
## Lab Report XRF 161

# S6 JAGUAR

- Quality Control of Limestone: Precision, Accuracy and Reliability with compact benchtop WDXRF

Limestone is one of the most important industrial commodities with numerous applications, ranging from steel production to construction materials, pharmaceuticals and food. The breadth of applications results in a wide range of quality requirements. Hence, quality control is crucial to ensure that the final product complies with market specific quality standards. This makes precise, accurate, and reliable monitoring of compositional changes a critical prerequisite for cost efficient limestone production.

The grade of a high-purity limestone, and thus, the value of the final product is application specific and depends on elements such as Fe, Mg, Mn, and Na. This report demonstrates the excellent analytical performance of the compact wavelength dispersive X-ray fluorescence (WDXRF) spectrometer S6 JAGUAR for precise and accurate quality control of limestone and similar materials.

Table 1: Analytical conditions – Vacuum mode, no filter, matrix: CO2

| Elements | Voltage [kV] | Current [mA] | Analyzer crystal | Filter | Detector |
|---|---|---|---|---|---|
| Na, Mg | 30 | 13.3 | XS-55 | none | FlowCounter |
| Al, Si | 30 | 13.3 | PET(002) | none | FlowCounter |
| P, S | 30 | 13.3 | Ge(111) | none | FlowCounter |
| K, Ca, Ti | 50 | 8 | LiF(200) | none | FlowCounter |
| Mn, Fe | 50 | 8 | LiF(200) | none | HighSense XE |

## S6 JAGUAR: Control Made Simple and Reliable

The S6 JAGUAR is the most powerful benchtop WDXRF spectrometer. Software and hardware of the S6 JAGUAR are ideally configured to enable best in-its-class results and optimal productivity for a multitude of industrial applications. Equipped with 400 W excitation power, the S6 JAGUAR offers outstanding sensitivity for a wide range of elements (F to U). The optimized sample excitation – especially for light elements – at low voltage and full 400 W power results in a 3-fold intensity improvement when compared to conventional 200 W systems.

The S6 JAGUAR sequential WDXRF is designed for industrial environments. Several key features guarantee an user-friendly operation and high instrument uptime, including:

- Ergonomic TouchControl™ interface for routine operation without PC peripherals
- SampleCare™ technology, protecting critical system components
- Intuitive interface of the SPECTRA.ELEMENTS with powerful user account control
- 24-position EasyLoad automated sample magazine for unattended analysis
- Sturdy design and robust, high quality components

## Preparation and Calibration

The preparation of pressed powder pellets is simple and fast. Careful pellet preparation enables high reproducibility and reliability, in particular for minor and trace element analyses. Thus, pressed pellets are typically preferred for rapid quality monitoring of limestone products and similar industrial commodities.

For this lab report, 8 g of milled specimen powder and 2 g of binding wax have been pressed for 20 s at 150 kN to produce 40 mm pellets (Figure 1).

The analytical conditions and the crystal setup used for this lab report are listed in Table 1. To account for the compositional variability of limestones, peak positions and optional background positions have been defined by fixed position.

Figure 2 shows a typical calibration curve. The calibration statistics listed in Table 2 reveal the excellent capabilities of the S6 JAGUAR for limestone analyses.

## Repeatability, Precision and Accuracy

A 21-hours repetition test demonstrates the impressive precision and stability of the S6 JAGUAR (Figure 3 and Table 3). After each analysis, the sample was unloaded from the sample chamber and re-loaded 1 hour later. Particularly remarkable are the very low standard deviations for Fe, Mg, Mn, and Na. A comparison of the measured concentrations to the certified composition reveals an excellent accuracy for these product critical elements (Table 4).

Figure 1: Pressed pellets.

Table 2: Compositional range and calibration statistics.

| [wt.%] | Concentration Range | $R^2$ |
|---|---|---|
| **$Na_2O$** | 0.003 – 0.46 | 0.991 |
| **MgO** | 0.24 – 17.88 | 0.999 |
| **$Al_2O_3$** | 0.05 – 2.4 | 0.972 |
| **$SiO_2$** | 0.45 – 12.4 | 0.999 |
| **$P_2O_5$** | 0.013 – 0.117 | 0.970 |
| **$SO_3$** | 0.016 – 0.13 | 0.981 |
| **$K_2O$** | 0.001 – 0.96 | 0.993 |
| **CaO** | 38.46 – 55.15 | 0.984 |
| **$TiO_2$** | 0.03 – 0.15 | 0.992 |
| **MnO** | 0.007 – 0.28 | 0.999 |
| **$Fe_2O_3$** | 0.04 – 2.43 | 0.996 |

