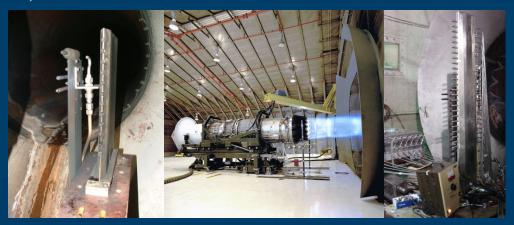


Development of calibration procedures for non-volatile particulate matter mass measurement methods--status report

John Kinsey, U. S. Environmental Protection Agency, Office of Research and Development, National Risk Management Research Laboratory, Research Triangle Park, NC





Research problem

- Three candidate methods were identified by the SAE E-31
 Committee for possible use in an Aerospace Recommended Practice (ARP) for the measurement of non-volatile particulate matter (PM) mass emissions during engine certification. A fourth technique was added later at the request of EPA's Office of Transportation and Air Quality.
- None of these techniques measure PM mass directly, have a standard procedure available to implement the method, or incorporates a process to adequately quality-assure the data collected.
- A program was designed to standardize each method, provide appropriate quality assurance/control procedures, and validate the measurements against a traceable standard method (filter gravimetric).



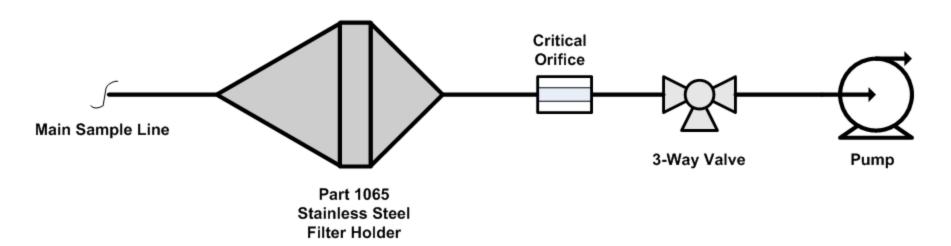
Program objectives

- Develop Standard Operating Procedures (SOPs), including quality assurance/quality control checks, for four near-real-time non-volatile particulate matter (black carbon) mass measurement techniques including:
 - Carbon burn-off [i.e., National Institute of Occupational Safety and Health (NIOSH) Method 5040]
 - Multi-Angle Absorption Photometer (MAAP)
 - Laser Induced Incandescence (LII)
 - Photoacoustic analysis (PA)
- Validate all four methods against the filter gravimetric technique using a known black carbon aerosol source (Mini-CAST) indicative of turbine engine exhaust in a controlled laboratory environment
- If possible, determine the sensitivity of these techniques to organic carbon in the test aerosol
- Provide the above information to the SAE E-31 Committee for inclusion in an Aerospace Recommended Practice for the measurement of non-volatile PM mass to be used in future engine certification



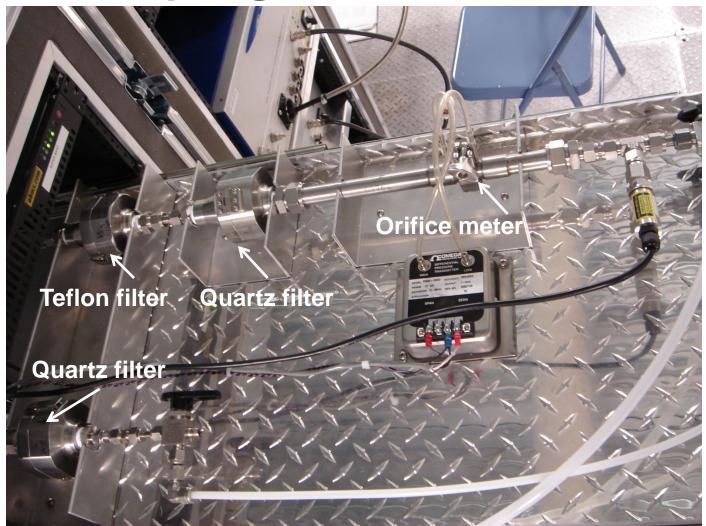
Modifications to NIOSH Method 5040

- Development of specialized sampling train for gas turbine exhaust
- Validation against filter gravimetric technique





