NC DAQ Source Test Observers Checklist
EPA Methods 1 - 5 & 26A (40 CFR 60 Appendix A)
METHOD 26A—DETERMINATION OF HYDROGEN HALIDE AND HALOGEN EMISSIONS FROM STATIONARY SOURCES - ISOKINETIC METHOD

Line by line explanation and references.

This method is applicable for determining emissions of hydrogen halides (HCl, HBr, and HF) and halogens (Cl_2, Br_2, and F_2) from stationary sources. Gaseous and particulate pollutants are withdrawn isokinetically from the source and collected in an optional cyclone, on a filter, and in absorbing solutions. The tester may use this sampling arrangement to also sample concurrently for filterable particulate matter.

This checklist is intended to point out stack testing procedures that can be easily checked to help ensure an accurate and valid test. It is not intended to replace familiarity with the reference test methods. Some items can be filled out prior to the test observation, such as the Facility ID #, source tested, and applicable regulations.

In addition, be certain to review the stack test protocol and the DAQ protocol approval letter prior to going on-site. These documents may indicate changes in methodology or special items you will need to verify during the observation. This information can be essential in answering questions on-site and determining if enough applicable data is being recorded. **Do not reject a test without consulting with the Stationary Source Compliance Branch (SSCB). If you have testing concerns discuss them immediately with the testing company and SSCB.**

This reference document is intended to be used in conjunction with the Methods 1-5 checklist reference document. **Items common to both checklists (most of pages 1 and 3 – i.e. flow, moisture, and particulate measurement) are explained only in the Methods 1-5 checklist reference document.** This reference document simply adds guidance for the new and modified checklist items from Method 26A.

Additional notes:

- This checklist includes simultaneous sampling for filterable particulate. This is not required for M26A and in many cases the company will not be sampling for particulate at the same time. If this is the case, the Method 5 filter will still be used and acetone cleanup will still be conducted but neither are weighed/analyzed. The isokinetic sampling principles and flow/moisture measurement still applies to Method 26A.
- If sampling is not being conducted for halogens (Cl_2, F_2, and Br_2) then the fourth and fifth impingers can be removed from the sampling train. If this is not clear in the protocol and protocol approval letter, be sure to confirm with the SSCB.

Changes/Notes for Page 1 from the Methods 1-5 (only) Observation Checklist

4.1) The impinger/moisture collection setup is very different for Method 26A versus a straight Method 4. This checklist item 4.1 now simply informs the observer to refer to page 2 for the M26A impinger and equipment requirements.

5.1) M26A is also required to be run concurrently with Methods 2-5.

5.6) M26A requires higher filter and probe temperatures than Method 5. The new requirement is described on page 2 (Section 26A.2.a). In addition, Method 26A requires a Teflon mat filter except as allowed in 26A.1.d below.

5.9) Clarification is added that these acetone cleanup procedures apply only to the "front half" (Method 5) portion of the sampling train. This is the equipment prior to and including the Method 5 filter. Cleanup for the "back half" (Method 26A) portion of the train is different and described on page 2 of the M26A checklist (Section 26A.3.d,e).

Method 26A Observation Checklist Guidance (for page 2 of 3 of the M5/26A Checklist)

26A.1. *Equipment and Reagents per Method 26A? (Impingers 4 & 5 optional if only testing for HCl, HF)*

The below Figure 1 gives a general overview of the required equipment. Specific equipment requirements are spelled out in the following section. If sampling is not being conducted for halogens (Cl_2, Fl_2, and Br_2) then the fourth and fifth impingers (0.1 N NaOH) can be removed from the sampling train.

