CTM-027 – DETERMINATION OF AMMONIA EMISSIONS IN STATIONARY SOURCES

Applicability

This method is applicable for the determination of ammonia emissions from stationary sources.

Principle

A stack gas sample is withdrawn isokinetically from the source and must be collected at the actual stack temperature, or slightly above, in order to minimize either negative or positive reactions that would bias the ammonia results. Samples pass through an in-stack filter and are collected in impingers containing sulfuric acid solution. All collected sample fractions are analyzed using ion chromatography.

In-Stack Detection Limits & Sample Times

CTM-027 sample recovery procedures are designed such that a sample containing 1 ppmV ammonium can be detected in the sample collection media. Actual in-stack method detection limits (ISDL) are based on actual source sampling parameters and analytical results. Actual detection limits can be improved through increased stack gas sampled (sample time).

For this source, the in-stack concentration is:

Allowable = _____ lbs/hr
Stack flow = _____ dscfm
MW = molecular weight.

ppm = (lb / hr) x 387E6 / (dscfm x MW x 60) = _____ ppm.

Therefore, the sample time will be _____ minutes.

Sample Train Equipment & Reagents

The CTM-027 sample train is based upon the Reference Method 17 sample train, a schematic of which is shown in Figure 17-1 of that method. The sample train will be constructed of components specified under CTM-027, Section 1.1, with the following highlights.

1) **Probe liner** and **nozzle** – constructed of borosilicate or quartz glass.
2) **In-stack filer holder** –constructed of borosilicate glass or Teflon, a filter support (with gaskets made of either Teflon, Viton, silicone rubber, each capable of handling actual stack temperatures).

3) **Filters –** glass fiber filter without organic binders, with collection efficiency of $\geq$ 99.95% for 0.3 um diameter particles.
4) **Dry Gas Metering System**
5) **Impinger train**
    - **Impingers #1 and #2 –** each of Greenburg-Smith (G-S) construction and each charged with 100 ml of 0.1N H_2SO_4.
    - **Impingers #3 –** modified Greenburg-Smith (MG-S) construction with the tip removed and is either left empty or charged with 100 ml of 0.1N H_2SO_4 (if needed to capture possible breakthrough from impinger #2 due to high ammonia concentrations and/or high sample flow rate requirement).
    - **Impinger #4** – modified Greenburg-Smith construction with the tip removed and charged with 200-300 grams of indicating silica gel.
6) **Silicone grease –** may be used for impinger assembly.
7) **Silica Gel**
8) **DI Water –** must be blank-checked for ammonium ion and other constituents of interest prior to testing.
9) **0.1N Sulfuric Acid Solution** – reagent grade

Sample Recovery Equipment

1) **Wash bottles –** Polyethylene (2 quantity). One containing DI water, the other reagent-grade acetone.
2) **Sample Storage Bottles –** Clean high-density polyethylene (HDPE) bottles with 250 or 500 ml capacity, having wide mouth construction and airtight seals. Used to store the 0.1N H_2SO_4 impinger solutions, rinses and spent silica gel.
3) **Graduated Cylinders –** glass or HDPE

Sample Train Preparation & Assembly

The sample train is assembled as per Section 2.0 of the method. Once the sample train is assembled and pre-heated (either with the heating system or the stack), a pre-test leak check is required, as stated below.

Leak Checks

Pre-test and post-test leak checks will be conducted following the procedures of Method 17. The pre-test leak check will be conducted at 15" Hg vacuum (or a lower rate not to be exceeded during sampling) and the post-test leak check will be conducted at or above the highest vacuum reached during sampling. A leakage rate in excess of 0.02 cfm is unacceptable and will void the test run.

Sampling

1) **The stack gas temperature will be determined**, then the in-stack filter, probe and area just prior to the entrance of the Impinger #1 will be pre-heated to a temperature at or slightly above the stack gas temperature. Note: The pre-heating is optional and is done to save time by getting the filter up to stack temperature. Alternatively, the train will be pre-heated in the stack.

2) The dry gas meter will be activated and **a pre-test leak check** will be conducted on the sample train as indicated above.
3) The preheated and leak checked sample train will be placed into the source stack and **the isokinetic sampling procedures in Method 17 will be followed.**
4) At end of sampling period a **post-test leak check** will be conducted as indicated above.
5) **Three valid test runs** will be conducted, consecutively.

