

LAB-X5000 for analysis of sulfur in fuels and petroleum products



BACKGROUND

There is a continuing and increasing demand for a rapid, simple and cost-effective measurement of sulfur in a wide range of petroleum products including automotive, marine and aviation fuels. Benchtop energy dispersive X-ray fluorescence (EDXRF) analysers such as the LAB-X5000 can be found in many refinery laboratories and petrochemical test laboratories, with the instruments often operated by production staff on a 24/7 basis, providing accurate results conforming to globally accepted test norms. EDXRF is well regarded for its excellent performance, ease of use, versatility, speed and cost effectiveness. Within the oil supply system from crude oil to refinery output, distribution, storage and usage, quality control and assurance is required at almost every stage of the process.

Air and water quality are becoming increasingly important concerns for the shipping and boating industries. As part of ongoing efforts to reduce accumulating pollution released from burning fossil fuels, the International Maritime Organization (IMO) recently announced that the Maritime Protection Committee (MEPC) agreed to implement the proposed 0.50% global sulfur cap on marine fuels on January 1st, 2020. This is a significant reduction from the current limit of 3.50% which has been in place since 2012. Within sulfur emission control areas (SECA), the sulfur limit remains at the 0.10% level established in 2015. Beginning January 1, 2018 companies will be required

to monitor emissions from impacted ships. Sulfur content in fuels reduces their heating values and increases their CO₂-to-heat contents. Taking on fuel with lower sulfur content and verifying its concentration can be part of an emissions reduction program. On-board sulfur testing is a simple, reliable and affordable way to ensure compliance to maritime emission regulations and avoid delays, fines, sanctions, vessel forfeiture or other penalties.

The LAB-X5000 can measure sulfur from the ppm range to high percent, covering all analytical requirements in a refinery laboratory, on a ship or in a testing laboratory.

CONFORM TO ASTM AND ISO METHODS AND STANDARDS

Analysis using the LAB-X5000 conforms to test methods and standards including ASTM D4294, ISO 8754, ISO 20847 and IP 336. Calibrations can be created by the user directly on the instrument or pre-loaded in the factory following stringent quality and performance criteria. Calibration maintenance is performed using the provided setting up samples (SUSs), which can also be used for tracking calibration stability.

| Concentration range | Unit | Standard error of calibration | Measurement time (s) | Limit of detection (3 σ) | Limit of quantification |
|---------------------|-------|-------------------------------|----------------------|----------------------------------|-------------------------|
| 0 - 150 | mg/kg | 3 | 300 | 4 | 12 |
| 0 - 1000 | mg/kg | 6 | 300 | 4 | 12 |
| 0.1 - 5 | % m/m | 0.02 | 50 | n/a | n/a |

Table 1. Typical calibration performance for sulfur in oils and fuels. Calibration was created using mineral oil standards.

| Analysis time (seconds) | Given sulfur concentration | Precision (2 σ) at given concentration | ASTM D4294-08 repeatability | ISO 20847-04 repeatability (for diesel fuels) |
|-------------------------|----------------------------|--|-----------------------------|---|
| 300 | 10 mg/kg | 2.7 mg/kg | Outside scope | Outside scope |
| | 50 mg/kg | 2.2 mg/kg | 5.4 mg/kg | 9 mg/kg |
| | 100 mg/kg | 2.9 mg/kg | 8.5 mg/kg | 10 mg/kg |
| | 500 mg/kg | 5.3 mg/kg | 24 mg/kg | 17 mg/kg |
| | 1000 mg/kg | 7.2 mg/kg | 37 mg/kg | Outside scope |
| 50 | 0.5 % m/m | 0.003 % m/m | 0.011 % m/m | Outside scope |
| | 1.0 % m/m | 0.005 % m/m | 0.016 % m/m | Outside scope |
| | 3.0 % m/m | 0.006 % m/m | 0.033 % m/m | Outside scope |
| | 5.0 % m/m | 0.014 % m/m | 0.150 % m/m | Outside scope |

Table 2. Typical repeatability data for sulfur calibrations.

| Concentration range | Unit | Standard error of calibration | Measurement time (s) | Limit of detection (3 σ) | Limit of quantification |
|---------------------|-------|-------------------------------|----------------------|----------------------------------|-------------------------|
| S: 0 - 1 | % m/m | <0.01 | 120 | 4 | 12 |
| Cl: 0 - 1 | % m/m | 0.02 | 120 | 4 | 12 |

Table 3. Typical calibration performance for sulfur and chlorine in oils and fuels. Calibration was created using mineral oil standards.

| Analysis time (seconds) | Given sulfur concentration (% m/m) | Precision (2 σ) at given concentration |
|-------------------------|------------------------------------|--|
| 120 | 0.100 | 0.001 |
| | 0.300 | 0.001 |
| | 0.400 | 0.003 |
| | 0.600 | 0.003 |
| | 0.800 | 0.003 |
| | 1.000 | 0.004 |

Table 4. Typical sulfur repeatability data for calibrations containing sulfur and chlorine.

| Analysis time (seconds) | Given chlorine concentration (% m/m) | Precision (2 σ) at given concentration (% m/m) | EPA 9075-97 repeatability (% m/m) |
|-------------------------|--------------------------------------|--|-----------------------------------|
| 120 | 0.000 | 0.000 | 0.000 |
| | 0.100 | 0.001 | 0.018 |
| | 0.500 | 0.002 | 0.040 |
| | 0.600 | 0.002 | 0.044 |
| | 0.800 | 0.003 | 0.051 |
| | 1.000 | 0.003 | 0.057 |

Table 5. Typical chlorine repeatability data for calibrations containing sulfur and chlorine.

OILS AND FUELS ANALYSIS MADE EASY

The Hitachi LAB-X5000 energy dispersive X-ray fluorescence (EDXRF) analyser makes sulfur analysis easy. This rugged, compact analyser is designed to provide reliable and reproducible results in laboratories, production environments and mobile inspection operations. The intuitive interface is displayed on a large, industrial touch screen. Streamlined software and one-touch measurement start function are inspired by our line of point-and-shoot handheld analysers so any operator can get high quality results. Built-in atmospheric compensation allows analysis without the need for helium or vacuum purge, minimizing the cost per analysis. The analytical method parameters have been optimised by applications engineers and include automatic compensation for changes in sample density due to varying carbon/hydrogen (C/H) ratios, enabling the measurement of multiple types of oils and fuels with a single calibration, simplifying analyser setup and operation.

The LAB-X5000 includes several features that help protect against damage caused by sample spills. Sample cups fit inside a secondary safety window that contains leaks from the cup. These windows

are re-usable and removable and do not require tools for assembly. The sample is inserted into an automatic turntable that positions it for analysis then moves the sample away from the X-ray tube and detector when the measurement is complete. While the risk of a leak escaping both the sample cup and secondary containment is small, should it occur it would happen away from the analytical components. To remind users that a sample should be removed after a measurement, an audible alert is generated when the analysis is complete.

Advanced data handling capabilities include connectivity to LiveConnect, a cloud-based service for storing and managing analysis data anytime, anywhere. Locally, 100,000 results and spectra are stored on-board the analyser. Results can be printed on the integrated printer and transferred via USB.

The LAB-X brand has been trusted by the petroleum industry for decades for its reliability, ease of use, stability and ruggedness.

Visit www.hitachi-hightech.com/hha for more information.

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