Analysis of Pb in gasoline according to ASTM D5059 norm with ARL PERFORM'X Sequential X-Ray Fluorescence Spectrometer

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Key Words
ARL PERFORM'X, XRF, WDXRF, X-ray fluorescence, lead, gasoline

Goal

In order to demonstrate the analysis of lead (Pb) in gasoline, a calibration curve has been constructed according to the ASTM D5059 norm.

Instrumentation

Thermo Scientific ARL PERFORM'X series spectrometer used in this analysis was a 4200 watt system. This system is configured with 6 primary beam filters, 4 collimators, up to nine crystals, two detectors, helium purge and our 5GN+ Rh X-ray tube for best performance from ultra-light to heaviest elements thanks to its 50 micron Be window. This new X-ray tube fitted with a low current filament ensures an unequalled analytical stability month after month.

The ARL PERFORM'X offers the ultimate in performance and sample analysis safety. Its unique LoadSafe design includes a series of features that prevent any trouble during sample loading. Liquid cassette recognition prevents any liquid sample to be exposed to vacuum by mistake. Over exposure safety automatically ejects a liquid sample if X-ray exposure time is too long.

The Secutainer system protects the primary chamber by collecting any drops in a specially designed container, easily removed and cleaned by any operator. For spectral chamber protection, the ARL PERFORM'X uses a helium shutter designed for absolute protection of your goniometer during liquid analysis under helium operation. In the "LoadSafe Ultra" optional configuration, a special X-ray tube shield provides total protection against liquid cell rupture.

Lead analysis in gasoline

The ASTM D5059 norm - test method C - has been chosen for low Pb levels. 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	50	Scintillator	Fine	LiF200
Pb Lα1	60	50	Scintillator	Fine	LiF200
Pb background	60	50	Scintillator	Fine	LiF200

A power of 3kW is used in order to avoid too much heating of the supporting mylar film which would cause sagging of the film and instability of analysis.

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

$$\text{Pb ratio} = (\text{Pb L}\alpha 1 - \text{Pb background}) / \text{Bi La1}$$

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Standard intensities are correlated to the Pb content in ppm, which results in the calibration curve shown in Figure 1. Table 1 gives the numerical results including 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

Repeatability test

A repeatability test for the analysis of Pb has been done using two samples with different Pb concentration: A and B.

Three liquid cells of each sample (A1, A2, A3, B1, B2 and B3) were prepared and analyzed. The results obtained are the following:

Sample	Pb concentration in ppm
A1	3.7
A2	3.7
A3	3.6
Average	3.7
SD	0.02

Table 2: Repeatability test on sample A

Sample	Pb concentration in ppm
B1	10.4
B2	10.4
B3	10.1
Average	10.3
SD	0.2

Table 3: Repeatability test on sample B

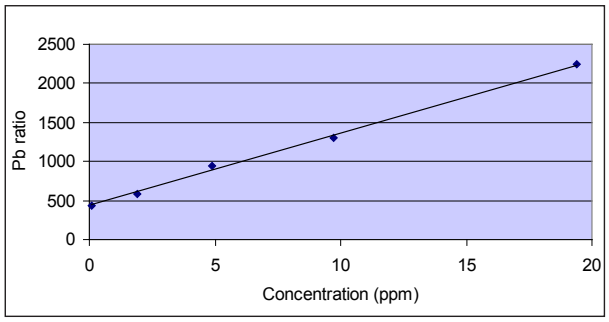

Figure 1: Pb calibration curve

Conclusion

Good calibration curves are obtained with the ARL PERFORM'X XRF for Pb determination in gasoline when applying the appropriate ASTM standard method.

Thanks to the reproducibility of loading and to the 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 very good accuracy and precision can be achieved with the ARL PERFORM'X sequential XRF instrument. This instrument is well suited for the analysis of Pb in petrochemical products.

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