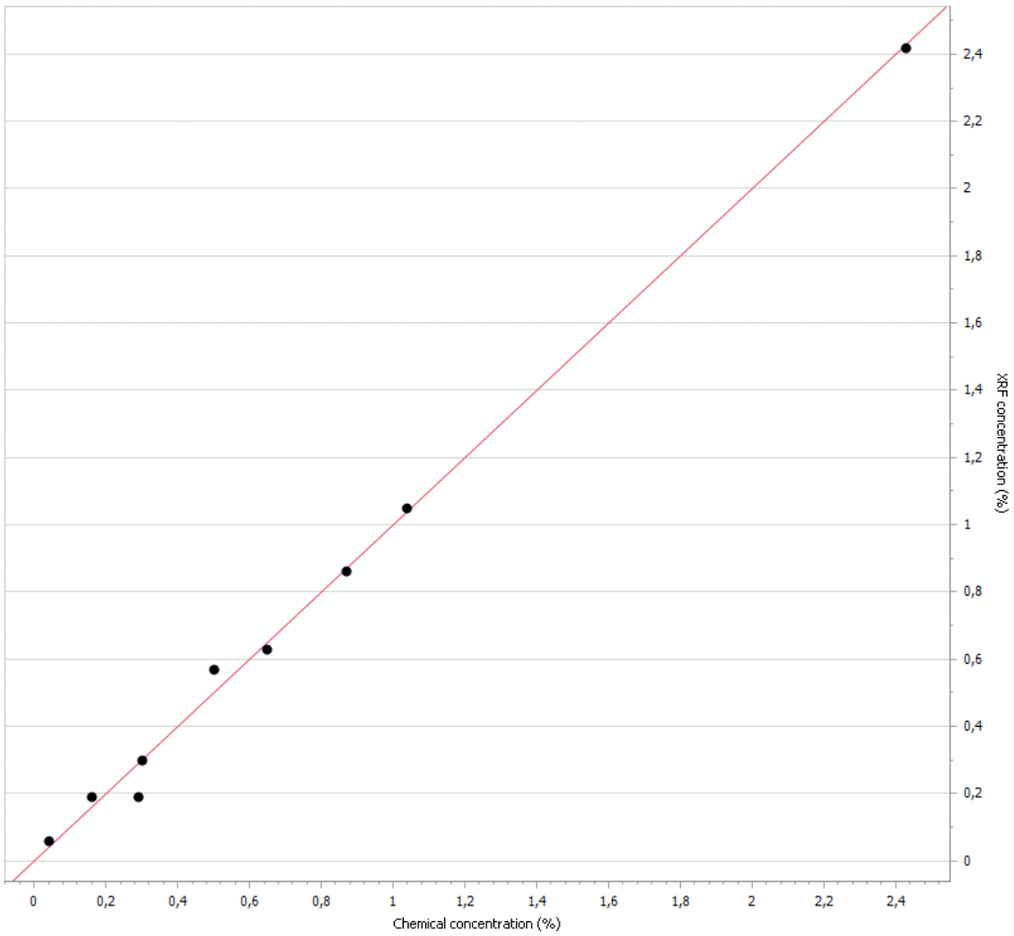


Figure 2: Calibration curve for Fe. Plot produced by Bruker's SpectraElements software.

Table 3: 21-hours Stability and Precision Test (sample not used for the calibration).

| [wt.%] | $Na_2O$ | MgO | $Al_2O_3$ | $SiO_2$ | $P_2O_5$ | $SO_3$ | $K_2O$ | CaO | $TiO_2$ | MnO | $Fe_2O_3$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Rep-1 | 0.327 | 2.08 | 5.75 | 19.72 | 0.057 | 0.252 | 0.964 | 36.65 | 0.281 | 0.046 | 3.23 |
| Rep-2 | 0.329 | 2.06 | 5.76 | 19.69 | 0.057 | 0.250 | 0.963 | 36.58 | 0.282 | 0.046 | 3.23 |
| Rep-3 | 0.328 | 2.07 | 5.73 | 19.69 | 0.056 | 0.250 | 0.965 | 36.57 | 0.281 | 0.045 | 3.23 |
| Rep-4 | 0.325 | 2.07 | 5.74 | 19.68 | 0.057 | 0.249 | 0.964 | 36.58 | 0.280 | 0.045 | 3.23 |
| Rep-5 | 0.332 | 2.07 | 5.74 | 19.66 | 0.056 | 0.250 | 0.965 | 36.67 | 0.284 | 0.046 | 3.23 |
| Rep-6 | 0.329 | 2.07 | 5.73 | 19.65 | 0.055 | 0.251 | 0.960 | 36.53 | 0.281 | 0.045 | 3.22 |
| Rep-7 | 0.324 | 2.07 | 5.72 | 19.67 | 0.056 | 0.248 | 0.964 | 36.60 | 0.283 | 0.045 | 3.23 |
| Rep-8 | 0.321 | 2.07 | 5.72 | 19.64 | 0.055 | 0.250 | 0.963 | 36.58 | 0.281 | 0.045 | 3.22 |
| Rep-9-21 | … | … | … | … | … | … | … | … | … | … | … |
| Rep-22 | 0.326 | 2.05 | 5.72 | 19.54 | 0.055 | 0.245 | 0.959 | 36.37 | 0.278 | 0.045 | 3.21 |
| **Average** | 0.326 | 2.07 | 5.73 | 19.64 | 0.056 | 0.248 | 0.962 | 36.56 | 0.281 | 0.045 | 3.22 |
| **Abs. Std. Dev.** | 0.003 | 0.010 | 0.015 | 0.040 | 0.001 | 0.002 | 0.002 | 0.063 | 0.001 | 0.001 | 0.006 |
| **Rel. Std. Dev. [%]** | 1.03 | 0.48 | 0.25 | 0.20 | 1.14 | 0.76 | 0.22 | 0.17 | 0.47 | 1.14 | 0.18 |

