BRUKER

S2 PUMA for Elemental Analysis in the Petrochemical Industry

- Quality Control for Lubricants and Fuels with Accuracy and Precision

EDXRF

Innovation with Integrity

Get all Information you Need for Fuels and Oils – Elemental Analysis with the S2 PUMA

To guarantee optimal engine and machine performance while maintaining the best-possible protection of our environment, today's fuels and lubricants are real high-tech blends. When producing, using, and recycling these substances it is important to have a clear and precise understanding of the content and composition of accompanying elements and additive metals. X-ray fluorescence (XRF) analysis is the ideal method to accomplish this: With this technique, an elemental analysis can be conducted quickly and precisely. This ensures that additives are used economically, the properties of lubricants and fuels are guaranteed, and that our environment is protected.

S2 PUMA Performs Elemental Analysis Reliably, Precisely, and Quickly

The range of uses for XRF in elemental analysis in the field of petrochemicals is very broad, ranging through the entire product life cycle of lubricants and fuels. For example, sulfur can be found in crude oil in amounts of up to five mass percent and above, but in lubricating oils only variable proportions ranging from the upper ppm range to percent are permitted, and in automobile fuels only a few ppm. In order to quickly and reliably determine the quality and quantity of the constituents in such blends, Bruker developed the S2 PUMA: An energy-dispersive X-ray spectrometer (EDXRF) in the form of an easy-to-operate, bench-top device, which can be ideally configured for the kind of analyses that are typical in the petrochemical industry. Thanks to its user-friendly TouchControl™ with intuitive user interface, users without any special training can perform precise analysis.

Our S2 PUMA is frequently used in the petrochemical industry in the following areas:

- Sulfur analysis in fuels such as car or ship diesel **(ASTM D4294, EN ISO 20847 (DIN), EN ISO 8754 (DIN)**
- Verification of additives in lubricants, particularly Ca, Zn, P, and Mg **(ASTM D6481, ASTM D7751)**
- Metal abrasion analysis (e.g. Fe, Cu, Pb) in lubricating oils
- Monitoring of bio-fuel production, for example through P or Fe measurements

S2 PUMA for Best Data Quality – Optimum Precision, Best Accuracy and Norm Compliant

Sulfur Analysis Done Quickly and Easily With the S2 PUMA

Instrument specifications and parameters for sulfur analysis in the petrochemical industry are specified in norms such as ASTM D4294, EN ISO 20847 (DIN), and EN ISO 8754 (DIN). The S2 PUMA easily satisfies the requirements of these norms.

A calibration of the instrument is carried out with prepared sulfur standards. Fig. 1 shows an overlaid spectra of various standards in the concentration range from 0 to 1000 ppm S and illustrates the possibility to use them for quantitative measurements.

S2 PUMA – Sulfur Measurements With Excellent Precision and Compliance to ASTM D4294

When dealing with the precision of sulfur measurements in petrochemical products, ASTM D4294 is a strict norm. To demonstrate that the data is sufficiently precise, a sample with 25 ppm sulfur was measured 42 times in series (see Figure 3). The sample must be prepared fresh each time. The deviation of the mean values of every two consecutive measurements here may not exceed certain values which are concentration-dependent. For sulfur concentrations of 25 ppm this value is 3.5 ppm. These criteria may only be exceeded in a maximum of one value in 20 cases.

ASTM D4294: Above all Doubts With S2 PUMA results.

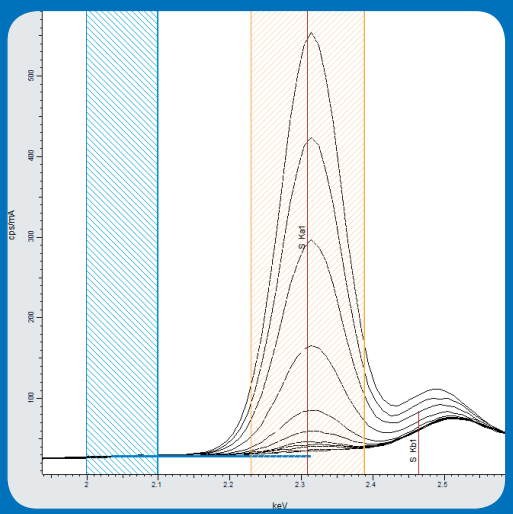


Figure 1: Superimposed spectra of sulfur in paraffin standards in the concentration range of 0 to 1000 ppm S. The peak is integrated over the yellow area; for background correction the blue shaded area is used.

