
## Lab Report XRF 169

# S2 PUMA Series 2

- Verify the quality of your Nickel Ore in 2 minutes!

### Introduction

Nickel is of critical importance for the production of many steel types. It is also used in non-ferrous alloys as well as in the electronics and battery industries. For a long time the main source for the nickel were magmatic sulfide ore deposits. Nickel laterites are so-called low-grade Ni ores, which became an important source of Ni due to the easy access compare to alternative ore deposits and the large size of the laterite deposits (i.e. long life time of the mining project). For cost efficient, profitable nickel production processes, monitoring of the chemical composition of the extracted ore and its associated materials is inevitable. Besides the major elements, such as Ni, Fe, and Si, the elements Mg, Al, Ca, Ti, Cr, Mn, Co, Cu, and Zn typically occur in minor and trace amounts.

All these elements can be quantified by using the S2 PUMA Series 2 energy-dispersive X-ray fluorescence (EDXRF) spectrometer. This instrument enables fast and accurate control of both the mining process and the subsequent beneficiation processes. This lab report shows the performance of the S2 PUMA for the analysis of nickel laterite ore, prepared as pressed pellets.

## Instrument

The S2 PUMA Series 2, a versatile, high-performing benchtop EDXRF spectrometer, is an excellent analytical solution for a wide range of applications. The optimized beam path and the new HighSense silicon drift detector (SDD) ensure high sample throughput in combination with outstanding precision and accuracy.

The ergonomic, easy-to-use TouchControl™ interface of the instrument allows for independent routine operation without external PC. Combined with the high-duty air filter, the S2 PUMA is ready for harsh and dusty mining environments. Bruker's unique SampleCare™ technology protects important system components, such as X-ray tube and the detector. This guarantees high instrument uptime and ensures low cost of ownership.

A S2 PUMA Series 2 with standard HighSense detector and a 50 Watt Pd target X-ray tube was used for the measurements.

## Sample preparation

To enable fast process control, the samples were prepared as pressed pellets. Due to the simple and straightforward sample preparation, the analytical results are available within minutes after taking the sample. For the pressed pellets 10.0 g of sample material has been mixed with 2.0 g of wax binder for XRF and pressed for 15 s at 15 tons.

## Measurement parameters

The analytical conditions were optimized for highest sample throughput (Table 1). The advanced spectrometer technology of the S2 PUMA Series 2 compared to its predecessor allows us to use one instead of two analytical ranges (= one kV setting) and still reduce the counting time by 40%.

The samples have been measured under vacuum, which avoids the use of expensive helium as purging gas. This minimizes dramatically the costs per sample.

| Tube voltage [kV] | Tube current [µA] | Filter | Measurement time [s] |
|---|---|---|---|
| 25 | 150 | none | 60 |

Table 1: Measurement parameters

## Calibration

A set of 14 international certified reference materials (CRMs) were used to prepare the calibration for the 12 elements. The CRMs were prepared as pressed powder pellets. Table 2 shows the concentration ranges of the different nickel laterite CRMs used to carry out the calibration. Figure 1 and 2 show the excellent spectral resolution of the overlaid spectra for the Ni standards and the calibration curve, respectively.

| | Minimum Concentration | Maximum Concentration |
|---|---|---|
| Ni | 0.31 % | 2.89 % |
| MgO | 1.7 % | 27.4 % |
| $Al_2O_3$ | 1.6 % | 13.08 % |
| $SiO_2$ | 31.97 % | 48.08 % |
| CaO | 0.13 % | 3.11 % |
| $TiO_2$ | 0.02 % | 0.36 % |
| $Cr_2O_3$ | 0.65 % | 1.75 % |
| $Fe_2O_3$ | 12.72 % | 39.42 % |
| MnO | 0.11 % | 0.68 % |
| Co | 222 ppm | 899 ppm |
| Cu | 21 ppm | 91 ppm |
| Zn | 77 ppm | 327 ppm |

