

Brick Kiln Emissions Sampling Protocol: Dilution sampling for climate-relevant particle emissions

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July 5, 2016

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1 Scope

This protocol is designed to measure climate relevant characteristics of particle and gas emissions from brick kilns using a dilution sampling system coupled with carbon balance calculations. This protocol is applicable for kilns with and without exhaust stacks and has been tested on bull's trench, zigzag, clamp, vertical shaft, and tunnel kilns in South Asia and Vietnam. General guidelines for selecting measurement methods for brick kilns can be found in the report: *Brick Kiln Measurement Guidelines: Black Carbon Emissions and Energy Performance* [9].

Particle characteristics included in this protocol include particulate matter mass smaller than 2.5 micrometers (PM_{2.5}), organic carbon (OC), elemental carbon (EC), optical scattering by particles, and optical absorption by particles. Gas species include carbon dioxide (CO₂), carbon monoxide (CO), and sulfur dioxide (SO₂). This protocol describes the equipment, sampling procedure, and calculations required to produce the output metrics listed in Table 1.

Table 1: Output emission metrics determined in this protocol. The numerators of the emission metrics are pollutant mass (in grams) and refers to the mass of each of the pollutants measured in the protocol (PM_{2.5}, BC, OC, CO, and SO₂). Modified combustion efficiency is not species specific.

Output Metrics for Species: PM_{2.5}, BC, OC, CO, and SO₂

Fuel based emission factor	g pollutant/kg fuel
Brick based emission factor	g pollutant/kg fired brick
Energy based emission factor	g pollutant/MJ
Emission rate	g pollutant/hr
Carbon emission ratio	g pollutant/g Carbon
Modified combustion efficiency	%

Fuel measurements are required to calculate emission metrics. This method requires continuous monitoring of fuel consumption during the emission sampling period, and fuel samples must be taken for laboratory analysis. See the *Introduction to Brick Kilns & Specific Energy Consumption Protocol for Brick Kilns* [2] for additional guidance on fuel and energy measurements.

2 Definitions

1. LPM: liters per minute
2. D₅₀ Cutpoint: This is a design specification for particle size separators. It is the particle diameter for which 50% of the particles are removed.
3. T₉₀ Response Time: This is a design specification for real-time sensors. It is the time that it takes for a sensor signal to reach 90% of its steady state value when the sensor is exposed to a step change of the target species.
4. Isokinetic sampling: Sampling that maintains a velocity through the nozzle that is equal to the velocity of the flue gases in an exhaust stack.

3 Safety

There are many potential hazards when sampling kiln emissions. You can really hurt yourself or someone else. So always remember, safety first!

3.1 Safe platform and scaffolding

Some kilns are not equipped with scaffolding for equipment and technicians, so temporary scaffolding may have to be built for the sampling duration. It is important that all platform structures, temporary or permanent, have secure railings and are built for the weight and space required for sampling. Scaffolding platforms that are greater than 1.5 x 1.5 meters are recommended. It is recommended that technicians use a safety harness with at least one safety rope attached to the structure at all times.

3.2 Electrical safety

At some sites, the AC power supply may not be well regulated. A voltage regulator is highly recommended. All cords and wiring should be off the platform floor, and tied up to the railing as much as possible. Make sure the connectors and junctions do not pose a risk of shock.

3.3 Moving safely at the kiln site

For most kilns, it is necessary to walk on the kiln to measure the emissions. The firing zone should be identified at the onset, so that all technicians are aware of safe paths to walk at the kiln site.

3.4 Toxic pollutants

Brick kilns produce toxic pollutants and technicians can be exposed to toxic gases and particles. Safely masks that are rated for SO₂ are recommended for all kilns sites, but are especially important for clamp and forced draft kilns. Sulfuric acid and nitric acid can form on the probes inserted into the stack. Gloves should be worn whenever handling the exposed probes.

3.5 Personal protection equipment

- Wear heat resistant gloves when handling the probe. The probe may be hot, and it also may be covered in sulfuric acid or other compounds that should not contact skin.
- Wear eye protection.
- A safety harness must be worn by all technicians on the platform. The harness must be attached by a safety cord to a secure structure.
- A face mask with acid gas adsorbent should be used to reduce exposure to acid gases such as SO₂.

3.6 Laboratory safety

The calibration gases contain hazardous concentrations of CO and SO₂. CO has acute health effects when inhaled. SO₂ is an acid gas which reacts with moisture on human skin and lungs to form sulfuric acid (H₂SO₄). The laboratory should have a proper ventilation hood to exhaust these gases from the laboratory. The NIOSH Pocket Guide to Chemical Hazards gives dose response information and appropriate personal protection equipment for different levels of exposure. Lab technicians should wear appropriate personal protection equipment, according to the Safety Data Sheet for the calibration gas.

4 Equipment

The sampling equipment (Figure 1) consists of a sample probe (Section 4.1), sensor sample train (Figure 4 and Section 4.2), and accessories (Section 4.5). The sample probe includes a dilution flow train that is required for all measurements. Two probe configurations are described, one for stack sampling which has a single point nozzle and isokinetic flow train (Figure 2), and one for calm air open-plume sampling which has a multi-point nozzle and no isokinetic flow train (Figure 3). In addition to sampling equipment, required laboratory equipment is listed in Section 5.1.

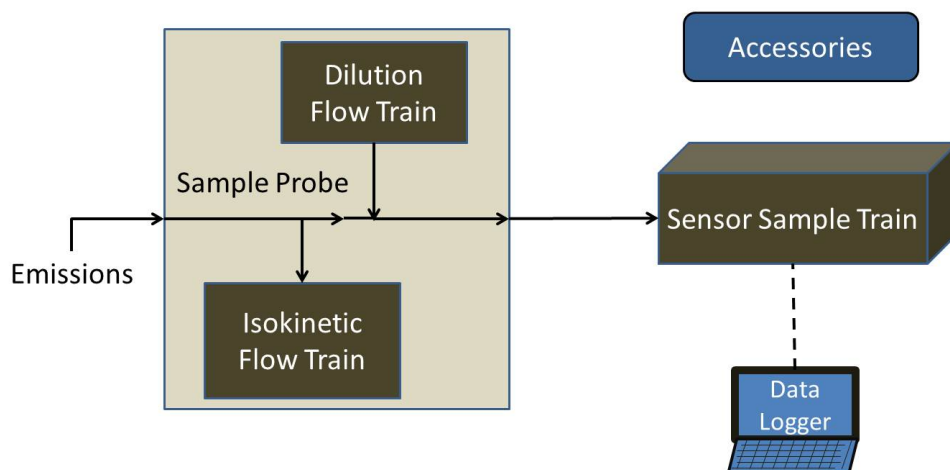


Figure 1: Overview of sampling configuration.

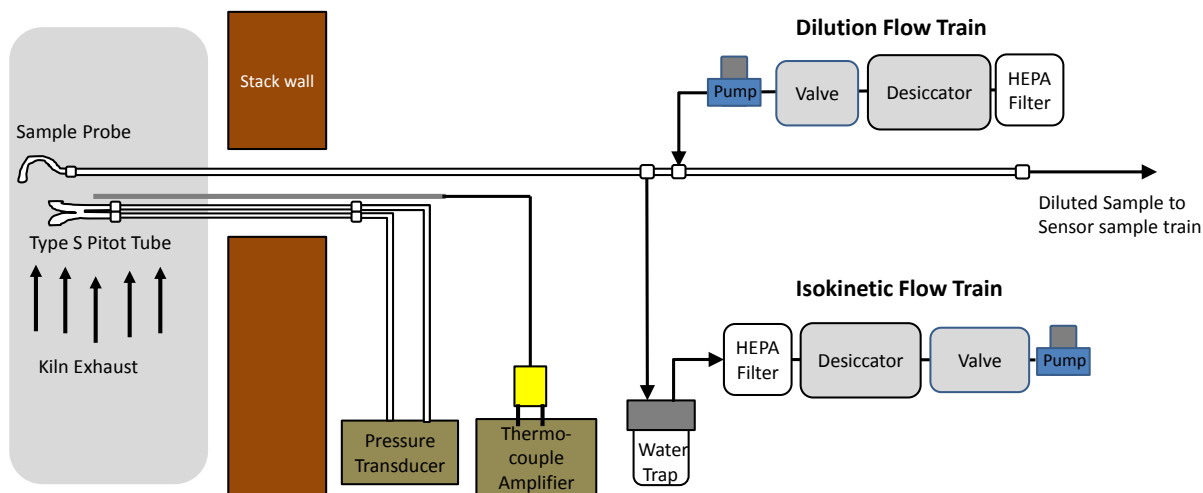


Figure 2: Isokinetic dilution probe for kilns with stacks, including the dilution flow train and isokinetic flow train (see Section 4.1.1).

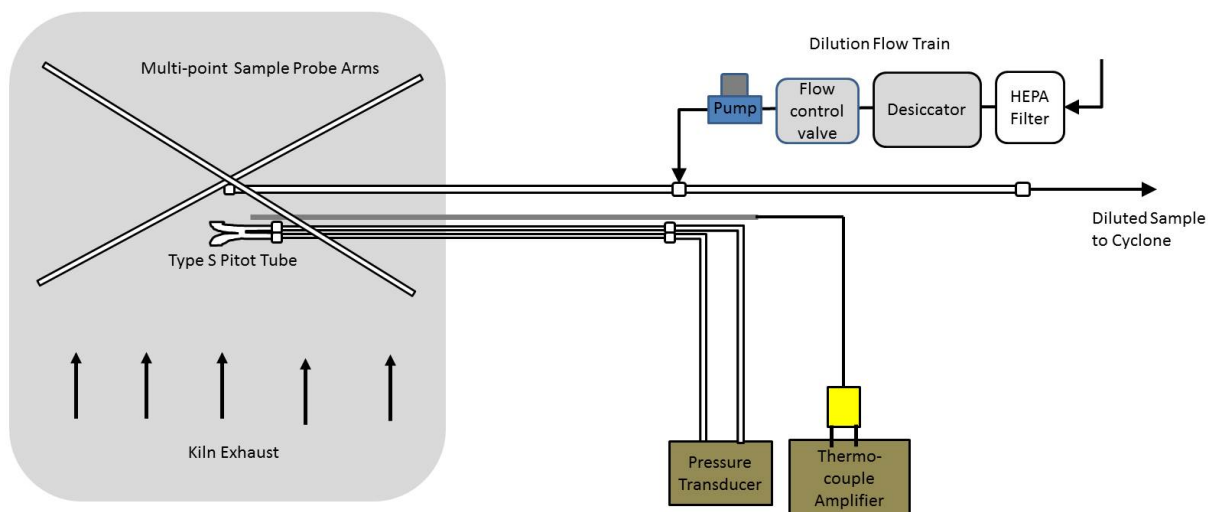


Figure 3: Open plume dilution probe for kilns without stacks, including the dilution flow train (see Section 4.1.2).

