then Current Data. The Save Current Data window will open.
 - b. Choose a folder for data storage.
 - c. Designate a filename for the data file(s).
 - d. Choose the data storage type.
 - e. Select OK.

3. In Microsoft Windows Explorer, locate the data file of interest and double-click on the file to display the file contents. This file is ready for data graphing or reduction.

A.5.6 Data Flow

The data files are produced by the Monitor in the form of Excel spreadsheets which includes the mass concentration averaged over 10 s and 1 min time intervals as well as other key instrument operating parameters. Other averaging times can also be added to the data output stream as described in the instrument manual.

A.5.7 Calculation of Results

Fuel-specific PM EIs shall be calculated from the TEOM data using the procedure outlined in TA D.

A.6 MICROBALANCE – QUARTZ CRYSTAL

An older instrument, which has recently been reintroduced to automotive engine testing, is the quartz crystal microbalance (QCM). The harmonic oscillator principal used in the QCM is similar to the TEOM except that the collected PM is actually deposited on the crystal element itself using an electrostatic precipitator (Ref. A.2.2.3). Agreement between QCM measurements and traditional PM mass methods was found in recent testing conducted in the UK (Ref. A.2.2.3).

A.6.1 Equipment

A quartz crystal microbalance (QCM) shall be used for the determination of total PM mass concentration. A QCM instrument such as the SEMTECH-QCM (Figure A5) or equivalent analyzer is acceptable. The following procedures are based on the SEMTECH-QCM and its associated User Manual.

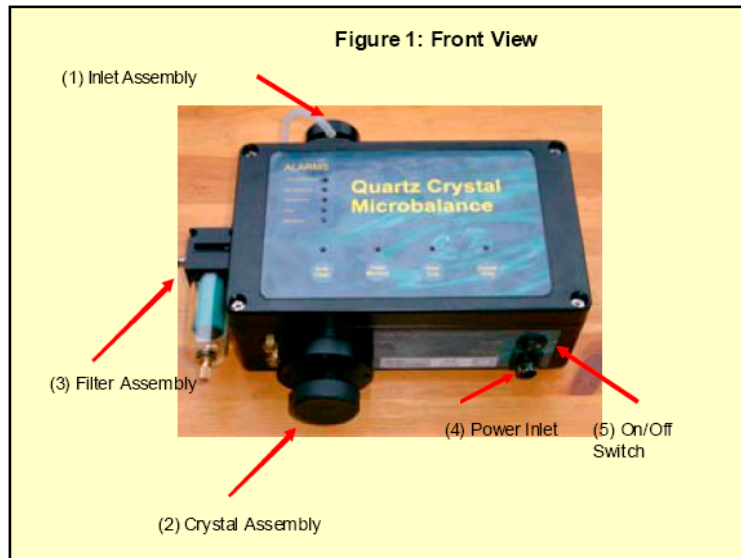


FIGURE A5 - SEMTECH-QCM INSTRUMENT

In the QCM, a piezoelectric crystal is used as a sensitive microbalance. The crystal is excited at its natural frequency, which decreases with increasing mass loading on its surface. The particulate mass collected on the crystal is measured by the change in the crystal's natural frequency. A point-to-plane electrostatic precipitator is used to deposit the material on the crystal surface.

A.6.2 Analyzer Routines

A.6.2.1 Instrument Zero

The instrument shall be zeroed before and after each sample collection period. This shall be accomplished by installing a High Efficient Particulate Air (HEPA) filter on the inlet of the QCM and monitoring the resulting concentration for a period of at least 10 min. Note that the QCM, unlike most instruments, will not achieve an absolute zero concentration. Instead, the average concentration measured with the HEPA filter installed shall be used to correct the data obtained during actual sampling as described below.

A.6.2.2 Crystal Cleaning

Before each use, and whenever needed during operation, the quartz crystal shall be cleaned using the “Basic Cleaning Procedure” outlined in Section 3.9 of the User Manual. At least once per year, or sooner if necessary, a major cleaning of the crystal and electrostatic precipitator shall be performed following the steps also described in Section 3.9 of the Manual under “Major Cleaning Procedure.”

