



Elemental Analysis for Petrochemical Applications - The Easy Way!

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In the last two decades elemental analysis has become one of the most important tasks in process and quality control in petrochemical production: Sulphur in automotive fuels has been a source of toxic air pollutants like sulphur dioxide and sulphates. Actual legislation enforces lower residual sulphur concentrations in fuels, like the today's limit of 10 ppm. Future regulations may drive these limits down even further. Therefore in all kind of fuels the sulphur concentration must be controlled continuously in refineries and tank farms.

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Modern car engines require accurately adjusted concentrations of all additives in lubricating oils to guarantee highest performance over a wide range of conditions. Here elemental analysis is used to ensure that the final product quality is the same, no matter on which days or locations the oil has been blended and bottled.

Analytical requirements, like detection limits, repeatability,... are typically defined in international standards, like ASTM or EN/ISO. Additional needs for reliability, ease of use, simple installation and robustness derive from the use close to production and along the supply chain - even in remote locations.

XRF (X-ray fluorescence spectrometry) has established itself as the method of choice offering lowest cost of operation due to the capability of direct analysis. The sample preparation is basic, just simple recipes instead of rocket science. If the instrument is setup to offer the easiest way of operation and ensures rock-solid technologies it is just dedicated to routine process analysis in industrial environments.

Petrochemical Applications – made easy

Understanding these requirements Bruker AXS has developed the S2 RANGER. The energy dispersive XRF spectrometer (EDXRF) applies latest Silicon drift detector technology, the XFlash detector, and combines it with the 50W direct excitation source. The S2 RANGER offers doubtless element identification due to the high



S2 RANGER – Benchtop EDXRF with TouchControl interface

spectral resolution and best analytical precision due to the capability analysing at high concentrations. Just regular electrical power connection and little Helium is required for the analysis. All components, e.g. vacuum pump, spectrometer, printer and computer are combined in one housing: Simple to connect, simple to relocate. The unique interface TouchControl enables users to operate the instrument right after some minutes of training since the workflow of the software is easy and intuitive.

The remaining question is about the application: ISO compliant analysis of fuels, contaminants in fuel oil from the process and additives in lubricants – experience is needed to run these applications reliable everyday in the industrial laboratory.

Sulphur in fuels

The analysis of higher sulphur concentrations in different fuel types and mineral oil by EDXRF is regulated in ISO 8754: Applying a calibration range up 5000 ppm, respectively 5% for the highest range. The final application must pass a repeatability test running one sample more than 20 times. The maximum deviation of the results must not exceed 0.01% for a sample with 0.1% sulphur.

The S2 RANGER was calibrated using 6 reference standards and a blank sample ranging from 0 – 5000 ppm. The achieved precision was 0.0005%, much lower than the required limit of 0.01%.

In refineries and tank farms the analysis of even lower sulphur concentrations in automotive fuels is performed to check if the actual requirements are fulfilled before shipment. WDXRF systems, like the S8 TIGER are mainly used in central laboratories for process control applying ASTM 2622 and achieving detection limits of less than 0.2 ppm. But along the supply chain and in remote locations EDXRF systems like the S2 RANGER are the instruments of choice.

ISO 20847 describes this application. The S2 RANGER has been calibrated according to the ISO standard and the final performance test has been done by running a sample 21 times. ISO 20847 demands that the repeatability stays within +/- 8 ppm. The result is shown in Figure 2.

The S2 RANGER easily achieves a result of 8.9 ppm +/- 0.9 ppm and passes the final compliance test. Once calibrated, drift correction standards defined, quality check procedure established by running a check sample every morning - the S2 RANGER is ready for the daily routine in tank farms, along the pipeline and on shipping sites.

More elements in petrochemical materials

During the product development of lubricants, in all production steps – blending and bottling, especially if done by contract blenders – XRF ensures that the same product quality is established worldwide. The S2 RANGER not only achieves a high precision for low

