



## Lab Report XRF 151

# S6 JAGUAR

- Trace element analysis in coke, coal and carbon products for energy, steel plants and aluminum by WDXRF (acc. to ISO 12980, ASTM D 6376-10 and YS/T 63.16)

### Introduction

Coal and pet coke are still playing an important role in energy generation and metal production: Coke is the reducing agent in blast furnace steel plants and it is used in form von anodes to reduce aluminum in electrolytic cells. In all these industrial processes and in power generation the trace element composition is an important factor. There is the need in aluminum to avoid cross contamination of the metal from impurities in coke. Higher chlorine concentration will lead to increased corrosion of equipment and if the sulfur concentration is getting too high, it will have a negative impact on the environment due to the SO<sub>x</sub> emission.

Wavelength dispersive X-ray fluorescence (WDXRF) is recommended in several international standards for the trace analysis of impurities in coal, coke and carbon products. The S6 JAGUAR with its 400 W excitation power, the closely coupled bam path and the HighSense goniometer combines low detection limits in a compact size. It will easily analyze the main trace elements in pressed pellets, SampleCare will protect system components from contamination coming from the pellets. For lower limits of detection the S6 JAGUAR can be equipped with the XS-400 analyzer crystal, which boost the intensity by typically more than 35%.

## Instrument

The S6 JAGUAR WDXRF spectrometer is a full-blown WDXRF spectrometer in compact size: Solid samples are analyzed under vacuum for optimal light element sensitivity and low cost of ownership. Liquid samples and loose powders will be analyzed the helium mode. The HighSense goniometer can be equipped with up to 4 analyzer crystals and two detectors to cover the entire element range for coal analysis.

For a few samples per day the S6 JAGUAR can be configured with manual sample loading, in case of higher sample throughput the system can be equipped with a 24 position EasyLoad sample magazine and TouchControl. So ensures the S6 JAGUAR optimal productivity. With its unique SampleCare technology and the added vacuum pump it offers lowest cost-of-operation and optimal instrument uptime. By adding TouchControl the S6 JAGUAR is easy to operate and ensures data integrity by its failsafe operation.

## Preparation

The standards and samples were ground to receive a very small grain size in a tungsten carbide vessel to avoid contamination with Cr and Fe. In addition one has to be careful with Na contamination. This can be avoided by using gloves. 7 g of anode coke powder was thoroughly mixed with 1.4 g of wax and pressed with an automated press in aluminum cups.

## Measurement

Each element is analyzed with an optimal set of instrument parameters: Light elements are excited with low voltage at maximum power, while all heavy elements starting from Ca upwards are best excited with 50 kV. For Na the XS 55 multilayer, for the elements Al, Si and Cl the PET crystal is used and for the element range K to Zn the LiF200 is applied. To answer the demand for better trace analysis and better line separation an XS-400 crystal can be used for K - Zn. This can be vital if traces are becoming important.



Figure 1: S6 JAGUAR with manual loading

## Calibration

A set of 15 international certified reference materials (CRMs) were used to prepare the calibration for the 12 elements. Each element was analyzed with up to 100 s for optimal detection of traces. Two calibration curves, one for S and one for Ni, are shown in figure 2 and 3.

Table 1: Concentration ranges and limits of detection for anode cokes analyzed with the S6 JAGUAR

Element	Lower Range [ppm]	Upper Range [ppm]	LOD [ppm]
Na	59	2401	12
Al	32	1524	10
Si	24	180	3
S	0.5%	4.69%	
Cl	100	200	2
K	5	35	1.5
Ca	25	387	1.1
Ti	2	28	1.1
V	32	698	1
Fe	99	1454	0.6
Ni	65	283	0.5
Zn	10	43	0.4