A.6.2.3 Flow Audit

A flow audit verifies that flow rates in the QCM meet specifications. A flow audit must be performed upon installation and before each measurement campaign. The flow audit shall be conducted by connecting a NIST-traceable flow meter to the instrument inlet and comparing the analyzer flow to the reference flow. If the two differ by >2%, a multipoint flow calibration must be conducted as outlined in Section A.6.3.2 below.

A.6.3 Calibration Standards

A.6.3.1 Sensor Validation

Standard calibration aerosols comprised of either fluorescent-tagged monodisperse polystyrene latex microspheres or polydisperse methylene blue particles shall be generated by nebulizing either a liquid suspension or dye solution, as appropriate. The aerosol shall then be dried and the charge neutralized before being supplied to the QCM sensor. The mass of particles collected shall be determined by wet solvent recovery from the crystal surface and subsequently analyzed using fluorescent spectro-photometry. These results shall then be compared to the change in frequency measured by the instrument to determine crystal sensitivity. (Also see Section 4.2 of the User Manual.)

A.6.3.2 Flow Calibration and Leak Check

Flow calibration shall be conducted at least annually as outlined in Section 4.3 of the SEMTECH-QCM User Manual. Due to its design, a typical leak check is not possible. Instead, the operator should check all gasketed surfaces prior to operation to assure integrity of the air flow path. If any gasket appears worn, these must be replaced prior to putting the instrument into service.

A.6.4 System Layout

The sampling system shall consist of a heated sample line connected to the QCM inlet. The heated sample line shall be set to the same operating temperature as that of the QCM crystal. Additional information on set-up of the sampling system can be found in the Operating Manual.

A.6.5 Test Procedure

A.6.5.1 Starting a Sampling Run

Refer to Sections 3 and 5 of the SEMTECH-QCM User Manual for more details on instrument operation and crystal change-out as well as the Software Manual-Version 3 for details on the installation and operation of the software.

1. Turn on the instrument without flow and allow it to equilibrate for at least 20 min.

2. Connect signal cables, if necessary, and activate the SEMTECH software. Change the instrument data collection parameters, as needed, by clicking on Instrument Set-up. Refer to the Software Manual for details.
3. Install a new crystal in the holder and replace the holder in the instrument. Also clean the electrode using the procedure outlined in Section 3.9 of the SEMTECH-QCM User Manual. Allow the instrument to warm up without air flow.
4. Start the air flow and allow the instrument readings to stabilize for a minimum of 10 to 15 min.
5. Activate the X-Y Display on the Run button of the software and click on Save File to begin data collection. The software will ask for a file name and location. Enter this information and note the time that data logging started on the data sheet or lab notebook.
6. Install a filter on the inlet and allow the analyzer to run for at least 10 min with the filter in-line. The instrument readings with the filter installed establishes the zero baseline which must be subtracted from the data collected without the filter. The zero baseline should be somewhere around 20 to 25 $\mu\text{g}/\text{m}^3$.
7. Remove the filter and note the time on the data sheet or lab notebook.
8. Begin the experiment as outlined in the approved test plan.
9. Monitor the alarms located at the bottom of the X-Y Display screen for "Frequency", "Corona Current", "Temperatures", and "Flow." If any alarm shows red, clean the crystal and allow at least 10 min for stabilization. If an alarm is still present, check instrument operation per the User Manual or Troubleshooting Guide provided by SENSORS, Inc.

A.6.5.2 Ending a Run and Data Retrieval

Follow these steps to end instrument operation and retrieve the test data:

1. After completion of the test run, stop the data collection by clicking on Save File. Note the time at which data collection was stopped on the data sheet or lab notebook. Also verify that the file was saved on the hard drive of the computer operating the instrument.
2. Retrieve the data file from the folder selected above and also save a copy on compact disk.
3. Press Return to Main Menu in the X-Y Display screen and exit the software.
4. The instrument may either be left on or turned off at the discretion of the operator. Do not change crystals if the instrument is left active.

