SPECTRO XRF Report

Nr. XRF-70, Rev. 2

SPECTRO xSORT XHH03

Analysis of Solid Metal Samples

SPECTRO Analytical Instruments GmbH, Kleve, Germany

Summary

Excitation of the fluorescence radiation in the sample by an X-ray tube has been optimized so that extremely short measuring times with a high sample throughput are possible. Specially developed detector technology, based on SDD, enables high signal throughput at high resolution; allowing for a unique analytical flexibility and a considerably accelerated signal processing.

With the SPECTRO xSORT it is possible to conduct laboratory-like analyses in a single measuring cycle that lasts only 2 seconds, except for the light elements Mg, Al, Si, P and S they require a longer measurement time. Only 10 additional seconds are necessary for the sorting of various aluminum and magnesium alloys. During this process even the light elements Al, Mg, Si, P and S are measured in air – without complicated additional techniques like helium flushing or vacuum, simplifying operation decidedly.

The analysis screen supplies the operator with all of the necessary information at a single glance and can display the measurement result in different views - also during measurement. The analytical results are stored and can be displayed later, printed and, after transfer to an external computer, processed with the “Result Manager” software.

1. Introduction

SPECTRO products are viewed around the world as standards for onsite metal analysis. No other company possesses comparable know-how and similarly comprehensive experience in this area.

As one of the leading international manufacturers of XRF instruments, laboratory analyses with extremely powerful X-ray fluorescence spectrometers is also an area in which SPECTRO excels.
With the new SPECTRO xSORT, SPECTRO introduces a compact energy dispersive X-ray fluorescence spectrometer for the continuous, fatigue-free handheld analysis and sorting of metals.

It makes use of innovative efficient components for excitation and detection. Together with other top quality modules, they are the basis for the unique precision, speed and safety of this system; combining the experience with onsite metal analysis and the demands for laboratory quality measurement results.

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2. Instrumentation

To test an alloy group, SPECTRO calibration modules are preinstalled on the SPECTRO xSORT.
There are three different modules available.
For example, the testing of stainless steels requires the “Standard” calibration module.
The measurement of magnesium or aluminum based alloys requires the calibration module “Light Elements” and the measurement of precious metal alloys, the module “Precious Metals”.

A single measuring cycle lasts only 2 seconds for the modules „Standard“ and „Precious Metals“. Only 10 seconds in addition are necessary for both the sorting of various aluminum and magnesium alloys and the analysis of different alloys including Al, Si, P and S with the module “Light Elements”.

3. Technical Data

Detector

- High resolution silicon drift detector (SDD)

Excitation

- X-ray tube with Rh anode
- up to 50 kV tube voltage

Operation

A sample name can be entered and then the start button can be released after the first result is displayed on the screen, or continue to press until the preset measurement time is elapsed.

Spectrometer Control

- ePC including Windows mobile operating system
- Automatic drift correction
- SD card slot
- Bluetooth, Wireless LAN, USB

Dimensions and Weight

- Height: 270 mm (10,7“)
- Width: 93 mm (3.7”)
- Depth: 230 mm (9.1“)
- Weight Instrument: 1.64 kg (3.62 lbs) including Battery pack

Power/ Electrical Connection

- Battery operation with Li-Polymer batteries, rechargeable (4 h typical duty cycle)
- 100 - 240 V, +/- 10 %, 50 / 60 Hz (AC adapter/ charger)
- 11 W during analysis
- 6 W Standby mode
- 3 W Offline mode

Options

- Additional battery pack
- Barcode reader (Bluetooth)
- Printer (Bluetooth)
- Wireless data transfer kit
- Small parts adapter
- Video camera
- Internal GPS receiver
- Docking station

Accessories (Included)

- Transport case
- Instrument/battery holster
- Two battery packs
- Charger/ AC adapter
- USB cable
- Consumables

Software

- Eval-Server for data treatment and fundamental parameter analysis
- ICAL (Intelligent Calibration Logic)
- Result Manager: post-processing of stored measurement values and protocols

Analysis Mode

- Automatic fingerprint analysis
- Automatic average calculation
- Calculation standard deviation and relative standard deviation

