

# Analysis of Pb in Oil according to ASTM D5059 Norm

ARL ADVANT'X Series with IntelliPower™  
Sequential X-Ray Fluorescence Spectrometers

## Key Words

- ARL ADVANT'X
- IntelliPower™
- Oil
- X-Ray Fluorescence
- XRF

## Introduction

This application note demonstrates the analysis of lead (Pb) in oil, using the Thermo Scientific ARL ADVANT'X, according to the ASTM D5059 norm.

## Instrumentation



The ARL ADVANT'X IntelliPower Series can be fitted with SmartGonio™ or with Universal Goniometer for a more extended range of elements, lower concentration limits, higher throughput and faster speed of analysis. With a clever management of power, the ARL ADVANT'X IntelliPower spectrometers can operate at 1200W and even 2500W without requiring external water cooling.

The analysis of liquids can be performed with ease and minimum sample preparation in XRF. The ARL ADVANT'X Series integrates an innovative shutter separating the primary chamber from the goniometer chamber. When liquid samples are analyzed, helium gas is introduced only in the primary chamber keeping the goniometer under vacuum. This allows a rapid change over from a vacuum to a helium environment (less than 2 min.) and permits the measurement of solids and liquids in the same batch without compromising the stability of analysis. In addition, it protects the goniometer from any liquid spillage. Helium consumption is also kept to a minimum.

## Lead analysis in oils



The ASTM D5059 norm - test method C - was chosen for low Pb in oil. Five standard samples were prepared in accordance with the norm in order to construct a calibration curve.

This norm proposes the use of bismuth (Bi) as an internal standard.

The instrument settings were the following:

	TUBE VOLT.[kV]	TUBE CURR. [mA]	DETECTOR	COLLIMATOR	CRYSTAL
Bi Lα1	60	41	Scintillator	Fine	LiF200
Pb Lα1	60	41	Scintillator	Fine	LiF200
Pb back-ground	60	41	Scintillator	Fine	LiF200

The Pb intensity ratios have been determined with the following formula:

$$\text{Pb ratio} = (\text{Pb } L\alpha 1 - \text{Pb background}) / \text{Bi } L\alpha 1$$

In each case the standard intensity was related to the Pb content in ppm, the resulting calibration curve is shown in Figure 1. Table 1 summarizes the measured concentrations achieved, together with the absolute difference between nominal and calculated concentrations, as well as the Standard Error of Estimate.

SAMPLE #	Pb RATIO	NOM. CONC. [PPM]	CALC. CONC. [PPM]	ABSOL. DIFF. [PPM]
00	437	0.1	0.0	0.1
02	588	1.9	1.7	0.2
05	950	4.9	5.5	0.6
10	1'304	9.7	9.4	0.3
19	2'240	19.4	19.4	0.0
Standard error of estimate (ppm):				0.4

Table 1: Calibration results for five standard samples with low Pb content

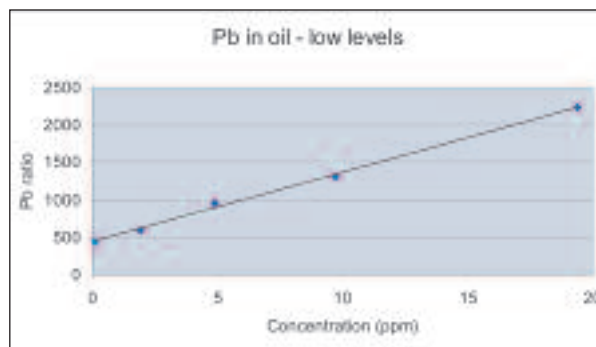


Figure 1: Pb calibration curve at 2500W

## Repeatability test on oil samples containing Pb

A repeatability test for the analysis of Pb was also performed using two oil samples with different Pb concentration: A and B.

Three liquid cells of each sample (A<sub>1</sub>, A<sub>2</sub>, A<sub>3</sub>, B<sub>1</sub>, B<sub>2</sub> and B<sub>3</sub>) were prepared and analyzed. The results obtained are shown in Table 2 on next page.

### Sample A

SAMPLE	Pb CONCENTRATION IN PPM
A1	3.7
A2	3.7
A3	3.6
Average	3.7
SD	0.02
%SD	0.64

### Sample B

SAMPLE	Pb CONCENTRATION IN PPM
B1	10.4
B2	10.4
B3	10.1
Average	10.3
SD	0.2
%SD	1.58

Table 2: Repeatability tests

## Conclusion

Good calibration curves can be obtained with the ARL ADVANT<sup>®</sup>X XRF for the determination of Pb in oil when applying the appropriate ASTM standard method.

Thanks to the reproducibility of loading and the ARL ADVANT<sup>®</sup>X helium shutter protecting the goniometer chamber from the helium environment, excellent repeatability of analysis can be demonstrated for Pb analysis.

The results obtained show that excellent accuracy and precision can be achieved with the ARL ADVANT<sup>®</sup>X XRF instrument, and that the instrument is ideally suited for the analysis of Pb in petrochemical products. Furthermore the new state-of-the-art OXSAS analytical software under Windows<sup>®</sup> XP Professional provides comprehensive analytical functions and ease of use.

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