Filter sampling trains



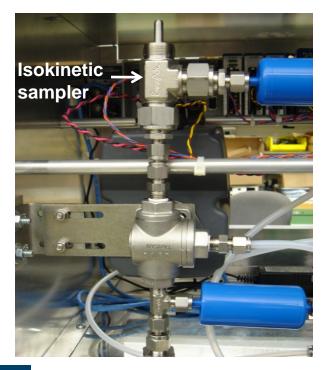


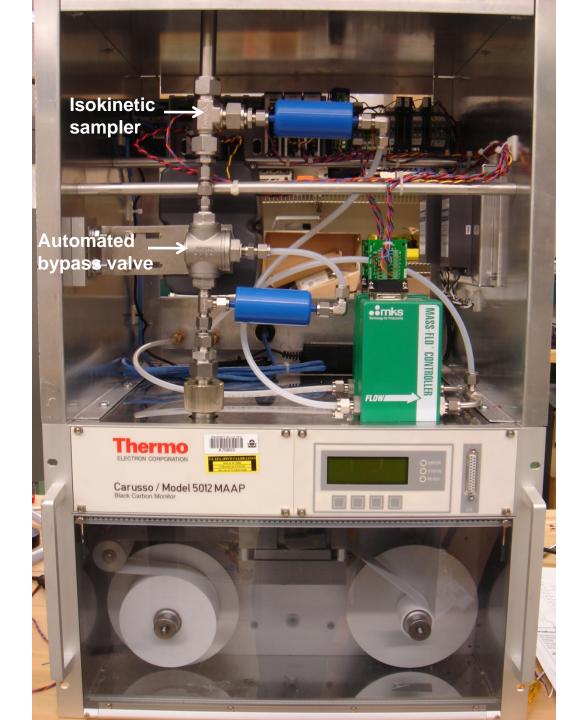
Modifications to Thermo Scientific Model 5012 MAAP for use in engine certification

- Reduce the flow through the filter tape to extend the time between filter changes
- Isolate the MAAP from the main sampling line during filter changes
- Perform the necessary calculations to determine black carbon (BC) concentration on a 1 Hz basis and log the data
- Calculate appropriate statistics from the calculated BC concentrations
- Provide the ability to implement a manual filter change
- Monitor the percent light transmission in real time so that the operator can determine when a filter change is about to take place
- Allow for and document some type of quality control check to tell the operator the instrument is working properly and ready for use
- Develop an add-on "package" incorporating the necessary changes for use in certification environments



Photos of SuperMAAP hardware modifications

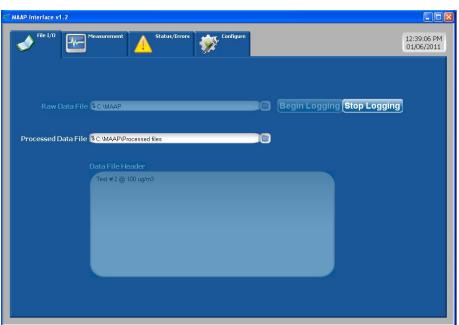


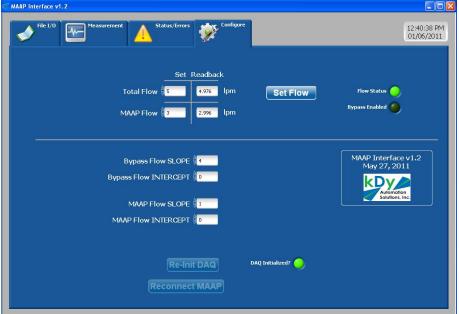




SuperMAAP user interface









Modifications to Artium Technologies LII 300 Instrument

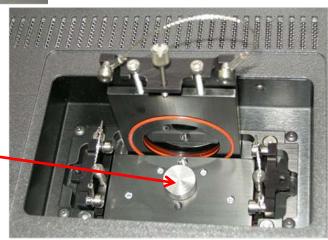
- Incorporation of a flow meter, filter, and external pump to monitor sample flow rate to the instrument
- Addition of an independent light source to be used as an independent QC check to verify proper instrument operation before starting measurements
- Ability to control instrument from a remote computer
- Procedure added to the firmware package for cell temperature and pressure calibration