A.6.6 Data Flow

The data files produced by the QCM are in the form of tab-separated, ASCII text files. These files can be imported into an Excel (or other) spreadsheet program, as desired for further analysis. The data files show the PM concentration as well as key instrument operating parameters.

A.6.7 Calculation of Results

Fuel-specific PM EIs shall be calculated from the QCM data using the procedure outlined in TA D.

A.7 MULTIANGLE ABSORPTION PHOTOMETRY

Aerosol absorption photometry is one specific set-up of optical absorption photometry. It analyzes the modification of filter optical properties such as transmittance or reflectance caused by the particles deposited on the filter matrix (Ref. A.2.2.6, A.2.2.8). Optical absorption methods are highly suitable for the measurement of combustion particles because the key component black carbon is a very efficient absorber of light in the visible spectral range. Since total particulate matter contains also a fraction of non-absorbing aerosol components, absorption photometry cannot be used for the measurement of total particle mass. However, black carbon makes up the largest and most stable fraction of aerosol components in the exhaust of a gas turbine engine (Ref. A.2.2.7). Besides its specific sensitivity to the main combustion product black carbon aerosol absorption photometry has the further advantage of being operated continuously such that time-resolved recordings of particle emissions are possible.

A.7.1 Equipment

Multi-Angle Absorption Photometry shall be used for the determination of the light-absorbing elemental or black carbon, respectively, in emission samples. BC is the key component with respect to combustion-related particulate emissions. It does not, however, represent the entire emitted particulate matter, since organic carbon and water-soluble components are not measured. An instrument such as the Model 5012 Multi Angle Absorption Photometer (MAAP) black carbon monitor offered by Thermo Electron measures ambient and source black carbon (BC) concentrations and aerosol light absorption properties. MAAP is based on the principle of aerosol-related light absorption and the corresponding atmospheric BC mass concentration. The Model 5012 uses a multi angle absorption photometer to analyze the modification of radiation fields in the forward and back hemisphere of a glass-fiber filter caused by deposited particles. The data inversion algorithm is based on a radiation transfer method and therefore takes multiple scattering processes inside the deposited aerosol and between the aerosol layer and the filter matrix explicitly into account. Figure A6 shows the Model 5012 MAAP instrument for continuous black carbon monitoring.

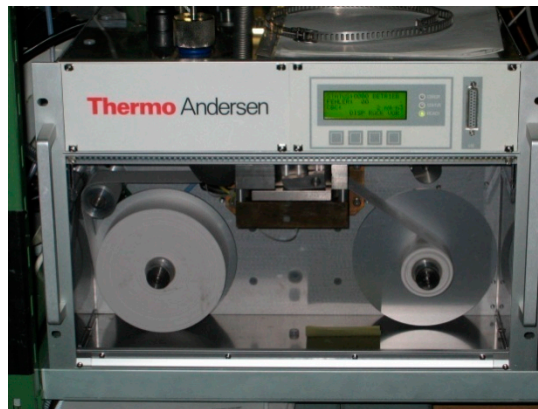


FIGURE A6 - MODEL 5012 MAAP FROM THERMO SCIENTIFIC

A.7.2 Analyzer Routines

A.7.2.1 System Operation

The sample is drawn into the Model 5012 through the inlet, as shown in Figure A7. The sample flows through the sampling tube and deposits onto the glass fiber filter tape. The filter tape will accumulate an aerosol sample towards a threshold value, whereupon the filter tape will automatically advance prior to reaching saturation. Within the detection chamber a 630 nm visible light source is aimed towards the deposited aerosol and filter tape matrix. The light transmitted into the forward hemisphere and reflected into the back hemisphere is measured by a series of photo-detectors, see Figure A8. During sample accumulation the light beam is attenuated from an initial reference reading from a clean filter spot. The reduction of light transmission, multiple reflection intensities, and air sample volume are continuously integrated over the sample run period to provide a real-time data output of black carbon concentration measurements.