APPLICATION NOTE - 01

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# LAB-X5000


The LAB-X5000 benchtop analyzers present many advantages:

- Compact and robust: ideally suited in a busy lab
- Easy to use: reliable results for all operators with minimum training
- Sample preparation to results in minutes: fast decision making for consistent product quality
- Conforms to international standard test methods

# LAB-X5000 for the rapid determination of sulfur in fuels and petroleum products

## INTRODUCTION

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) analyzers such as Hitachi's LAB-X5000 can be found in many refinery laboratories and petrochemical testing laboratories, with the instruments often operated by production staff on a 24/7 basis, providing accurate results that conform 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 are required at almost every stage of the process.

The LAB-X5000 can measure sulfur from the parts per million (ppm) range to high percent, covering all analytical requirements in a refinery laboratory, on a ship, or in a testing laboratory, including a mobile one.

## CONFORM TO ASTM, ISO, IP, JIS, GB, EPA METHODS AND STANDARDS

Analysis with the LAB-X5000 conforms to sulfur test methods and standards including ASTM D4294, ISO 8754, ISO 20847, IP 336, IP 496, JIS K 2541-4 and GB/T 17040.

For the determination of total chlorine in new and used oils, fuels, and related materials, the LAB-X conforms to EPA Method 9075. With this test, operators can quickly check if the oil is hazardous/non-hazardous, and if it can be recycled and

used as fuel, lubricant, or combustible for heating and power generation.

## OILS AND FUELS ANALYSIS MADE EASY

The Hitachi LAB-X5000 benchtop EDXRF analyzer makes fuels analysis easy. This rugged, compact analyzer is designed to provide reliable and reproducible results in laboratories, production environments and mobile inspection operations. The intuitive user interface is displayed on a large, industrial touchscreen, and the streamlined software ensures that any operator can get high quality results. Built-in atmospheric compensation removes the need for helium or vacuum purge, minimizing the cost per analysis while delivering repeatable results. The analytical method parameters have been optimized 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 analyzer setup and operation.

Calibrations can be created by the user directly on the instrument or done in our 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.

The LAB-X includes several features that help protect against potential damage caused by sample spills or leaks, minimizing downtime and preventing costly repairs: sample cups fit inside a secondary safety window that will contain accidental leaks from the cup. These windows are easy to replace 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 window is small, should it occur it would happen away from the analytical components. Finally, to remind users that a sample should be removed after a measurement, an audible alert is generated when the analysis is complete.

With up to 100,000 results stored on the analyzer itself, operators can view new and old results on the built-in screen, print them on the optional integrated printer for a hard-copy record, download them on a USB memory device as a CSV file, and upload them to our ExTOPE Connect cloud service or a local server via Wi-Fi for real-time data access anytime, anywhere.

## SAMPLE PREPARATION

The sample preparation is very simple and only takes a few seconds: operators pour the oil to be tested into a sample cup fitted with Poly-M film, place the cup in a safety window (also fitted with Poly-M film) in the LAB-X5000's analysis port, and press the Start button. Preliminary results are displayed within seconds on the analyzer's screen and updated until the end of the analysis.

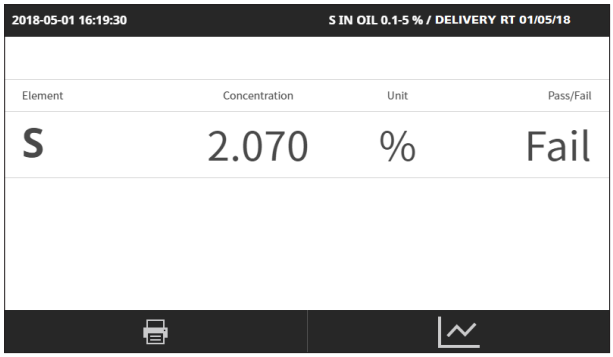
## PERFORMANCE AND RESULTS

Tables 1 to 4 show the typical performance the LAB-X delivers for the determination of sulfur and chlorine in petroleum products.

The limits of detection (LOD) are calculated from the results of 10 repeat measurements of a base mineral oil sample and the precision from 10 repeats of a sample containing the elements of interest.