Artium LII 300 independent light source

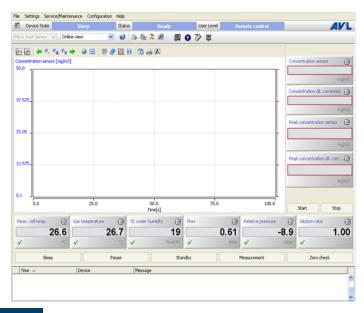


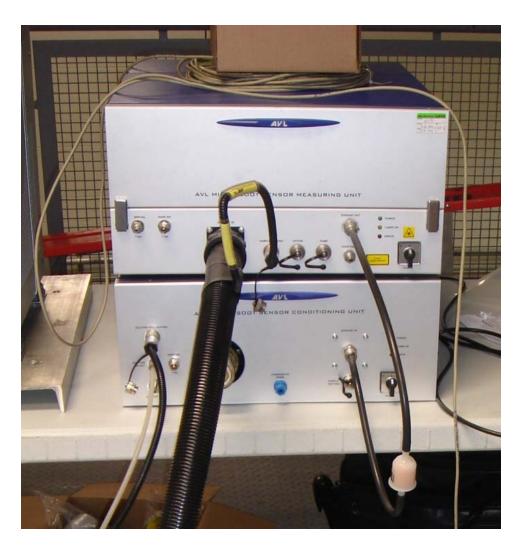
Access port for independent light source





AVL 483 Micro-Soot Sensor (MSS) photoacoustic analyzer





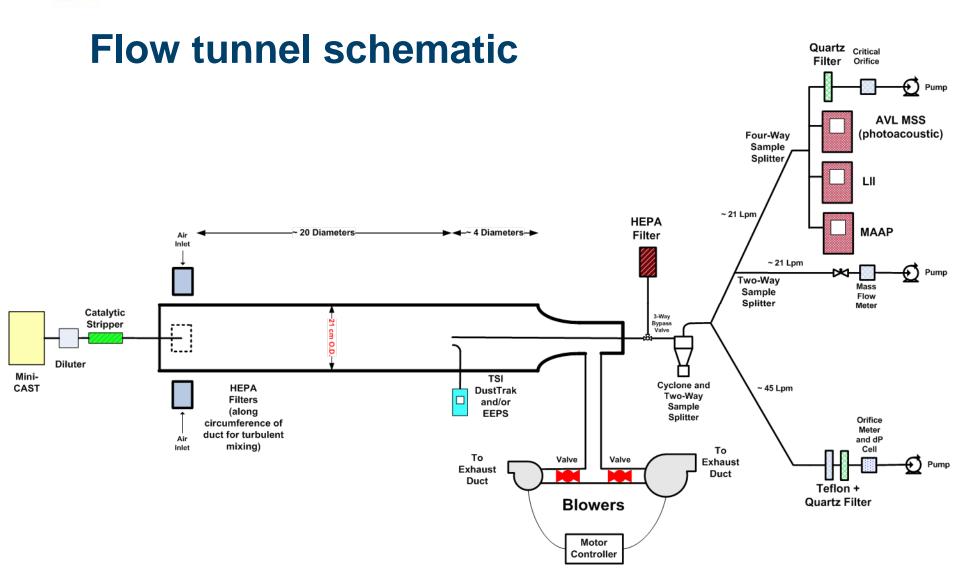
Note that Conditioning Unit and heated line/diluter are not being used



