Analysis of Sulfur (UV-F) in Fatty Acids, Animal Fats and Vegetable Fats by UV-Fluorescence

Application note:

Sulfur (UV-F) in Fatty Acids, Animal Fats and Vegetable Fats

Trace Elemental Instruments

The amount of sulfur in fatty acids, animal fats and vegetable fats is measured because some of these materials, for example methylester, are used to produce biodiesel. Processors of biodiesel need to monitor and control the Total Sulfur content in the feedstock.

SAMPLE INFORMATION

Sample Type	Fats
Component	Sulfur
Matrix	Fatty Acids, Animal Fats, Vegetable Fats
Concentration	0.1 – 100 mg S/L
Method Applicable	-

SUMMARY

The XPLORER-S elemental combustion analyzer, equipped with boat introduction module, has been used to perform the analysis of Sulfur in all kind of fat samples like fatty acids, animal fats, vegetable fats and palm oil. The various samples were introduced by the ARCHIE liquids autosampler equipped with heated sample tray into the XPLORER-S. All samples have been analyzed with a RSD well below 5%.

RESULTS

Sample	Concentration S (mg/L)	RSD (%) n=5
Methylester	0.21	4.95
Methylester blend	0.08	4.47
Mascol Lauryl Myristyl Alcohol	0.11	2.59
Crude Palm Oil	2.26	3.48
Animal Fat 1	1.19	3.72
Animal Fat 2	10.27	1.00
Fat A	6.61	1.40
Fatty Acid B	0.54	0.66

Detailed results can be found in the Appendix.

CONCLUSION

The XPLORER-S equipped with boat introduction module is able to measure the total amount of sulfur in all kind of fat samples like fatty acids, animal fats, vegetable fats and palm oil with excellent repeatability (RSD). The heated sample tray and specially developed boat program are key to proper sample introduction and a complete combustion.

CONFIGURATION

XPLORER-S with boat introduction module

Collision flow tube with boat cooling

ARCHIE liquids autosampler with conditioned sample tray

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METHODS

TE Instruments carried out an application to demonstrate that the XPLORER-S is an excellent solution for the analysis of Total Sulfur content in fatty acids, animal fats and vegetable fats. This elemental combustion analyzer fully complies with, but is not limited to, the following international norms:

For Sulfur:

- ASTM D5453
- ASTM D6667
- ASTM D7183
- SH/T 0689

SYSTEM DESCRIPTION

All samples were introduced automatically into the Boat Module of the XPLORER-S by the ARCHIE liquids XYZ autosampler. The ARCHIE picks up sample from the assigned vial position and delivers it into the boat 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 proprietary easy-to-use TEIS software controls sample introduction from the sample queue, processes the detector signal and calculates the Total Sulfur concentration from the calibration curves.

The heated sample tray (50 °C) of the ARCHIE assure that the fat samples can be aspirated and injected as a liquid. The boat module is utilized for samples that are difficult to oxidize or with a final boiling point higher than 420 °C. A custom made boat program facilitates a complete and controlled combustion of these high viscous samples.

The XPLORER-S is fitted with a dual-zone furnace, which enhances combustion performances. The temperature is adjustable up to 1150 °C. The Collision Flow combustion tube with boat cooling has a secondary oxygen flow to assures 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 improved cooling mechanism for boat introduction makes it unique. It suits all industrial applications; the most all-round combustion tube available in today's market.

Since moisture can affect the operation of the detector(s) a scrubber is placed between the furnace and the detectors. The PermaPure scrubber removes water vapor from the gas stream. Particles are blocked by a re-usable, cleanable filter, no longer need for expensive disposable PFTE fiber filters. Therefore only dry and clean gas will enter the sulfur reaction chamber.

After conditioning, the amount of sulfur is detected by pulsed UV-fluorescence. Sulfur dioxide (SO_2) is formed during the oxidation and transferred to the reaction chamber. The UV light excites the sulfur dioxide which in its turn emits a certain amount of UV light detected in a photomultiplier tube. The amount of light emitted reflects the total amount of SO_2 present in the gas, which corresponds to the total amount of sulfur in the sample.

*System settings & Boat program can be found in the Appendix.

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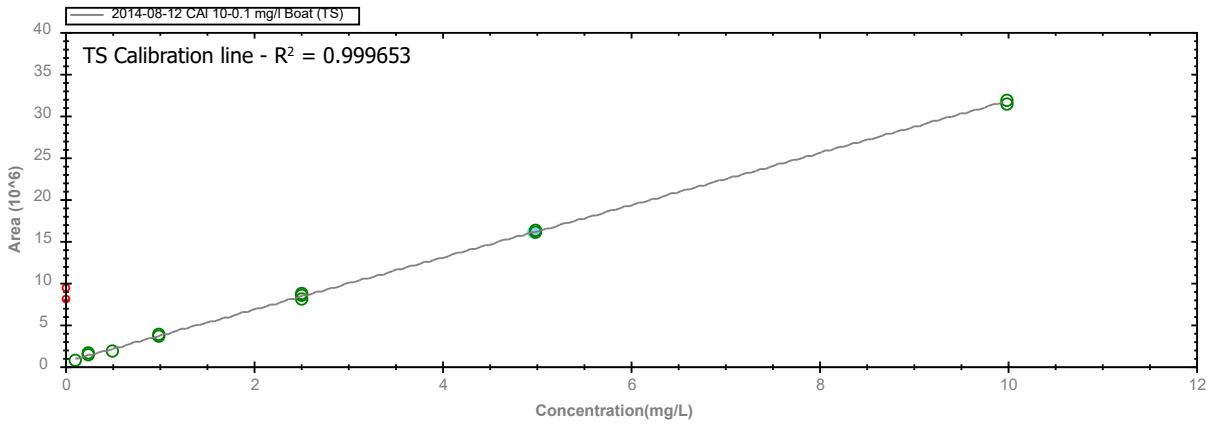
APPENDIX A CALIBRATION

The standards used for the calibration are made out of Xylene and prepared by the ARCHIE liquids auto sampler. The XPLORER-S is calibrated in two ranges, from 0.1 - 10 mg/L and from 1 - 100 mg/L.

