



## LAB-X5000 for the rapid analysis of lubricating oil to ASTM D6481 and D7751

### INTRODUCTION

Lubricating oils are a blend of highly refined base oils and chemical additives. The additives impart the final product properties to meet specific requirements, and can be antioxidants, corrosion inhibitors, anti-foam agents, detergents/dispersants etc. Additive packages combine several additives that work together to perform desired tasks.

To ensure product quality, it is necessary to monitor the content of additive elements such as phosphorus (P), sulfur (S), calcium (Ca) and zinc (Zn) in the additive packages and in the end lubricating oil at the manufacturing/blending location.

X-ray fluorescence (XRF) spectrometry is one of the simplest instrumental techniques for oil analysis because the sample preparation is simple, and the analysis is fast. For many years, benchtop energy-dispersive XRF (EDXRF) spectrometers have been used successfully as a quality control tool to analyse the additive packages as well as the final blended oil. Hitachi High-Tech have a long and highly respected reputation within the lubricating oil industry with instruments such as the Lab-X3000 series, Twin-X and X-Supreme providing simple to use, accurate, cost-effective and dependable 24/7 analysis.

### MEETING ASTM D6481 AND D7751 REQUIREMENTS

Now Hitachi High-Tech's LAB-X5000 EDXRF analyser includes the latest detector technology to deliver the high performance needed for the stringent quality control of lubricating oils. The pre-loaded calibration parameters have been optimised to ensure that the LAB-X meets and even surpasses the performance requirements of standard test method ASTM D6481 (Standard Test Method for Determination of Phosphorus, Sulfur, Calcium, and Zinc in Lubrication Oils by EDXRF), and also fully conforms with D7751 (Standard Test Method for Determination of Additive Elements in Lubricating Oils by EDXRF Analysis).

### LUBRICATING OIL ANALYSIS MADE EASY

With the LAB-X5000, the analysis of additives packages and lubricating oils 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, showing the contents for all key elements. Pass/Fail messages can be setup to quickly identify if a sample does not meet specifications.

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 Poly-4 film. They are re-usable and the film can be changed in seconds (no need for a tool). The LAB-X also includes an automated turntable that 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.

The automatic atmospheric compensation delivers reliable analysis without the need for helium or vacuum purge, minimising the cost per analysis without compromising results quality. Helium is used only when strictly necessary, for example when measuring low energy elements such as Mg.

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 to our ExTOPE Connect cloud service to manage them remotely. With the LAB-X connected to WiFi, the results are uploaded to your cloud account automatically, so you do not need to be near the analyser to access your data!

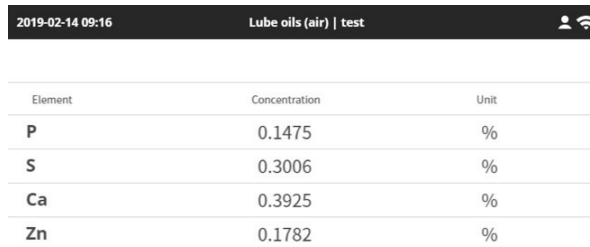
Users can set-up a QC routine with concentration targets and lower and upper limits for each control element; the displayed control graphs clearly show if the test is in control.

## SAMPLE PREPARATION

The sample preparation is simple: just pour the oil into a sample cup fitted with Poly-4 film, place it in the safety window (also fitted with Poly-4 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-X delivers. A simple empirical calibration was created for each method by measuring a series of standard reference materials to establish the relationship between the elements' contents and their X-ray signal. The calibrations used the pre-defined parameters included in the LAB-X5000 Lube Oil packages (pre-loaded in the analyser). The limits of detection were calculated from the results of 10 repeat measurements of a blank oil standard.



Element	Concentration	Unit
P	0.1475	%
S	0.3006	%
Ca	0.3925	%
Zn	0.1782	%



Clear results display



**Table 1:** Typical calibration performance for lubricating oil analysis (air path)

Analyte	Calibration range (% m/m)	Standard error of calibration (% m/m)	Limit of detection (3 $\sigma$ ) (% m/m)	Limit of quantification (10 $\sigma$ ) (% m/m)	Precision (95% confidence) (% m/m)	Total analysis time (seconds)
P	0 – 0.25	0.004	0.0008	0.0027	0.0024	100
S	0 – 0.60	0.010	0.0003	0.0011	0.0021	
Ca	0 – 0.60	0.005	0.0001	0.0003	0.0006	
Zn	0 – 0.25	0.004	0.0005	0.0018	0.0014	

Note: The precision was calculated from the results of 10 repeat measurements of a standard containing 0.15% P, 0.30% S, 0.40% Ca and 0.18% Zn.

**Table 2:** Typical calibration performance for the rapid analysis of lubricating oil (helium path)

Analyte	Calibration range (% m/m)	Standard error of calibration (% m/m)	Limit of detection (3 $\sigma$ ) (% m/m)	Limit of quantification (10 $\sigma$ ) (% m/m)	Precision (95% confidence) (% m/m)	Total analysis time (seconds)
P	0 – 0.25	0.003	0.0007	0.0023	0.0021	100
S	0 – 0.60	0.003	0.0002	0.0005	0.0010	
Ca	0 – 0.60	0.004	0.0002	0.0007	0.0009	
Zn	0 – 0.25	0.003	0.0003	0.0010	0.0006	

Note: the precision was calculated from the results of 10 repeat measurements of a standard containing 0.15% P, 0.30% S, 0.40% Ca and 0.18% Zn.

**Table 3:** Typical calibration performance for the rapid analysis of lubricating oil to ASTM D7751

Analyte	Calibration range (% m/m)	Standard error of calibration (% m/m)	Limit of detection (3 $\sigma$ ) (% m/m)	Limit of quantification (10 $\sigma$ ) (% m/m)	Precision (95% confidence) (% m/m)	Total analysis time (seconds)
Mg	0 – 0.40	0.01	0.007	0.023	0.018	320
P	0 – 0.25	0.01	0.001	0.003	0.001	
S	0 – 1.55	0.03	0.001	0.004	0.002	
Cl	0 – 0.40	0.007	0.0003	0.001	0.003	
Ca	0 – 1	0.02	0.0001	0.0004	0.003	
Zn	0 – 0.25	0.007	0.0002	0.0006	0.001	
Mo	0 – 0.05	0.003	0.001	0.004	0.0005	

Note: the precision was calculated from the results of 10 repeat measurements of a standard containing 0.1% Mg, 0.013% P, 0.65% S, 0.36% Cl, 0.52% Ca, 0.19% Zn and 0.027% Mo.

## SUMMARY

Once calibrated, Hitachi High-Tech's LAB-X5000 provides reliable lubricating oil analysis, enabling operators to make process decisions fast. Its ease of use and ruggedness make it an ideal tool at the blending plant, delivering results within minutes for maximum productivity.

Visit [www.hitachi-hightech.com/hha](http://www.hitachi-hightech.com/hha) for more information.



## ORDERING INFORMATION

The minimum needed for this application is:

- LAB-X5000 Lube Oil package, which includes: the analyser, user manual (on USB memory device), pre-loaded method parameters and method sheet (calibration instructions), setting up samples, and a liquids accessories pack.
- LAB-X5000 Lube Oil Maxi package; includes all the above, plus a helium purge for enhanced performance, and pre-loaded method parameters and method sheets for the analysis of lubricating to ASTM D7751.
- A set of calibration standards can be ordered as an option (Part Number 10003360 for D6481, and 10022254 for D7751).



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