



Lab Report XRF 97

S2 RANGER

The Analysis of Metal Samples Using EQUA ALL

Introduction

The quick material identification and monitoring of the elemental composition is an important job in daily routine. Various parts and materials must be checked whether they are compliant to the specification or not. The fast analysis will help to solve quickly quality problems and to improve substantially the production quality. What would be more obvious than using a X-ray fluorescence spectrometer for this task?

The energy dispersive X-ray fluorescence spectrometer S2 RANGER (EDXRF) comes precalibrated with the standardless quantification solution EQUA ALL. This program allows the quick analysis of different kind of samples, no matter if the sample is a liquid, powdered or bulk sample. Without performing a calibration the sample composition is analyzed covering all elements starting from sodium to uranium. EQUA ALL is therefore a unique versatile toolbox for every analytical job beside the routine. This report demonstrates the analytical performance of the S2 RANGER for the analysis of metal samples using EQUA ALL.

Instrument

The S2 RANGER is an EDXRF benchtop spectrometer with TouchControl™, the easy-to-use touch screen interface. In the compact design of the S2 RANGER everything is integrated avoiding a mess of cables and pipe work. With more than 50 W excitation power and the direct excitation beam path the S2 RANGER fully utilizes the capabilities of the XFlash® silicon drift detector. Based on latest detector technology the XFlash detector provides excellent resolution of less than 129 eV for Mn $K\alpha_1$ at 100,000 cps without the need of external cooling or liquid nitrogen. Due to its high countrate capability the S2 RANGER either requires only short measurement times or achieves a very good analytical precision due to the excellent counting statistics.

The S2 RANGER is equipped with an integrated vacuum pump enabling the analysis of light elements in solid samples without the need of helium. As option the S2 RANGER can be equipped with an XY autosampler for the analysis of large samples series.

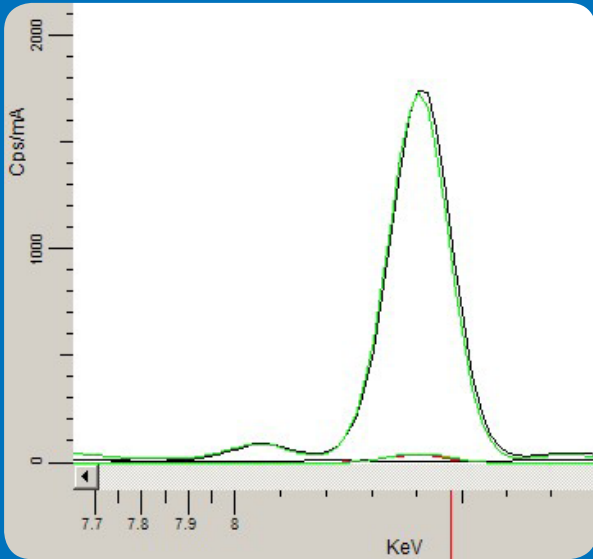


Figure 1: Example of a fitted spectrum

EQUA ALL with Direct Correlation

EQUA ALL is the solution for the standardless quantification of all different kind of samples analyzed with the S2 RANGER. During the evaluation, a theoretical spectrum is calculated based on the knowledge of the tube output, sensitivity, absorption and concentration of the elements in the sample. The software compares now the theoretical and measured spectra and modifies the element concentrations. With the new set of concentrations a new theoretical spectrum is calculated until a perfect match is achieved.

This brings essential benefits:

- Simple setup and calibration possible before installation, no measurement of pure element spectra needed
- Automatic and quick element identification for qualitative analysis
- All element lines are handled by only one free parameter – the element concentration
- Small number of free parameters (the element concentrations) to handle all parameters, such as line overlays and matrix corrections
- High degree of analytical flexibility

The quality of the fit and the calculated result can be checked graphically by displaying the measured and calculated spectra and the correlation factor R, shown in figure 1.

Preparation of the Samples

The surface of the metal sample influences significantly the quality of the results. It should be clean, flat and free of inclusions. Typically the samples have been polished using a 1000 mesh SiC grinding paper. Typical metal samples with the freshly prepared surface are shown in figure 2.



Figure 2: Example of prepared samples

Measurement Parameters

The measurement parameters in EQUA ALL are predefined depending on the sample preparation technique and well adjusted for the different sample types. The S2 RANGER runs the analysis of solid bulk samples under vacuum for enhanced light element performance.

Table 1: Measurement parameters

| Voltage [kV] | Beam filter | Life time [s] |
|-----------------|-------------|------------------|
| 20 | None | 100 |
| 40 | Al 500 µm | 30 |
| 50 | Cu 250 µm | 30 |

Table 1 shows the measurement parameters for metal samples. All samples are analyzed with three different conditions:

- Low kV and no filter for the analysis of light elements
- Medium kV and a 500 µm aluminium filter for heavier elements
- High kV and 250 µm copper filter for all element lines next to the tube lines. The filter improves significantly the peak to background ratio.