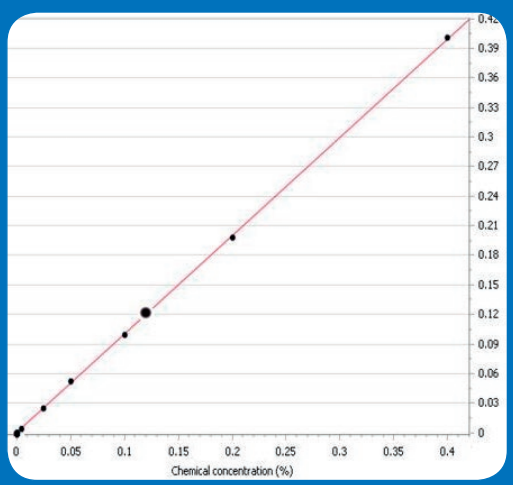


Figure 2: Calibration line for sulfur in paraffin in the range of 0 to 1000 ppm

Table 1: Calibration results for sulfur in paraffin

Element	Concentration range [ppm]	Detection limit (300 s, 3σ) [ppm]	Correlation (R^2)
Sulfur	0-1000	0.6	0.99998

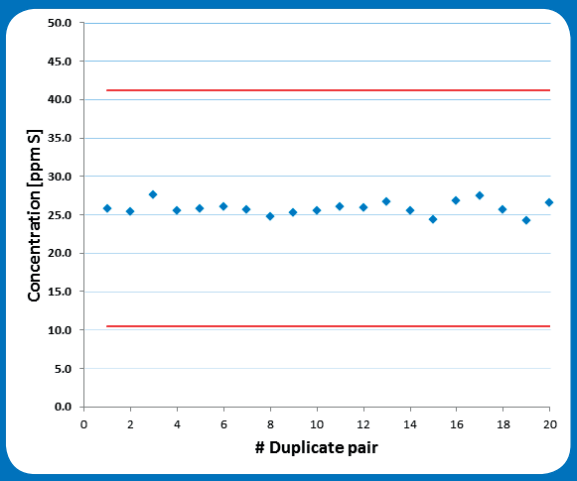


Figure 3: Repeatability of duplicate measurements of 25 ppm sulfur standards in accordance with ASTM D4294. The horizontal red lines denote the allowed limits for reproducibility according to the norm. The high requirements placed on the determination of sulfur in fuels, such as those contained in the ASTM D4294 norm, but also the requirements of EN ISO 20847 (DIN) and EN ISO 8754 (DIN), are met by the S2 PUMA.

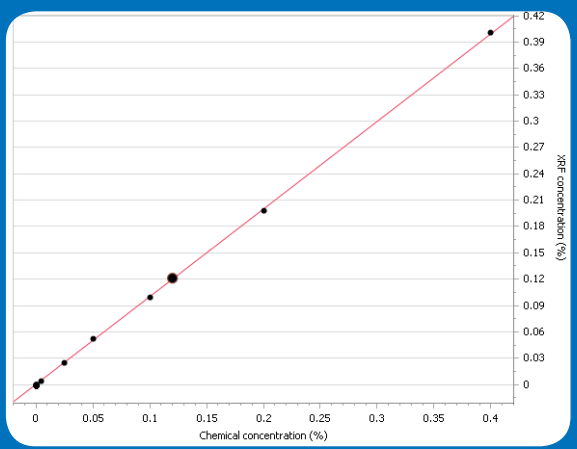


Figure 4: Calibration line for zinc in a lubricating oil matrix

Table 2:Calibration results for zinc in a lubricating oil matrix

Element	Concentration range [ppm]	Detection limit (100 s, 3σ) [ppm]	Standard deviation [ppm]	Correlation (R^2)
Zinc	0 - 0.4	0.3	8	0. 99997