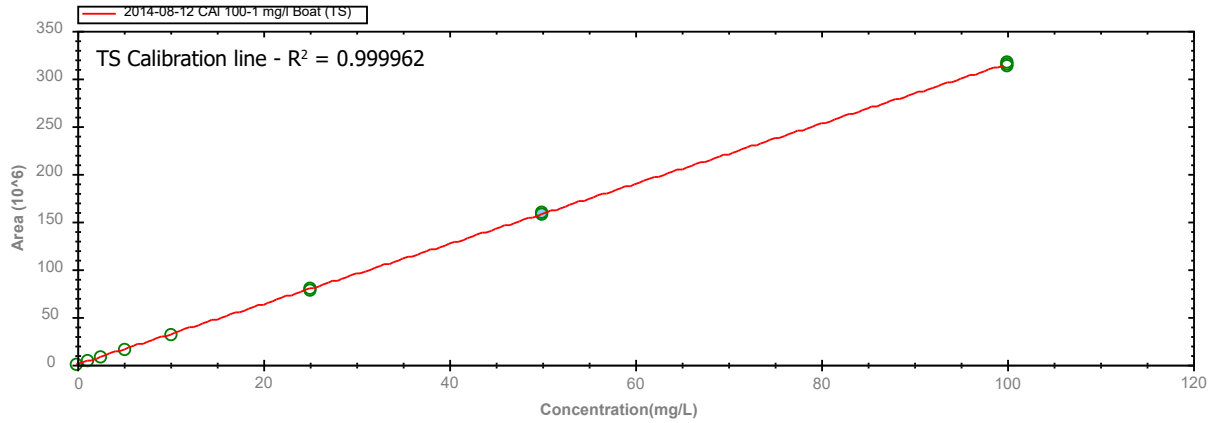
RANGE 0.1 - 10 mg/L

Concentration S (mg/L)	Average Area TS n=5
0.1	790,157
0.25	1,490,823
0.5	1,800,596
1	3,655,027
2.5	8,390,155
5	16,191,577
10	31,698,655



RANGE 0 - 100 mg/L

Concentration S (mg/L)	Average Area TS n=5
0	369,035
1	3,983,581
2.5	8,674,778
5	16,258,135
10	31,683,660
25	78,904,056
50	158,442,807
100	315,365,562



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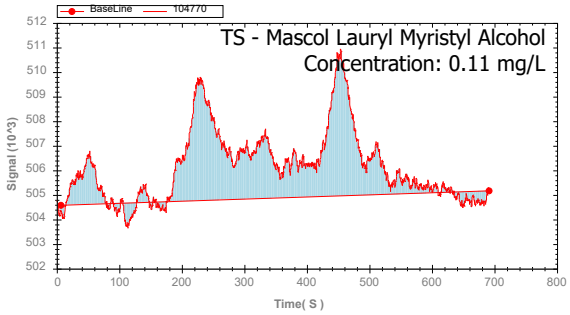
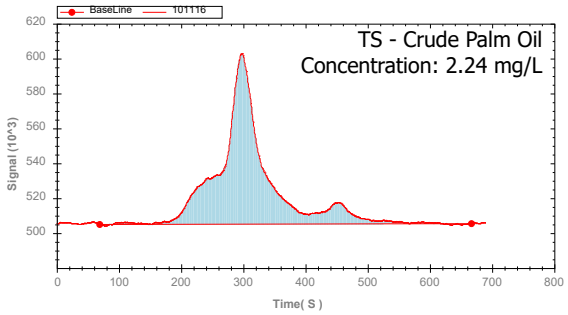
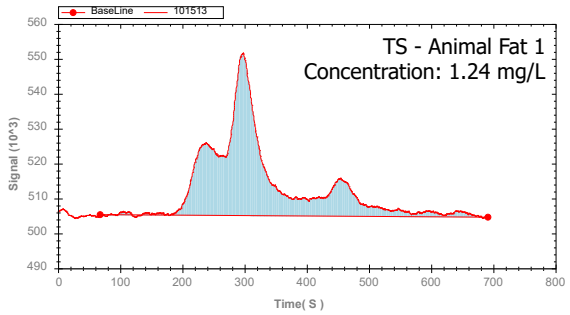
APPENDIX B EXAMPLE PEAKS







APPENDIX C BOAT PROGRAM

Position (mm)	Speed (mm/s)	Pause (s)
0	5	0
75	5	30
95	5	30
100	5	60
120	5	45
150	5	30
200	5	60
0	10	240

APPENDIX D SYSTEM SETTINGS

System settings	
Oxygen Injection	300 mL/min
Argon Injection	100 mL/min
Oxygen Collision Flow	100 mL/min
Furnace Temperature I	1000 °C
Furnace Temperature II	1000 °C
Internal System Temperature	30 °C
Sample Pick-up Speed	3 µL/s
Injection Speed	5 µL/s
Injection Volume	30 µL

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