



Application note:

Sulfur (MCT) in Refinery Products

Methods:

- ASTM D3120
- ASTM D3246

Residual sulfur in fuel oils will influence emissions of sulfur dioxide (SO₂), resulting in plant corrosion and atmospheric pollution if this level is too high. In Europe the maximum allowable level for sulfur in diesel has been set at 10 ppm.

Sulfur is tested in a variety of petroleum products after the hydro desulfurization process, because of the risk of catalyst poisoning.

SAMPLE INFORMATION

Sample Type	Oils & Fuels
Component	Sulfur
Matrix	Base oil, Methanol, Biofuel, Diesel
Concentration	1 – 10 mg S/L
Method Applicable	ASTM D3120 ASTM D3246

SUMMARY

The XPLOER-TS elemental combustion analyzer, equipped with Liquids Introduction Module, has been used to perform the analysis of sulfur in Refinery Products according the international norms ASTM D3120 and ASTM D3246. The various samples were introduced by the ARCHIE liquids autosampler into the XPLOER-TS. The achieved results show a RSD well below 3%.

RESULTS

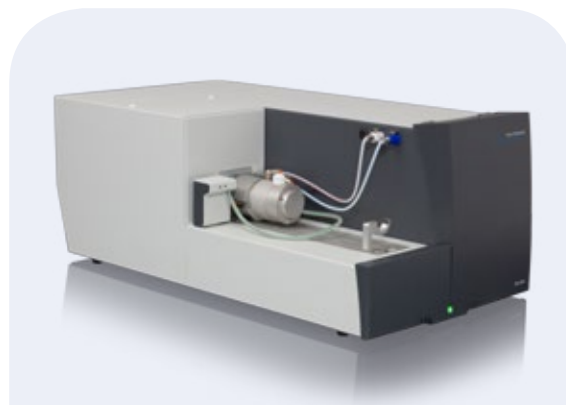
Sample	Conc. S (mg/L)	RSD (%) n=5
Standard 10 mg/L	9.53	0.18
Base oil	2.75	1.3
Methanol	1.72	0.89
Biofuel	1.47	2.7
Diesel ULSD	7.04	0.90

Detailed results can be found in the Appendix.

CONCLUSION

The XPLOER-TS equipped with Liquids Introduction Module is able to measure the amount of Total Sulfur in methanol, biofuel and diesel with an excellent repeatability (RSD), in reference to ASTM D3120 and ASTM D3246.

CONFIGURATION



XPLOER-TS



Collision Flow Tube



Liquids Introduction Module



ARCHIE liquids autosampler with conditioned sample tray

Trace Elemental Instruments





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METHODS

TE Instruments carried out an application to demonstrate that the XPLOER-TS is an excellent solution for the analysis of sulfur in Refinery Products. This elemental combustion analyzer fully complies with, but not limited to, the following international norms:

For Sulfur:

- ASTM D3120
- ASTM D3246
- ASTM D3961
- GBT 11061
- SHT 0253

The amount of sulfur in Refinery Products has been measured according to:

ASTM D3120 – “Standard Test Method for trace sulfur in light liquid petroleum hydrocarbons by oxidative microcoulometry.”

ASTM D3246 – “Standard Test Method for sulfur in petroleum gas by oxidative microcoulometry.”

SYSTEM DESCRIPTION

All samples were introduced automatically into the XPLOER’s heated Liquids Module by the ARCHIE liquids XYZ autosampler. The ARCHIE picks up sample from the assigned vial position and delivers it into the heated Liquids Module. In between analysis of samples, standards, and blanks, the syringe and needle are cleaned in a special dual wash & drain station tray to avoid cross contamination. The Liquids Module is specifically designed for the introduction of liquid samples with a final boiling point up to 420 °C. The proprietary easy-to-use TEIS software controls sample introduction from the sample queue, processes the detector signal and calculates the Total Sulfur concentration.

The XPLOER-TS is fitted with a dual-zone furnace, which enhances combustion performances. The temperature is adjustable up to 1150 °C. The Collision Flow combustion tube has a secondary oxygen flow to assure the sample is fully combusted. Collision of the combusted gases creates a dynamic turbulence of the oxidizing gas stream and replaces some of the depleted oxygen. Resulting in more oxidation power for samples which are difficult to oxidize.

