[APPLICATION NOTE – 31 v2]

X-MET8000

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ELEMENT	% ↑
S	0.076

X-MET8000 for fast on-site sulfur in marine fuel analysis to ISO8754, ASTM D4294 international test methods

BACKGROUND

The International Maritime Organisation (IMO) has been working for many years to reduce the harmful impact of shipping to the environment. As part of ongoing efforts to reduce air pollution from the burning of marine fuels, the IMO recently announced that the proposed 0.5% global sulfur cap on marine fuels will come into effect on 1st January 2020. This is a significant reduction from the current maximum sulfur concentration of 3.50%. In addition within sulfur emission control areas (SECA's), the sulfur limit remains at the 0.1% level established in 2015.

To ensure compliance with global marine fuel legislation, ship owners must ensure they are burning the correct specification fuel for all geographical locations.

In addition, port authorities perform on-the spot checks on ships in SECA area to ensure compliance. In this case the analytical equipment used for the test needs to be fully portable.

KEY BENEFITS

- Rapid, easy to use on-site sulfur analysis
- Portable system for ship to ship measurements
- Conformance to ISO and ASTM test methods
- Rugged, field proven analyser
- Automatic correction for temperature and pressure ensures correct results
- Embedded GPS to pinpoint where analysis is performed
- Real time data sharing and management via ExTOPE Connect cloud service
- 45 years experience in portable X-ray fluorescence (XRF) analysis

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ANALYTICAL SOLUTION

Unlike other techniques available (e.g. titration), X-ray fluorescence (XRF) is rapid, simple, and does not require the use of chemicals. It is a field proven technique that is widely used in the oil industry. With result available in seconds, it is a non-destructive technique and can be used by any operator with minimal training.

Our field portable XRF analyser, the X-MET8000, combines a high performance X-ray tube and state of the art silicon drift detector delivering the speed and performance needed for the routine determination of sulfur in marine fuels.

Robust (IP54 rated to ensure minimal ingress of dust and splash water; tested to MIL-STD810G for ruggedness) and compact, the X-MET8000 is versatile and can be used on board a ship, in a laboratory, or taken wherever sulfur analysis needs to be performed.

SAMPLE PREPARATION AND MEASUREMENT

Sample preparation is simple and follows the same MARPOL* procedure as for laboratory sulfur analysis (Annex VI).

For a homogenous** fuel oil sample, simply pour (approx. three quarters full) the fuel oil into a sample cup which is fitted with a thin polyester film (3,5um Mylar). Note: No weighing or sample volume measurement is required.

Place the assembled sample cup into the safety window (also fitted with thin film), which minimises the risk of sample leakage onto the analyser avoiding potentially costly repairs.

The cup plus safety window are placed either:

- into the light stand and safety shield (which fit into the X-MET's compact transport case)
- or benchtop stand. Note: When not in use, the stand can be folded up and safely put away, requiring minimum storage space

*MARPOL- : International Convention for the prevention of pollution from ships

**Homogenous - The measured sample needs to be homogenous for a valid result, i.e. the fuel oil should not contain any particles, be of a single phase, and may require heating to pour the sample into the sample cup. In addition, taking two sub-samples and comparing the results will give a good indication of sample homogeneity.

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SAMPLE PREPARATION AND MEASUREMENT (Cont…)

Pressing the X-MET8000 trigger starts the analysis, and initial results are available in seconds, updated until the end of the analysis. A typical analysis time is 60 seconds. Note: The MARPOL procedure specifies that a second sub sample should be prepared and measured in the same way,

CALIBRATION

The X-MET8000 “Sulfur application package” comes pre-loaded with optimised calibration parameters covering the concentration range of 0 to 5% sulfur. For optimum accuracy the calibration is split into two ranges, i.e. 0 - 0.75 % sulfur and 0.75 - 5 % sulfur.