Sample Recovery

Note: Acetone will not be used to dry glassware.

1) The nozzle is removed and the in-stack filter holder is disassembled. The filter is discarded (not used for analysis). Using DI water, front half of filter holder and nozzle are cleaned and dried to prepare them for the next run.
2) **Sample Container #1** (250 ml HDPE bottle):
   A) The back half of the in-stack filter holder and the glass probe liner with any glassware attaching it to the first impinger are each rinsed three times with 10 ml portions of 0.1 H_2SO_4 and the rinses added to Sample Container #1.
   B) Next, the same components are each rinsed three times with water and these water rinses are added to Sample Container #1 for analysis. Note: Although the method has conflicting information regarding the fate of these water rinses, since this sample is then diluted with water (see next step), BTS requires the water rinses be recovered in this step and not discarded.
   C) The final volume of Sample Container #1 will be brought to exactly 230 ml using DI water.
3) **Sample Container #2, Sample Container #3,** and **Sample Container #4** (250 ml HDPE bottles):
   A) The Impinger #1 solution will be poured into a graduated cylinder; the volume recorded, then the contents poured into Sample Container #2. The impinger stem, impinger body and graduated cylinder will be each be rinsed with 5 ml portions of water and the rinses added to Sample Container #2. The final sample volume will be brought to exactly 230 ml using DI water.
   B) The above procedure will be repeated for Impinger #2 and Impinger #3, placing the impinger contents and rinses into Sample Container #3 and Sample Container #4, respectively.
4) **NOTE**: The total volume in each Sample Container should not exceed 230 ml so that 20 ml of DI water can be used when transferring the contents in the laboratory for analysis. The final volume of each Sample Container to be analyzed should be 250 ml. If the final volume of any of the fractions is different from 250 ml, then the CTM-027 Equation 2 conversion factor must be modified accordingly (i.e. if 260 ml is the final volume in one of the sample fractions, the conversion would be 0.26 instead of 0.25).
5) Impinger #4 contents (silica gel) will be transferred into a 250 ml HDPE bottle to determine moisture weight gain.
6) A field blank of the 0.1N sulfuric acid impinger solution will be collected, recovered and analyzed, per Section 3.6 of CTM-027.

Note: All samples will be kept refrigerated (not frozen) at 39ºF and warmed slowly prior to analysis.

Note: Sample analysis must be conducted **within two weeks** after their collection date in the field.

Sample Preparation & Analysis

1) Impinger Solutions –The contents of each of the Sample Containers and the field blank will be poured from their HDPE bottles into separate 250 ml volumetric flasks. The interior of each emptied HDPE bottle will be rinsed twice with 10 ml portions of DI water and the rinses added to their respective flasks. The final volume in each flask will be brought to 250 ml with DI water if necessary.
   NOTE: Sample Container #4 (Impinger #3 contents and rinses) need not be prepared for analysis unless ammonia breakthrough has occurred as indicated by analysis of Sample Containers #2 and #3. The general rule for determining breakthrough has occurred is when the concentration of Container #3 (Impinger #2 contents and rinses) is greater than 10% of the concentration of Container #2 (Impinger #1 contents and rinses).
2) Follow the conditions outlined in the method for Ion Chromatography conditions, calibration and QA/QC.

Calculations

All calculations must be performed in accordance with CTM-027, including any blank corrections. Detailed sample calculations will be included in the final report.

Special Situations Section

In limited situations (ie: moisture saturated stacks or extremely high temperature stacks) an in-stack filter may not be viable. **Subject to BTS approval**, an out-of-stack filter will be used in these situations, with the probe and filter temperature maintained:

1) At a temperature sufficient to prevent moisture condensation for a moisture saturated stack.

2) At a temperature of 350 ºF or greater for extremely high temperature stacks.

If either of these situations is present and an out-of-stack filter is being proposed, justification **must** be presented and additional details **must** be provided in the following "Proposed Deviations from this BTS Template or the Method" section. Otherwise, an in-stack filter will be used per the method.

Proposed Deviations from this BTS Template or the Method

(Insert any proposed deviations here)