

# S4 PIONEER

## ANALYSIS OF OXYGEN IN COPPER

### Introduction

During the copper melting and refining process, the oxygen concentration in the produced copper must be reduced gradually to a very low level. Along with the oxygen concentration, material properties such as weakness and conductivity can be controlled. Therefore, a fast and reliable analytical method for process control and material testing is required. In this field, XRF presents itself as a fast and reliable tool, even for the daily routine analysis of oxygen in metals. This report demonstrates the analytical performance of the S4 PIONEER for this task.

### Instrument

The innovative concept of the S4 PIONEER (Fig. 1), is a new milestone in the development of wavelength dispersive XRF-instruments (WD-XRF). It combines the flexibility of a modern spectrometer with up to 10 primary beam filters, 4 collimators and 8 analyzer crystals with the advantage of 4 kW-excitation reduced to a very small amount of required floor space ( $\sim 0.8\text{m}^2$ ). The consequent compact design leads to a very high sensitivity, even for light elements like Oxygen, and, therefore, the lowest LLD's can be reached in a very short measuring time. For the determination of Oxygen in copper, the analyzer crystal OVO-55 was combined with the fine  $0.46^\circ$ -collimator. For excitation, the Rh-end-window X-ray tube with a standard  $75\mu\text{m}$  ultrathin Be-window was employed with 27 kV and 150 mA.



Figure 1: S4 PIONEER

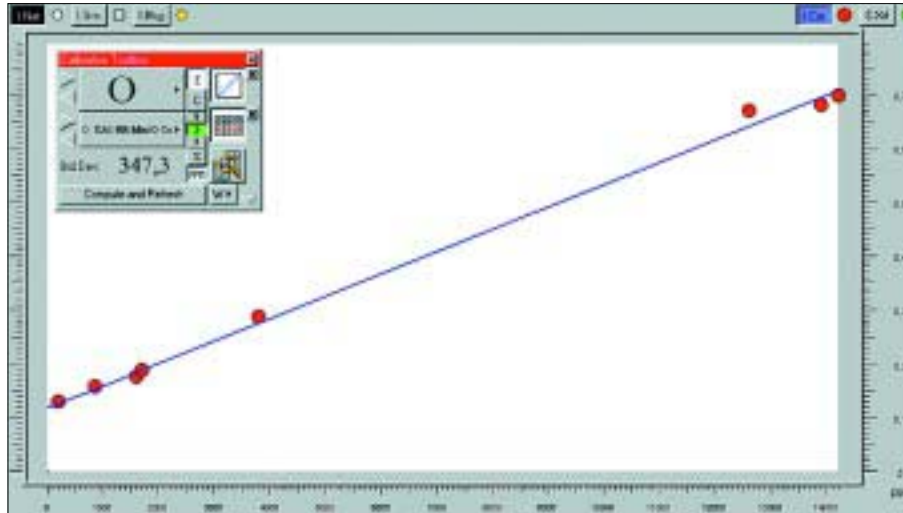


Figure 3: Calibration Curve for Oxygen K $\alpha$

### Sample Preparation

For the correct determination of Oxygen in copper samples (Fig. 2), it is necessary to achieve - after milling, grinding and polishing - a smooth and flat surface. To remove residuals from the polishing paste, it is useful to rinse the sample with acetone. It is important to measure the samples directly after preparation, otherwise the Oxygen concentration will increase due to oxidation on the surface.



Figure 2: Copper samples

### Calibration

During the measuring method, the Oxygen-K $\alpha$ -Line (Fig. 3) was carefully adjusted. The calibration was performed by measuring 8 copper samples with Oxygen concentrations from 0.02 to 1.42 %. The evaluation of the calibration was done with the unique comprehensive calibration tools of SPECTRA<sup>plus</sup> and a total measuring time of 100 s. was chosen.

Table 1: Details of Oxygen Calibration

Std.	Chem. Conc. (ppm)	XRF Conc. (ppm)	A.S.D. (ppm)	R.S.D. (%)
Cu 1	200	260	60	27
Cu 2	860	930	70	8
Cu 3	1600	1350	- 250	16
Cu 4	1700	1670	- 30	2
Cu 5	3800	4050	250	6
Cu 6	12600	13250	650	5
Cu 7	13900	13500	- 400	3
Cu 8	14200	14000	- 200	2

Table 2: Reproducibility for one sample with different measuring times (20 s., 60 s. and 100 s. total measuring time)

<b>Std.</b>	<b>M.T. 20 s Conc. (%)</b>	<b>M.T. 60 s Conc. (%)</b>	<b>M.T. 100 s Conc. (%)</b>
Repetition 1	1.41	1.42	1.41
Repetition 2	1.42	1.42	1.42
Repetition 3	1.41	1.43	1.42
Repetition 4	1.36	1.44	1.42
Repetition 5	1.41	1.43	1.41
Repetition 6	1.43	1.42	1.41
Repetition 7	1.43	1.47	1.44
Repetition 8	1.40	1.40	1.40
Repetition 9	1.40	1.39	1.40
Repetition 10	1.39	1.41	1.40
Repetition 11	1.43	1.40	1.40
Repetition 12	1.42	1.42	1.40
Repetition 13	1.43	1.41	1.42
Repetition 14	1.44	1.43	1.42
Repetition 15	1.37	1.43	1.44
Repetition 16	1.42	1.43	1.42
Average	1.41	1.42	1.42
Std. Dev abs.	0.02	0.02	0.01
Std. Dev rel.	1.7	1.3	0.9

## Results

Details of the calibration are shown in Table 1. The corrected net intensities were used for the evaluation. Figure 2 presents the regression curve, whereby the corrected intensities are displayed with red dots. The Lower Limit of Detection (LLD) is 100 ppm ( $3\sigma$ , 100 s.). To evaluate how fast and reproducible the S4 PIONEER is for the daily routine analysis of Oxygen, a sample was analysed 16 times with different total measuring times of 20 s., 60 s. and 100 s. The results are presented in Table 2.

Even for very short measuring times, the S4 PIONEER enables the reliable determination of Oxygen

in copper. Depending on the requirements of accuracy and precision for process control in daily routines, the measuring time is determined and directly influenced by the counting statistics as can be seen from the results.

## Conclusions

The S4 PIONEER provides analytical performance with a high analyzing speed, high accuracy and the best reproducibility - even for the light element Oxygen - in a metal matrix. The S4 PIONEER presents itself as an important tool in process control and material testing to determine even light elements in addition to other traces in metal.

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