

AGILENT GAS CHROMATOGRAPHY AND SULFUR-SELECTIVE DETECTION

ANALYSIS OF SULFUR COMPOUNDS ACCORDING TO ASTM D5504



The presence of sulfur in natural gas and gaseous fuels can be corrosive, poisonous, and odorous. There are many ways to measure sulfur, with each technique offering its own strengths. The Agilent 8355 Sulfur Chemiluminescence Detector has been designed to fulfill and exceed all testing requirements, specifically in the areas of:

- Linear response
- Non quenching performance
- LOD/LOQ
- Ease-of-use
- Uptime readiness

Gas chromatography with sulfur chemiluminescence detection provides a rapid means of identifying and quantifying sulfur impurities or sulfur based odorants in natural gas and gaseous fuels. Examples include sulfur compounds in air, methane, propane, digester gas, and refinery fuel gases.

With the increased use of lower grade ingredients, and tighter global regulation on pollution, sulfur testing is gaining more importance for both up and downstream activities. As with any process, there are significant throughput and profitability impacts if the process is not controlled properly. One of the most reliable detection methods for a variety of sulfur compounds is by chemiluminescence (light-producing reaction). A sulfur chemiluminescence detector (SCD) is comprised of three main components:

- Dual plasma
- Burner, Reaction cell
- Detector

Each component needs to work dependently to demonstrate robustness, ease-of-use, and analytical performance.



When using an SCD to test gaseous fuels, it is imperative that the analyzer is capable of providing the high quality data required to optimize a process and ensure regulatory compliance.

Technology overview

The 8355 Sulfur Chemiluminescence Detector (SCD) is a sulfur-selective detector for gas chromatography. As the industry leader in SCD technology, Agilent reimagined how an SCD could be even better. Although there are numerous advances, the 8355 SCD epitomizes robustness and simplicity.

Operation of the SCD is based on the chemiluminescence from the reaction of ozone with sulfur monoxide (SO) produced from combustion of the analyte: Sulfur compound (analyte) \rightarrow SO + H₂O + other products SO + O₃ \rightarrow SO₂ + O₂ + h ν (< 300–400 nm).

Method parameters

GC conditions

Column: Agilent DB-Sulfur, 320 μ m \times 60 m, 4.2 μ m (G3903-63001)

Sample introduction: 2-Valve system

Carrier gas: Constant pressure at 14.5 psi

Oven: 30 °C (1.5 min)
then 15 °C/min to 250 °C (3 min)

SCD conditions

Base: 250 °C

Furnace: 800 °C

Air flow (oxidation): 60 mL/min

H₂ flow (oxidation/lower): 38 mL/min

H₂ flow (upper): 8 mL/min

O₂ (ozone gen): 40 mL/min

Table 1. Practical detection limits for Detected Analytes

No.	Analyte	Detection limit (pg/sec)
1	Hydrogen sulfide	0.096
2	Carbonyl sulfide	0.20
3	Methyl mercaptan	0.49
4	Ethyl mercaptan	1.04
5	Dimethyl sulfide	0.20
6	Carbon disulfide	0.094
7	2-Propanethiol	1.42
8	<i>tert</i> -Butyl mercaptan	1.62
9	1-Propanethiol	4.3*
10	Thiophene	0.21
11	<i>n</i> -Butyl mercaptan	0.22
12	Diethyl sulfide	6.0*
13	Methyl ethyl sulfide	0.30
14	2-Methyl-1-propanethiol	4.84
15	1-Methyl-1-propanethiol	9.43

* Values were calculated from the 7.5 ppm standard due to low responses.

Results and Discussion

When analyzing impurities or additives in natural gas or gaseous fuels, the ability to qualify as well as quantify can be equally important. We determined the limits of detection (LOD) under normal operating conditions on a standard test mix. The performance demonstrated impressive LOD on the key compounds of interest.

Sensitivity

Detection limits were calculated for the analytes from the 2.3 ppm standard (Table 1). Over half of the detected analytes yielded a detection limit of <0.5 pg/sec.

Conclusion

The Agilent 8355 Sulfur Chemiluminescence Detector is a sulfur-selective detector for gas chromatography. The 8355 SCD epitomizes robustness and simplicity. It is capable of providing the high quality data required to optimize a process, and ensure regulatory compliance.

These data represent typical results.

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Published in USA, January 27, 2016
5991-6552EN