Grade ID Mode

- Grade identification based on stored library of grade specifications
- Grade verification, based on selected grade specification
- Optional display of analysis and/or grade identification
- Grade library easily edited and expanded

Sorting Mode

- Fast pass/fail assessment based on a reference sample
- Symmetrical and asymmetrical tolerances allowed
- Three modes of operation to suit different sorting tasks
- Log reports

Documentation

- Basic operator manual
- Back up on USB-flash drive

4. Sample Preparation

To enhance accuracy samples can be prepared with grinding paper (approx. 60 or 80 grit aluminum or zirconium oxide) or a milling machine can be used.
For grade identification in most cases no sample preparation is necessary.
On most finished parts sample preparation is not necessary.

5. Detection Limits

In EDXRF the detection limits are sample specific. They are influenced by matrix effects, line overlaps as well as signal processing of the detector.

The following data are related to measurements of pure single element standards at a measuring time of 10 seconds for elements Z ≥ 21 respectively 20 seconds for Mg, Al, Si, P and S using a 4 µm polypropylene film between sample and measuring head.

To estimate the detection limits at a measuring time of 2 seconds (instead of 10 seconds) the given values have to be multiplied by a factor of 2.2. The detection limits of Mg, Al, Si, P and S have to be multiplied by 1.4 to estimate the detection limits at a measuring time of 10 seconds.
In consideration of line overlaps, pile ups and other detector artifacts the Eval-Server calculates detection limits and displays them as analysis result.

The LOD is calculated as 3 times standard deviation (SD).

Table 1: Iron base

Element	LOD [%]
Al	0.4
Cd	0.02
Co	0.09
Cr	0.01
Cu	0.01
Mn	0.02
Mo	0.007
Nb	0.007
Ni	0.02
P	0.03
Pb	0.02
S	0.01
Sb	0.02
Si	0.08
Sn	0.03
Ti	0.02
V	0.01
W	0.03
Zn	0.007
Zr	0.005

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Table 2: Nickel base

Element	LOD [%]
Ag	0.03
Al	0.3
Cd	0.03
Co	0.01
Cu	0.06
Cr	0.02
Fe	0.01
Hf	0.05
Mn	0.01
Mo	0.01
Nb	0.01
Pb	0.04
Re	0.06
Sb	0.04
Si	0.07
Sn	0.04
Ta	0.06
Ti	0.01
V	0.01
W	0.04
Zn	0.04

Table 3: Titanium base

Element	LOD [%]
Al	0.4
Cd	0.02
Co	0.02
Cr	0.01
Cu	0.007
Fe	0.006
Mn	0.009
Mo	0.007
Nb	0.003
Ni	0.009
Pd	0.02
Ru	0.01
Si	0.08
Sn	0.02
Ta	0.07
V	0.1
Zn	0.005
Zr	0.004

Table 4: Cobalt base

Element	LOD [%]
Cd	0.03
Cr	0.01
Cu	0.01
Fe	0.02
Mn	0.01
Mo	0.007
Nb	0.007
Ni	0.07
Pb	0.03
Si	0.07
Sn	0.04
Ta	0.07
Ti	0.01
V	0.01
W	0.04
Zn	0.009
Zr	0.009

Table 5: Copper base

Element	LOD [%]
Ag	0.03
Al	0.3
As	0.01
Au	0.04
Bi	0.03
Cd	0.03
Co	0.007
Fe	0.01
Mn	0.008
Ni	0.01
P	0.02
Pb	0.04
Sb	0.06
Se	0.009
Si	0.07
Sn	0.05
Te	0.04
Ti	0.01
V	0.01
Zn	0.08

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Table 6: Aluminum base

Element	LOD [%]
Ag	0.003
Bi	0.003
Cd	0.004
Co	0.003
Cr	0.004
Cu	0.002
Fe	0.005
Mg	0.4
Mn	0.002
Mo	0.001
Nb	0.001
Ni	0.008
Pb	0.003
Sb	0.005
Sc	0.01
Si	0.05
Sn	0.003
Ti	0.01
V	0.006
Zn	0.01
Zr	0.001