Additives in Lubricants – There is Simply no Better Way than the S2 PUMA

Sulfur isn't the only element that plays a central role in lubricants and fuels. That is why the S2 PUMA is also capable of determining the quality and quantity of additives used in the refinement of lubricants. These additives, such as abrasion minimizers or pitting protection additives, are added to lubricating oils in small quantities in order to achieve or improve certain properties. Due to their complex formulations, these special chemicals can sometimes be very expensive, so the quantities added are decisive for the quality, efficiency, but also the environmental friendliness of the product.

S2 PUMA – Norm Compliance for Mg, P, S, Cl, Ca, Zn, and Mo

In addition to the norm ASTM D6481 now also ASTM D7751 is applied for these measurements. This norm takes into account the important additive elements magnesium, phosphorus, sulfur, chlorine, calcium, zinc, and molybdenum in concentration ranges typical for their use in lubricants.

Using the example of zinc as abrasion-reducing additive, Figure 4 depicts the calibration line on the S2 PUMA. Table 2 shows the calibration results for zinc in lubricants on the S2 PUMA, and Table 3 gives typical repeatabilities of ten measurements of additive elements in lubricants.

The S2 PUMA meets all requirements of ASTM D7751 and D6481 on the analysis of additives. Calibration over a wide range of concentrations, the low detection limit, the small standard deviation, and very good accuracy make the S2 PUMA the ideal partner when it comes to the analysis of oil additives. The S2 PUMA can also be used to examine used oils. Here the amounts of heavy metals, halogens, etc. contained determine the type of recycling or disposal.

No matter which lubricating oil you need to specify: The S2 PUMA delivers spot-on ASTM compliance for D6481, D7751 and other.

Table 3: Repeatability data for 10 measurements of a QC sample for additives in lubricants according to ASTM D7751

Measurement	Mg [%]	P [%]	S [%]	Cl [%]	Ca [%]	Zn [%]	Mo [%]
Rep. 01	0.099	0.113	0.514	0.0238	0.203	0.124	0.0100
Rep. 02	0.105	0.114	0.517	0.0238	0.204	0.124	0.0099
Rep. 03	0.103	0.112	0.514	0.0249	0.202	0.123	0.0101
Rep. 04	0.100	0.113	0.516	0.0249	0.204	0.124	0.0100
Rep. 05	0.105	0.113	0.515	0.0238	0.204	0.124	0.0100
Rep. 06	0.103	0.113	0.512	0.0237	0.202	0.123	0.0100
Rep. 07	0.105	0.113	0.514	0.0236	0.203	0.124	0.0100
Rep. 08	0.104	0.114	0.514	0.0248	0.202	0.123	0.0101
Rep. 09	0.097	0.114	0.518	0.0240	0.204	0.124	0.0099
Rep. 10	0.100	0.113	0.516	0.0237	0.203	0.124	0.0099
Mean value	0.102	0.113	0.515	0.0241	0.203	0.124	0.0100
Abs. standard deviation	0.0030	0.0004	0.0018	0.0005	0.0009	0.0005	0.0001
Rel. standard deviation	3.4 %	0.5 %	0.4 %	2.1 %	0.5 %	0.5 %	0.7 %
Certified value	0.100	0.110	0.500	0.025	0.200	0.120	0.010

Technological Innovations as key to Best Results and Reliable Evaluation

The S2 PUMA is eminently suitable for elemental analysis when examining engine and machinery lubricants as well as fuels. The analyzer works cost-effective and quick, but still offers a high degree of sensitivity and precision. It is just as possible to precisely analyze important additive elements such as calcium, zinc, phosphorus, and molybdenum as it is to analyze other additive and trace elements like magnesium, potassium, silicon, sulfur, or barium. Even the main indicator elements for abrasion conditions, such as iron, copper, and lead, can be determined with the same high level of precision.