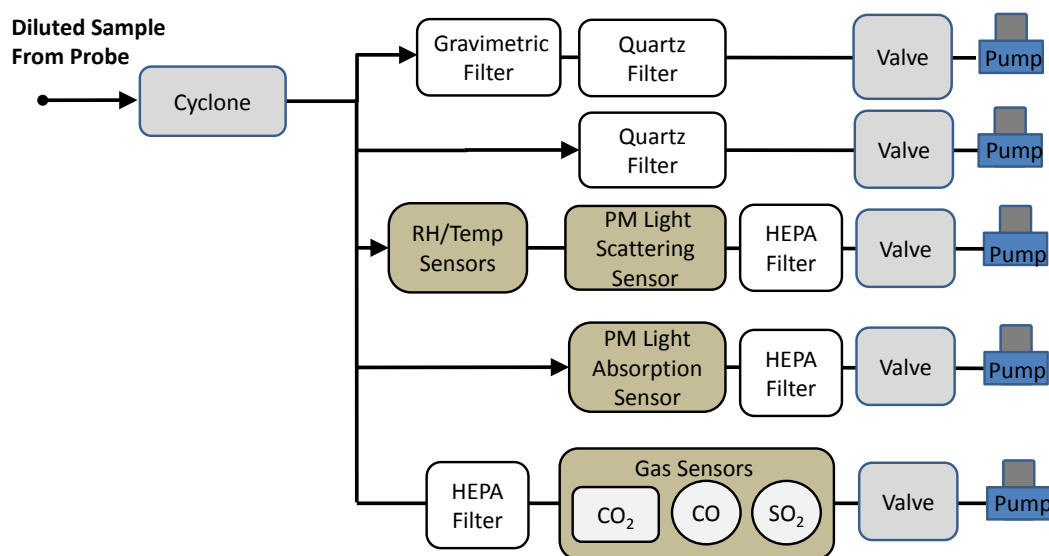


Figure 4: Sensor sample train (see Section 4.2)

4.1 Sample probe

Two sample probe configurations are described, one for single point isokinetic stack sampling (Figure 2, Section 4.1.1), and one for calm air, open-plume sampling (Figure 3, Section 4.1.2).

4.1.1 Isokinetic dilution probe for stack sampling

The probe assembly consists of a probe tube, nozzle, type S pitot tube, thermocouple, dilution flow train, and isokinetic flow train and is shown in Figure 2.

4.1.1.1 Sampling probe nozzle The probe nozzle should meet the requirements of EPA Method 5 [6]. A set of nozzle sizes with inlet diameters ranging from 3/32 - 1/4 inch is recommended. The procedure for choosing an appropriate nozzle size is given in Section 6.4.

4.1.1.2 Tube The probe tube should be made of stainless steel with a minimum inner diameter of 1/4 in. Larger diameter tubing allows for reduced particle wall losses, but adds weight to the probe. The probe tube must be an appropriate length to position the nozzle at the center of the stack. In order to be versatile to accommodate a range of stack diameters, a variable length probe tube 0.5 - 3.5 meters is recommended. This can be achieved by using 1 meter long probe tube segments that can be connected together with stainless steel compression fittings. The tube should connect to the isokinetic flow train and dilution flow train no more than 2 inches outside the kiln wall to avoid condensation inside the sample tube. The stainless steel tubing should continue at least 20 cm downstream of the dilution inlet 'T' to allow thorough mixing of the sample and dilution air.

4.1.1.3 Pitot tube The pitot tube is used to measure the flue gas velocity at the probe tip and is used to determine the correct sample flow rate for isokinetic sampling conditions. A type S pitot tube is recommended. Compared to a standard pitot tube, the type S has larger ports to prevent interference from particle accumulation. The type S pitot also produces a higher pressure differential than a standard pitot tube, making it more appropriate for measuring low velocities seen in natural draft kilns. The type S pitot tube should meet the specifications of EPA Method 2 [5] with regard to the shape, dimensions, calibration, and attachment to the sample probe. The real-time pitot tube pressure should be recorded by a pressure transducer with resolution of 0.1 Pa (0.01 mm H₂O), a range of at least -250 to +250 Pa (25 mm H₂O), and an accuracy and precision of 3% of the reading. The pressure transducer should be calibrated according to Section 7.4.6.

4.1.1.4 Thermocouple The thermocouple measures the flue gas temperature at the probe tip. Thermocouple specifications and placement guidelines should refer to EPA Method 2 [5].

4.1.1.5 Back-purge A probe back-purge system is recommended to periodically flush the probe and pitot tube of particles and condensation which can accumulate in the pitot tube, probe nozzle, and probe tube. Accumulation of particles and condensation can cause particle loss and erroneous velocity measurement by the pitot tube. To back-purge the probe and pitot tube, a burst of compressed air must be blown into the probe in the reverse direction to force particles and condensation out of the inlet. A back-purge system includes a compressed air source, such as a compressed air tank, and isolation valves and/or connections that isolate the probe from the rest of the testing kit.

4.1.2 Multi-point probe for open plume sampling

For kilns without exhaust stacks, a multi-point sample probe must be used to capture a representative cross-section of the plume in free air above the kiln. In this case, the plume generally has a low velocity (less than 1 m/s) that meets the calm air sampling criteria for PM_{2.5}, and isokinetic sampling is not required [8]. The probe has the same specifications as in Section 4.1.1 above, except the isokinetic flow train is not needed, and the probe nozzle is replaced with an array of multi-point probe arms (Figure 3). The pitot tube is not necessary but can be used to verify that calm air sampling criteria is met (exhaust velocity less than 1 m/s).

4.2 Sensor sample train

The sensor sample train contains real-time sensors and filters for particle collection. The sample train is pictured in Figure 4. Sub-assemblies of sample train components can be found as manufactured stand-alone assemblies. For example, gas sensors might be combined in a gas analyzer that includes a HEPA filter, pump, and flow control mechanism. The entire sample train can even be combined in one manufactured assembly. A total sample flow in through the cyclone of 1.5 - 3.0 LPM has been demonstrated to result in good ranges of filter loading, low pressure drop, and low power consumption to allow the equipment to be small and portable. Larger system flows might be required for some sensors and sub-assemblies.

4.2.1 Cyclone

The cyclone (or impactor) should have a D_{50} cutpoint of $(2.5\mu\text{m})$ at a flow rate that matches the total design flow rate of the sensor sample train (Equation 1). A total sample flow (cyclone flow) of 1.5 - 3.0 LPM has been demonstrated to match the requirements of the sample train. An impactor is also acceptable instead of cyclone.

4.2.2 Filter holders and filter media

Two filter holders are placed in parallel flow configuration (see Figure 5). Filter holders should have a diameter of 37 mm to 100 mm and be capable of holding two filters in series. A filter diameter of 37 mm is appropriate for lower flows and shorter sampling time, while a filter diameter of 100 mm allows for greater flexibility of filter loading ranges, sampling time, and dilution ratios. 47mm filters have been successfully used for kiln sampling. The filter holders should be made of a material that is compatible with an organic cleaning solvent such as acetone or isopropyl alcohol. A two-stage filter holder (two plates in series) may be used to hold both the gravimetric and backup quartz filter in a single filter holder.

PM is collected on the gravimetric filter to determine PM mass concentration. Several types of filter media can be used for gravimetric analysis, including PTFE membranes, Millipore, Pall Emfab, and Pall Pallflex. Organic carbon and elemental carbon mass concentrations are collected on Pall Tissuequartz quartz fiber filter media.

4.2.3 Relative humidity sensor

Capacitance-based relative humidity (RH) sensors are appropriate for this purpose. The RH sensor should be located in the sample train upstream of any HEPA filter because the HEPA filter media can accumulate and release moisture.

4.2.4 Temperature sensor

Many types of temperature sensors are appropriate, including thermocouples, thermistors, and LM35s. The temperature sensor should have a resolution of $1\text{ }^{\circ}\text{C}$ or less, a range of at least $0 - 100\text{ }^{\circ}\text{C}$, and an accuracy of $2\text{ }^{\circ}\text{C}$. The temperature sensor calibration should be checked using the procedure described in Section 7.4.3.

4.2.5 PM light scattering sensor

The PM light scattering sensor measures real-time particle light scattering, which is used as a proxy for real-time PM mass emissions. A family of suitable sensors exist on the market, which includes basic passive optical sensor modules to commercial indoor air quality and personal exposure monitors with built-in touch screen interfaces, pumps, batteries, and flow control. The sensor wavelength should be within the visible or infrared spectrum. Optical particle counters, which process the scattering signal to estimate particle concentration, are also acceptable if they meet the calibration criteria. The sensor should be calibrated against a standard integrating nephelometer as described in Section 7.4.4. The sensor should have a resolution of 100 Mm^{-1} (inverse megameters) or better, and a range of at least $0 - 100,000\text{ Mm}^{-1}$.

4.2.6 PM light absorption sensor

The PM light absorption sensor measures real-time particle light absorption, which is used as a proxy for real-time BC mass emissions. Current sensor technologies are filter-based (Aethalometer, Micro-aethalometer, Particle Soot Absorption Photometer (PSAP)), photoacoustic (PAX), or extinction-scattering cell. The sensor should be calibrated following the manufacturer's instructions. The sensor should have a resolution of 100 Mm^{-1} (inverse megameters) or better, and a range of at least $0 - 100,000\text{ Mm}^{-1}$.

4.2.7 Gas sensors

The CO_2 , CO , and SO_2 sensors can be stand-alone sensors or combined in a single instrument. Particulate matter must be removed from the sample stream with a HEPA filter before reaching the sensors. In figure

4, the gas sensors are located on their own branch of the sample train, but they can also be located on any other of the sample train branches downstream of the filter or downstream of the pump. The sensors should be calibrated following the manufacturers instructions and the calibration should be checked with a bump test described in Section 7.4.1.

4.2.7.1 CO₂ sensor The sensor should have a resolution of 10 ppm or better, a range of at least 0 - 30,000 ppm (3%) CO₂, and a T₉₀ response time of 60 seconds or less.

4.2.7.2 CO sensor The sensor should have a resolution of 1 ppm or better, a range of at least 0 - 5,000 ppm CO, and a T₉₀ response time of 60 seconds or less.

4.2.7.3 SO₂ sensor The sensor should have a resolution of 1 ppm or better, a range of at least 0 - 2,000 ppm SO₂, and a T₉₀ response time of 60 seconds or less.

4.2.8 HEPA filter

High Efficiency Particulate Arrestance (HEPA) filters remove particles from the gas stream. They should be located in the sample train where necessary to protect sensors, pumps, and valves from particle contamination.

4.2.9 Flow control valve

The flow control valves are used to adjust the flow rates in the sample train. In Figure 4, the flow control valves are located immediately upstream of the pumps. No sensors should be located in the low pressure area between the valve and the pump. There are several other flow control options. The flow control valves may be located immediately downstream of the pumps if the pumps are designed for pressure and not vacuum. A flow control valve can be replaced by a non-adjustable flow orifice if the flow should be constant. Flow control valves can be fine adjustment metering (needle) valves with manual adjustment, or mass flow controllers with automated set points. Another option is to control the flows with pump speed controllers, in which case flow control valves are not needed.

4.2.10 Pump

Vacuum pumps are used to draw the sample through the sensors. In Figure 4 the pumps are located immediately downstream of the flow control valves, and they pull a vacuum against the valve. If pressure pumps are used instead of vacuum pumps the location of the pump and valve can be reversed. Pumps with speed controllers (manual or automated) can be used to control the flow rates instead of the flow control valves. Fewer pumps are required by connecting one pump to multiple parallel branches of the sample train. In fact, all pumps in Figure 4 could be replaced by one larger pump, but flow adjustment in each branch becomes difficult. The pumps should always be protected from particle contamination with upstream HEPA filters.

4.2.11 Sample tubing

All sample tubing that transports particles should be electrically conductive (stainless steel or conductive silicon) to dissipate static charges to prevent particle loss on tube walls. Particle laden flow paths should be as short as possible with minimum bends to minimize particle loss. All sample tubing should be resistant to SO₂ reactivity; aluminum or non-stainless steel should not be used.

4.2.12 Optional equipment in sampling train

Real-time flow sensors can be used in the sampling system for better quality control. Additional species such as methane, hydrocarbons, and NO_x, can be measured by adding additional gas sensors. Gas species can also be measured by collecting integrated bag samples at the pump outlet, and then transporting the sample to the laboratory for analysis.

4.3 Dilution flow train

The dilution flow train draws in ambient air, removes moisture and particles, and forces the air into the sample probe to dilute the sample stream. The dilution flow train contains a HEPA filter, desiccant chamber, flow control valve, and pump as described in Sections 4.2.8, 4.2.9, and 4.2.10. The desiccant chamber removes moisture from gases that pass through it. The desiccant should be a material that indicates when it is saturated by changing color. Silica gel beads are recommended as a desiccant, because they provide a clear visual indication when saturated, and do not shed dust particles like anhydrous calcium sulfate. Once saturated, the silica gel can be regenerated in an oven at 100°C. The dilution flow train must be able to produce flow rates that range from 10 to 110% of the cyclone flow rate. Flow rates greater than the cyclone or system flow rate are used for easy purging of the system with clean, dry air.