Table 7: Magnesium base

Element	LOD [%]
Ag	0.003
Al	0.2
Cd	0.003
Ce	0.02
Co	0.01
Cr	0.002
Cu	0.007
Fe	0.004
La	0.02
Mn	0.002
Mo	0.0008
Nb	0.001
Nd	0.04
Ni	0.008
Pb	0.009
Sb	0.004
Si	0.01
Sn	0.004
Th	0.003
Ti	0.008
Y	0.003
Zn	0.009
Zr	0.001

Table 8: Lead base

Element	LOD [%]
Ag	0.1
As	0.1
Bi	0.2
Cd	0.02
Co	0.03
Cu	0.02
Fe	0.03
Cr	0.03
Mn	0.04
Ni	0.03
Pd	0.1
Sb	0.04
Sn	0.3
Ti	0.05
V	0.04
Zn	0.02

Table 9: Tin base

Element	LOD [%]
Ag	0.08
Al	0.1
Cd	0.03
Co	0.01
Cr	0.01
Cu	0.008
Fe	0.01
Ge	0.01
Mn	0.01
Mo	0.004
Nb	0.004
Ni	0.02
Pb	0.02
Pd	0.02
Sb	0.06
Si	0.1
Ti	0.06
V	0.02
Zn	0.008

Table 10: Zinc base

Element	LOD [%]
Ag	0.04
Al	0.4
Au	0.04
Bi	0.04
Cd	0.04
Co	0.01
Cr	0.01
Cu	0.02
Fe	0.008
Mn	0.01
Ni	0.02
P	0.04
Pb	0.04
Sb	0.05
Se	0.01
Si	0.08
Sn	0.05
Ti	0.02
V	0.02

Table 11: Zirconium base

Element	LOD [%]
Ag	0.08
Al	0.4
Cd	0.07
Co	0.02
Cu	0.02
Cr	0.02
Fe	0.03
Hf	0.03
Mn	0.02
Mo	0.1
Nb	0.03
Ni	0.02
Pb	0.03
Sb	0.08
Si	0.1
Sn	0.08
Ta	0.03
Ti	0.04
V	0.04
W	0.03
Zn	0.008

Table 12: Gold base

Element	LOD [%]
Ag	0.04
Co	0.02
Cu	0.02
Cr	0.04
Fe	0.03
Mn	0.02
Ni	0.02
Pd	0.2
Pt	0.2
Rh	0.05
Ru	0.05
Ti	0.05
V	0.04
Zn	0.05

Table 13: Platinum base

Element	LOD [%]
Ag	0.03
Au	0.3
Co	0.01
Cu	0.02
Cr	0.03
Fe	0.02
Mn	0.03
Ni	0.02
Pd	0.04
Rh	0.05
Ru	0.05
Ti	0.04
V	0.05
W	0.6
Zn	0.01

6. Some Typical Measurement Examples

Analysis pauses are used to correct possible gain shifts. This procedure guarantees reproducible and accurate results as shown in the tables at different and changing ambient temperatures.
(ASD: Analytical Standard Deviation)
All data in mass percentage

Table 14: Aluminum base

3003 AL				
		Analysis [%]	ASD [%]	Certificate [%]
Al	10s	97.18		97.77
Cu	5s	0.084	0.002	0.085
Fe	5s	0.632	0.006	0.64
Mn	5s	1.06	0.01	1.10
Si	10s	0.31	0.04	0.25
	30s	0.32	0.02	
Zn	5s	0.019	0.001	0.020

4145 AB				
		Analysis [%]	ASD [%]	Certificate [%]
Al	10s	84.80		84.24
Cr	5s	0.038	0.003	0.050
Cu	5s	4.00	0.01	4.12
Fe	5s	0.526	0.005	0.54
Mg	10s	<0.7		0.050
	30s	<0.5		
Mn	5s	0.042	0.002	0.050
Si	10s	10.20	0.11	10.66
	30s	10.26	0.06	
Zn	5s	0.051	0.002	0.058