Apparatus for methods validation

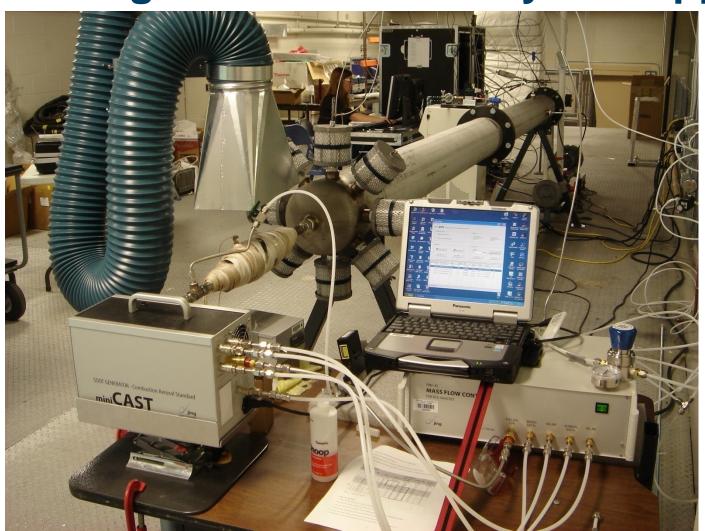
- Low speed flow tunnel (one pass, unheated)
- Mini-CAST burner (black carbon aerosol generator)
- Catalytic stripper (to remove volatile PM)
- ~ 1.7μm cut-point cyclone pre-separator (to eliminate large agglomerates shed from walls)
- Sample splitters
- Sampling trains for Teflon and quartz filters
- LII 300, SuperMAAP, and AVL MSS





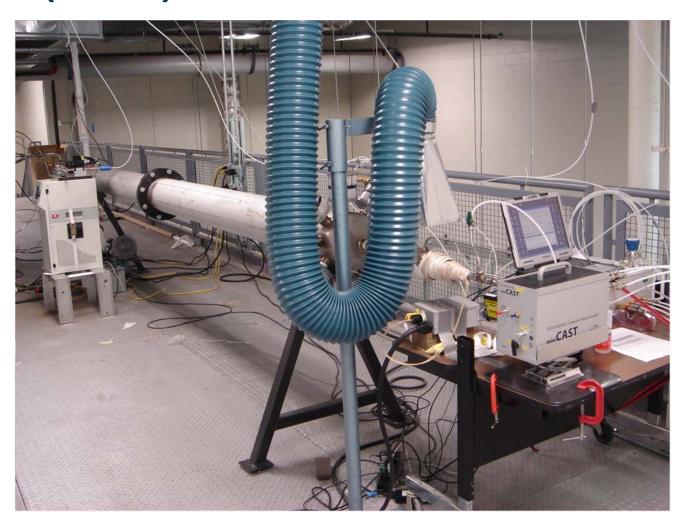


Aerosol generator and catalytic stripper



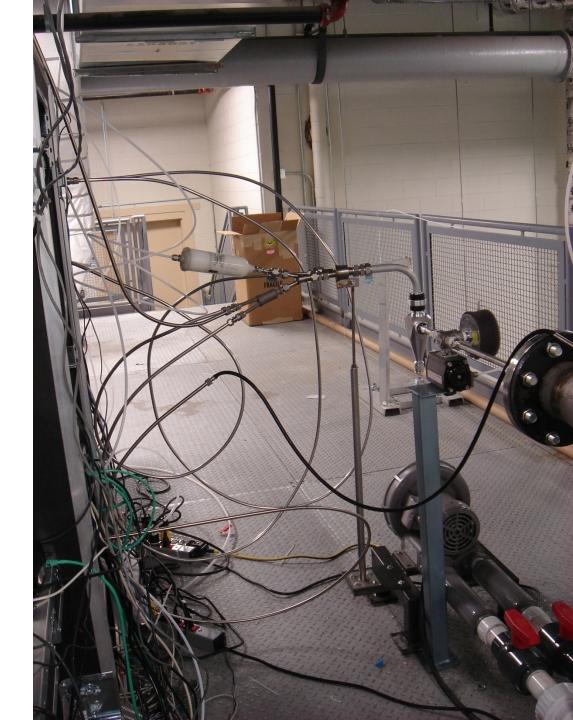


Flow tunnel and Engine Exhaust Particle Sizer (EEPS)



