INFICON

APPLICATION NOTE

Rapid Analysis of SO_2 to Determine Catalyst Efficiency using Micro GC Fusion®

INTRODUCTION

Sulfuric acid is one of the most produced chemicals in the world. Almost 180 million tons are consumed per year on a worldwide basis.[1] In the United States alone, billions of pounds are produced and sold for a variety of uses including the formulation of fertilizers, insecticides and detergents. To generate sulfuric acid, sulfur dioxide (SO_2) is oxidized to generate sulfur trioxide (SO_3), which when reacted with water, forms sulfuric acid (H_2SO_4). The majority of sulfuric acid is manufactured using this process.

Catalysts facilitate the oxidation of SO_2. The analysis of SO_2 at the inlet and outlet of the catalytic bed determines the conversion efficiency and performance of the catalyst. A percent level of SO_2 is introduced at the catalytic bed inlet. After the catalytic conversion of SO_2 to sulfuric acid, the concentration of the SO_2 exiting the bed outlet is often around 100 ppm. Since sample integrity may be compromised due to the delay encountered transporting the sample to an analysis lab, it is preferable to obtain accurate results quickly and reliably on-site.

Gas chromatography (GC) can be used to accurately analyze a wide concentration range of SO_2. Micro GC Fusion is a small, transportable GC capable of analyzing SO_2 across a broad linear range. The microelectromechanical systems (MEMS) based thermal conductivity detector (TCD) in Micro GC Fusion is able to accurately measure a wide concentration range of compounds in less than 60 seconds.

EXPERIMENTAL

Micro GC Fusion is configured with a 12 m Rt-Q-Bond column and a variable volume injector. The Rt-Q-Bond column was selected due to the excellent separation of the SO_2 peak from the neighboring water peak. A variable volume injector allows a broad concentration range of samples to be analyzed.

Two calibration gas standards from Air Liquide® are used. The first standard contains 12% SO_2 in air to mimic the initial concentration of SO_2 at the catalytic bed inlet. The second standard contains 100 ppm of SO_2 in air to mimic the concentration of SO_2 exiting the bed. The method was designed to elute SO_2 quickly, while maintaining separation of the water peak.

The 12% SO_2 and 100 ppm SO_2 calibration gases were introduced using a 100 mL gas tight syringe. The syringe was then used to dilute the 12% SO_2 calibration gas to 6% and 0.96%, which were also introduced to the instrument. (See Table 1.) Multiple injections of each concentration were analyzed. A calibration curve was created by plotting the average area counts and concentrations of each of the four calibration standards.

Ten consecutive runs were conducted using the 12% SO_2 calibration gas standard to calculate the relative standard deviation (%RSD) for peak area and retention time. This calibration gas was connected directly to the sample inlet with 1/16 in. Restek SilcoNert® tubing.

RESULTS

In less than 60 seconds, SO_2 is separated from the solvent peak (air) and water. (See Figure 1.) Chromatograms corresponding to the four calibration standard concentrations are displayed in Figure 2.

The calibration curve shows excellent linearity of SO_2 concentrations ranging from 100 ppm to 12%. (See Figure 3.)

The %RSD calculations for both retention time and area show excellent repeatability. Over ten runs, the %RSD is 0.14% for retention time and 0.43% for area count. (See Table 2.)

CONCLUSION

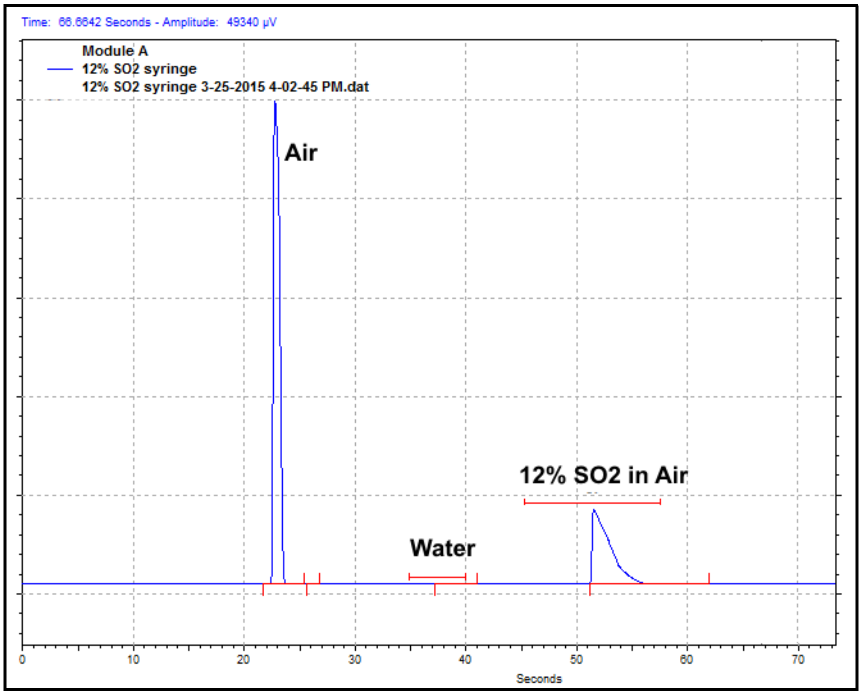
The 12 m Rt-Q-Bond column analyzes 100 ppm to 12% SO_2 at the inlet and outlet of the catalytic bed with excellent linearity and precision. Analyses can be conducted within 60 seconds directly on-site to monitor the conversion efficiency and performance of the catalyst to optimize sulfuric acid production efficiency.