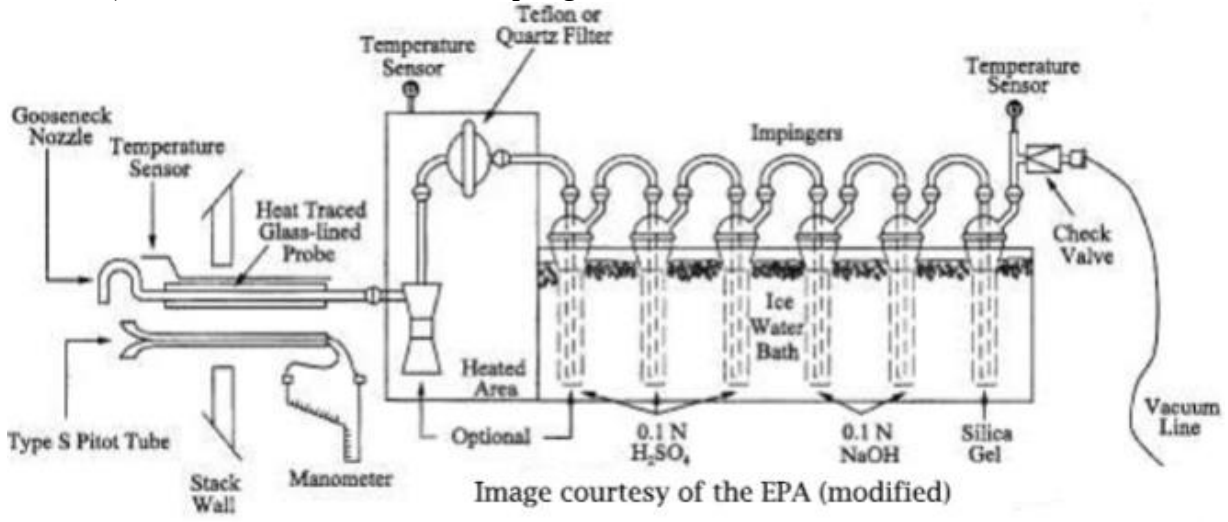

Figure 1 – Schematic of M26A Sampling Train (Courtesy of the EPA)

26A.1.a *Probe nozzle and probe liner borosilicate or quartz glass?*

Use a borosilicate or quartz glass nozzle; constructed and calibrated according to Method 5, sections 6.1.1.1 and 10.1, and coupled to the probe liner using a Teflon union. A stainless steel nut is recommended for this union.

26A.1.b *Cyclone (optional) between probe liner and filter holder?*

The use of the cyclone is required only when the sample gas stream is saturated with moisture; however, the cyclone is recommended to protect the filter from any liquid droplets present. The cyclone may be glass or Teflon. The cyclone is not typically used.

26A.1.c *Teflon mat filter used?*

A Teflon mat (e.g., Pallflex TX40HI45) filter should be used unless the stack gas temperature exceeds 210 °C (410 °F). Then a quartz fiber filter may be used.

26A.1.d *Stack temp > 410 Deg F? If so, quartz filter may be used and one-piece glass nozzle/ liner mandatory*

When the stack temperature exceeds 210 °C (410 °F), a one-piece glass nozzle/liner assembly must be used and a quartz fiber filter may be used

26A.1.e *Impinger #1 (Optional knockout or condensate impinger; shortened stem) 50 ml of 0.1 N H2SO4*

This optional impinger is recommended as a water knockout trap for use under high moisture conditions. If used, this impinger should be constructed with a shortened stem (see Image No. 1 below) and contain 50 mL of 0.1 N H_2SO_4.

Image No. 1 – Shortened Stem Impinger

26A.1.f,g *Impingers #2 and #3 (Greenburg-Smith Standard Tip & 100 ml of 0.1 N H2SO4) (Acid Impinger)*

Impingers #2 and #3 should each contain 100 ml of 0.1 N H_2SO_4 and shall be of the Greenburg-Smith design with the standard tip (Method 5, section 6.1.1.8; see Image No. 2 below).

Image No. 2 – Greenburg-Smith Standard Tip Impinger

26A.1.h,i *Impingers #4 and #5 (Modified Greenburg-Smith & 100 ml of 0.1 N NaOH) (Alkaline Impinger)*

Impingers #4 and #5 shall be of the modified Greenburg-Smith design (Method 5, section 6.1.1.8; see Image No. 3 below) and each shall contain 100 ml of 0.1 N NaOH.

Image No. 3 – Modified Greenburg-Smith Impinger

26A.1.j *Impinger #6 - silica gel (See item 4.3 on page 1)*

Self-explanatory.

26A.1.k *Acidic and Alkaline absorbing solutions prepared per Method?*

Acidic Solution Preparation: To prepare 1 liter of the 0.1 N H_2SO_4 solution, slowly add 2.80 ml of concentrated 17.9 M H_2SO_4 to 900 ml of water while stirring, and adjust the final volume to 1 liter using additional water. Shake well to mix the solution.

Alkaline Solution Preparation: To prepare 1 liter of the 0.1 N NaOH solution, dissolve 4.00 g of solid NaOH in 900 ml of water and adjust the final volume to 1 liter using additional water. Shake well to mix the solution.

26A.2.a *Probe and filter temperatures between 248 and 273 Deg F?*

The heating system used must be able to maintain the temperature range between 248 to 273 °F. Probe and filter temperatures should be recorded at each sampling point.

26A.3.a *200 ml blanks prepared for each absorbing solution? (250 ml of acidic sol. if optional impinger used)*

A separate blank solution of each absorbing reagent should be prepared for analysis with the field samples. These are referred to as Container Nos. 6 through 9 (Reagent Blanks) in the method. Save portions of the absorbing reagents (0.1 N H_2SO_4 and 0.1 N NaOH) equivalent to the amount used in the sampling train; dilute to the approximate

volume of the corresponding samples using rinse water directly from the wash bottle being used. Add the same ratio of sodium thiosulfate solution used in container No. 4 to the 0.1 N NaOH absorbing reagent blank. Also, save a portion of the rinse water alone and a portion of the acetone equivalent to the amount used to rinse the front half of the sampling train. Place each in a separate, prelabeled sample container.