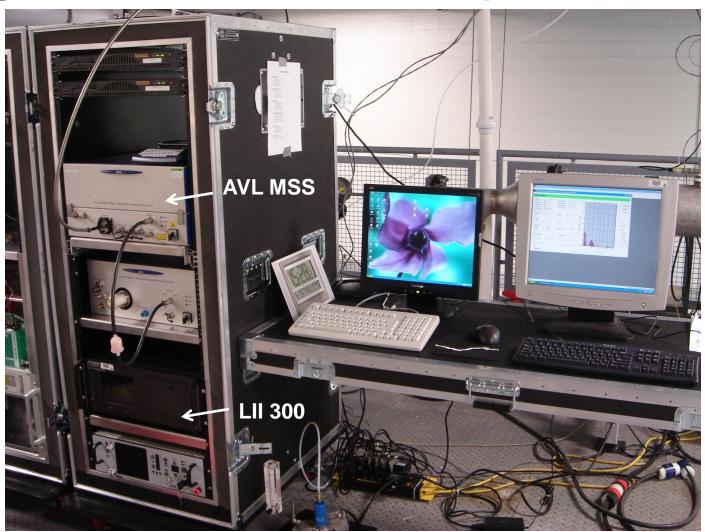


Sample distribution system



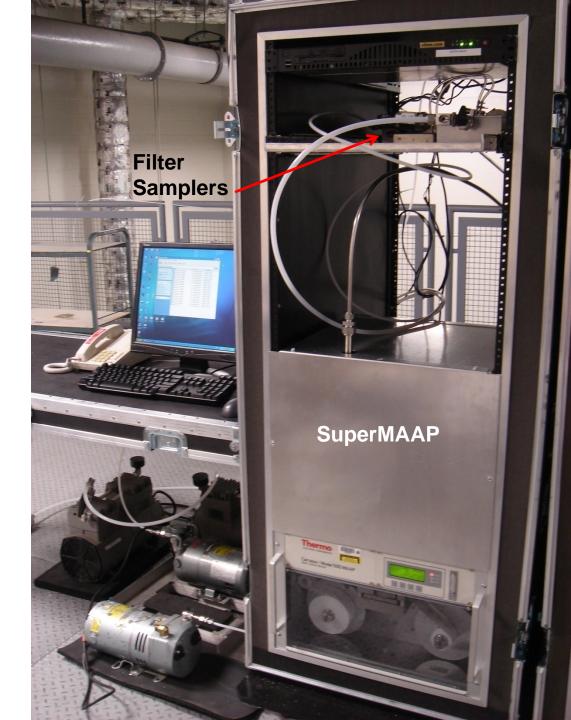


Right instrument rack and operator's station



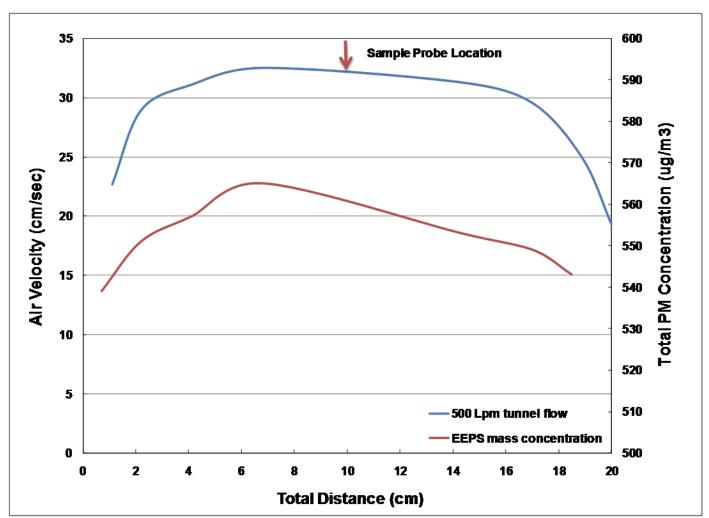


Left instrument rack and operator's station



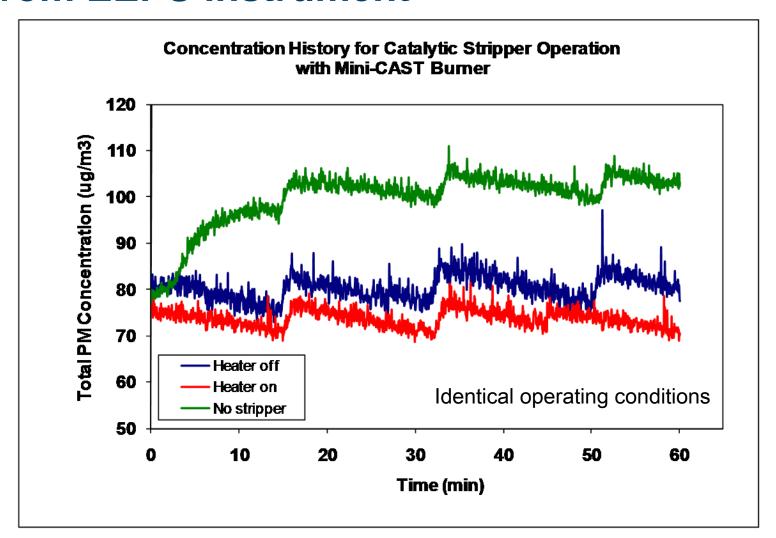


Typical tunnel velocity and concentration profile



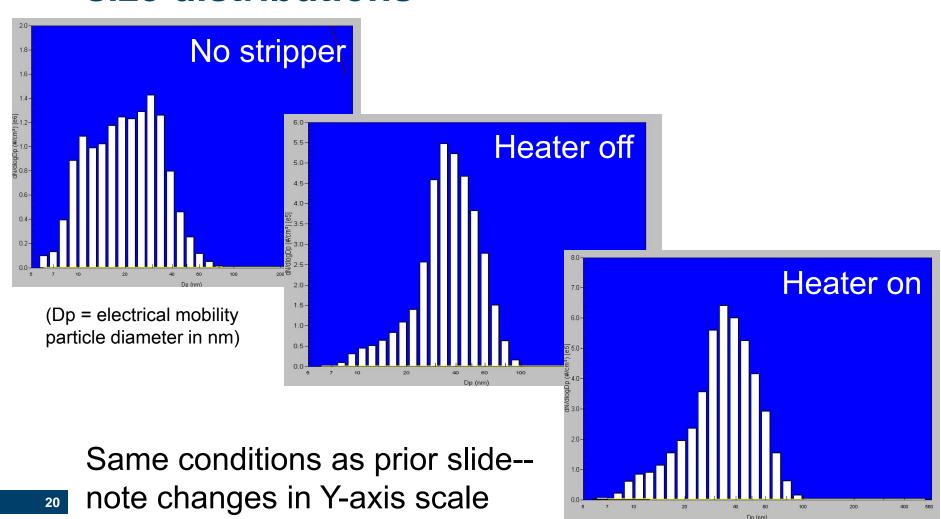


Typical CAST mass concentration output from EEPS instrument





Typical CAST differential number particle size distributions





Experimental matrix

Aerosol Type ^a	Sampling Condition	Target Soot Concentration (μg/m³)	No. of Runs	Teflon Filter	Quartz Filter	MAAP	LII	* AVL MSS
Non-volatile PM	1	10	6	X	X	X	X	X
	2	50	6	X	X	X	X	X
	3	100	6	X	X	X	X	X
	4 ^b	500	6	X	X	X	X	X
	5	1000	6	X	X	X	X	X
Total PM⁵	1	10	6	X	X	X	X	X
	2	50	6	X	X	X	X	X
	3	100	6	X	X	X	X	X
	4 ^c	500	6	X	X	X	X	X
	5	1000	6	X	X	X	X	X

^a Non-volatile PM = Mini-CAST® with catalytic stripper; Total PM = Mini-CAST without catalytic stripper (will include volatile organics).

^b Conducted only if time and resources permit.

^c Lowest priority tests.



Current program status

- LII and MAAP instrument workshops were held at EPA
- SOPs have been developed for all methods and instruments which are currently undergoing EPA Quality Assurance review
- Flow tunnel and associated apparatus has been completed and all instruments and sensors have now been calibrated by the APPCD Metrology Laboratory
- All modifications have now been completed for the SuperMAAP and its performance exceeds all expectations
- Operational data have been collected on the flow tunnel and the Mini-CAST burner both with and without the catalytic stripper
- Two preliminary tests were conducted with all instruments except the LII which malfunctioned and is currently undergoing repairs



Tentative work schedule

	Jun	Jul	Aug	Sep	Oct	Nov	Dec	2012		
Activity								Jan	Feb	Mar
Conduct Non-Volatile PM Tests			l							
Conduct Total PM Tests										
Data Reduction/Lab Analyses										
Draft Report Preparation										
Final Report Preparation										