5083 AF				
		Analysis [%]	ASD [%]	Certificate [%]
Al	10s	93.62		93.49
Cr	5s	0.156	0.004	0.15
Cu	5s	0.071	0.002	0.078
Fe	5s	0.341	0.005	0.34
Mg	10s	4.8	0.3	4.85
	30s	5.0	0.2	
Mn	5s	0.710	0.007	0.74
Si	10s	0.16	0.03	0.17
	30s	0.20	0.02	
Ti	5s	0.031	0.003	0.027
Zn	5s	0.051	0.001	0.050

6061 SS				
		Analysis [%]	ASD [%]	Certificate [%]
Al	10s	96.84		97.81
Cr	5s	0.249	0.005	0.229
Cu	5s	0.308	0.003	0.30
Fe	5s	0.348	0.004	0.35
Mg	10s	1.4	0.3	1.00
	30s	1.1	0.2	
Mn	5s	0.047	0.003	0.052
Si	10s	0.63	0.04	0.64
	30s	0.65	0.02	
Ti	5s	0.042	0.003	0.037
Zn	5s	0.081	0.002	0.080

Table 15: Stainless and heat resisting steel

AISI 303				
		Analysis [%]	ASD [%]	Certificate [%]
Mn	2s	1.86	0.07	1.87
P	10s	0.06	0.03	0.032
	30s	0.07	0.02	
S	10s	0.38	0.02	0.378
	30s	0.33	0.01	
Si	10s	0.70	0.08	0.634
	30s	0.55	0.05	
Cr	2s	17.5	0.1	17.35
Ni	2s	8.97	0.11	8.64
Mo	2s	0.57	0.02	0.58

AISI 416				
		Analysis [%]	ASD [%]	Certificate [%]
Mn	2s	0.37	0.06	0.35
P	10s	<0.05		0.026
	30s	0.03	0.01	
S	10s	0.29	0.02	0.29
	30s	0.32	0.01	
Si	10s	0.39	0.07	0.37
	30s	0.41	0.04	
Cr	2s	12.22	0.08	12.25

Table 16: Aerospace materials

17-7PH				
		Analysis [%]	ASD [%]	Certificate [%]
Mn	2s	0,.62	0.07	0.76
P	10s	0.05	0.02	0.023
	30s	<0.03		
S	10s	<0.04		0.0019
	30s	<0.02		
Si	10s	0.43	0.07	0.361
	30s	0.27	0.04	
Cr	2s	17.2	0.1	16.94
Ni	2s	7.4	0.1	7.22
Al	10s	1.3	0.3	1.19
	30s	1.0	0.2	

Table 17: Materials with special physical properties

Nitronic 60				
		Analysis [%]	ASD [%]	Certificate [%]
Mn	2s	8.7	0.1	8.58
Si	10s	3.53	0.13	3.67
	30s	3.88	0.08	
Cr	2s	16.3	0.1	16.37
Ni	2s	8.6	0.1	8.5
Fe	2s	61.7	0.2	61.71

Table 18: 11% Manganese steel

SS-CRM No. 493/1				
		Analysis [%]	ASD [%]	Certificate [%]
Mn	2s	11.4	0.1	11.24
P	10s	<0.061		0.095
	30s	0.11	0.02	
S	10s	<0.05		0.022
	30s	0.04	0.02	
Si	10s	0.81	0.09	0.86
	30s	0.83	0.05	
Cr	2s	0.12	0.02	0.14
Ni	2s	2.92	0.08	2.97
Mo	2s	0.90	0.02	0.94

Table 19: Copper base

CDA 623				
		Analysis [%]	ASD [%]	Certificate [%]
Al	10s	9.37	0.58	9.19
	30s	9.84	0.35	
Mn	2s	0.166	0.015	0.16
Fe	2s	2.10	0.04	2.13
Ni	2s	0.07	0.02	0.075
Cu	2s	87.8		88.4

CDA 706				
		Analysis [%]	ASD [%]	Certificate [%]
Mn	2s	0.596	0.023	0.62
Fe	2s	1.35	0.040	1.30
Co	2s	<0.029		0.013
Ni	2s	10.16	0.09	10.03
Cu	2s	87.6		87.9
Zn	2s	0.15	0.05	0.082