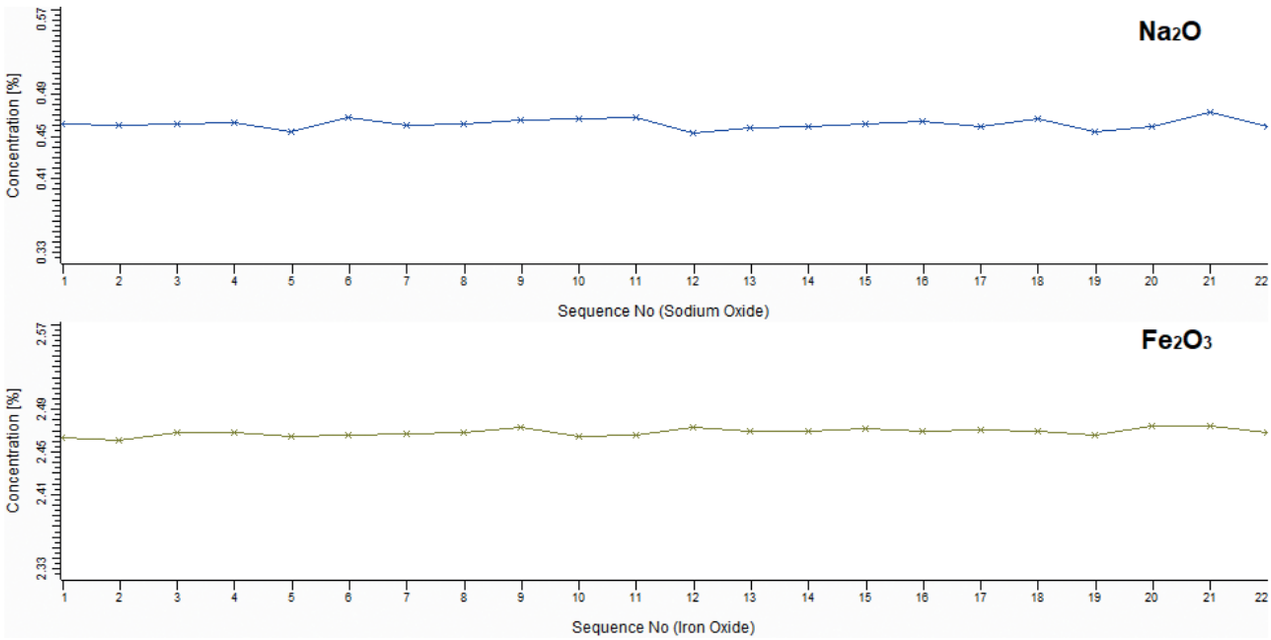


Figure 3: Repeatability for Na and Fe (t = 21 hours). Plots produced by Bruker's SpectraElements software.

Table 4: Analytical Accuracies for Product Critical Minor Elements.

| [wt.%] | $Na_2O$ | MgO | MnO | $Fe_2O_3$ |
|---|---|---|---|---|
| Certified | 0.460 | 5,97 | 0.280 | 2,43 |
| Measured | 0.461 | 5.87 | 0.286 | 2.47 |
| **Abs. Deviation** | 0.001 | 0.10 | 0.006 | 0.04 |

## Conclusion

The benchtop S6 JAGUAR is a powerful sequential WDXRF, which achieves excellent analytical results for critical minor and trace elements in limestone production. It can compete with full-size WDXRF systems on many levels, while the compact size of the S6 JAGUAR is comparable to energy dispersive XRF (EDXRF) units.

The outstanding analytical capabilities of the S6 JAGUAR stem from a smart combination of cutting-edge technical components and innovative geometric design, including the HighSense XE detector, the closely coupled beam path, and the 400 W X-ray tube. Low cost of operation is achieved by using the integrated vacuum mode instead of expensive helium. Ease-of-use and high system uptime are ensured, e.g. by the intuitive TouchControl interface and Bruker's SampleCare™ technology.

The S6 JAGUAR outperforms benchtop EDXRF systems and provides significantly better precision for most minor and trace elements (Mg, Al, P, K, Ti, Mn, and Fe) (Figure 3 and Table 3). Hence, if is important and precision is mission critical, the S6 JAGUAR is your system of choice!



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