4.3.1 Optional equipment in dilution flow train

4.3.1.1 Gas sensors Gas sensors can be added to the dilution flow train to monitor real-time background concentrations. Fugitive emissions from the kiln and human respiration can cause changes in background concentrations during the sampling event that would not be detected with a procedure that uses only pre- and post-test background periods. However, in open-air sampling, the difference is small.

4.3.1.2 Adsorption cartridges Adsorption cartridges for organic gases and acid gases can be added to the dilution flow train to remove gases from the dilution air that are measured in the sampling kit.

4.3.1.3 Flow sensors Real-time flow sensors can be added to the dilution flow train to determine the dilution ratio in real-time.

4.4 Isokinetic flow train

The isokinetic flow train is used to maintain an isokinetic sampling flow rate at the nozzle. The isokinetic flow train has the same components as the dilution flow train, but with an added water trap to collect condensed water. The water trap is a container with inlet and outlet ports in the lid. It should be located at the lowest point in the system. The isokinetic flow train should be connected to the stainless steel sampling probe tube with a stainless steel 'T', and the tubing from the junction to the isokinetic flow train should be long enough to allow the exhaust gas to cool to ambient temperatures before the gas reaches the water trap. The isokinetic flow train should be capable of producing a flow range of at least 0 to 2.5 LPM.

4.4.1 Optional equipment in isokinetic flow train

Gas sensors can be added to the isokinetic flow train to monitor undiluted stack concentrations. These concentrations can be used to determine the dilution ratio and molecular weight of the flue gas. Real-time flow sensors can be added to the isokinetic flow train to determine the probe nozzle velocity in real-time.

4.5 Required accessories

4.5.1 HEPA filter

A HEPA filter is required to connect to the nozzle inlet during background air measurement periods to prevent dust and other PM from collecting on the analysis filters. The filter should have low resistance and should connect easily to the sampling system inlet.

4.5.2 Primary flow calibrator

A primary flow calibrator such as a thin film (bubble) meter or piston type meter is required to measure system flow rates. Bubble meters can malfunction in direct sunlight and around other sources of infrared light, such as hot kilns.

4.5.3 Vacuum gauge

A hand pumped vacuum gauge should be used to test the sample train for leaks. This tool is also used for bleeding automotive brake lines (Mityvac, Actron). The gauge must have a range at least as large as the maximum system vacuum pressure.

4.5.4 Sample train cleaning kit

The cleaning kit should include a high purity organic solvent that evaporates completely from surfaces without leaving a residue (such as acetone, isopropyl alcohol, or ethanol). Deionized water should also be included for cleaning surfaces that are not compatible with organic solvent. A solution of baking soda and water works well for cleaning the probe after sampling coal fuel emissions. Fabric wiping sheets should be used that do not shed fibers, such as Kimwipes. Push rods and/or pull strings should be used to force cleaning plugs through the inside passages of the sample train. The cleaning plugs should not shed fibers, and may contain soft brush bristles. A compressed air source is useful for blowing out soft tubing that cannot be cleaned with a plug.

4.5.5 Fuel measurement equipment

One or more scales are required to weigh fuel. Hanging scales have proven sufficient for this purpose. A high range scale (about 0 - 50 kg) and a low range scale (0 - 5 kg) are recommended.

4.5.6 Fuel collection materials

Fuel samples must be collected for laboratory analysis. Sealable plastic bags or other seal-able containers are required and well as a log book and labeling system. Laboratory equipment for fuel analysis is in Section 5.1.

4.5.7 Ambient pressure/temp/RH meter

A handheld environmental monitor is required for measuring ambient pressure, temperature, and relative humidity.

4.5.8 Data logging equipment

The real-time data must be logged to a data storage device. The sensor equipment may have a built-in data logger or data can be logged to a file using computer software. Logging on a computer allows observation of real-time data while sampling.

4.5.9 Electricity power source

The sampling equipment must have a reliable power source, either batteries, AC power adapter, or both.

4.5.10 Rags

Rags are useful for plugging the port hole around the sample probe and handling a hot probe.

5 Facilities

5.1 Laboratory facilities

Laboratory facilities described in this section are required to calibrate sensors, prepare and analyze filter samples, analyze fuel samples, regenerate desiccant, and calibrate the pitot tube. It is not necessary for one laboratory to have all required facilities. It is common for specialized laboratories with the required equipment to offer specific analysis services.

5.1.1 Gravimetric analysis facilities

A gravimetric balance is required and should be housed in a controlled environment in accordance with US code of regulations (EPA 40 CFR 1065.190) [7].

5.1.2 Thermal optical OC-EC analysis facilities

Refer to OC-EC Protocol NIOSH 5040 [3].

5.1.3 Sensor calibration facilities

Laboratory facilities must have a fume hood, calibration gases, and a nephelometer or similar optical scattering reference sensor. Calibration gas specifications are provided in Section 7.4.1.

5.1.4 Fuel analysis facilities

A bomb calorimeter is required for determining the energy content of fuel samples. The gross calorific value should be determined using test method ASTM D5865. Refer to test method ASTM D3176 for required laboratory equipment to determine the elemental composition of fuel samples.

5.1.5 Pitot tube calibration facilities

Refer to EPA Method 2 Section 10 [5].

5.1.6 Oven facilities

An oven capable of maintaining a temperature of 550°C is required to prepare quartz filters (see Appendix A) and a temperature near 100°C is required to regenerate saturated desiccant. A metal tray to hold the desiccant and heat resistant gloves to handle the tray are also useful. The oven can also be used to determine the moisture content of fuel samples as an alternate method to ASTM D3176.

5.2 Kiln site facilities

5.2.1 Sample porthole

Kilns with stacks should have a porthole in the stack wall that is a minimum of 10 cm in diameter. The height of the porthole should follow EPA Method 1 guidelines, if safe and practical, and be “at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion or contraction in the stack” [4].

5.2.2 Sampling access platform

The platform floor should be about 1 meter below the porthole. The surface should be at least two square meters and allow comfortable space for two technicians and all equipment. The platform length along the axial direction of the porthole should be equal to the stack diameter to allow walking space to insert and remove the sample probe. A canopy on the platform can help protect the equipment from direct sun and bad weather.

5.2.3 Electrical power requirements

If the sampling equipment uses AC power, check that the power supplied at the kiln site does not fluctuate and that power can be maintained on the platform throughout the sampling period. A generator may be required.

6 Emission sampling procedure

6.1 Kiln site preparations

In the days prior to a sampling event, visit the kiln site and make sure the site requirements in Section 5.2 are met.

6.2 Laboratory preparation before a series of measurements

6.2.1 Prepare gravimetric filters

Gravimetric filters should be weighed using a microbalance and placed in numbered petri dishes according to the procedure in Appendix C.

6.2.2 Prepare quartz filters

Quartz filters are baked at 550°C for a minimum of 4 hours to remove organic carbon and then placed in numbered petri dishes. A detailed description of the procedure can be found in Appendix A. Quartz filters are placed in baked foil-lined petri dishes, sealed with Teflon tape, enclosed in plastic bags and stored at -4°C. When loading and unloading filters, they are to be handled only about 2mm from the edge with tweezers rinsed with acetone. Filters are not to come in contact with anything except the baked foil in the original petri dish and the filter holder used for testing.

6.2.3 Calibrations

All sensors should be calibrated according to the manufacturer's instructions. The sensor calibrations should be checked following the procedures described in Section 7.4. Calibration must be done for gas sensors, optical PM sensors, flow sensors, temperature sensors, RH sensors, and pressure transducers.

6.3 Laboratory preparation before each sampling event

6.3.1 Equipment check

If necessary, clean the sample train, including nozzles, probes, filter holders, and cyclone. Check the probe nozzles for defects as described in EPA Method 5 Section 10.1 [6]. Also inspect the pitot tube in a similar manner for defects or changes in shape according to EPA Method 2 Section 10.1 and recalibrate or replace it defects are observed [5]. Replace the desiccant the color indicates saturation and charge batteries for all equipment and accessories.

6.3.2 Load filters into filter holders

The filter analysis in this method requires two quartz filters for every test, one primary quartz filter that collects the PM sample, and one backup quartz filter downstream of the gravimetric filter that is used to correct for gaseous organic carbon adsorption artifact.

The filters should be loaded into the filter holder in a clean environment free of dirt, dust, wind, and airborne background emission. Load a gravimetric filter, primary quartz filter, and backup quartz filter into the filter holders as in Figure 5. Put caps on the filter holder connection ports and store in sealed bag and at -4°C until the sampling event.

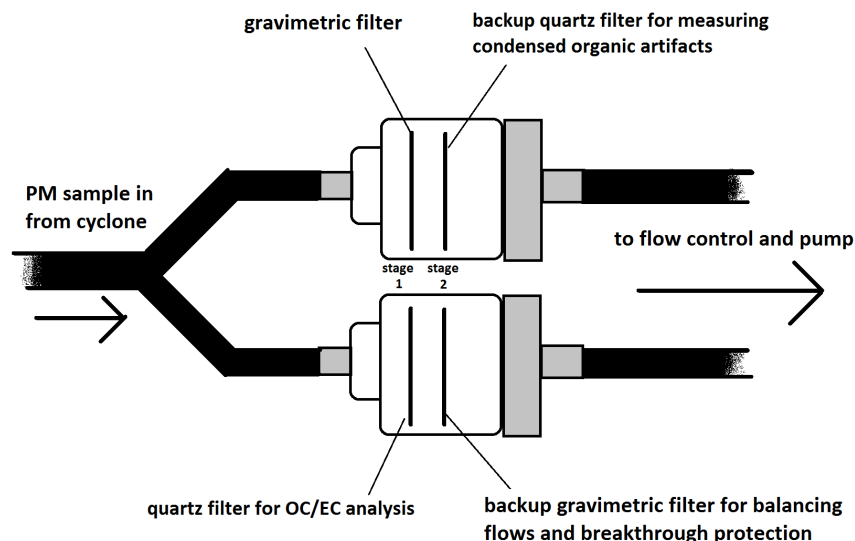


Figure 5: Filter loading diagram.

6.4 Kiln site measurement procedure

6.4.1 Start a new datasheet

There should be one datasheet that includes all the information for each test. Write down the number of the test, site information, and technicians performing the test in the datasheet. Record the numbers of the filters that have been installed in the filter holders. An example datasheet is shown in Appendix D.

6.4.2 Start fuel monitoring

In a continuous kiln, fuel loading must be monitored during the entire sampling event. When fuel is added to the kiln, the time, fuel type, and fuel mass must be recorded in a log book. Usually, a technician that is separate from emissions testing will be assigned to this task.

6.4.3 Collect fuel samples

Place approximately 1 kg sample of each fuel type in a sealed bag or container for subsequent laboratory analysis of heating value, moisture content, and carbon content. Label each bag with the name of the fuel, site, date, time, and technician who collected the sample.

6.4.4 Monitor brick process

In a continuous kiln, the number of green bricks loaded per hour and the number of fired bricks removed per hour from the kiln must be determined. Bricks should be counted for a duration of 15 minutes and the total should be recorded in a log book. Four 15 minute periods should be monitored for both loading and unloading bricks.

6.4.5 Weigh bricks

Randomly select and weigh 24 bricks. Record the weight of each in the logbook.

6.4.6 Determine diameter of the stack

Use a long thin rod and push it through the porthole until it reaches the far wall of the stack. Mark the length of rod at the outer wall of the stack and measure this length after removal. Use a rod with a hooked tip to determine the width of the stack wall. The inner diameter of the stack is the length measured on the long rod minus the thickness of the wall. The outside diameter of the stack can be determined by measuring the circumference with a rope if the stack is circular or measuring the length of each side if the stack is rectangular. All measurements should be done at the height of the porthole. Record the stack diameter in the datasheet and draw a dimensional cross section of the stack.