Table 2: Concentration ranges used for the nickel laterite calibration

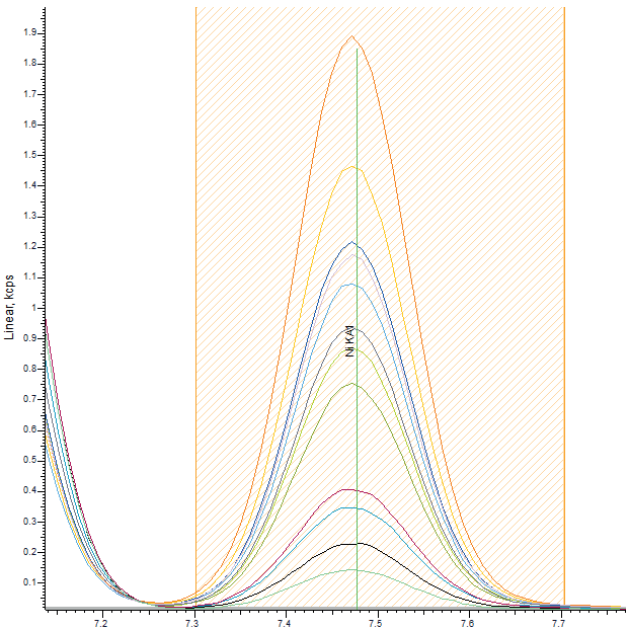


Figure 1: Overlaid spectra at the Ni Kα peak

Figures 2 and 3 show the calibration curves for Ni and $Fe_2O_3$, respectively.

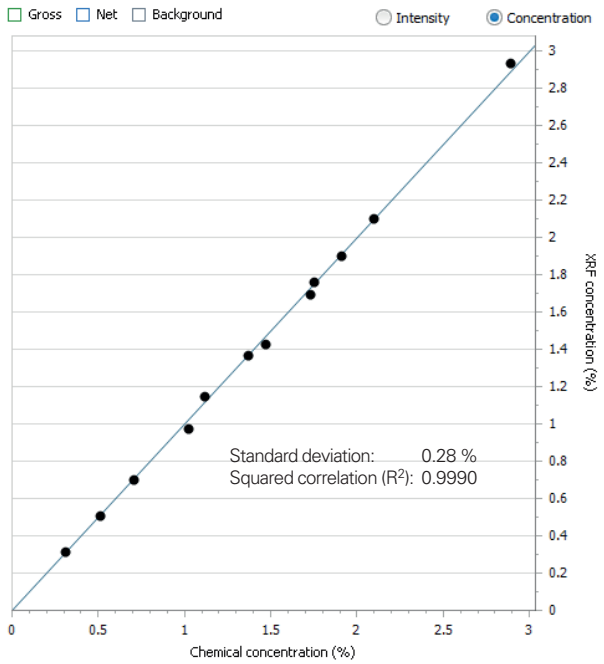


Figure 2: Calibration curve for Ni

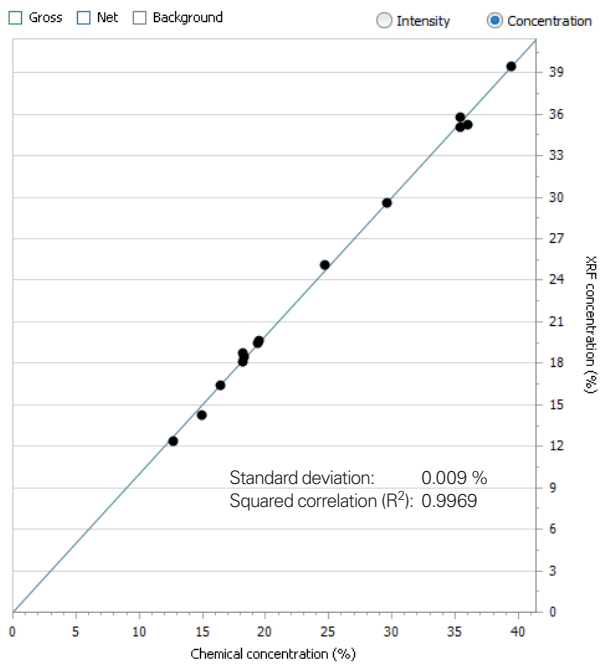


Figure 3: Calibration curve for $Fe_2O_3$

## Results

The precision of the S2 PUMA Series 2 is demonstrated by a repeatability test of the same nickel ore sample. For each measurement the sample was loaded into and un-loaded from the measurement chamber. Figure 4 graphically shows the repeatability of Ni measurements for a sample prepared as pressed pellet. The dotted lines show threefold standard deviations (3 sigma) of the measurements. For process control such threshold values can be defined for each element within the instrument software SPECTRA.ELEMENTS. These values indicate immediately if the result for a particular sample is out of specification.

