

LAB-X5000

LAB-X5000 for the rapid determination of Sulfur and Chlorine in oil – Helium path

INTRODUCTION

Within the petroleum industry, routine 24/7 quality control analysis for sulfur in a range of petroleum products, e.g. crude oil, diesel fuel, kerosene, jet fuel, naphtha, etc. is often carried out using benchtop Energy Dispersive X-ray Fluorescence (EDXRF) analysers such as the LAB-X5000 and X-Supreme8000 instrument.

As well as measuring the sulfur content, it is also important to monitor the content of chlorine. Chlorides can react with other compounds to form hydrochloric acid (HCl) which is highly corrosive and can damage process equipment. Chlorine is naturally present in crude oil in the form of inorganic chlorides and is washed out of the crude during the desalting process. However, low levels of organic chlorides can be introduced in the crude oil and refinery streams through the cleaning of pipelines and tanks which uses chlorinated solvents. To prevent costly damages and potentially catastrophic failures, it is essential to monitor chlorine levels in the crude, but also in the refined products.

SULFUR AND CHLORINE IN OIL ANALYSIS MADE EASY

With the LAB-X5000, the analysis of sulfur and chlorine in oil couldn't be easier. Routine analysis is carried out by pouring the oil into a sample cup, placing the cup in the LAB-X's analysis port, and pressing a button to start the measurement. Initial results are displayed within seconds on the large, industrial LCD touch screen. Pass/Fail messages can be setup to quickly identify if a sample is out of specification.

The combination of a high-resolution detector and optimised calibration parameters ensure that you get results you can trust. For most analyses, built-in atmospheric compensation delivers reliable analysis without the need for helium, minimising the cost per analysis while retaining optimum stability. Helium is used only when strictly necessary, for example when measuring low levels of sulfur or chlorine (<20 mg/kg).

The LAB-X includes several features that help protect against damage caused by sample spills or leaks, thus minimising downtime and preventing costly repairs. Sample cups fit inside a secondary safety window that contains potential leaks from the cup. These windows are fitted with film. They are re-usable, and the film can be changed in seconds (no need for a tool). The LAB-X's automated turntable only places the sample above the X-ray tube and detector for the duration of the analysis, minimising the risk of damage or contamination to critical components. An audible alert is generated when the analysis is finished to remind users that the sample should be removed.



With up to 100,000 results stored on the analyser itself, operators can view new and old results easily, print them on the integrated printer for a hard-copy record, download them on a USB memory device, and even upload them automatically to our ExTOPE Connect cloud service to manage them remotely.

SAMPLE PREPARATION

The sample preparation is simple: just pour the oil (for total chlorine determination) into a sample cup fitted with Poly-M film, place it in the safety window (also fitted with Poly-M film) in the LAB-X's analysis port, and press the Start button.

PERFORMANCE AND RESULTS

The data shown in this section highlights the typical performance that the LAB-X5000 delivers. For each concentration range, a simple empirical calibration was created by measuring a series of standard reference materials (mineral oils) to establish the relationship between the sulfur and chlorine contents and their X-ray signal. The analytical method parameters 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.



Sample introduction



Starting the analysis

Table 1: Typical calibration performance for low sulfur and chlorine in oil (Helium path)

Analyte	Calibration range (mg/kg)	Standard error of calibration (mg/kg)	Limit of detection (3 σ) (mg/kg)	Guaranteed limit of detection (3 σ) (mg/kg)	Limit of quantification (10 σ) (mg/kg)	Precision (95% confidence) (mg/kg)
S	0 – 150	2.2	1.3	2.0	4.3	1.4 at 50
Cl	0 – 50	0.9	0.6	1.0	3.3	0.7 at 30

Table 2: Typical calibration performance for sulfur and chlorine in oil (Helium path)

Analyte	Calibration range (% m/m)	Standard error of calibration (% m/m)	Limit of detection (3 σ) (% m/m)	Guaranteed limit of detection (3 σ) (% m/m)	Limit of quantification (10 σ) (% m/m)	Precision (95% confidence) (% m/m)
S	0 – 1	0.007	0.0008	0.0011	0.0025	0.001 at 0.6
Cl	0 – 1	0.006	0.0002	0.0003	0.0006	0.001 at 0.5

Note: the limits of detection and quantification were calculated from the results of 10 static repeat measurements of a blank oil standard. These will increase with the amount of the other element present.

The LAB-X5000's precision at different sulfur and chlorine levels was calculated from the results of 10 repeat measurements of known mineral oil standards. Precision will vary with the amount of the other element present.

Table 3: Typical repeatability for low chlorine determination (Helium path)

Analysis time (seconds)	Given chlorine concentration (mg/kg)	Precision at given concentration (95% confidence) (mg/kg)
300	3 (with 6 mg/kg S)	1.1
	5 (with 150 mg/kg S)	0.7
	10 (with 75 mg/kg S)	0.8

Note: the typical performance for the determination of sulfur and chlorine in oil (air path) can be found in Application Note 01.

SUMMARY

Once calibrated, Hitachi High-Tech's LAB-X5000 provides reliable sulfur and chlorine in oil analysis, enabling operators to make process decisions fast and verify that products meet specifications. Its ease of use and ruggedness make it an ideal tool for oil testing, delivering results within minutes for maximum productivity.



ORDERING INFORMATION

The minimum required for this application is:

LAB-X5000 Low Sulfur & Chlorine in oil (Helium) package, which includes: the analyser, user manual (on USB memory device), pre-loaded method parameters and method sheets (calibration instructions), setting-up samples and a liquids accessories pack.

Options:

Factory calibrations:

- We have a comprehensive range of factory calibrations for the determination of sulfur and chlorine in petroleum products. Please speak with your local Hitachi High-Tech representative to discuss which calibrations meet your testing requirements.

Calibrations standards sets:

- Low S and Cl (0-150 ppm S, 0-50 ppm Cl in mineral oil), Part Number 10003368
- S and Cl (0-1%) in mineral oil, Part Number 10011743.

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



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