Application Note

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# Sulfur Dioxide Analysis Using the Agilent 990 Micro GC


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## Introduction

Sulfuric acid is one of the most important chemicals in the world. It is widely used for manufacturing fertilizers, pigments, dyes, drugs, explosives, detergents, and inorganic salts and acids. It is also used in petroleum refining and metallurgical processes.

The most important process for making sulfuric acid is the contact process. During this process, sulfur is burned in air to make sulfur dioxide ($SO_2$). Then, the $SO_2$ is converted to sulfur trioxide ($SO_3$) by oxygen ($O_2$). The reaction of $SO_2$ and $O_2$ is reversible, and usually a catalyst is used to speed up the reaction to increase the amount of $SO_3$ created.

During the contact process, the concentration of $SO_2$ should be monitored before and after its reaction with $O_2$ to track the conversion rate of $SO_2$. The $O_2$ concentration also needs to be monitored to ensure that $SO_2$ and $O_2$ are in the right ratio to make $SO_3$. The Agilent 990 Micro GC provides fast and accurate measurement of $SO_2$ and $O_2$, which helps control the $SO_3$ manufacturing process.

## Experimental

**Channel 1:** A 10 m, Agilent CP-Molesieve 5 Å, backflush channel, with retention time stability (RTS) option for $O_2$ analysis. The backflush option and RTS is used to protect the Molesieve 5 Å column from moisture, $CO_2$, $SO_2$, and other contaminants. This is beneficial to the long term RT repeatability and column performance of Molesieve 5Å column.

**Channel 2:** A 12 m, Agilent CP-Sil 19CB straight channel for $SO_2$ analysis.

Figure 1 shows the chromatogram of the $O_2$ analysis on channel 1. Figure 2 shows the chromatogram of the $SO_2$ and moisture ($H_2O$) analysis on channel 2. During manufacturing, $H_2O$ is present as moisture in the gas mixture of $O_2$ and $SO_2$. Figure 2 shows that $SO_2$ and $H_2O$ can effectively be separated on the CP-Sil 19CB column at the tested concentration. Their peak resolution in this chromatogram is 3.6, and $SO_2$ can accurately be quantitated. In the reaction gas, the concentrations of $SO_2$ and $H_2O$ are sometimes as high as 10%. With such high concentration, the peaks broaden, and the resolution gets worse. Under these circumstances, it is necessary to use a filter to remove the moisture from the reaction gas before analyzing $SO_2$. The signal-to-noise ratio (S/N) for 35 ppm $SO_2$ under the applied test conditions is 98, and the calculated detection limit is 1.1 ppm.

**Table 1.** Test conditions for the Agilent CP-Molesieve 5 Å and the Agilent CP-Sil 19CB channels.

| Channel Type | 10 m, Agilent CP-Molesieve 5 Å, Backflush | 12 m, Agilent CP-Sil 19CB, Straight |
|---|---|---|
| Injector Temperature | 110 °C | 110 °C |
| Column Pressure | 200 kPa | 220 kPa |
| Column Temperature | 80 °C | 50 °C |
| Carrier Gas | Helium | Helium |
| Backflush Time | 7 seconds | NA |
| Injection Time | 40 ms | 40 ms |