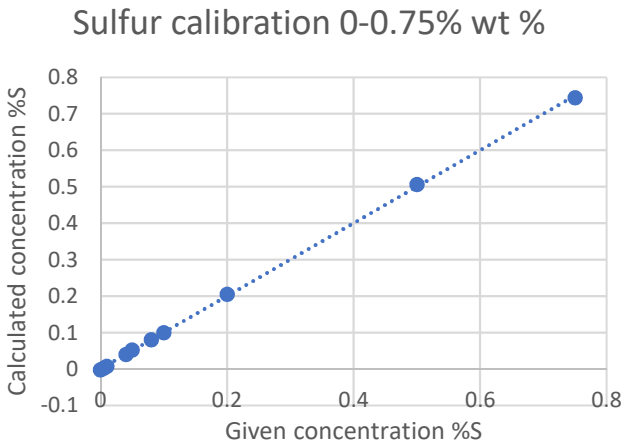
Following good laboratory practise and the procedure specified in the standard test methods ISO8754 and ASTM D4294, a series of certified sulfur in mineral oil calibration standards were measured. These standards evenly spanned the concentration range of interest and each standard was measured using an analysis time of 60 seconds.

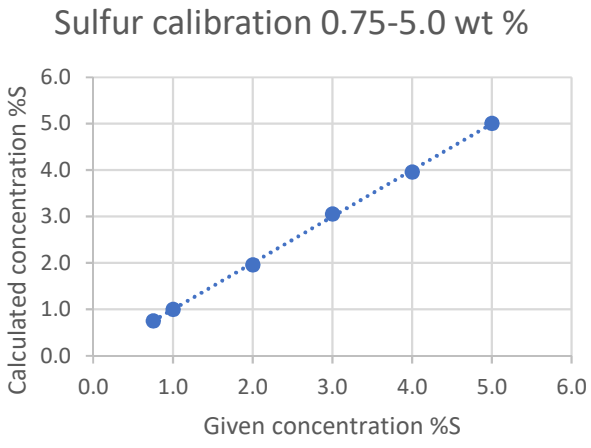
Note: The calibration standards are measured in “air path” and the X-MET8000 is fitted with automatic temperature and pressure compensation to automatically correct for any variation. Without this compensation, changes in air density (pressure or temperature) caused by atmospheric changes or for example the measurement of hot fuel oil samples could affect the results.

Full portability with the light stand.

Sample cups measured in the benchtop stand.

Benchtop stand, folded for transport and storage.





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CALIBRATION (Cont…)

	Concentration (%S)	Analysis time (seconds)	Standard Error of calibration (%S)	Precision (95% confidence) %S
Low range	0 - 0.75	60	0.004	0.002 @ 0.1%S
				0.004 @ 0.5%S
High range	0.75 - 5.00	60	0.046	< 0.02 @ 2.0 %S

Table 1: Typical calibration performance for sulfur in oil

The Standard Error of the calibration is an indication of accuracy of analysis, i.e. for measurements of fuel oil samples that are prepared and measured in the same way as calibration standards, and of the same oil matrix type, a typical accuracy of analysis will be plus or minus 2 x the standard error of calibration.

Instrument repeatability

To demonstrate instrument repeatability three fuel oil samples of known and different sulfur concentration were each measured 10 times. The results are shown in table 2 demonstrating excellent X-MET8000 instrument stability

	Sample 1 (%S)	Sample 2 (%S)	Sample 3 (%S)
Given concentration	0.08	0.5	3.00
X-MET result 1	0.082	0.495	3.01
X-MET result 2	0.083	0.505	3.01
X-MET result 3	0.082	0.506	3.01
X-MET result 4	0.083	0.507	3.01
X-MET result 5	0.083	0.508	3.01
X-MET result 6	0.084	0.508	3.01
X-MET result 7	0.082	0.507	3.01
X-MET result 8	0.083	0.507	3.00
X-MET result 9	0.083	0.507	3.00
X-MET result 10	0.081	0.507	3.02
Average (%S)	0.083	0.506	3.00
Standard deviation. (%S)	0.001	0.004	<0.01

Table 2: Repeatability measurements on the X-MET8000

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Validation results

To validate the calibration, a series of marine fuel oils with known sulfur concentrations, and from different geographical locations were measured.

The proposed MARPOL procedure of measuring two sub samples was used and the individual values and average sulfur concentration values measured on the X-MET are shown in Table 3.