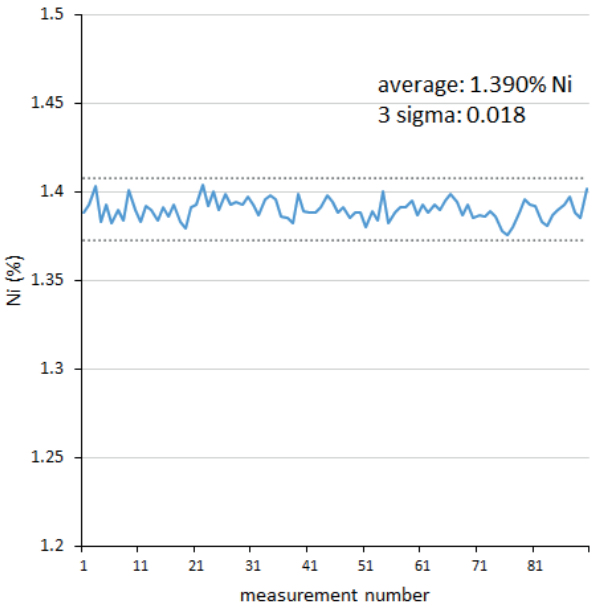


Figure 4: Repeatability for Ni in a typical nickel ore sample prepared as pressed pellet

| Sample ID | Gross measurement time* | Ni (%) | MgO (%) | $Al_2O_3$ (%) | $SiO_2$ (%) | CaO (%) | $TiO_2$ (%) | $Cr_2O_3$ (%) | MnO (%) | $Fe_2O_3$ (%) | Co (PPM) | Cu (PPM) | Zn (PPM) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Rep-1 | 00:01:37 | 1.388 | 24.05 | 2.094 | 48.24 | 0.32 | 0.03 | 0.77 | 0.21 | 14.02 | 375 | 32 | 119 |
| Rep-2 | 00:01:39 | 1.393 | 24.19 | 2.077 | 48.17 | 0.32 | 0.03 | 0.77 | 0.21 | 14.00 | 377 | 31 | 125 |
| Rep-3 | 00:01:39 | 1.403 | 24.00 | 2.102 | 48.10 | 0.32 | 0.03 | 0.77 | 0.22 | 14.05 | 416 | 35 | 119 |
| Rep-4 | 00:01:31 | 1.383 | 24.08 | 2.102 | 48.19 | 0.32 | 0.03 | 0.77 | 0.21 | 14.00 | 376 | 32 | 116 |
| Rep-5 | 00:01:39 | 1.393 | 24.00 | 2.107 | 48.13 | 0.32 | 0.03 | 0.77 | 0.22 | 14.05 | 374 | 33 | 116 |
| Rep-6 | 00:01:32 | 1.382 | 24.28 | 2.086 | 48.35 | 0.32 | 0.03 | 0.77 | 0.22 | 13.96 | 400 | 34 | 124 |
| Rep-7 | 00:01:39 | 1.39 | 24.18 | 2.102 | 48.26 | 0.32 | 0.03 | 0.77 | 0.22 | 13.97 | 369 | 33 | 122 |
| Rep-8 | 00:01:32 | 1.384 | 24.09 | 2.094 | 48.19 | 0.32 | 0.02 | 0.77 | 0.21 | 14.03 | 336 | 33 | 123 |
| … | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. |
| Rep-90 | 00:01:38 | 1.402 | 24.12 | 2.099 | 48.15 | 0.33 | 0.03 | 0.77 | 0.21 | 14 | 361 | 32 | 115 |
| **Average measure value** | **00:01:35** | **1.390** | **24.10** | **2.101** | **48.21** | **0.32** | **0.03** | **0.77** | **0.22** | **14.01** | **354** | **33** | **120** |
| **Abs. standard deviation** | | **0.006** | **0.07** | **0.013** | **0.09** | **0.00** | **0.00** | **0.00** | **0.00** | **0.03** | **21** | **1** | **5** |
| **Rel. Standard deviation** | | **0.43** | **0.28** | **0.60** | **0.18** | **1.43** | **7.82** | **0.23** | **2.29** | **0.24** | **6.03** | **3.27** | **4.09** |

Table 4: Results of the repeatability test. *Gross measurement time includes all steps from starting a measurement to seeing the results on the screen (e.g., loading, pumping, measuring, venting, unloading, processing).

## Conclusion

The achieved high precision demonstrates the excellent suitability of the S2 PUMA Series 2 to determine the elemental composition of nickel laterite ores.

The new detector technology of the S2 PUMA Series 2 allowed us to switch from a 2-range to a 1-range setting and still reduce the counting time by 40%. In combination with an optimized workflow and a faster software the gross measurements time was reduced from ~5 minutes down to 2 minutes per sample – without compromise on analytical performance (e.g., precision, accuracy).

In comparison to other spectroscopic techniques such as atomic absorption spectroscopy (AAS) or inductively coupled plasma optical emission spectrometry (ICP-OES) the sample preparation required for XRF is not time consuming and does not require any chemical sample digestion steps.




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