6.4.7 Equipment setup

6.4.7.1 Select nozzle For stack sampling, a nozzle size must be chosen for isokinetic sampling. The appropriate nozzle size depends on the stack velocity, the system flow rate through the cyclone, and the dilution flow. Table 2 can be used for guidance for a 1.5 LPM sampling system. A 1/8 in diameter nozzle will be sufficient in most practical cases. However, if stack velocities are low and dilution ratios are low, larger nozzle sizes are required. If system flows are higher than 1.5 LPM, larger size nozzles may also be required.

Table 2: Guidance chart for nozzle selection (based on 1.5 LPM sampling system).

Stack Velocity m/s	Nozzle Diameter in	Dilution Ratio Range
0.5	1/8	30:1 - 6:1
0.5	3/16	30:1 - 2:1
0.5	1/4	30:1 - 1:1
1	1/8	30:1 - 3:1
1	3/16	30:1 - 1:1
2-10	1/8	30:1 - 1:1

If the appropriate nozzle size is unknown, a 1/8 inch (3.175 mm) diameter nozzle should be selected. The probe nozzle can be changed, if necessary, after the stack velocity measurement.

6.4.7.2 Assemble probe For stack sampling, assemble a probe that will reach to stack center. For open-plume sampling, choose a probe length that will reach to the center of the plume, and use the multi-point probe nozzle (refer to section 4.1.2). It is recommended to assemble the probe on the ground and then bring the assembled probe to the measurement location, rather than assemble on a stack platform.

6.4.7.3 Assemble Equipment Bring sampling equipment to platform or open plume measurement location. Install the filter holders into the sample train and attach tubing to dilution, and isokinetic sampling systems. Do not attach the probe to the sampling system.

6.4.7.4 Leak check Perform a leak check on the sample train as described in Section 7.1.

6.4.7.5 Set dilution flow Adjust the dilution flow so the dilution ratio is appropriate for the kiln (see Equation 3). If an appropriate dilution ratio is not known, use the procedure in Section 6.4.7.6 to set a dilution ratio.

6.4.7.6 Determine appropriate dilution flow If an educated guess cannot be made for the dilution ratio, use this method to determine an appropriate dilution flow.

Set the flows to the following criteria:

1. Filter flows = 0 ccm (off or closed) to prevent PM from accumulating on the filter before the sample period.

2. Cyclone flow = PM_{2.5} cutpoint flow. Adjust the flow through light scattering, light absorption, and gas sensors to achieve this (see Equation 1).
3. Isokinetic flow = 0 ccm (off or closed)
4. Dilution flow: Adjust the dilution flow so the dilution ratio is 15:1 for stack sampling, or a dilution ratio of 1:1 for open plume sampling (see Equation 3). This is an approximation; the dilution ratio is checked and adjusted in the following step.

Insert the probe assembly into the kiln exhaust stream so the nozzle is located at the center of the stack or plume. Connect the probe to the sampling system. Sample emissions for a minimum of 5 minutes. For kilns with intermittent fuel feeding, continue sampling for one full fuel feeding cycle. Inspect the real-time sensor data to make sure the emissions are in an appropriate range for the sensors. Increase the dilution flow if any sensor readings are above 80% of the maximum specification for that sensor, or if the relative humidity reading is above 50%. Decrease the dilution flow if the minimum CO₂ concentration reading is below 2000 ppm. If the dilution flow is adjusted, record the change and recalculate the dilution ratio (Equation 2).

Disconnect the probe from the sampling system.

6.4.7.7 Set filter flows Connect a HEPA filter to the sampling system inlet to prevent dust and other background PM from accumulating on the gravimetric and OC-EC filters in the sample train. Set the flow rates through filter holders 1 and 2. A flow rate of 100 - 500 ccm is recommended, depending on the PM concentrations, dilution ratio, and expected sample period length.

Table 3 shows a few scenarios for reference when selecting the filter flow rate (for a 47mm filter). Long sampling periods require low flow rates, and short samples should have higher flow rates. Note that the flow rate should be set the same for both filter holders.

Table 3: Relationship between filter flow rate and the maximum recommended sampling time.

Stack Concentration g/m ³	Dilution Ratio	Filter Flow rRate ccm	Maximum Sampling Time hr
0.05	30	100	13.8
0.05	30	250	5.5
0.05	30	500	2.8
0.05	15	100	7.1
0.05	15	250	2.9
0.05	15	500	1.4
0.01	15	100	35.7
0.01	15	250	14.3
0.01	15	500	7.1

6.4.7.8 Set flow through cyclone Once the filter flows have been set, adjust the other parallel flows in the sample train (light scattering, light absorption, or gas sensor) to maintain the correct PM_{2.5} cutpoint flow rate through the cyclone (see Equation 1).

6.4.7.9 Measure stack velocity This section is only applicable to kilns with stacks. For open plume sampling, this section should be ignored. Insert the probe assembly (with pitot tube and thermocouple) into the sample port so the nozzle is located at stack center. Connect the pitot tube and thermocouple to the sampling system. Do not connect the sampling nozzle to the sampling system. Measure the stack velocity for at least 5 minutes by recording the pitot tube pressure and stack temperature. Calculate the average stack velocity (Equation 4).

6.4.7.10 Set isokinetic sampling flow Calculate the required flow rate of the isokinetic flow train for isokinetic sampling conditions (Equations 5 and 6). If the required isokinetic flow rate is negative, then use a larger diameter nozzle and repeat the calculations in Equations 5 and 6. If the required isokinetic flow

rate is near or above the maximum flow of the isokinetic flow train, then use a smaller diameter nozzle and repeat the calculations in Equations 5 and 6.

6.4.7.11 Measure system flows Use a primary flow calibrator to measure and record all sample train flows (cyclone, gravimetric filter holder, OC-EC filter holder, light scattering, light absorption, gas sensors, dilution, and isokinetic flows). The nozzle should not be connected to the sampling system in this step. Only clean or background air should be drawn through the primary flow calibrator.

6.4.7.12 Measure ambient conditions Use a handheld temperature, pressure, and relative humidity sensor and record these environmental parameters at the start of the test.

6.4.8 Emission sample

6.4.8.1 Pre-test background period With the HEPA filter still connected to the sample inlet, sample ambient background air for 10 minutes. Record the start and end times of this background period in the datasheet.

6.4.8.2 Test period Remove the HEPA filter from the probe inlet. Connect the sample inlet to the probe. Note: the probe should already be in the stack, (Section 6.4.7.9). Record the current time in the datasheet. The minimum test length is one complete burn cycle or fuel feeding cycle, however a longer test period over several burn cycles will result in a more representative sample. A sample that is 3-12 hours is recommended. At the end of the test period, record the time and remove the sample probe from the stack. If the real-time light absorption sensor is filter-based, monitor the absorption filter transmissivity during the test period and replace the filter when necessary according to the manufacturer's instructions.

6.4.8.3 Post-test background period Connect the HEPA filter to the probe inlet. Sample ambient background air for 10 minutes. Record the start and end times of this background period in the datasheet.

6.4.9 After emission sample

6.4.9.1 Measure ambient conditions Use a handheld temperature, pressure, and relative humidity sensor and record these environmental parameters in the datasheet at the end of the test.

6.4.9.2 Post-test flow check Measure and record all sample train flows (cyclone, gravimetric filter holder, OC-EC filter holder, light scattering, light absorption, gas sensors, dilution, and isokinetic flows).

6.4.9.3 Post-test leak check Repeat the leak check procedure in Section 7.1.

6.5 Off-site procedures after sampling

6.5.1 Remove filters from filter holders

Use tweezers to remove the filters from the filter holders and return the filters to their original petri dishes. Only touch the tweezers to the outside rim on the filters that is outside of the PM collection area.

6.5.2 Clean sample train

Clean the sample train using the cleaning kit described in Section 4.5.4. The parts of the sample train that are exposed to PM emissions should be cleaned after every sampling event. This includes the sample probe, nozzle, pitot tube, sample tubing, cyclone, and filter holders. Other system components may also need to be cleaned after every sampling event if specified by the equipment manufacturer.

6.5.3 Gravimetric analysis

See the example gravimetric analysis procedure in Appendix C.

6.5.4 Thermal-optical analysis

See NIOSH Method 5040 and the example OC-EC analysis procedure in Appendix B.

6.5.5 Fuel analysis

Perform ultimate analysis of the fuel samples following ASTM D3176. Determine the heating value following ASTM D5865.

6.6 Off-site procedures after a series of measurements

6.6.1 Calibration

The sensor calibrations should be checked following the procedures described in Section 7.4. This includes gas sensors, optical PM sensors, flow sensors, temperature sensors, RH sensors, and pressure transducers.

7 Quality assurance

7.1 Leak testing

Sample train leaks can cause erroneous measurements. Leak testing should be performed at the sampling site before and after each sampling event and before calibrations. To perform a leak test, inlet and outlet ports must be plugged and sealed to form a closed system. When the equipment is off, connect a vacuum gauge to one of the ports. Apply a vacuum to the sample train by turning on the pumps momentarily or using a hand pump vacuum gauge. The vacuum pressure should be equal to the maximum vacuum pressure that the train is exposed to during operation. Do not draw a higher vacuum than any system components are rated for. The vacuum pressure must remain constant for 30 seconds to pass the leak test. If the leak test fails, the leak must be located and sealed until the system passes the leak test. Individual sections of the sample train may need to be leak tested separately to isolate the leak.

7.2 Reduce particle losses

Particle loss can be minimized by using a sample train that follows appropriate design considerations. The tubing that transports the PM sample from the probe to the PM measurement components should be as short as possible and made of electrically conductive material to prevent electrostatic deposition. The tubing should have as few bends as possible, and any bends that do exist should have as long of a radius as possible. The particle loss can be measured in an aerosol laboratory with appropriate equipment, or estimated using PM sampling theory [1].

7.3 PM sampling conditions

The real-time sample temperature data provides a record that the sample was adequately cooled to near-ambient temperature. The real-time RH sensor provides a record that the sample was adequately dried to an RH below 50%.

7.4 Calibration

7.4.1 Gas sensor calibration

Gas sensors should be calibrated according to the manufacturers' specifications. In addition, the sensor calibration should be checked before and after a series of sampling events. In the calibration check, the gas sensors are exposed to zero and span calibration gases and the sensor output is recorded. The difference between the measured concentration and the reference concentration is used to estimate sensor uncertainty.

7.4.1.1 Calibration gas concentrations The zero and span concentrations should be at approximately 0% and 100% of the measured (or expected) diluted kiln emission concentrations. Zero gas should contain 0 ppm CO, 0 ppm CO₂, 0 ppm SO₂, balance air. A recommended span gas concentration, if the range of concentrations is not known, is approximately 1000 ppm CO, 20000 ppm CO₂, 100 ppm SO₂, with balance air. (The balance gas is air because electrochemical sensors require the presence of O₂ to complete their chemical reaction).

7.4.1.2 Bump test procedure

1. Setup the sensor sample train in a fume hood that will collect all gases that exit the sample train.
2. Perform a leak test (Section 7.1)
3. Connect the zero point calibration gas source to the sample train inlet as shown in Figure 6

The calibration gas source is connected to the sample train inlet with a T connector that has an open port to the surrounding atmosphere to ensure that the sample train pressure is equal to ambient pressure as during normal operating conditions. Connect the T to the positive pressure port of the pitot tube pressure transducer.

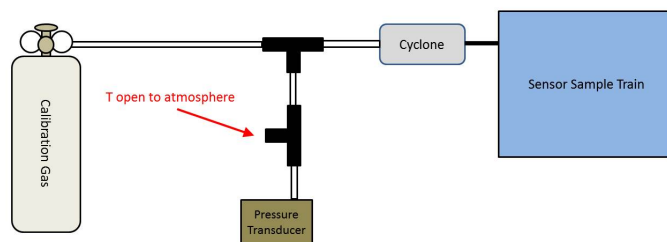


Figure 6: Gas sensor bump test setup.

