

# S4 EXPLORER

## ANALYSIS OF LEAD IN PETROCHEMICAL PRODUCTS – ASTM 5059

### Instrumentation

The S4 EXPLORER XRF (Fig. 1) spectrometer was used to derive the following results. It was fitted with the Bruker AXS Rh-anode X-ray tube with the ultrathin 75  $\mu\text{m}$  Be-window. For an optimum analysis a Helium environment in the sample chamber was used. The S4 EXPLORER is equipped with a state-of-the-art vacuum seal to keep a vacuum in the spectrometer chamber while flushing Helium gas into the sample chamber. This permits a minimum of change-over time from vacuum to Helium and reduced Helium consumption. In addition, all analytical components of the instrument are kept stable under vacuum.

### Sample Preparation

To each 10 ml of a set of premixed standards, which were prepared according to the ASTM Standard 5059, 1 ml of the Bi-Internal Standard Solution was added. The samples were then mixed and poured into a standard Bruker AXS sample cup (Fig. 2). The latter was sealed with a 6  $\mu\text{m}$  Mylar™ foil.

### Analytical Procedure

Helium gas was used to flush the sample chamber while maintaining a stable vacuum in the spectrometer chamber itself. X-ray tube excitation was set to 50 kV and 20 mA.



Fig 1. Plug 'n analyze S4 EXPLORER

The measurement of 7 standards allowed the creation of a calibration curve, which was used to calculate sensitivities and limits of detection. The whole calibration and the resulting standard deviation of the calibration curve was calculated using the 32bit Software SPECTRA<sup>plus</sup>.

One blank Toluene with an added internal standard was used as a blank according to ASTM 5059.

The standard suggests collecting at least 100.000 counts for each sample to allow for 0.3 % deviation of the counting statistics. This is reflected in the measuring time. One sample takes less than 5 minutes to measure with the SPECTRA<sup>plus</sup> automatic measurement time optimization set to check for a max 0.3 % deviation of counting statistics.

### Results

- The S4 EXPLORER can fully meet the ASTM 5059 Standard. The results are shown in Tab. 1
- For Lead in Petrol a range from 0.0 g / US gallon to 0.3 g / US gallon was calibrated.
- Overall calibration deviation was 0.002 g / US gallon resulting in a detection limit of 0.4 ppm Pb.
- Since one US Gallon equals 3.78 liters, the standard deviation of the calibration is 0.0005 g/l for the calibration range.
- Reproducibility was performed using a lead free petrol sample from a filling station and was found to be better than 1 ppm or 0.0001 g/l and repeatability was 1.1 ppm.

Author: Alexander Seyfarth, Bruker AXS, Inc.,  
Madison, WI, USA



Fig. 2. Standard liquid cups

**Table 1**

Calibration range	0.0 - 0.3 g / US gallon
Calibration standard deviation	0.002 g / US gallon or 0.0005 g / liter
Lower Limit of Detection	0.4 ppm

BRUKER AXS GMBH

D-76187 KARLSRUHE  
GERMANY

TEL. (+49) (721) 595-2888  
FAX (+49) (721) 595-4587  
<http://www.bruker-axs.de>  
Email: [info@bruker-axs.de](mailto:info@bruker-axs.de)

BRUKER AXS, INC.

5465 EAST CHERYL PARKWAY  
MADISON, WI 53711-5373  
USA

TEL. (+1) (800) 234-XRAY  
TEL. (+1) (608) 276-3000  
FAX (+1) (608) 276-3006  
<http://www.bruker-axs.com>  
Email: [info@bruker-axs.com](mailto:info@bruker-axs.com)