DATA

Table 1 Air Liquide SO_2 calibration gas standard concentration information

Component	Calibration Gas 1	Calibration Gas 2	Dilution 1 (50 mL 12% SO2, 50 mL air)	Dilution 2 (8 mL 12% SO2, 92 mL air)
SO_2	12%	100 ppm	6%	0.96%
Air	Balance	Balance	Balance	Balance

Figure 1 Chromatogram of 12% SO_2 in air



Module: 12 m Rt-Q-Bond column, Variable Volume Injector, TCD
Column Temperature: 75°C (hold 10 s) --> 200°C*; Ramp Rate:* 1.5°C/s*; Column Head Pressure*: 35 psi, Helium
Inject Time: 75 ms, *Injector Temperature:* 90°C

Figure 2 Chromatogram overlay of four concentrations of SO_2

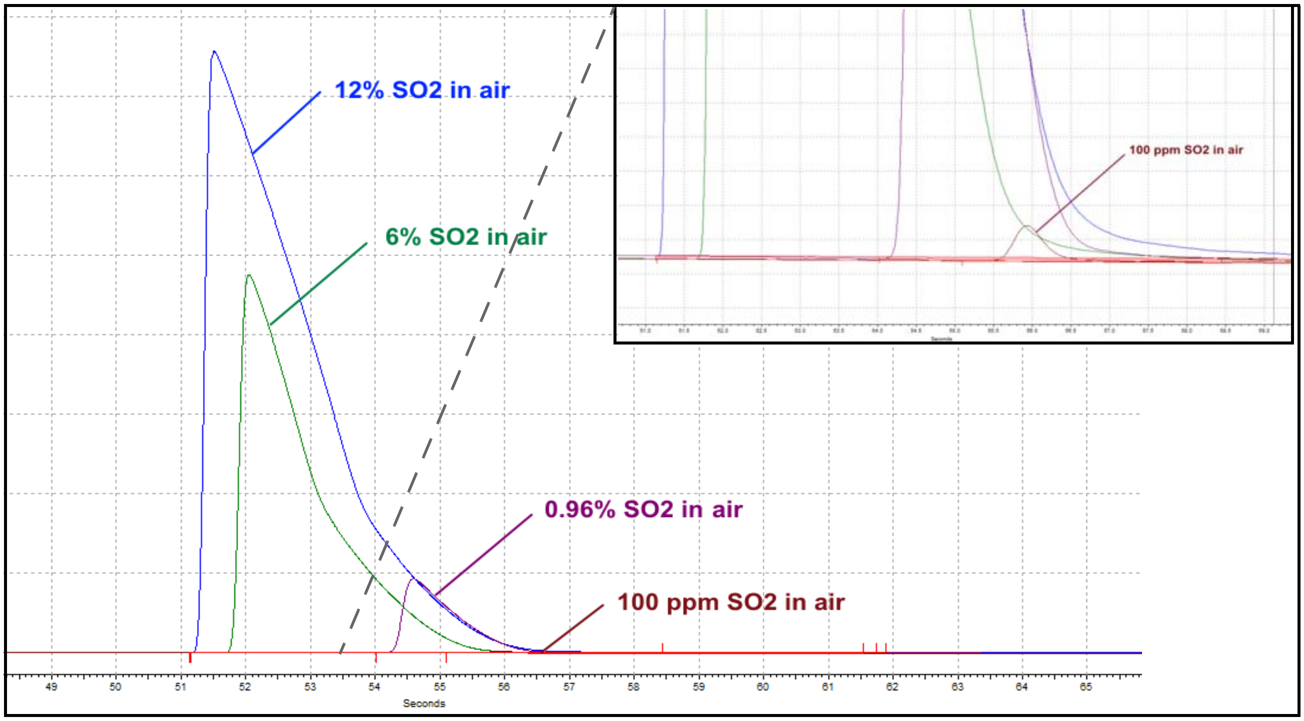


Figure 3 Calibration curve for SO_2

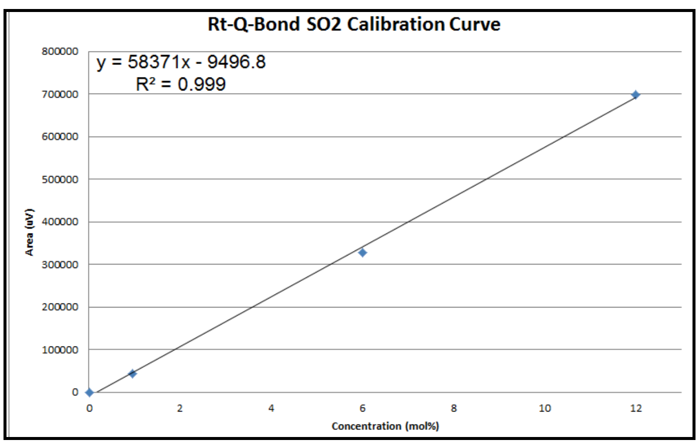


Table 2 Repeatability data for the 12% SO_2 calibration gas standard - ten runs

Compound	Retention Time (s)	RT %RSD	Area %RSD
12% SO2	51.3	0.14	0.43

REFERENCES

1 Davenport, W.G.; M.J. King; Sulfuric Acid Manufacture. Elsevier. 2006. https://books.google.com/books?id=tRAb2CniRG4C&printsec=frontcover#v=onepage&q&f=false (accessed August 17, 2015)