4. Set the flows in the sample train to normal operating flow rates.
5. Turn on the supply of zero point calibration gas so the flow is slightly above the sample train flow.
When calibration gas is flowing, the pressure in the T should be slightly above atmospheric pressure (positive differential pressure) to ensure that excess calibration gas is flowing out of the port to atmosphere, and no ambient air is being drawn into the sample train.
6. Sample the calibration gas until the sensors readings reach steady state values (about 5 - 10 minutes).
7. Turn off the zero point calibration gas and disconnect from the sample train.
8. Repeat Steps 3 - 7 with the span point calibration gas instead of the zero gas.
9. Inspect the real-time sensor data and record the reference gas concentrations (zero and span) and actual sensor readings (zero and span) for each gas sensor.

7.4.2 RH sensor calibration

Calibrate according to the manufacturer's specifications. The calibration can be checked by sampling dry desiccated air and saturated air (steam).

7.4.3 Sample temperature sensor calibration

Calibrate according to the manufacturer's specifications. If the sensor is submersible in water, the calibration can be checked by placing the sensor in an ice bath and in boiling water. If the sensor is not submersible in water, the sensor calibration can be checked against a reference temperature sensor.

7.4.4 PM light scattering sensor calibration

7.4.4.1 Setup The PM scattering sensor should be calibrated against a standard integrating nephelometer, or other reference instrument that measures the optical scattering coefficient. The sampling system setup is the same as normal operation. The flow through the cyclone must be set for the PM_{2.5} cutpoint. The reference meter should also have a PM_{2.5} particle separator on the inlet.

A smoke chamber can be used to supply a constant concentration of smoke to the instruments. A mixture of combustion smoke and dilution air can be drawn into the smoke chamber, or a small amount of combustion smoke can be generated inside the smoke chamber. In order to ensure the sample inside the smoke chamber is uniform, the smoke source should be removed, the smoke chamber should be completely sealed, and the smoke should age for several minutes before sampling. The volume of the smoke chamber should be about 1 m³.

7.4.4.2 Calibration The calibration should include a minimum of at least 3 calibration points: one zero point, one point at the mid range of the reference meter, and one point at approximately 90% of the range of the reference meter. The zero point is achieved by sampling HEPA filtered air through both the optical sensor and the reference instrument. The mid and high range calibration points can be achieved by sampling from the smoke chamber simultaneously through both the optical sensor and the reference instrument. Preparatory testing should be done to determine the combustion and dilution conditions required to achieve the desired concentrations.

7.4.4.3 Linearity check The optical sensor should be checked for linearity over the full measurement range (at least 0 - 100,000 Mm⁻¹). If the reference instrument has a smaller measurement range (which is true for standard integrating nephelometers), then an additional step is required in the calibration process to check the linearity of the optical sensor beyond the range of the reference instrument.

Disconnect the reference instrument from the smoke chamber. Assemble the sample train of the sampling instrument including the dilution probe. Connect the dilution system to the sampling probe as when sampling from a stack. Note that the isokinetic flow train, temperature sensor and pitot tube are not used. Set the dilution flow to be greater than the cyclone flow so the sensor sample train is sampling pure dilution air and no smoke from the chamber. Generate a smoke concentration in the smoke chamber that is higher than the measurement range of the optical sensor. Decrease the dilution flow to increase the concentration of smoke exposed to the optical sensor. Measure several calibration points over the range of the optical sensor. For each calibration point, adjust the dilution flow to set the concentration, then measure the dilution ratio (Equation 2) and optical sensor reading. The sensor reading should be linearly proportional to the parameter $\frac{DR}{1+DR}$, where DR is the dilution ratio (see Equation 2). Generate a plot with $\frac{DR}{1+DR}$ on the x axis and the sensor reading on the y axis to verify that the relationship is linear.

7.4.5 PM light absorption sensor calibration

Calibrate according to the manufacturer's specifications.

7.4.6 Pressure transducer calibration

The pressure transducer should be calibrated against an inclined reference manometer using a 3 point calibration over the full range of expected pressure readings as described in EPA Method 2 Section 6.2.

7.4.7 Pitot tube calibration

The pitot tube should be calibrated according to EPA Method 2 Section 10.1. Since the pitot calibration coefficient is dependent on calibration velocity, the calibration should be performed at a velocity that is near the stack velocity of the kiln(s) being measured.

7.5 Uncertainty propagation

Measurement uncertainties should be propagated in the calculations and expressed as confidence intervals of the resulting emissions metrics.

8 Data analysis and calculations

8.1 Variables

a = fuel type index

BC_{CER} = BC carbon emission ratio ($\frac{g_{BC}}{g_C}$)

$BC_{EF,mass}$ = BC emission factor, mass based ($\frac{g_{BC}}{kg_{fuel}}$)

$BC_{EF,energy}$ = BC emission factor, energy based ($\frac{g_{BC}}{MJ}$)

$BC_{EF,brick}$ = BC emission factor, brick based ($\frac{g_{BC}}{kg_{brick}}$)

BC_{ER} = BC emission rate ($\frac{g_{BC}}{hr}$)

$brick_{rate}$ = brick production rate ($\frac{kg_{brick}}{hr}$)

C = total carbon concentration ($\frac{g}{m^3}$)

C_p = pitot tube calibration coefficient (-). For uncalibrated type S pitot use 0.84.

$CO_{ppm,n}$ = CO concentration for n th real-time data series point (ppm)

$CO_{ppm,prebkg}$ = average CO concentration during pre-test background period (ppm)

$CO_{ppm,test}$ = average CO concentration during test period (ppm)

$CO_{ppm,postbkg}$ = average CO concentration during post-test background period (ppm)

$CO_{ppm,bkg}$ = average background CO concentration (ppm)

CO_{ppm} = average CO concentration (background subtracted) (ppm)

CO = average CO concentration ($\frac{g}{m^3}$)

CO_{CER} = CO carbon emission ratio ($\frac{g_{CO}}{g_C}$)

$CO_{EF,mass}$ = CO emission factor, mass based ($\frac{g_{CO}}{kg_{fuel}}$)

$CO_{EF,energy}$ = CO emission factor, energy based ($\frac{g_{CO}}{MJ}$)

$CO_{EF,brick}$ = CO emission factor, brick based ($\frac{g_{CO}}{kg_{brick}}$)

CO_{ER} = CO emission rate ($\frac{g_{CO}}{hr}$)

$CO2_{ppm,n}$ = CO₂ concentration for n th real-time data series point (ppm)

$CO2_{ppm,prebkg}$ = average CO₂ concentration during pre-test background period (ppm)

$CO2_{ppm,test}$ = average CO₂ concentration during test period (ppm)
 $CO2_{ppm,postbkg}$ = average CO₂ concentration during post-test background period (ppm)
 $CO2_{ppm,bkg}$ = average background CO₂ concentration (ppm)
 $CO2_{ppm}$ = average CO₂ concentration (background subtracted) (ppm)
 $CO2$ = average CO₂ concentration ($\frac{g}{m^3}$)
 D_{nozzle} = nozzle tip inside diameter (mm)
 $D_{f,b}$ = diameter of exposed area of backup OC-EC filter (cm^2)
 $D_{f,p}$ = diameter of exposed area of primary OC-EC filter (cm^2)
 DR = dilution ratio (-)
 $EC_{density,b}$ = EC density on the backup OC-EC filter measured by the Sunset Analyzer ($\frac{\mu g}{cm^2}$)
 $EC_{density,p}$ = EC density on the primary OC-EC filter measured by the Sunset Analyzer ($\frac{\mu g}{cm^2}$)
 $EC_{mass,b}$ = EC mass on the backup OC-EC filter (μg)
 $EC_{mass,p}$ = EC mass on the primary OC-EC filter (μg)
 EC_b = average EC concentration on backup OC-EC filter ($\frac{g}{m^3}$)
 EC_p = average EC concentration on primary OC-EC filter ($\frac{g}{m^3}$)
 EC = average EC concentration ($\frac{g}{m^3}$)
 EC_{CER} = EC carbon emission ratio ($\frac{gEC}{gC}$)
 $EC_{EF,mass}$ = EC emission factor, mass based ($\frac{gEC}{kg_{fuel}}$)
 $EC_{EF,energy}$ = EC emission factor, energy based ($\frac{gEC}{MJ}$)
 $EC_{EF,brick}$ = EC emission factor, brick based ($\frac{gEC}{kg_{brick}}$)
 EC_{ER} = EC emission rate ($\frac{gEC}{hr}$)
 $filter_{mass,i}$ = initial gravimetric filter mass (μg)
 $filter_{mass,f}$ = final gravimetric filter mass (μg)
 $flow_{abs}$ = PM absorption sensor flow rate (ccm)
 $flow_{cyclone}$ = cyclone flow rate (ccm)
 $flow_{dil}$ = dilution flow rate (ccm)
 $flow_{filter1}$ = filter holder 1 (gravimetric) flow rate (ccm)
 $flow_{filter2}$ = filter holder 2 (OC-EC) flow rate (ccm)
 $flow_{gas}$ = gas sensor flow rate (ccm)
 $flow_{iso}$ = isokinetic sample train flow (ccm)
 $flow_{nozzle,stk}$ = isokinetic nozzle flow rate (ccm)
 $flow_{scat}$ = PM scattering sensor flow rate (ccm)
 $fuel_{CF,a}$ = fuel carbon fraction, dry basis, for fuel type a ($\frac{kgC}{kg_{fuel}}$)

$fuel_{CF,eff}$ = effective fuel carbon fraction, dry basis ($\frac{kg_C}{kg_{fuel}}$)
 $fuel_{LHV,a}$ = lower heating value of dry fuel for fuel type a ($\frac{MJ}{kg_{fuel}}$)
 $fuel_{LHV,eff}$ = effective fuel heating value ($\frac{MJ}{kg_{fuel}}$)
 $fuel_{mass,a}$ = moist fuel consumed for fuel type a (kg)
 $fuel_{MC,a}$ = moisture content, wet basis for fuel type a (-)
 $fuel_{rate}$ = dry fuel consumption rate ($\frac{kg}{hr}$)
 $K_p = 129$ = pitot velocity equation constant ($\frac{m^3 * Pa * g}{mol * K * kg}$)^{0.5}
 MCE = modified combustion efficiency (-)
 $MW_C = 12.01$ = carbon molecular weight ($\frac{g}{mol}$)
 $MW_{CO} = 28.01$ = CO molecular weight ($\frac{g}{mol}$)
 $MW_{CO_2} = 44.01$ = CO₂ molecular weight ($\frac{g}{mol}$)
 $MW_{SO_2} = 64.07$ = SO₂ molecular weight ($\frac{g}{mol}$)
 MW_{stak} = flue gas molecular weight ($\frac{g}{mol}$). Assume 29 or measure flue gas composition.
 n = real-time data series index
 $n_{prebkgstart}$ = real-time data series index for pre-background start time
 $n_{prebkgstop}$ = real-time data series index for pre-background stop time
 $n_{teststart}$ = real-time data series index for test start time
 $n_{teststop}$ = real-time data series index for test stop time
 $n_{postbkgstart}$ = real-time data series index for post-background start time
 $n_{postbkgstop}$ = real-time data series index for post-background stop time
 $OC_{density,b}$ = OC density on the backup OC-EC filter measured by the Sunset Analyzer ($\frac{\mu g}{cm^2}$)
 $OC_{density,p}$ = OC density on the primary OC-EC filter measured by the Sunset Analyzer ($\frac{\mu g}{cm^2}$)
 $OC_{mass,b}$ = OC mass on the backup OC-EC filter (μg)
 $OC_{mass,p}$ = OC mass on the primary OC-EC filter (μg)
 OC_b = average OC concentration on backup OC-EC filter ($\frac{g}{m^3}$)
 OC_p = average OC concentration on primary OC-EC filter ($\frac{g}{m^3}$)
 OC = average OC concentration ($\frac{g}{m^3}$)
 OC_{CER} = OC carbon emission ratio ($\frac{gOC}{gC}$)
 $OC_{EF,mass}$ = OC emission factor, mass based ($\frac{gOC}{kg_{fuel}}$)
 $OC_{EF,energy}$ = OC emission factor, energy based ($\frac{gOC}{MJ}$)
 $OC_{EF,brick}$ = OC emission factor, brick based ($\frac{gOC}{kg_{brick}}$)
 OC_{ER} = OC emission rate ($\frac{gOC}{hr}$)

