Lab Report XRF 125

S2 PUMA

● Analysis of High- and Low-Si Aluminum

Introduction

Aluminum is one of the most widely used metals worldwide because of its sought after properties. It has a low specific weight, it is corrosion resistant, and has a high conductivity and hence is used in transportation, packaging, and for high-voltage power lines.

Alloying aluminum with other metals such as silicon or magnesium is necessary for the reduction of melting temperatures and for heat-treatability to yield higher strengths. Although the metal generally is used alloyed, some impurities are undesired. Particularly iron is a troublesome addition, because of the formation of its intermediate phases during cooling which reduce ductility and machinability of the material. Iron contamination becomes even more significant for recycled aluminum because this process is often a down-cycling.

Innovation with Integrity

XRF

Fast and reliable quality control with the S2 PUMA

The S2 PUMA is the high-performing benchtop energy-dispersive X-ray fluorescence (EDXRF) spectrometer for a wide range of applications. It is ideally suited for quality control of aluminium samples due its XY Autochanger for unattended analysis of large series of process samples. Its HighSense™ beam path geometry with the XFlashTM SDD detector ensures high sample through-put in combination with best precision, optimal accuracy, and high spectral resolution (see Figure 1). Even higher sample loads and continuous process control can be achieved by the Automation option which allows for the professional integration in automated laboratory environments.

The instrument is perfectly equipped for industrial environments due to its ergonomic TouchControl™ interface for the independent routine operation without any PC peripherals. The dedicated instrument protection system SampleCare™, the powerful user account control of the spectrometer's software SPECTRA. ELEMENTS, and sturdy design guarantee a high instrument uptime.

Compound	Concentration Range [wt.-%]	Calibration Standard Deviation 3σ [wt.-%]
Mg	0.19 - 8.32	0.0916
Si	0.62 - 12.89	0.1422
Ti	0.040 - 0.360	0.0042
Cr	0.030 - 0.170	0.0067
Mn	0.020 - 0.400	0.0038
Fe	0.380 - 1.090	0.0217
Ni	0.060 - 0.660	0.0141
Cu	0.070 - 2.56	0.0361
Zn	0.030 - 0.880	0.0060
Sn	0.040 - 0.190	0.0009
Pb	0.010 - 0.120	0.0022

Table 1: Details on calibration data

Calibration Data

For this report ten aluminium reference samples have been used to set-up a calibration on the S2 PUMA XY Autochanger with Ag LE X-ray tube. The samples have been obtained with a coquille and have been cast as a cylinder with a diameter of 40 mm, and then cut into disk of 4 - 13 mm height. The surfaced to be analyzed was mill-cut and polished.

Analysis measurement time was 300 s and all analyses have been performed under vacuum for optimal light element detection and lower operating costs not requiring helium flushing. Peak and background positions have been defined by fixed position, integration (see Figure 1),

and peak height or lower envelope, respectively. See Table 1 for the ranges and the standard deviations of the elements of the reference samples used for calibration.

The exemplary calibration curve of Si (see Figure 2) shows the excellent analytical abilities of the S2 PUMA with a strong linear association (R^2=0.99941) and 3σ standard deviation of 0.1422 % for a concentration range of 12.3 %. Aluminium was calculated as balance.
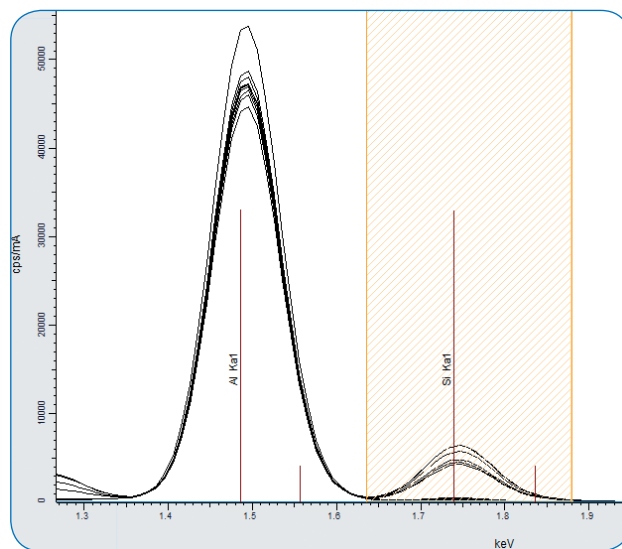

Figure 1: Excellent spectral resolution of the Al and Si Kα peaks

Analytical Precision

To demonstrate the impressive analytical stability of the S2 PUMA, the measurement of one sample was repeated 10 times every 14 minutes (see Table 2 for the measurement conditions) whereby the sample was unloaded from the sample chamber after every analysis. The analytical statistics of this repetition test on the S2 PUMA reveal very low relative standard deviations and indicate very precise measurements. The statistics together with the analysis results are given in Table 3.