The instrument control software SPECTRA.ELEMENTS can handle blanks and duplicates easily and allows for the fully automatic evaluation of those. Oxygen is an important component in many petroleum-based products, but usually too light for EDXRF analysis. However, Aut-O-matic Matrix included in SPECTRA.ELEMENTS enables the user to determine the concentration of compounds such as oxygen automatically by Compton peak optimization.

We offer the S2 PUMA in a range of different device configurations. The S2 PUMA Single and S2 PUMA XY Autochanger models are popular in the petrochemical industry. Whereas the Single model is manually fed with one sample at a time, the XY Autochanger can automatically analyze up to 20 samples in one sequence. Here, new samples can be added to the queue at any time – even while the measurement process is running!

The S2 PUMA provides you with top-notch elemental analysis in a compact device: With its optimized optical construction with 50 Watt X-ray tube and silicon-drift detector (SDD), the HighSense™ technology developed by Bruker offers high count rates and excellent repeatability which guarantees the best in analysis results. The optional use of the HighSense™ LE detector ensures high transmission rates due to its ultra-thin entry window to quantify light elements and increases the S2 PUMA's capabilities even more.

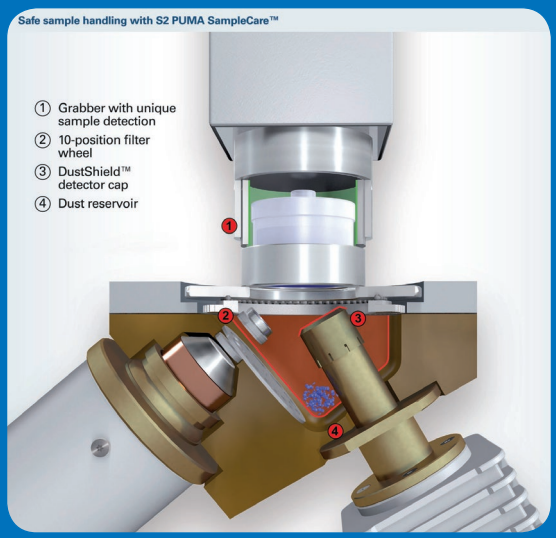
- Unmatched detection limits, precision, and speed with **HighSense™ technology**
- **Helium mode** for best analytical performance of volatile samples; cost-effective **reduced Helium** pressure mode for non-volatile samples
- S2 PUMA XY Autochanger with failsafe **recognition of liquid cups**
- **Nitrogen mode** for most cost-efficient analysis of oil samples

Analytical Performance Comes in Handy With Ease of use and Lowest Cost of Ownership

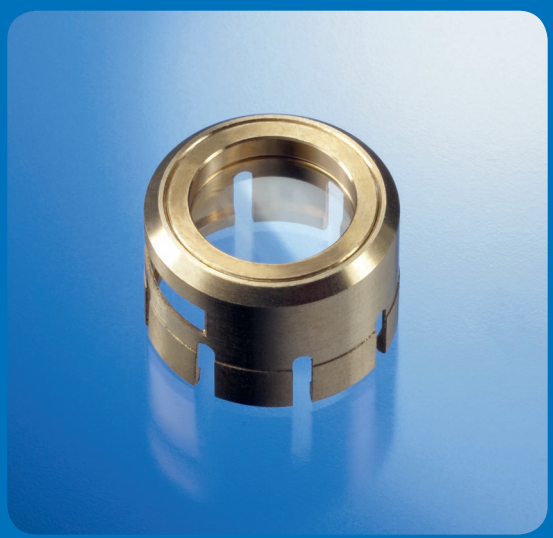
With the analysis of light elements in particular, their low-energy radiation is absorbed by the surrounding air. Consequently, such measurements are carried out in a helium atmosphere or under vacuum. Since such vacuum analyses may not be conducted with highly volatile samples, such as fuels, it pays off that the S2 PUMA XY Autochanger automatically detects these samples when loaded and prevents the formation of a vacuum. A sample-chamber that is reduced to the smallest possible dimensions translates into minimal consumption of helium, which means very low operating costs but high analytical capability.