ΔP = average differential pitot tube pressure (Pa)
 P_{amb} = ambient barometric pressure (Pa)
 $P_{amb,pre}$ = ambient pressure during the pre-test flow measurement (Pa)
 $P_{amb,post}$ = ambient pressure during the post-test flow measurement (Pa)
 P_{stak} = absolute stack pressure (Pa). Assume $P_{stak} = P_{amb}$ or measure.
 $P_{std} = 101325$ = standard pressure (Pa)
 PM_{mass} = mass of PM collected on the gravimetric filter (μg)
 PM = average $PM_{2.5}$ concentration ($\frac{g}{m^3}$)
 PM_{CER} = PM carbon emission ratio ($\frac{g_{PM}}{g_C}$)
 $PM_{EF,mass}$ = $PM_{2.5}$ emission factor, mass based ($\frac{g_{PM_{2.5}}}{kg_{fuel}}$)
 $PM_{EF,energy}$ = $PM_{2.5}$ emission factor, energy based ($\frac{g_{PM_{2.5}}}{MJ}$)
 $PM_{EF,brick}$ = $PM_{2.5}$ emission factor, brick based ($\frac{g_{PM_{2.5}}}{kg_{brick}}$)
 PM_{ER} = $PM_{2.5}$ emission rate ($\frac{g_{PM_{2.5}}}{hr}$)
 PM_{abs} = PM optical absorption coefficient (Mm^{-1})
 PM_{scat} = PM optical scattering coefficient (Mm^{-1})
 $Q_{f,p,pre}$ = pre-test flow rate through primary OC-EC filter at actual conditions ($\frac{l}{min}$)
 $Q_{f,p,post}$ = post-test flow rate through primary OC-EC filter at actual conditions ($\frac{l}{min}$)
 $Q_{f,p,pre,std}$ = pre-test flow rate through primary OC-EC filter at standard conditions ($\frac{sl}{min}$)
 $Q_{f,p,post,std}$ = post-test flow rate through primary OC-EC filter at standard conditions ($\frac{sl}{min}$)
 $Q_{f,p,std}$ = average flow rate through primary OC-EC filter at standard conditions ($\frac{sl}{min}$)
 $Q_{grav,pre}$ = pre-test flow rate through gravimetric filter at actual conditions ($\frac{l}{min}$)
 $Q_{grav,post}$ = post-test flow rate through gravimetric filter at actual conditions ($\frac{l}{min}$)
 $Q_{grav,pre,std}$ = pre-test flow rate through gravimetric filter at standard conditions ($\frac{sl}{min}$)
 $Q_{grav,post,std}$ = post-test flow rate through gravimetric filter at standard conditions ($\frac{sl}{min}$)
 $Q_{grav,std}$ = average flow rate through gravimetric filter at standard conditions ($\frac{sl}{min}$)
 $R = 8.314$ = ideal gas constant ($\frac{m^3 \cdot Pa}{K \cdot mol}$)
 SEC = specific energy consumption ($\frac{MJ}{kg_{brick}}$). See Specific Energy Consumption Protocol [cite].
 $SO2_{ppm,n}$ = SO_2 concentration for n th real-time data series point (ppm)
 $SO2_{ppm,prebkg}$ = average SO_2 concentration during pre-test background period (ppm)
 $SO2_{ppm,test}$ = average SO_2 concentration during test period (ppm)
 $SO2_{ppm,postbkg}$ = average SO_2 concentration during post-test background period (ppm)
 $SO2_{ppm,bkg}$ = average background SO_2 concentration (ppm)

$SO2_{ppm}$ = average SO_2 concentration (background subtracted) (ppm)

$SO2$ = average SO_2 concentration ($\frac{g}{m^3}$)

$SO2_{CER}$ = SO_2 carbon emission ratio ($\frac{g_{SO2}}{g_C}$)

$SO2_{EF,mass}$ = SO_2 emission factor, mass based ($\frac{g_{SO2}}{kg_{fuel}}$)

$SO2_{EF,energy}$ = SO_2 emission factor, energy based ($\frac{g_{SO2}}{MJ}$)

$SO2_{EF,brick}$ = SO_2 emission factor, brick based ($\frac{g_{SO2}}{kg_{brick}}$)

$SO2_{ER}$ = SO_2 emission rate ($\frac{g_{SO2}}{hr}$)

SSA = single scattering albedo (-)

t_n = time stamp of the n th data point of the real-time data series (hh:mm:ss)

$t_{teststart}$ = start time of test period (hh:mm:ss)

$t_{teststop}$ = stop time of test period (hh:mm:ss)

Δt_{test} = length of time of test period = PM sample time (min)

T_{samp} = sample temperature (K)

$T_{samp,pre}$ = sample temperature during the pre-test flow measurement (K)

$T_{samp,post}$ = sample temperature during the post-test flow measurement (K)

T_{stak} = average stack temperature (K)

$T_{std} = 293$ = standard temperature (K)

v_{stak} = stack velocity ($\frac{m}{s}$)

V_{grav} = volume of air (at standard conditions) sampled through the gravimetric filter (m^3)

$V_{f,b}$ = volume of air (at standard conditions) sampled through the backup OC-EC filter (m^3)

$V_{f,p}$ = volume of air (at standard conditions) sampled through the primary OC-EC filter (m^3)

8.2 Sample train flows

Assuming the pressure is constant throughout the sample train, the cyclone flow is:

$$flow_{cyclone} = flow_{filter1} + flow_{filter2} + flow_{scat} + flow_{abs} + flow_{gas} \quad (1)$$

The dilution ratio is:

$$DR = \frac{flow_{dil}}{flow_{cyclone} - flow_{dil}} \quad (2)$$

Rearranging to determine the dilution flow rate to achieve a desired dilution ratio:

$$flow_{dil} = \frac{DR}{1 + DR} flow_{cyclone} \quad (3)$$

8.3 Isokinetic flow calculations

The stack velocity is calculated by:

$$v_{stak} = C_p K_p \sqrt{\frac{\Delta P T_{stak}}{P_{stak} MW_{stak}}} \quad (4)$$

The required volumetric sample flow rate in through the nozzle to meet isokinetic conditions is given by:

$$flow_{nozzle,stak} = v_{stak} \frac{\pi D_{nozzle}^2}{4} 60 \quad (5)$$

The required isokinetic sample train flow to produce the correct nozzle velocity for isokinetic sampling conditions is given by:

$$flow_{iso} = flow_{nozzle,stak} \frac{T_{samp}}{T_{stak}} + flow_{dil} - flow_{cyclone} \quad (6)$$

8.4 Gas species concentrations

The real-time sensor data has the following form:

Table 4: Real-time gas sensor data format

n	time	CO	SO ₂	CO ₂
1	t_1	$CO_{ppm,1}$	$SO2_{ppm,1}$	$CO2_{ppm,1}$
2	t_2	$CO_{ppm,2}$	$SO2_{ppm,2}$	$CO2_{ppm,2}$
3	t_3	$CO_{ppm,3}$	$SO2_{ppm,3}$	$CO2_{ppm,3}$
...				
n	t_n	$CO_{ppm,n}$	$SO2_{ppm,n}$	$CO2_{ppm,n}$
...				

If the sample conditions (temperature and pressure) are different than the gas sensor calibration conditions, apply corrections to the real-time data points, if necessary, according to the sensor manufacturer's specifications.

Determine the index n for the start and stop times of the pre-test background period, test period, and post-test background period.

Table 5: Start and stop indexes of real-time data series for background and test periods

period	start	stop
pre-test bkg	$n_{prebkgstart}$	$n_{prebkgstop}$
test	$n_{teststart}$	$n_{teststop}$
post-test bkg	$n_{postbkgstart}$	$n_{postbkgstop}$

The following equations 7 - 12 are used to determine the average CO concentration. The same set of equations are used to determine the average SO₂ and CO₂ concentrations.

The average concentration of CO during the pre-test background period is given by:

$$CO_{ppm,prebkg} = \frac{\sum_{n_{prebkgstart}}^{n_{prebkgstop}} CO_{ppm,n}}{n_{prebkgstop} - n_{prebkgstart} + 1} \quad (7)$$

The average concentration of CO during the test period is given by:

$$CO_{ppm,test} = \frac{\sum_{n_{teststart}}^{n_{teststop}} CO_{ppm,n}}{n_{teststop} - n_{teststart} + 1} \quad (8)$$

The average concentration of CO during the post-test background period is given by:

$$CO_{ppm,postbkg} = \frac{\sum_{n_{postbkgstart}}^{n_{postbkgstop}} CO_{ppm,n}}{n_{postbkgstop} - n_{postbkgstart} + 1} \quad (9)$$

The average CO background concentration is calculated as the average of the pre-test and post-test background concentrations:

$$CO_{ppm,postbkg} = \frac{CO_{ppm,prebkg} + CO_{ppm,postbkg}}{2} \quad (10)$$

The average concentration of CO (background subtracted) is:

$$CO_{ppm} = CO_{ppm,test} - CO_{ppm,bkg} \quad (11)$$

The ideal gas law is used to convert the CO concentration units from ppm to g/m³ at standard temperature and pressure:

$$CO = \frac{CO_{ppm} P_{std} MW_{CO}}{10^6 RT_{std}} \quad (12)$$

8.5 PM_{2.5} concentration

The mass of PM collected on the gravimetric filter is the difference in the final and initial filter mass:

$$PM_{mass} = filter_{mass,f} - filter_{mass,i} \quad (13)$$

The volumetric flow rate through the gravimetric filter is measured at the beginning and end of the sampling procedure. The flow measurements are converted from actual to standard conditions by:

$$Q_{grav,pre,std} = Q_{grav,pre} \frac{P_{amb,pre}}{P_{std}} \frac{T_{std}}{T_{smp,pre}} \quad (14)$$

$$Q_{grav,post,std} = Q_{grav,post} \frac{P_{amb,post}}{P_{std}} \frac{T_{std}}{T_{smp,post}} \quad (15)$$

The average gravimetric filter flow rate is:

$$Q_{grav,std} = \frac{Q_{grav,pre,std} + Q_{grav,post,std}}{2} \quad (16)$$

The PM sample time is the length of time of the test period:

$$\Delta t_{test} = t_{teststop} - t_{teststart} \quad (17)$$

The total volume of air that was sampled by the gravimetric filter is:

$$V_{grav} = \frac{Q_{grav,std} \Delta t_{test}}{1000} \quad (18)$$

The average PM_{2.5} mass concentration is:

$$PM = \frac{PM_{mass}}{V_{grav} 10^6} \quad (19)$$

The PM background concentration is assumed to be negligible because the background air that is mixed with the emission sample is clean HEPA filtered dilution air.

8.6 OC and BC concentrations

EC is treated as equivalent to BC. OC and EC concentrations are calculated from the OC and EC density (in $\mu\text{g}/\text{cm}^2$) on the filters, as determined in OC-EC analysis systems. The concentration of OC and EC are determined for both the primary quartz filter and backup quartz filters. The OC and EC on the backup quartz filter is a measurement of the positive adsorption artifact, and is subtracted from the primary quartz filter.

