

Controlled Condensation Method For Flue Gas SO₃ Measurements

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Background

Accurate SO₃ measurements are needed to diagnose and solve a wide variety of operating and emissions problems in fossil fuel-fired utility plants, such as visible plume problems, cold-end corrosion, and precipitator performance. There are two basic manual SO₃ sampling techniques: an impinger method that is based on SO₃ adsorption in a liquid medium, and a condensation method which is based on SO₃ collection due to its dew point. The major advantage of the condensation method is that it provides reliable and reproducible SO₃ and SO₂ values with minimal interference from high SO₂ concentrations. CONSOL R&D has used a modified condensation method for the past 15 years in a wide variety of applications. We made a number of modifications to this procedure to facilitate one-person operation with a rapid turn-around time and in-field data reduction. We have found this procedure to be reliable, reproducible, and sensitive at the 2 ppmv level. In this presentation, the CONSOL controlled condensation sampling method is described, and data are presented demonstrating reproducibility.

System Design

A schematic of the sampling system is shown in Figure 1. The flue gas sample is pulled through a temperature controlled (500 °F) quartz-lined probe fitted with a quartz wool plug to remove particulate matter. The sample then passes through a hot-water-cooled condenser that is loosely packed with glass wool. The condenser assembly is maintained at 140 °F. Sulfur trioxide (SO₃), in the presence of water vapor, will selectively condense on the glass wool while the flue gas, containing SO₂ and water vapor, passes through the condenser and into the miniature impingers. These impingers are similar to those used in EPA Method 6; the first two are filled with 20 mL of 3% hydrogen peroxide to capture the SO₂. These impingers are followed by one empty and one silica gel-filled impinger. After exiting the impingers, the gas is conveyed through a rotometer, through a pump, and, finally, through a dry gas test meter.

Sampling Procedure

The sampling components are assembled, heated and leak-checked. The probe is positioned in the flue gas duct. Care is taken to ensure an air-tight fit. The sampling pump is started, and the sampling rate of 3 LPM is maintained for 45-60 min. Sampling data are recorded at regular

intervals. They include:

Starting Volume	Flow Meter Reading
Interval Volume	Gas Meter Temperatures
Final Volume	System Vacuum
Probe Temperature	Sample O ₂ Concentration
Condenser Temperature	Barometric Pressure
Stack Temperature	Sampling Time Period

After completing the sampling protocol, the probe is removed from the stack and leak-checked. The sampling components are disassembled and transported to the recovery and analysis area.

Sample Recovery and Analysis

The quartz plug is removed and extracted with isopropyl alcohol (IPA). The solids are filtered and the filtrate is diluted to 50 mL and retained for analysis.

The sample probe is rinsed with IPA. The risings are collected, diluted to 50 mL, and retained for analysis.

The condenser is extracted with IPA. The IPA is collected, diluted to a known volume, and retained for analysis.

The impinger solution is recovered, and the impingers are rinsed with deionized water. The risings are combined with the spent impinger solution, diluted to a known volume, and retained for analysis.

The samples are analyzed by BaCl₂ titration, as described in EPA Method 6. The thorin end point is more visible when the titrating solution is >80% IPA. For this reason, IPA is used in all the recovery steps. The breakdown of SO_x species provided in this method follows:

Quartz Plug	-	SO ₃ Absorbed onto Solid Particles
Probe	-	Gas Phase SO ₃
Condenser	-	Gas Phase SO ₃
Impingers	-	SO ₂

In our test work, the sum of the probe and condenser represents the gas phase SO₃ concentration. In almost all cases, the majority of the measured SO₃ is collected in the condenser. The SO₃ values are reported in ppmv at duct conditions and at 0% flue gas oxygen. The percent of SO_x present as SO₃ is calculated.

Reproducibility

The reproducibility of the method was evaluated in a pilot-scale combustor and at a 650 MW coal-fired utility plant. In the pilot-scale combustor, the average SO₃ concentration was 9.0 ppmv with a standard deviation of 0.8 ppmv for a series of eight repetitive tests. This is a relative standard

deviation was 8.8%. The average SO₂ concentration for these test was 2293 ppmv, with a standard deviation of 51 ppmv. At the coal-fired utility, the average SO₃ concentration was 21 ppmv and the standard deviation was 2 ppmv for a series of six repetitive tests. This is a relative standard deviation of 10%. The average SO₂ concentration for these tests was 2904 ppmv, and the standard deviation was 79 ppmv.

Figure 1. Sampling System Schematic