26A.3.b *Blanks diluted to same volume of field samples (see d,e below) using blank sample of DI rinse water?*

Dilute 200 ml of each absorbing solution (250 ml of the acidic absorbing solution, if a condensate impinger is used) to the same final volume as the field samples using the blank sample of rinse water. If a particulate determination is conducted, collect a blank sample of acetone.

26A.3.c *Post-test moisture removal (optional and typically not conducted) - required when the optional cyclone is used or when liquid is visible on the filter at the end of the sample run.*

When the optional cyclone is included in the sampling train or when liquid is visible on the filter at the end of a sample run even in the absence of a cyclone, perform the following procedure. Upon completion of the test run, connect the ambient air conditioning tube at the probe inlet and operate the train with the filter heating system between 120 and 134 °C (248 and 273 °F) at a low flow rate (e.g., $\Delta H = 1$ in. H_2O) to vaporize any liquid and hydrogen halides in the cyclone or on the filter and pull them through the train into the impingers. After 30 minutes, turn off the flow, remove the conditioning tube, and examine the cyclone and filter for any visible liquid. If liquid is visible, repeat this step for 15 minutes and observe again. Keep repeating until the cyclone is dry. NOTE: It is critical that this procedure is repeated until the cyclone is completely dry.

26A.3.d *Acid Impinger Catch - Measure liquids from impinger #'s 1-3; rinse impingers and connecting glassware with DI water; and add all liquids (impinger catch and rinse water) to one storage container.*

Container No. 1 - (Optional; Filter Catch for Particulate Determination). Same as Method 5, section 8.7.6.1, Container No. 1. i.e. the filter.

Container No. 2 - (Optional; Front-Half Acetone Rinse for Particulate Determination). Same as Method 5, section 8.7.6.2, Container No. 2.

Container No. 3 - (Knockout and Acid Impinger Catch for Moisture and Hydrogen Halide Determination). Disconnect the impingers. Measure the liquid in the acid and knockout impingers (impingers #1-3) to ±1 ml by using a graduated cylinder or by weighing it to ±0.5 g by using a balance. Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Transfer this liquid (from all three impingers) to a leak-free sample storage container (Container #3). Rinse these impingers and connecting glassware including the back portion of the filter holder (and flexible tubing, if used) with water and add these rinses to the same storage container. Seal the container, shake to mix, and label. The fluid level should be marked so that if any sample is lost during transport, a correction

proportional to the lost volume can be applied. Retain rinse water and acidic absorbing solution blanks to be analyzed with the samples.

26A.3.e *Alkaline Impinger Catch - Measure liquids from impingers #4 & #5; rinse impingers and connecting glassware with DI water; and add all liquids (impinger catch and rinse water) to one container.*

Container No. 4 - (Alkaline Impinger Catch for Halogen and Moisture Determination). Measure and record the liquid in the alkaline impingers as described in section 8.2.3. Transfer this liquid to a leak-free sample storage container. Rinse these two impingers and connecting glassware with water and add these rinses to the container. Add 25 mg of sodium thiosulfate per ppm halogen anticipated to be in the stack gas multiplied by the volume (dscm) of stack gas sampled (0.7 mg/ppm-dscf). Seal the container, shake to mix, and label; mark the fluid level. Retain alkaline absorbing solution blank to be analyzed with the samples.

NOTE: 25 mg sodium thiosulfate per ppm halogen anticipated to be in the stack includes a safety factor of approximately 5 to assure complete reaction with the hypohalous acid to form a second Cl ion in the alkaline solution.

Container No. 5 - (Silica Gel for Moisture Determination). Same as Method 5, section 8.7.6.3, Container No. 3.

26A.3.f *Sodium thiosulfate added to alkaline impinger catch per Method 26A?*

See 26.A.3.e above.

26A.3.g *DI rinse water blank prepared?*

A portion of the rinse water alone and a portion of the acetone equivalent to the amount used to rinse the front half of the sampling train should be saved. Each should be placed in a separate, prelabeled sample container.

26A.3.h *Is the rinse water deionized, distilled water that conforms to American Society of Testing and Materials (ASTM) Specification D 1193-77 or 91, Type 3*

Self-explanatory.

26A.3.i *Record the analytical lab to be used for analysis:* Self-explanatory.

26A.3.j *Audit sample obtained (if required and commercially available)?*

An audit sample analysis is required when conducting EPA Reference Method 26A if there are two or more independent accredited audit sample providers (AASP) to have blind audit samples available for purchase. **Note:** Currently (as of December 2019), there are no longer the minimum two providers, therefore, the requirement to obtain these audit samples is no longer in effect until such time as another independent AASP has audit samples available for purchase.