The total mass of OC and EC on the filter is determined by measuring the diameter of the filter area that was exposed to the emission sample (exclude the clean filter area on the edge of the filter where it was clamped in the filter holder):

$$OC_{mass,p} = OC_{density,p} \frac{\pi D_{f,p}^2}{4} \quad (20)$$

$$EC_{mass,p} = EC_{density,p} \frac{\pi D_{f,p}^2}{4} \quad (21)$$

$$OC_{mass,b} = OC_{density,b} \frac{\pi D_{f,b}^2}{4} \quad (22)$$

$$EC_{mass,b} = EC_{density,b} \frac{\pi D_{f,b}^2}{4} \quad (23)$$

Since the backup OC-EC filter is inline with the gravimetric filter, the total volume sampled by the backup OC-EC filter is the same as the total volume sampled by the gravimetric filter calculated in Equation 18.

$$V_{f,b} = V_{grav} \quad (24)$$

The volumetric flow rate through the primary OC-EC filter is measured at the beginning and end of the sampling procedure. The flow measurements are converted from actual to standard conditions by:

$$Q_{f,p,pre,std} = Q_{f,p,pre} \frac{P_{amb,pre}}{P_{std}} \frac{T_{std}}{T_{samp,pre}} \quad (25)$$

$$Q_{f,p,post,std} = Q_{f,p,post} \frac{P_{amb,post}}{P_{std}} \frac{T_{std}}{T_{samp,post}} \quad (26)$$

The average primary OC-EC filter flow rate is:

$$Q_{f,p,std} = \frac{Q_{f,p,pre,std} + Q_{f,p,post,std}}{2} \quad (27)$$

The OC-EC sample time is the length of time of the test period calculated in Equation 17. The total volume of air that was sampled by the primary OC-EC filter is:

$$V_{f,p} = \frac{Q_{f,p,std} \Delta t_{test}}{1000} \quad (28)$$

The average OC and EC mass concentration on the primary and backup OC-EC filter are:

$$OC_p = \frac{OC_{mass,p}}{V_{f,p} 10^6} \quad (29)$$

$$EC_p = \frac{EC_{mass,p}}{V_{f,p} 10^6} \quad (30)$$

$$OC_b = \frac{OC_{mass,b}}{V_{f,b} 10^6} \quad (31)$$

$$EC_b = \frac{EC_{mass,b}}{V_{f,b} 10^6} \quad (32)$$

The reported OC and EC concentrations are the primary filter minus the backup filter:

$$OC = OC_p - OC_b \quad (33)$$

$$EC = EC_p - EC_b \quad (34)$$

8.7 MCE

The modified combustion efficiency metric is calculated as:

$$MCE = \frac{CO2_{ppm}}{CO_{ppm} + CO2_{ppm}} \quad (35)$$

8.8 Carbon Emission Ratios

The total carbon concentration in the emission sample is the sum of the carbon concentrations from each species. The carbon concentration of a species is the species concentration multiplied by the mass fraction of carbon in that species. OC and EC have a carbon mass fraction of 1.

$$C = CO \frac{MW_C}{MW_{CO}} + CO2 \frac{MW_C}{MW_{CO2}} + OC + EC \quad (36)$$

The carbon emission ratio of each species (CO, SO₂, PM_{2.5}, OC, and EC) is the species concentration divided by the total carbon concentration. The CO Carbon Emission Ratio equation is shown below.

$$CO_{CER} = \frac{CO}{C} \quad (37)$$

BC is treated as equivalent to EC, so $BC_{CER} = EC_{CER}$.

8.9 Fuel mass based emission factor

The effective fuel carbon fraction is used to perform a carbon mass balance to relate carbon emitted with fuel mass consumed. Then mass based emission factors are calculated from carbon emission ratios in grams of pollutant per kg dry fuel.

The effective fuel carbon fraction is the carbon released from the fuel divided by the total dry fuel consumed during the emission measurement period. The calculation requires measurement (or estimation) of the fuel mass, moisture content, and carbon content for each type of fuel consumed during the emission measurement period. Fuel mass is measured by observation during the sampling event. Fuel moisture content and carbon content are measured by ultimate analysis.

$$fuel_{CF,eff} = \frac{\sum^a (fuel_{mass,a} (1 - fuel_{MC,a}) fuel_{CF,a})}{\sum^a (fuel_{mass,a} (1 - fuel_{MC,a}))} \quad (38)$$

The equation for the mass based emission factor for CO is shown below. Other species follow the same formula:

$$CO_{EF,mass} = CO_{CER} fuel_{CF,eff} 1000 \quad (39)$$

8.10 Energy based emission factor

Energy based emission factors are calculated from mass based emission factors and the effective fuel heating value.

The effective fuel heating value is the energy released from the fuel divided by the total dry fuel consumed during the emission measurement period. The calculation requires measurement (or estimation) of the fuel mass, moisture content, and LHV for each type of fuel consumed during the emission measurement period.

Fuel mass is measured by observation during the sampling event. Fuel moisture content is measured by ultimate analysis or gravimetrically, and LHV is determined by ASTM 5865:

$$fuel_{LHV,eff} = \frac{\sum^a (fuel_{mass,a}(1 - fuel_{MC,a})fuel_{LHV,a})}{\sum^a (fuel_{mass,a}(1 - fuel_{MC,a}))} \quad (40)$$

The equation for the energy based emission factor for CO is shown below. Other species follow the same formula.

$$CO_{EF,energy} = CO_{EF,mass}fuel_{LHV,eff} \quad (41)$$

8.11 Brick based emission factor

The brick based emission factor is the product of the energy based emission factor and the specific energy consumption. The equation for CO is shown below. Other species follow the same formula.

$$SEC = \frac{fuel_{rate}}{brick_{rate}}fuel_{LHV,eff} \quad (42)$$

$$CO_{EF,brick} = CO_{EF,energy}SEC \quad (43)$$

8.12 Emission rates

Emission rates are calculated from fuel based emission factors and fuel consumption rate. The fuel consumption rate calculation requires measurement (or estimation) of the fuel mass and moisture content for each type of fuel consumed during the emission measurement period:

$$fuel_{rate} = \frac{\sum^a (fuel_{mass,a}(1 - fuel_{MC,a}))}{\Delta t_{test}}60 \quad (44)$$

The equation for CO emission rate is shown below. Other species follow the same formula:

$$CO_{ER} = CO_{EF,mass}fuel_{rate} \quad (45)$$

8.13 Real-time data

The real-time sensor data series should be plotted to illustrate the emission patterns during the sampling period.

8.13.1 Real-time optical scattering and absorption

These sensors show the temporal variability of PM emissions. To get a rough estimate of particle darkness, the single scattering albedo (SSA) can be calculated:

$$SSA = \frac{PM_{scat}}{PM_{abs} + PM_{scat}} \quad (46)$$

The average SSA can be calculated for a test, or for individual PM events to see relative changes in particle darkness within a test.

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Appendix A Preparing quartz fiber filters: Procedure

The following is an example protocol used by the University of Illinois for quartz filter preparation.

1. Rinse the porcelain bowls with acetone, and let them dry in the exhaust flow hood.
2. Place about 10 quartz fiber filters per bowl, arranged around the walls rather than stacked one on top of the other. Place the filters loosely rather than jamming them together.
 - a. DO NOT BAKE QUARTZ FILTERS FROM DIFFERENT LOTS IN THE SAME BATCH
 - b. DO NOT PAIR (Q04F-2xxx, Q04B-6xxx) QUARTZ FILTERS FROM DIFFERENT BATCHES/LOTS!!
 - c. The Lot # is printed on the box in which the filters are supplied (e.g. 54053).
3. Cover the bowls with aluminum foil so that no dust particles can fall into the bowl, but leaving a small gap for air to flow in and out of the bowl during baking.
4. Print out the aluminum disc template (one template with twelve circles per 48 discs, or 24 filters) on an 8" x 11" sheet of paper.
5. Fold aluminum foil into four layers, each layer the size of the template sheet, separating the foil layers with blank sheets of paper (this helps separate the discs later).
6. Align the layered foil with the paper template, and cut out the aluminum discs with scissors. Separate the aluminum discs from the paper discs; discard the paper discs.
7. Make an aluminum envelope and place the aluminum discs into it. The envelope should be covered to prevent dust from falling on the discs, but cracked open slightly to allow air to flow in and out during baking.
8. Place the quartz filter-loaded porcelain bowls and the disc-loaded aluminum envelope together into the muffle furnace (this should be set at 550 °C).
9. Close the oven door and turn on the oven if required. Wait for at least four hours, though an overnight baking procedure may be convenient.
10. When the baking time has completed, turn off the oven, and crack open the door slightly (this helps the filters cool faster though you should not leave the door wide open for a too-rapid cool-down). Cool-down typically takes about 2 hours.
11. Once the oven has cooled to about 100 °C, remove the porcelain bowls and the aluminum envelope.
12. Prepare self-adhesive labels with the filter numbers and Lot #, and attach the labels to polystyrene petridishes (both).
13. Line pre-labeled petridishes with the baked aluminum foil liners using the dies to ensure the foil sits properly. The foil liner ensures the quartz filter does not touch the sides of the polystyrene petridish (avoiding potential contamination).
14. Place the freshly-baked quartz filters in the petridishes, close the petridishes, and wrap Teflon tape around the edges (this helps prevent gases from seeping in/out).
15. Bag the quartz filters in series (this helps future shipment), and place the bags in the freezer. Label the bags with the filter numbers inside the bag, as well as the Lot #.
16. Complete the entries in the Master Logbook (Filter #, when prepared, etc.)
 - a. On the front page, write the range of quartz filter numbers that were prepared by you, along with the Lot # and the baking conditions (temperature, time in hours, furnace location).

Preparing quartz fiber filters: QA

- a. Analyze one filter set (Q04B/Q04F) from each freshly-baked lot of filters
- b. Make sure to note down the date when each group of filters was baked in the Master Logbook, along with the conditions, and the filter numbers.
- c. After the filters have been baked and stored in the freezer, analyze one blank filter set from the Not-Sent lot every month.
- d. Make sure each filter number is accompanied by a LOT number. NEVER mix filters from different lots in one group/set of baked filters.

Appendix B OCEC analysis of quartz filters: Procedure

The following is an example procedure used by the University of Illinois. This protocol uses a Sunset Laboratories OC/EC Analyzer.

CAUTIONS:

1. No two analyses can have the same name!
2. Do not take filters out of aluminum foil-lined petri-dishes. Punch them inside the petridish. But don't punch through the aluminum foil liner.
3. Do not punch the filter with the sampled face face down on the aluminum foil.
4. Check the PAR file, the FID flame, and the pressure before each analysis.
5. Be very gentle with the valves turn down the valves only till you begin to feel resistance, DO NOT FORCE THE VALVES.
6. Duplicate analysis for 10% of the filters of each type
7. In case of static (e.g. punch clings to the oven, doesn't sit on the boat), wave the Polonium strip (near the OC/EC analyzer) close to and over the oven a few times (do not touch the filter or sample boat with it).

Daily Procedures

Must be performed before each day of analysis or in-between analyses as specified.