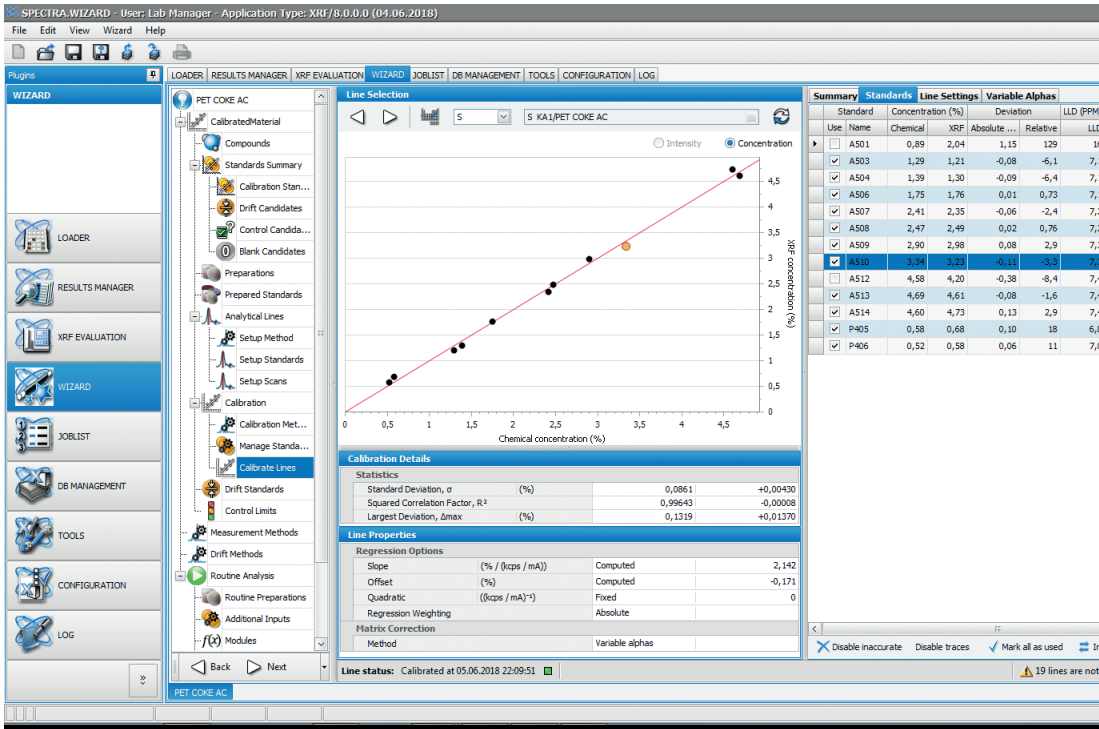


Figure 2: Calibration curve for S covering the range from 0.5 % to 4.69 %

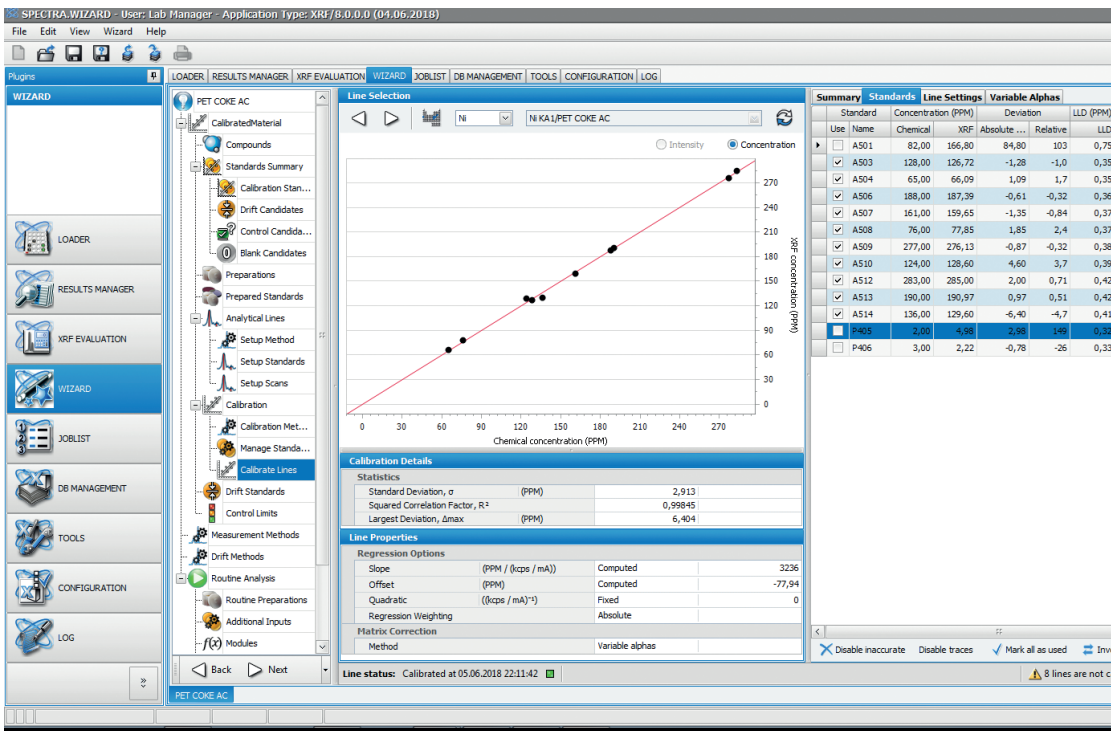


Figure 3: Calibration curve for Ni covering the range from 65 to 283 ppm

Table 2: Accuracy test for anode cokes, comparing certified with analyzed concentrations, as well as a 10 fold precision test

Element	XRF conc.	Cert. Conc.	Abs Std Dev.	Rel. Std. Dev. [%]
S [%]	3.40	3.30	0.02	0.72
Ni [ppm]	128.00	124.00	4.48	3.50
Si [ppm]	24.00	28.00	1.15	4.80
Fe [ppm]	276.00	266.00	2.65	0.96
Na [ppm]	623.00	645.00	43.61	7.00
Al [ppm]	153.00	150.00	5.66	3.70
Ca [ppm]	112.00	107.00	2.35	2.10
K [ppm]	17.00	17.00	1.84	10.80
Cl [ppm]	100.00	n.a.		
Ti [ppm]	5.00	4.00	0.31	6.10
Zn [ppm]	40.00	41.00	0.10	0.25
V [ppm]	302.00	300.00	2.33	0.77

## Conclusion

The control of ash forming elements and other impurities requires a spectrometer with high sensitivity in order to cope with the low concentration levels of some elements. The S6 JAGUAR with 400 W excitation power and its closely coupled beam path offers high analytical performance for the analysis of coke, coal and carbon products. It also delivers high analytical precision which makes the routine analysis an easy job. The sample throughput of the S6 JAGUAR is perfectly suited for a wide range of laboratories, equipped with an Easy-Load autosampler is also runs large batches unattended.

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