The combustion gas, SO₂ is led into a sulfuric acid scrubber for rapid water and interference removal. Particulates are blocked by a re-usable, cleanable filter. The clean and dry gas is led into

the temperature controlled titration cell which detects the amount of Total Sulfur.

Measuring sulfur, by use of Microcoulometry is an absolute technique. No calibration required. The sulfur dioxide (SO₂), carried by the combustion gas, reacts with the tri-iodine present in the titration cell. The amount of charge (the integral of the regeneration current over the measuring time) used to regenerate the lost tri-iodine, is directly related to the Total Sulfur content of the sample.

*System settings can be found in the Appendix.



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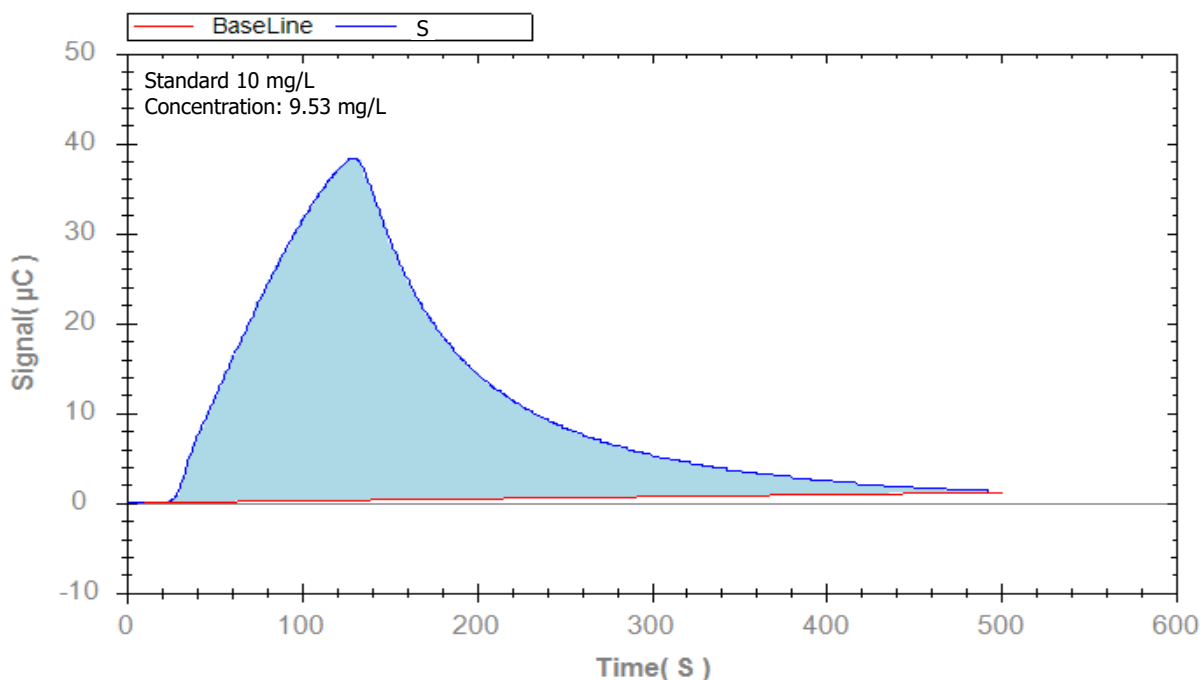
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APPENDIX A RESULTS

Sample	Concentration S (mg S/L)	SD (mg S/L)	RSD (%) n=5
Standard 10 mg/L	9.53	0.02	0.18
Base oil	2.75	3.6	1.3
Methanol	1.72	0.015	0.89
Biofuel	1.47	0.040	2.7
Diesel ULSD	7.04	0.063	0.90



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APPENDIX B SYSTEM SETTINGS

System settings	
Oxygen Injection	300 mL/min
Argon Injection	100 mL/min
Oxygen Collision Flow	100 mL/min
Oxygen Bypass	50 mL/min
Furnace Temperature I	1000 °C
Furnace Temperature II	1000 °C
Liquids Module	500 °C
Tracing	200 °C
Internal System Temperature	30 °C
Gain	Auto gain
Cell Cooling	15 °C
Sample Pick-up Speed	3 µL/s
Injection Speed	1 µL/s
Injection Volume	100 µL

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