Sample introduction

Starting the analysis




Results screen

Table 1: Typical calibration performance for sulfur in oils and fuels. Calibrations were created using mineral oil standards.

| Concentration range | Concentration unit | Standard error of calibration | Guaranteed limit of detection (3σ) | Limit of quantification (10σ) | Precision (95% confidence) | Measurement time |
|---|---|---|---|---|---|---|
| 0 - 150 | mg/kg | 3 | 6 | 12 | 2.2 at 50 | 300 seconds |
| 0 - 1000 | mg/kg | 6 | 6 | 12 | 5.3 at 500 | 300 seconds |
| 0.1 - 5.0 | % m/m | 0.02 | n/a | n/a | 0.005 at 1 | 50 seconds |

Table 2: Typical repeatability data for sulfur determination

| Given sulfur concentration | Concentration unit | Precision (95% confidence) at given concentration | ASTM D4294 repeatability | ISO 20847 repeatability (diesel fuels) | Measurement time |
|---|---|---|---|---|---|
| 10 | mg/kg | 2.7 | Outside scope | Outside scope | 300 seconds |
| 50 | mg/kg | 2.2 | 5.4 | 9 | |
| 100 | mg/kg | 2.9 | 8.5 | 10 | |
| 500 | mg/kg | 5.3 | 24 | 17 | |
| 1000 | mg/kg | 7.2 | 37 | Outside scope | |
| 0.5 | % m/m | 0.003 | 0.011 | Outside scope | 50 seconds |
| 1.0 | % m/m | 0.005 | 0.016 | Outside scope | |
| 3.0 | % m/m | 0.006 | 0.033 | Outside scope | |
| 5.0 | % m/m | 0.014 | 0.046 | Outside scope | |

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

| Analyte | Concentration range | Concentration unit | Standard error of calibration | Guaranteed limit of detection (3σ) | Limit of quantification (10σ) | Precision (95% confidence) | Measurement time |
|---|---|---|---|---|---|---|---|
| S | 0 - 1 | % m/m | < 0.01 | 6 | 12 | 0.001 at 0.1<br>0.003 at 0.4 | 120 seconds |
| Cl | 0 - 1 | % m/m | 0.02 | 6 | 12 | 0.001 at 0.1<br>0.002 at 0.5 | |

Table 4: LAB-X5000 repeatability for chlorine at 120 s analysis time versus EPA Method 9075 repeatability

| Given concentration (% m/m) | LAB-X5000 precision (95% confidence) (% m/m) | EPA 9075 repeatability (% m/m) |
|---|---|---|
| 0.02 | < 0.001 | 0.008 |
| 0.05 | < 0.001 | 0.013 |
| 0.1 | 0.001 | 0.018 |
| 0.2 | 0.001 | 0.026 |
| 0.4 | 0.002 | 0.036 |
| 0.6 | 0.002 | 0.044 |

## SUMMARY

Once calibrated, Hitachi High-Tech's LAB-X5000 provides cost-effective, accurate and repeatable determination of sulfur (and chlorine) in a wide variety of petroleum products, enabling operators to make process decisions fast and ensure products meet stringent specifications. The analyzer's ease of use and ruggedness make it an ideal quality control tool at the refinery and any testing facility, delivering results within minutes for maximum productivity.

## ORDERING INFORMATION

The minimum required for this application is:

- LAB-X5000 Sulfur package, which includes: the analyzer, user manual, pre-loaded optimized method parameters, method sheets (calibration instructions), setting-up samples, and a liquids accessories pack

**Optional items:**

- Factory calibrations:
  - We have a comprehensive range of factory calibrations for the determination of sulfur, and sulfur and chlorine in oil. Please speak with your local Hitachi High-Tech representative to discuss which calibrations meet your testing requirements
- Calibration standards sets:
  - S in mineral oil standards (0 - 1000 mg/kg), part number 10003362
  - S in mineral oil standards (0 - 5 % m/m), part number 10011742
  - S and Cl in mineral oil standards (0 - 1 % m/m), part number 10011743

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