The excitation conditions are automatically adjusted to ensure an optimal analysis. The S2 RANGER current was automatically set to a count rate of 100,000 cps to avoid detector saturation maintaining the optimum spectral resolution. The measurements were performed using the predefined evaluation method EQUA METALS solution of the S2 RANGER with a total measuring time of 160 s.

Results

The tables 2 to 6 show the results for major and minor elements in different kind of metals applying the EQUA METALS evaluation method. These examples indicate the typical accuracy of the standardless evaluation solution EQUA ALL.

The precision of EQUA METALS and the S2 RANGER was determined by a 10-times measurement of the SS303 and STELLITE 6B samples. Tables 7 and 8 show the obtained precision data.

Table 7: Nickel alloy HAST C-4

| | Average | Standard deviation |
|-------|---------|--------------------|
| Si(%) | 0.56 | 0.02 |
| S(%) | 0.37 | 0.10 |
| Cr(%) | 17.30 | 0.09 |
| Mn(%) | 1.73 | 0.07 |
| Fe(%) | 70.35 | 0.09 |
| Co(%) | 0.40 | 0.04 |
| Ni(%) | 8.10 | 0.02 |
| Cu(%) | 0.38 | 0.01 |
| Mo(%) | 0.55 | 0.002 |

Table 8: Cobalt alloy STELLITE 6B

| | Average | Standard deviation |
|-------|---------|--------------------|
| Si(%) | 0.72 | 0.03 |
| Cr(%) | 30.28 | 0.14 |
| Mn(%) | 1.66 | 0.05 |
| Fe(%) | 0.78 | 0.08 |
| Co(%) | 58.68 | 0.09 |
| Ni(%) | 2.65 | 0.04 |
| Mo(%) | 0.81 | 0.003 |
| W(%) | 3.94 | 0.02 |

Discussion

The S2 RANGER with EQUA ALL is the solution for the quick characterization of all different kind of samples. Within less than three minutes the sample composition is clear. EQUA ALL can be easily adapted to own materials and is therefore a versatile tool for all non-routine tasks.

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Table 2: Stainless steel sample SS303

| | Certified | Measured |
|-------|-----------|----------|
| Si(%) | 0.58 | 0.54 |
| S(%) | 0.309 | 0.41 |
| Cr(%) | 17.21 | 17.24 |
| Mn(%) | 1.68 | 1.76 |
| Fe(%) | 70.88 | 70.4 |
| Co(%) | 0.16 | 0.44 |
| Ni(%) | 8.14 | 8.12 |
| Cu(%) | 0.36 | 0.38 |
| Mo(%) | 0.55 | 0.55 |

Table 3: Cr-Mo steel sample 1-1/4Cr-1/2Mo

| | Certified | Measured |
|-------|-----------|----------|
| Si(%) | 0.63 | 0.55 |
| Cr(%) | 1.22 | 1.36 |
| Mn(%) | 0.485 | 0.707 |
| Fe(%) | 96.700 | 96.4 |
| Cu(%) | 0.133 | 0.12 |
| Mo(%) | 0.47 | 0.467 |

Table 4: Tool steel sample TSS-5

| | Certified | Measured |
|-------|-----------|----------|
| Si(%) | 2.02 | 1.66 |
| V(%) | 0.2 | 0.197 |
| Cr(%) | 0.26 | 0.303 |
| Mn(%) | 0.82 | 1.06 |
| Fe(%) | 95.80 | 95.6 |
| Mo(%) | 0.31 | 0.306 |

Table 5: Nickel alloy HAST C-4

| | Certified | Measured |
|-------|-----------|----------|
| Si(%) | 0.52 | 0.70 |
| V(%) | 0.08 | 0.12 |
| Cr(%) | 21.9 | 21.5 |
| Mn(%) | 0.63 | 0.66 |
| Fe(%) | 18.35 | 18.2 |
| Co(%) | 1.53 | 1.62 |
| Ni(%) | 47.5 | 48.5 |
| Cu(%) | 0.12 | 0.26 |
| Nb(%) | 0.1 | 0.12 |
| Mo(%) | 8.6 | 8.03 |

Table 6: Cobalt alloy STELLITE 6B

| | Certified | Measured |
|-------|-----------|----------|
| Si(%) | 0.65 | 0.78 |
| Cr(%) | 29.9 | 30.15 |
| Mn(%) | 1.45 | 1.72 |
| Fe(%) | 0.73 | 0.91 |
| Co(%) | 59.0 | 58.54 |
| Ni(%) | 2.24 | 2.68 |
| Mo(%) | 0.78 | 0.81 |
| W(%) | 4.05 | 3.91 |



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