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Table 20: Nickel base

Monel R405				
		Analysis [%]	ASD [%]	Certificate [%]
Si	10s	0.089	0.041	0.046
	30s	0.067	0.022	
Mn	2s	1.03	0.03	1.03
Fe	2s	1.26	0.03	1.31
Co	2s	<0.042		0.02
Ni	2s	65.2		64.8
Cu	2s	32.3	0.2	32.3

RENE 142				
		Analysis [%]	ASD [%]	Certificate [%]
Al	10s	5.18	0.47	6.13
	30s	5.81	0.28	
Cr	2s	6.58	0.080	6.98
Co	2s	12.02	0.10	11.81
Ni	2s	58.9		57.61
Mo	2s	1.53	0.03	1.51
Hf	2s	1.22	0.11	1.34
Ta	2s	6.01	0.18	6.3
W	2s	5.03	0.19	4.92
Re	2s	2.46	0.16	2.6

Table 21: Cobalt base

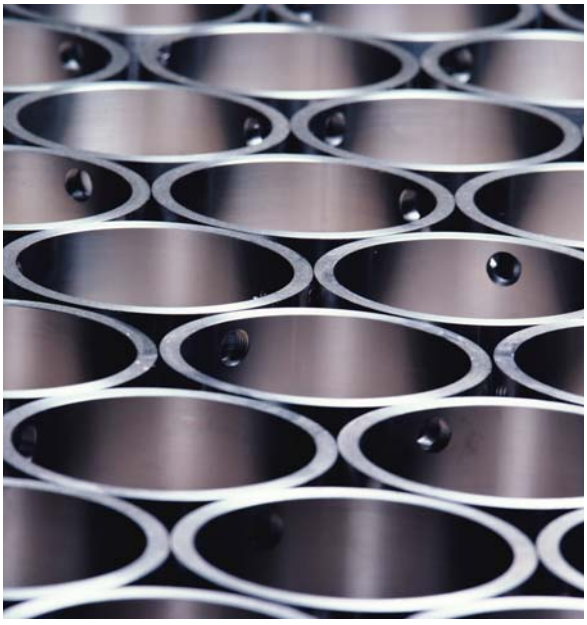
Ultimet 1233				
		Analysis [%]	ASD [%]	Certificate [%]
Cr	2s	25.4	0.20	25.4
Mn	2s	0.780	0.061	0.79
Fe	2s	3.30	0.070	2.99
Co	2s	53.8		53.4
Ni	2s	9.57	0.10	9.47
Nb	2s	0.020	0.010	0.016
Mo	2s	4.80	0.05	4.72
W	2s	2.34	0.10	2.39

Table 22: Titanium base

Ti 6-6-2				
		Analysis [%]	ASD [%]	Certificate [%]
Al	10s	4.70	0.38	5.57
	30s	5.20	0.21	
Ti	2s	85.7		85.5
V	2s	5.74	0.12	5.57
Fe	2s	0.55	0.02	0.56
Ni	2s	0.02	0.01	0.015
Cu	2s	0.53	0.02	0.51
Mo	2s	0.008	0.004	0.008
Sn	2s	2.23	0.05	1.99

Table 23: Magnesium base

Mg 468 HL				
		Analysis [%]	ASD [%]	Certificate [%]
Mg	10s	95.24		96.14
	30s	95.48		
Mn	2s	0.26	0,01	0.2
Cu	2s	0.105	0,003	0.09
Zn	2s	2.63	0,01	2.52
La	2s	0.22	0,03	0.23
Ce	2s	0.59	0,04	0.6



7. Conclusion

With the SPECTRO xSORT it is possible to conduct laboratory-like analyses in a single measuring cycle that lasts only 2 seconds. For integration of the light elements Mg, Al, Si, P and S within a complete analysis or for sorting various aluminum and magnesium alloys only 10 additional seconds are necessary. During this process even the light elements are measured in air – without complicated techniques like helium flushing or vacuum, simplifying operation decidedly.

The Eval-Server corrects line-overlaps and all detector artifacts and determines the sample composition using fundamental parameter.

The LODs listed in this report show excellent sensitivity of the SPECTRO xSORT. The typical analysis results demonstrate outstanding precision and accuracy.

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