**Table 2.** $SO_2$ calibration gas standard.

| Compounds | Concentration |
|---|---|
| $SO_2$ | 0.1% |
| $N_2$ | Balance |

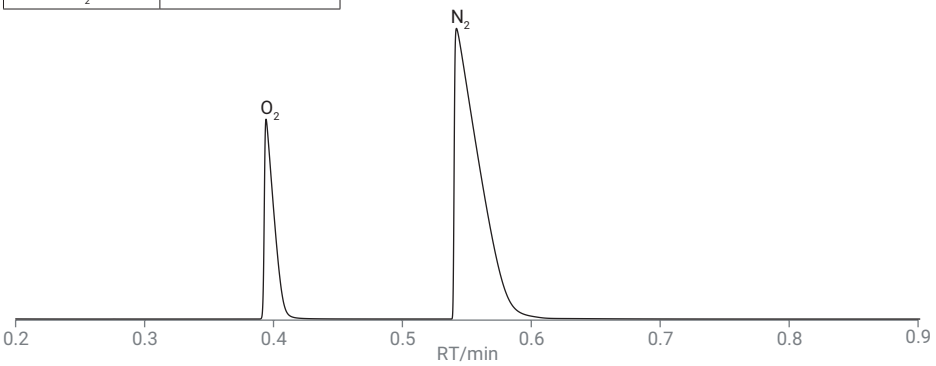


**Figure 1.** $O_2$ analysis on the Agilent CP-Molesieve 5 Å channel.

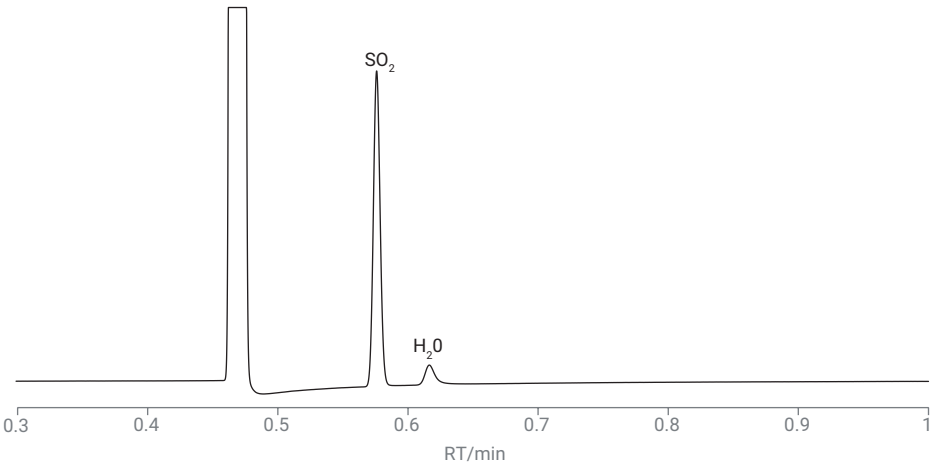


**Figure 2.** $SO_2$ and $H_2O$ analysis on the Agilent CP-Sil 19CB channel.

The instrument repeatability was evaluated by 10 injections of calibration standard (1,000 ppm $SO_2$ and lab air). Table 3 shows the RT and area RSD for $O_2$ and $SO_2$. The RT RSD% is less than 0.1% and the area RSD% is lower than 1%.

## Conclusion

This study demonstrates the application of the Agilent 990 Micro GC for $SO_2$ and $O_2$ analysis, which can be used for performance evaluation of the catalyst or the process control in sulfuric acid manufacturing. With the specially selected column channel, Agilent CP-Sil 19CB, 0.1% $SO_2$ and $H_2O$ can be resolved with resolution greater than 3. Oxygen is analyzed on a molecular sieve channel with backflush option. The quantitation precision was evaluated by 10 consecutive analyses of calibration standard and lab air with RT repeatability less than 0.1% and area repeatability less than 1%, demonstrating excellent instrument performance for reliable qualification and quantitation of $SO_2$ and $O_2$.

**Table 3.** RT and area repeatability of $SO_2$ and $O_2$ on two analytical channels.

| | $SO_2$ | | $O_2$ | |
|---|---|---|---|---|
| | RT (min) | Area (mv × s) | RT (min) | Area (mv × s) |
| | 0.576 | 1.689 | 0.395 | 74.520 |
| | 0.576 | 1.704 | 0.395 | 74.622 |
| | 0.575 | 1.700 | 0.395 | 74.598 |
| | 0.575 | 1.721 | 0.395 | 74.616 |
| | 0.575 | 1.697 | 0.395 | 74.596 |
| | 0.575 | 1.694 | 0.395 | 74.608 |
| | 0.576 | 1.669 | 0.395 | 74.592 |
| | 0.576 | 1.684 | 0.395 | 74.568 |
| | 0.576 | 1.680 | 0.395 | 74.568 |
| | 0.575 | 1.716 | 0.395 | 74.617 |
| Average | 0.576 | 1.695 | 0.395 | 74.592 |
| RSD% | 0.09 | 0.93 | 0.002 | 0.041 |

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