

SULFUR IN GASOLINE: NIST REFERENCE STANDARDS



Technology Advantage: Agilent Intuvo 9000 GC with SCD



Introduction

The petrochemical industry relies heavily on measuring sulfur throughout its various processes. Being able to monitor discrete sulfur-containing compounds is invaluable for process control, because these compounds are odorous and corrosive.

ASTM D5623 provides guidelines for the determination of sulfur-containing compounds in light petroleum¹. Total sulfur is often reported as estimated from the total area sum. NIST reference standards were used to demonstrate the sulfur-measuring capabilities of the Agilent Intuvo 9000 Gas Chromatograph equipped with an Agilent 8355 Sulfur Chemiluminescence Detector (SCD) and a 30 m Agilent Intuvo DB1 column.

The Intuvo 9000 GC provides a unique solution for sulfur analysis of light petroleum with additional advantages over conventional gas chromatographs:

- Smaller footprint
- Increased stability
- Easier maintenance

At only 27 cm, the Intuvo 9000 GC is approximately half the size of a conventional GC. A proprietary flow path and connection paradigm yields a more robust analysis, while enabling faster column changes for method development.



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Experimental

NIST standard reference material 2299 Sulfur in Gasoline, and NIST standard reference material 2298 Sulfur in Gasoline (High Octane) were used. An internal standard solution (ISTD) of diphenyl disulfide was spiked into the reference standards at 10 ppm for quality control. According to the reference material, the total sulfur in NIST 2299 is $13.6 \pm 1.5 \mu\text{g/g}$, and the total sulfur in NIST 2298 is $4.7 \pm 1.3 \mu\text{g/g}$.

Split injections (10:1) were performed, and the analysis was achieved with a 30 m Intuvo DB1 column following a standard oven program. The SCD was maintained with recommended temperatures and flow rates. Complete method details are available in a separate Application Note².

Results and Discussion

The NIST standard reference materials were used to provide a controlled sample for instrument/detector evaluation. Figure 1 shows chromatograms from NIST SRM 2299 (Figure 1A) and NIST SRM 2298 (Figure 1B). Total sulfur (averaged from five injections) for NIST 2299 was determined to be $14.4 \pm 0.6 \mu\text{g/g}$. Total sulfur (averaged from four injections) for NIST 2298 was determined to be $4.0 \pm 0.1 \mu\text{g/g}$. Both were found to be within the expected tolerances. Peak shape and resolution were excellent for these reference samples.

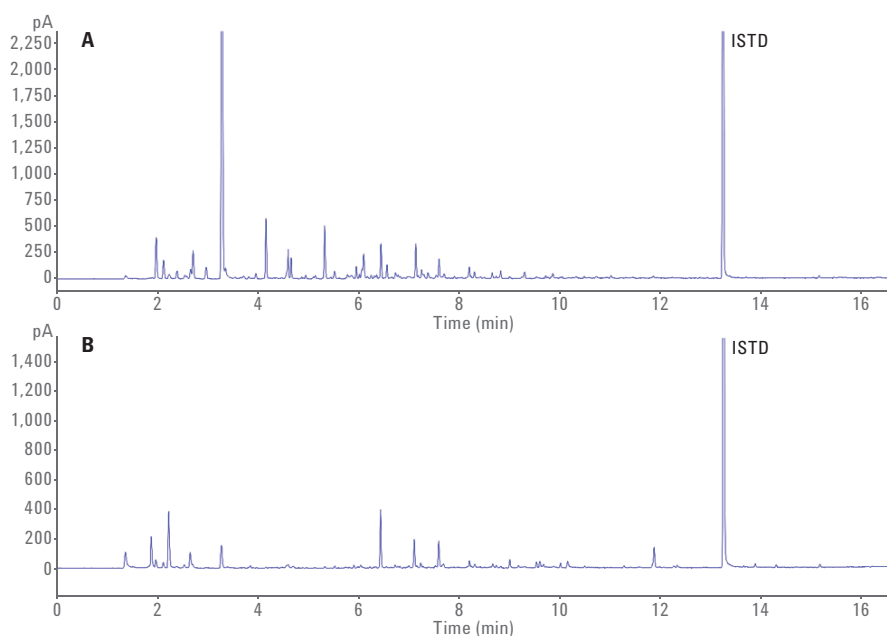


Figure 1. Chromatograms for NIST SRM 2299 (A) and 2298 (B). Diphenyl disulfide (13.25 minutes) was used as the ISTD.

Conclusion

The Agilent Intuvo 9000 GC, equipped with an Agilent 8355 SCD and a 30 m Agilent Intuvo DB1 column, yields equivalent performance to that of a conventional GC equipped with the same detector and column chemistry. The instrument was challenged with samples similar to that expected when following ASTM D5623, and was found to provide measurements within the specifications of NIST standard reference materials 2299 and 2298. This critical application from the petrochemical industry demonstrates the analytical capabilities of the Intuvo 9000 GC system.

References

1. ASTM 5623, Standard Test Method of Sulfur Compounds in Light Petroleum Liquids by Gas Chromatography and Sulfur Selective Detection.
2. Veeneman, R. Detection of Sulfur Compounds in Gasoline According to ASTM D5623 with Agilent's Intuvo GC and Dual Plasma Sulfur Chemiluminescence Detector, *Agilent Technologies Application Note*, publication number 5991-7215EN, **2016**.



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