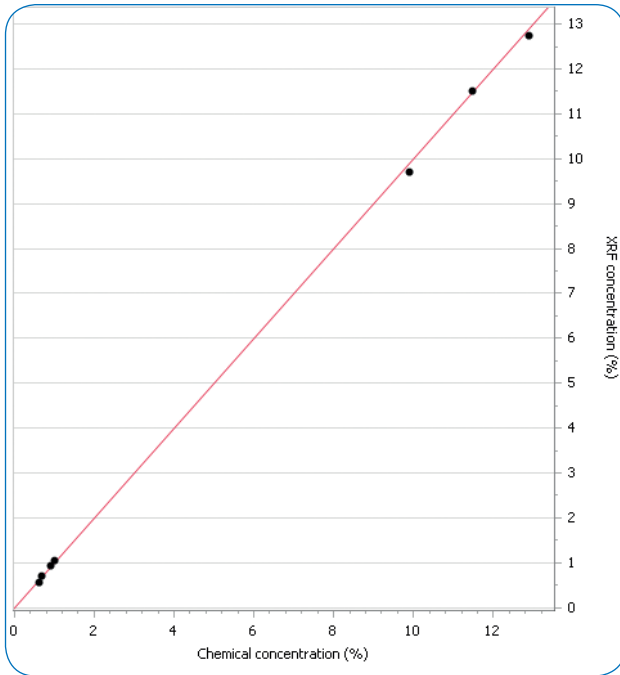| Voltage [kV]	Analyzed Elements	Filter	Measurement Time [s]
20	Mg, Si	none	100
40	Ti, Cr, Mn, Fe, Ni, Cu, Zn, Pb	Al (500 µm)	100
50	Sn	Cu (250 µm)	100

Table 2: Measurement conditions

Figure 2: Calibration curve for Si in SPECTRA.ELEMENTS

Measurement Accuracy

The superior analytical accuracy of the S2 PUMA is demonstrated below. The results of a measurement of an aluminium sample and the reference values are listed in Table 4.

The values listed show the very high accordance of measured and reference concentrations of the analyzed sample. For the aluminium alloys the important elements Mg, Si, and Fe are depicted particularly well.

	Mg [wt.-%]	Al [wt.-%]	Si [wt.-%]	Ti [wt.-%]	Cr [wt.-%]	Mn [wt.-%]	Fe [wt.-%]	Ni [wt.-%]	Cu [wt.-%]	Zn [wt.-%]	Sn [wt.-%]	Pb [wt.-%]
Rep-1	0.24	85.48	11.54	0.045	0.032	0.400	0.603	0.079	0.933	0.584	0.014	0.050
Rep-2	0.24	85.54	11.50	0.040	0.029	0.398	0.603	0.079	0.932	0.582	0.014	0.050
Rep-3	0.24	85.47	11.54	0.049	0.038	0.400	0.604	0.078	0.933	0.582	0.015	0.050
Rep-4	0.24	85.51	11.52	0.037	0.031	0.397	0.606	0.078	0.935	0.583	0.014	0.051
Rep-5	0.23	85.50	11.53	0.040	0.033	0.398	0.608	0.079	0.936	0.583	0.013	0.050
Rep-6	0.24	85.50	11.51	0.041	0.033	0.398	0.608	0.079	0.936	0.584	0.013	0.050
Rep-7	0.24	85.54	11.49	0.044	0.028	0.396	0.602	0.079	0.935	0.584	0.016	0.051
Rep-8	0.24	85.54	11.49	0.038	0.032	0.399	0.604	0.078	0.931	0.582	0.013	0.050
Rep-9	0.25	85.50	11.52	0.042	0.028	0.397	0.606	0.080	0.933	0.585	0.014	0.051
Rep-10	0.24	85.51	11.51	0.037	0.029	0.397	0.606	0.079	0.933	0.583	0.015	0.051
Avg.	**0.24**	**85.51**	**11.52**	**0.041**	**0.031**	**0.398**	**0.605**	**0.079**	**0.934**	**0.583**	**0.014**	**0.050**
Abs. Std. Dev.	**0.00**	**0.02**	**0.02**	**0.004**	**0.003**	**0.001**	**0.002**	**0.001**	**0.002**	**0.001**	**0.001**	**0.001**
Rel. Std. Dev.	**1.96**	**0.03**	**0.16**	**9.28**	**9.77**	**0.34**	**0.35**	**0.80**	**0.18**	**0.18**	**7.05**	**1.02**

Table 3: Excellent results of the precision test on the S2 PUMA

	Mg [wt.-%]	Al [wt.-%]	Si [wt.-%]	Ti [wt.-%]	Cr [wt.-%]	Mn [wt.-%]	Fe [wt.-%]	Ni [wt.-%]	Cu [wt.-%]	Zn [wt.-%]	Sn [wt.-%]	Pb [wt.-%]
Reference	0.37	85.95	9.90	0.220	0.170	0.020	1.090	0.660	0.720	0.670	0.120	0.110
Measured	0.37	85.99	9.81	0.227	0.191	0.062	1.105	0.644	0.704	0.676	0.121	0.100
Abs. Dev.	**0.00**	**0.04**	**0.09**	**0.007**	**0.021**	**0.042**	**0.015**	**0.016**	**0.016**	**0.006**	**0.001**	**0.010**

Table 4: Accuracy test of the certified reference material measured against the calibration

Summary

The high-power X-ray tube together with the HighSense beam path and our reliable vacuum pump deliver excellent analytical performance with lowest measurement times and operating cost. Industry-leading features such as the XY Autochanger sample handling with optional integration into automated environments and TouchControl make continuous process control as well as product quality assurance easy and quick.

Links
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