EDXRF together with the S2 PUMA provides you with everything you need for monitoring and optimizing lubricants and fuels:

- The highest in analytical accuracy and precision in **compliance with international standards**
- **HighSense technology** for excellent detection limits, with high spectral resolution
- Outstanding results for light elements such as magnesium, due to the **HighSense™ LE detector** with its thin, coated X-ray entry window
- The **SampleCare** technology protects important system components against contamination, ensuring high system up-time with no maintenance effort
- With **TouchControl** any user can quickly and efficiently measurements – in nine user languages
- **Island Mode:** TheS2 PUMA is fully network-capable, so it requires no peripherals: After installation no PC, mouse, keyboard, printer, etc. are necessary for measurements and results output
- The integrated **SPECTRA.ELEMENTS** software makes data acquisition, analysis, and visualization of the results a breeze



S2 PUMA with SampleCare™: Unique protection against contamination of important system components ensures the best in device availability



The S2 PUMA detector cap provides the detector with additional protection against dust and liquids

Technical Data

	S2 PUMA Single	S2 PUMA XY Autochanger
Element range	Sodium to americium (Na – Am) Carbon to americium (C – Am) with light element (LE) detector	
Concentration range	From ppm to 100 %	
X-ray tube	Pd or Ag anode; max. power 50 W; max. voltage 50 kV (low kV version: 30 kV max.)	
Primary beam filters	10-position automatic filter changer for wide range elemental analysis	
Detector	Silicon Drift Detector: Peltier cooled (no need for liquid nitrogen) with DustShield™; Also available as fast HighSense™ LE version with super-high count rates	
Collimator masks *	For small spot analysis: 1, 3, 8, 12, 18, 23 and 28 mm	
Sample observation *	Integrated HD video camera, for exact sample positioning and documenting the measurement position of a sample	
Atmosphere Mode	Air mode: For heavier elements in all sample types Helium mode*: Best performance for light elements in liquids and loose powders Nitrogen mode*: Cost-saving analysis of liquid samples Vacuum mode*: Enhanced light elements performance for stable samples and lowest cost of operation	
TouchControl™	Integrated 12.1" TFT touchscreen, adjustable, multilingual user interface, optional	
Connectivity	Built-in Ethernet port RJ45, 3x USB ports for mouse, keyboard, printer	
Power requirements	100-240 V, 50/60 Hz, max. power consumption 600 VA	
Sample types	Loose powders, granules, solids, pressed pellets, fused beads, and liquids	
Sample rotation *	Spins all sample types and sizes for better measurement statistics with inhomogenous samples	
Sample sizes	Up to 51.5 mm (2.03") Ø; Liquids, loose powders, and smaller samples in liquid cups up to 50 ml	Up to 40 mm (1.56") Ø and 38 mm (1.49") height in sample holders with max. 200 g sample weight, or 51.5 mm Ø as sample ring; Liquids, loose powders, and smaller samples in liquid cups up to 20 ml
Sample loader	Single position, manual loading	EasyLoad™ XY sample tray with 20-positions, removable; Grabber with automatic liquid sample detection
Dimensions (width x depth x height; weight)	67 x 71 x 37 cm; 87 kg (26.2 x 27.8 x 14.6"; 192 lbs)	67 x 71 x 61 cm; 108 kg (26.2 x 27.8 x 23.8"; 238 lbs)
Quality & safety	DIN EN ISO 9001:2008 2006/42/EC (CE-certified Machinery directive) 2014/35/EC (Electrical equipment) 2014/30/EC (Electromagnetic Compatibility) German Type Approval and Vollschutz according to BfS RöV Fully radiation-protected system; radiation <1 µSv/h (H*) Compliant to ICRP, IAEA, EURATOM	

* optional packages

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