The results demonstrates good agreement between the given concentration and the measured concentration on the X-MET8000 for a wide range of fuel oils, enabling rapid, simple on site analysis and screening.

Sample location	Laboratory Sulfur concentration %S	X-MET 1st sub sample %S	X-MET 2nd sub sample %S	X-MET average %S value (Result reported to 2 decimal places as MARPOL procedure)
Russia	0.012	0.011	0.013	0.01
Estonia	0.092	0.096	0.095	0.10
Russia	0.091	0.089	0.091	0.09
USA	0.006	0.003	0.004	0.00
Russia	0.097	0.09	0.087	0.09
Germany	0.093	0.095	0.095	0.09
Malta	0.083	0.091	0.091	0.09
Bahamas	0.08	0.088	0.087	0.09
South Korea	0.039	0.037	0.038	0.04
UK	0.088	0.089	0.091	0.09
Puerto Rico	<0.03	0.001	0.001	0.00
Gibraltar	0.097	0.103	0.102	0.10
USA	0.083	0.086	0.087	0.09

Table 3: Validation data.

Note (i): All results are within the Reproducibility* (R) value of the ISO 8754 test method, i.e. at 0.1% Sulfur R=0.016 %S.

(ii): All samples were well mixed and in some cases heated to ensure a homogenous sample.

*Reproducibility (R) : The difference between two single and independent test results obtained by different operators working in different laboratories.

Validation results (cont..)

Global fuel oils at the new 0.5% sulfur concentration

Sample location	Laboratory Sulfur concentration %S	X-MET 1st sub sample %S	X-MET 2nd sub sample %S	X-MET average %S value (Result reported to 2 decimal places as MARPOL procedure)
Gibraltar	0.472	0.472	0.472	0.47
USA	0.506	0.500	0.501	0.50
Spain	0.458	0.429	0.451	0.44
Russia	0.477	0.467	0.467	0.47
Angola	0.506	0.480	0.470	0.48
Brazil	0.524	0.514	0.516	0.52
Italy	0.496	0.487	0.483	0.48
Russia	0.452	0.440	0.454	0.45
Brazil	0.470	0.444	0.453	0.45
Belgium	0.476	0.471	0.475	0.47
Gibraltar	0.504	0.491	0.492	0.49
Singapore	0.450	0.436	0.418	0.43
UAE	0.459	0.458	0.452	0.45
Brazil	0.474	0.473	0.473	0.47
Argentina	0.570	0.552	0.552	0.55

Table 4: Validation data.

Note (i): All results are within the reproducibility (R) value of the ISO 8754 test method, i.e. at 0.5% sulfur R = 0.04 %S

(ii): All samples were well mixed and in some cases heated to ensure a homogenous sample.

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SUMMARY

Once calibrated (a procedure that can be carried out by the user or the analyser can be pre-calibrated on request), Hitachi-High Tech's X-MET8000 provides accurate and repeatable sulfur analysis in marine fuel oil. The X-MET8000's ease of use, ruggedness and portability makes it an ideal tool for sulfur analysis on ships, at the ports, or in the laboratory.

ORDERING INFORMATION, MINIMUM REQUIRED:

- X-MET8000 Optimum Sulfur in Oil Package (P/No 10009712)

Factory calibrated for Sulfur in oil (0-5%S) and Alloy FP applications.

Includes the compact and rugged carrying case, a wrist strap and lanyard, 2 Li-ion batteries, a battery charger, a USB cable to connect to a PC/laptop, 5 replacement thin film windows, Bluetooth and Wifi connectivity, and the user manuals. Also includes, a small sample tray, light stand and safety shield, sample cups, safety windows and film, setting up samples (SUS) and check samples.

- Or X-MET8000 Expert Customised Package

Accessories and calibrations individually ordered.

X-MET8000 in transport case

OPTIONAL EXTRAS:

- Benchtop stand (P/No 10001042)
- Rack for sample cells (P/No 10003439, holds 10 cups)
- Single open ended sample cups (P/No 10000941 pack of 100)
- Portable Bluetooth printer (Part/No 10006550)
- Factory calibration (P/No10009262); for Customised package - will be calibrated as per this application note

Hitachi High-Tech Analytical Science

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