1. Check and log pressure in cylinders and regulators before the start of each analysis session.
 - a. Order new tank if cylinder pressure is < 500 psi (800 psi for the Methane/Helium, which takes longer to order).
 - b. Set HeOx, He Regulators @ ~20psi and H2 at 18-20 psi.
 - c. Set Air and He-CH4 (calibration gas) regulators @ ~14 psi (both equal)
2. Set gas flows and light FID (flame ionization detector) by pushing the RED button.
 - a. First, set the air flow (~290 cc/min).
 - b. Initially, for lighting the FID, set the Hydrogen gas flow to ~90 cc/min, then push the RED button. Once you have confirmed the flame is lit (e.g. water condensation on a metal surface; also, if you hit the RED button a second time, there should not be an audible "pop"), reduce the Hydrogen to the desired range (65 ± 2 cc/min). The Hydrogen inlet pressure should be about 18-20 psi.
 - c. Next, adjust the Helium, He/Ox, and Cal. The flows should be about:

Air: ~290 cc/min
H2: 65 ± 2 cc/min
He1: 54-58 cc/min
He2: 12-13 cc/min
He3: 67-70 cc/min

He/Ox: 14-15 cc/min
Cal. Gas: 10-12 cc/min

3. Run instrument blank with NIOSH870-Bond-v1.par parameter file (you can use the quartz punch already in the oven from the previous analysis session)
 - a. Clean the oven by selecting "Run\CleanOven\" from the OCEC program menu.
 - b. Name the rawdata file as yymmdd.txt (select the correct directory e.g. C:\SunsetOCEC\Rawdata\bangkok or C:\SunsetOCEC\Rawdata\kimoanh)
 - c. Name file inst_blank_yymmdd (e.g. "inst_blank_050331"); subsequent instrument blanks can be named inst_blank_yymmdd_2, and so forth. REMEMBER: No two analyses have the same name.
 - d. OC should be less than 0.5 $\mu\text{g}/\text{cm}^2$; if not, repeat the run with the same punch inside the oven. (Do not worry if the first blank is $>0.5 \text{ g}/\text{cm}^2$, but if three successive are, then there is a problem.)
 - e. Log all analyses on calibration log sheet.
4. Taking punches from filters (every analysis run):
 - a. Make sure the punch, forceps, and punch-removing-tool (a.k.a. the paper clip) are cleaned with acetone and the acetone has evaporated off the tool surfaces before punching/handling the filter.
 - b. Usually, all filters are stored in aluminum foil-lined polystyrene petridishes. Filters should not be taken out of the petridish.
 - c. Take a punch from the quartz filter, with the sampled face (either showing a grey/black deposit or the side without any noticeable uniform hashing/lines on the filter) upward facing the punch. NEVER TAKE A PUNCH WITH THE SAMPLED FACE DOWNWARD PRESSING INTO THE ALUMINUM FOIL. Be gentle do not punch through the aluminum foil liner.
 - d. If the filter is stuck face down in the petridish, remove it by gently nudging the edges with clean forceps and place it sampled face-up in the petridish.
 - e. After taking a punch from the filter, close the petridish.
 - f. Immediately after loading the punch in the oven and starting the analysis, clean the punch, forceps, and punch-removing-tool (a.k.a. the paper clip) with acetone, to ensure the tools are dry before you have to take the next punch.
5. Analyze regular samples/blanks using the NIOSH870-Bond-v1.par parameter file.
 - a. If the sample is heavily loaded (e.g. you see the carbon the green FID response tailing into the calibration mode with the NIOSH870-Bond-v1.par protocol), use the NIOSH870-Bond-v2.par (Not yet created) protocol (analyze another punch).
 - b. A replicate analysis may not be always feasible, e.g. with Kim Oanhs kyq samples. For this, if you have a doubt (e.g. filter is really dark or black), use the NIOSH-Bond-v2.par (Not created yet) file for the analysis.
6. Make sure the FID flame is burning before each analysis.
7. Check the (FID1) "Calibration area" after each sample this should be relatively constant (now, about 240,000 units on FID1, about 1/4 that on FID2). Potential problems double calibration peaks, short calibration peaks. Make sure there are no leaks in the system (check system pressure and FID flame).
 - a. In case of double or half calibration peaks, see #8(d) below.
8. IMPORTANT: Run duplicate analyses:
 - a. For Bangkok DIESEL samples (Q04F-2xxx or Q04B-6xxx), run replicate analysis on 10% of the filters:
If the filter label number ends in 5 (i.e. Q04F-2xx5), analyze two punches from the Q04F-2xx5 filter only. If the filter label number ends in 0 (i.e. Q04B-6xx0), analyze two punches from the Q04B-6xx0 filter only. MAKE SURE YOU ANALYZE SAMPLES AS SEQUENTIALLY AS POSSIBLE.
 - b. For all other samples (e.g. kimoanhs kyq), run duplicate analysis of every tenth filter.

c. If difference between duplicates is more than combined measurement uncertainty, troubleshoot using the Sunset S.O.P.

d. In case of any major problems during the analysis, e.g. double-or-half calibration peak area, sudden leaks, FID flame-outs, etc. make a note in the logbook, and analyze another punch from the filter. If the Calibration Area is 10-15% off the usual value during that analysis session (as of writing, ~240,000 units on FID1), make a note in the “Comments” section; we can reanalyze this filter later.

9. At the end of each analysis session:

a. Click on “Standby”

b. IMPORTANT: Turn down all the needle valves to their zero position, till you begin to feel resistance. Turning the valves down tight can damage them.

Weekly Procedure

1. Run 3-Peak calibration with (Not yet created) parameter file (once every analysis session).

a. Name file `cal3peak_yymmddde.g.cal3peak_050331`

b. Check the results:

- Integrate HeOx and He peaks using OCECCALC (140 325 seconds for Helium-mode, 325 670 seconds for He/Ox)

- Both peaks should be within 5% of calibration area. Enter this offset in the “+/-” column, e.g. +5%

- If not, run again and troubleshoot using Sunset S.O.P.

- Log calibration area and percent differences of HeOx and He peaks from the calibration area

c. CHANGE THE PAR FILE TO NIOSH-Bond-v1.par AFTER 3-PEAK CALIBRATION

2. Run 1 point calibration with sucrose standard, aim for about 10 μg of carbon on filter.

3. For Sucrose Standard Calibration procedure refer to page 8 in Sunset Laboratory Standard Operating Procedure (SOP): For the Analysis of Organic and Elemental Carbon on Quartz Fiber Filters

Monthly Procedure

1. Run 3 point calibration with sucrose standard aiming for 10, 25 and 40 μg carbon on each filter. Check linearity of instrument response vs. carbon input.

Quarterly Procedure

1. Run temperature calibration according to the “Using the Temperature Calibration Device” in the OCEC binder.

2. The temperature calibration should also be performed after changes to the main over or heating coil, or if the insulation is adjusted.

Biannual procedure: Flow calibration

1. Turn off main ovens and methanator ovens (methanator oven temperature must be ≥ 100 C or the oven can be ruined due to exposure to air)

2. Setup flow connector from flow box exhaust vents (1/8 fingertight fittings) to tubing for connection to the Gilibrator.

3. Follow procedure in the Sunset Manual (page 14) Installation Section 5-8.

4. Use Gilibrator, averaging measurements.

Other Occurrences

1. If a filter should happen to fall off the glass tray in the machine
 - a. Use a hooked piece of SS wire to remove the filter (e.g. long paper clip).
 - b. Clean oven: Run -> Clean Oven
2. If the reflectance signal is literally “off the chart” it can be adjusted by carefully rotating the black nob directly above the laser.
3. If you should happen to break the glass boat:
 - a. Report immediately (one boat should be in reserve at all times, so we need to order a replacement if one is broken)

References: Sunset Laboratory Standard Operating Procedure (SOP) for the Analysis of Organic and Elemental Carbon on Quartz Fiber Filters.

Acknowledgments: Andrew Grieshop, Carnegie Mellon University

Appendix C Gravimetric filter analysis: Procedure

The following is an example procedure used by the University of Illinois. The protocol is written using a Cahn Microbalance.

General Operating Principles

1. NEVER turn off the instrument if the power goes out, warm up the instrument for at least two hours before use. The power switch is at the right-hand backside of the instrument.
2. Warm up (i.e. POWER ON BUT DO NOT USE) for at least 2 hours if the instrument was just Switched On (see 1).
3. Press RANGE to select the weight range (this is normally the middle “A” LED 250 mg).
4. The “SAMPLE” pan is the bigger, flat pan.
5. The BRAKE raises the pans so that when you place a sample (or calibration weight) on the pan, there is minimal pan-swing. If you do engage the BRAKE, after placing the weight, remember to release the BRAKE by pressing the BRAKE button again before taking a reading. We haven’t found the BRAKE to be as efficient as just being gentle when placing the filter on the pan.
6. If the SAMPLE pan comes unhooked, re-hook it using a Kim-Wiped pencil (see the Manual). (Just try not to do this!)

Weighing filters General procedure: EQUILIBRATION, CALIBRATION, MEASUREMENT.

1. Always wear gloves when weighing or otherwise handling filters. Wear one long glove, and over it, a pink anti-static glove. Typically the glove is worn on the right (or dominant) hand, and only the right glove port is opened.
2. EQUILIBRATION: Filters must equilibrate to the box temperature and relative humidity for 24 hours before weighing. They must be in the glove box, lying in the open petri dishes.
3. MEASUREMENT of one filter:
 - a. Make sure the weighing chamber window is closed.
 - b. Wait for the empty sample pan to stop moving, and for the weight reading to stabilize.
 - c. The instrument should read 0.0000.003 mg; if not, press TARE to zero the display. (You should not need to TARE often if the balance is stable.) (Hint: press TARE with the blunt end of the forceps to avoid disturbing the balance.)
 - d. Open the weighing chamber window.

- e. De-static the filter (see STATIC below)
 - f. Place the filter carefully on the sample pan.
 - g. Close the weighing chamber window.
 - h. Wait for the sample pan plus filter to stop moving, and for the weight reading to stabilize. It may fluctuate up and down, but should not be continuously changing in the same direction.
 - i. Record the weight.
 - j. Open the weighing chamber window, remove the filter, and put it back in the petri dish.
 - k. Close the window and make sure the weight returns to 0.000 ± 0.003 mg.
4. SEQUENCE: Process the filters in batches of FIVE: weigh each of five filters once, and repeat the sequence of five three times. The three readings for each filter should be within 0.005 mg (max - min). If not, take a fourth reading for that filter and use the best-three-of-four (ONLY IF WITHIN 0.005 mg). If you still do not get a good reading, discard the filter (if clean) and make a note in the logbook. Do not discard dirty filters, but make a note in the logbook.
 5. CALIBRATION is done at the beginning of each day. The procedure is similar to MEASUREMENT except that this is done with a weight.
 - a. Use the 200 mg weight to calibrate the "A" range.
 - b. Use nylon or Teflon forceps (not metal) to handle the calibration weight.
 - c. Tare, weigh the weight, and re-tare as described under MEASUREMENT.
 - d. Always store the filter weight in its container. Scratching the weight may cause its mass to decrease.
 - e. Weighing the calibration weight like a regular filter (thrice). Repeat this calibration check after two or three five-filter sessions.
 6. STATIC: Millipore, PVC and other filters may carry a static charge, causing the scale to read incorrectly. This shows up as problems with the reading (noisy read-out, drift, and/or sudden read-out shifts). Before placing the filter on the SAMPLE pan, pass each side of the filter by the Polonium strips (move it in circles 3-5 times) to remove the static charge. Do not touch the strips, or the sample may be damaged.
 7. CONTROL FILTER: One filter should be kept in the glove box and equilibrated with the others. This filter should be weighed once in every 15 filters. It should be the first filter of the day. If this filter's weight changes more than 0.005 mg, the balance should be re-calibrated.

Appendix D Example datasheet

Example Kiln Data Sheet

Date	
Time	
Technician	
Computer	
Site	
Event	
Probe	
Nozzle #	
Nozzle Diam. (mm)	
Fuel	

Holder #		Filter ID
	Grav	Stage 1
		Stage 2
	OCEC	Stage 1
		Stage 2

Initial Conditions							
	AethFlow	IsoFlow	F1Flow	F2Flow	GasFlow	DilFlow	Cyclone
Sensor (sccm)							
Bubble (ccm)							
Dilution Ratio		Ambient Temperature (C)				Ambient RH (%)	
Stack Velocity (m/s)		Ambient Pressure (hPa)					

Final Conditions							
	AethFlow	IsoFlow	F1Flow	F2Flow	GasFlow	DilFlow	Cyclone
Sensor (sccm)							
Bubble (ccm)							
Dilution Ratio			Ambient Temperature (C)			Ambient RH (%)	
Stack Velocity (m/s)			Ambient Pressure (hPa)				

Time	Pre-background		Sample Period		Post-background	
	start	stop	start	stop	start	stop

[illegible]

Make dimensional drawing of stack cross-section and sample points on back side