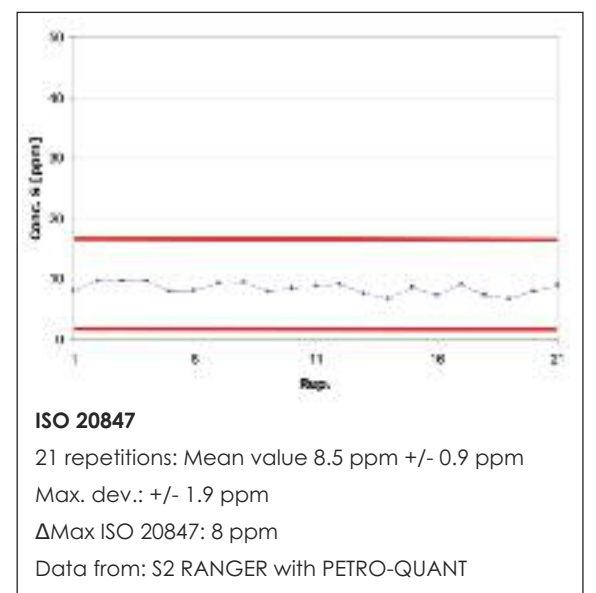


Figure 2: Control chart for ISO 20847 performance test

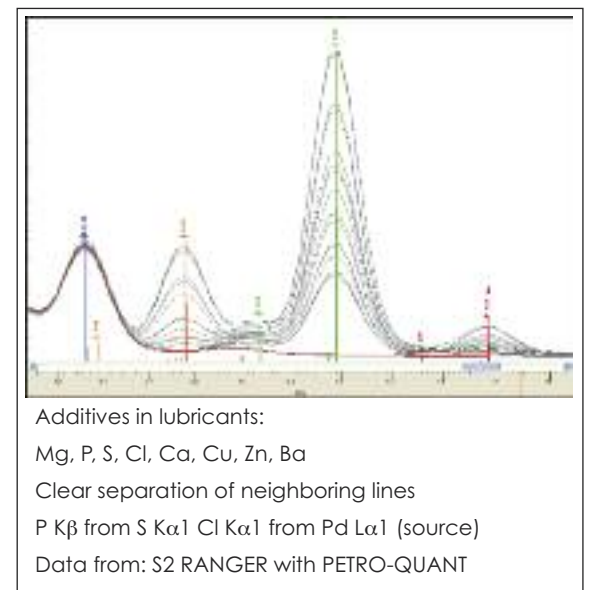


Figure 3: Sample Spectra of the S2 RANGER recorded with the XFlash detector

concentrations values of a single element, it is also setup to analyse several elements simultaneously. The XFlash detector records the sample spectra with a high resolution, neighbouring element lines are clearly separated, shown in Figure 3.

Commonly used elements as additive to prepare lube oils are Mg, P, S, Cl, Ca, Cu, Zn and Ba. The S2 RANGER has been calibrated with 23 standards having randomly distributed concentrations. A quality check sample has been analysed 21 times, the results are shown in Table 1.

The achieved detection limits, standard deviations and precision values show impressively that the S2 RANGER is the ideal instrument ensuring highest quality

in the blending process. Remembering about additional requirements for industrial quality control the S2 RANGER fulfils not only the analytical requirements it also offers the intuitive TouchControl interface: Operation made simple with step by step workflows and a clear graphical user interface just to be operated with a finger tip.

Start up made easy

Calibration standards, quality samples and drift correction monitors – the setup of norm compliant calibrations are time and money consuming. Not everybody is XRF expert and has the experience to achieve the maximum performance from an analytical

instrument. Therefore Bruker AXS offers in addition to the S2 RANGER PETRO-QUANT. This package consists of a sample preparation kit with liquid cups, foils and balance as well as a set of predefined applications. Several ready-to-analyse solutions can be installed on the S2 RANGER directly in the factory:

- ISO 8754 in gasoline, diesel, mineral oil
- ISO 20847 in gasoline, diesel, mineral
- Additives in lube oils (Mg, P, S, Cl, Ca, Cu, Zn, Ba)
- Contaminants in fuel oil (S, V, Fe, Ni)

The requirements of process and quality control in the petrochemical industry are perfectly met by the S2 RANGER. Delivered as ready-to-analyse solution offering high analytical performance with easy operation, rocksolid technology, less installation and maintenance effort, the S2 RANGER is the perfect choice.

LUBRICANT-8	Mg(%)	P(%)	S(%)	Cl(%)	Ca(%)	Cu(%)	Zn(%)	Ba(%)
Calibration Range [%]	0.2	0.15	0.755	0.15	0.505	0.05	0.15	0.2
Detection limits [ppm]	80	5.5	2.8	2.7	4.2	1.2	0.7	23
Precision Test								
Average [%]	0,0831	0,1407	0,6514	0,1524	0,0706	0,0202	0,152	0,0784
Abs.Std.Dev. [%]	0,0054	0,001	0,0039	0,0011	0,0005	0,0002	0,002	0,0011

Table 1: Summary of analytical results for additives in lube oils