SPECTRO XRF Report

No. XRF-73, Rev. 4

SPECTRO xSORT XHH03

Analysis of Rock, Sediment and Soil

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 allows unique analytical flexibility. With the SPECTRO xSORT it is possible to conduct laboratory-like analyses for all the important elements during analysis of rock, sediment and soil. The installed processing and calibration modules ensure accuracies typical for SPECTRO's laboratory XRF instruments, even for the SPECTRO xSORT. An analysis can be conducted within 30 seconds.

1. Introduction

For many years now, X-ray fluorescence analysis (XRF) has been an established method in the laboratory for rapid screening analyses of rocks and contaminated soil. Laboratory XRF instruments from SPECTRO are considered around the world to have set the standard for the analysis of completely unknown samples.
With the xSORT, SPECTRO offers a portable, compact XRF analyzer for the onsite analysis of rock and soil. Its calibration is based on the successful calibration models utilized in the laboratory instruments.

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2. Instrumentation

Calibration modules from SPECTRO were installed on the SPECTRO xSORT for the elemental analysis of rock and soil samples. The instrument is available in two variations: The basic variant and the SPECTRO xSORT ECM, the variation that enables using shorter measuring times.

With the SPECTRO xSORT ECM, only 30 seconds are necessary for the analysis of the elements Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Br, Rb, Sr, Y, Zr, Nb, Mo, Ag, Cd, In, Sn, Sb, Te, I, Cs, Ba, La, Ce, Pr, Nd, Ta, W, Au, Hg, Tl, Pb, Bi, Th and U in a sample. In a second sub measurement, the elements Mg, Al, Si, P, S, Cl, K and Ca can be analyzed; a measuring time of 30 s is also sufficient for these elements. Precision and accuracy of the analytical results as well as detection limits are improvable by increasing the measuring time.

For the analysis of rock and soil, the analysis of several subsamples in order to determine the average analytical result for this generally inhomogeneous sample material is recommended; making the short measuring times very advantageous. Additionally, the SPECTRO xSORT automatically calculates the average of the individual analyses.

3. Technical Data

Detector

- High resolution silicon drift detector (SDD)

Excitation

- X-ray tube with Rh anode
- Up to 50 kV tube voltage
- Up to 2.5 W tube power
- Radiation filter for optimal excitation conditions

Operation

The start button can be released when the first measured value, including any measurement errors, is displayed on the screen. The button can also be held until the preset measurement duration has been reached. The analytical results displayed after about 2 seconds are usually stable after 15 seconds, with the statistical measurement error decreasing as the analysis continues.

Spectrometer Control

- ePC including Windows mobile operating system
- SD card slot
- Bluetooth, WiFi, USB

Dimensions and Weight

- Height: 270 mm (10.7”)
- Width: 93 mm (3.7”)
- Depth: 230 mm (9.1”)
- Weight: 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)

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

Software

- Eval-Server for data treatment and fundamental parameter analysis
- iCAL (Intelligent Calibration Logic)

Documentation

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

4. Sample Preparation

For the accurate analysis of rock and soil samples, it is necessary to take representative samples and to adhere to the sample preparations requirements for the respective analytical technique.
Rock and soil samples can, for example, be prepared as fine loose powder. The samples should, as far as possible, be previously dried. For this report, approx. 4 g sample material was poured into a sample cup with an outer diameter of 32 mm and measured.

5. Analytical Results

The lower limits of detection (LLD) are summarized for various elements in rocks and soils for a measuring time of 60 s in Table 1.

$$LLD = \frac{3}{S}\sqrt{\frac{\dot{B}}{t}}$$

$\dot{B}$ = Background count rate [cps]
S= Sensitivity [cps/(µg/g)]
t= Measuring time [s]

Table 1: Detection limits for various elements in a 100 % SiO_2 matrix with a measuring time of 60 s

Element	LLD [µg/g]	Element	LLD [µg/g]	Element	LLD [µg/g]
Ti	100	Zr	2	Ta	10
V	30	Nb	2	W	6
Cr	20	Mo	2	Au	5
Mn	15	Cd	7	Hg	2
Fe	10	In	8	Tl	2
Co	5	Sn	9	Pb	3
Ni	3	Sb	9	Bi	2
Cu	3	Te	15	Th	2
Zn	2	I	30	U	3
As	1	Cs	40		
Se	1	Ba	45		
Br	1	La	60		
Rb	1	Ce	85		
Sr	2	Pr	110		
Y	2	Nd	150		

In the following tables 2 to 4, the analytical results for standard reference materials are listed as examples. The samples were analyzed using two sub measurements of 30 s each. The analytical error given for the SPECTRO xSORT corresponds to the statistical error with a confidence level of 95%.

Table 2: Analytical results for the basalt sample: BHVO-2

Element	Unit	Given	xSORT
Mg	%	4.36	3.8 ± 1.5
Al	%	7.15	6.7 ± 0.3
Si	%	23.32	21.7 ± 0.1
P	%	0.12	0.10 ±0.02
K	%	0.43	0.40 ±0.01
Ca	%	8.15	8.33 ±0.03
Ti	%	1.64	1.57 ±0.03
Fe	%	8.56	8.01 ±0.02
V	µg/g	317	280 ± 60
Cr	µg/g	280	290 ± 40
Mn	µg/g	1290	1210 ± 50
Co	µg/g	45	< 100
Ni	µg/g	119	147 ± 8
Cu	µg/g	127	129 ± 8
Zn	µg/g	103	107 ± 6
Rb	µg/g	9.8	9 ± 2
Sr	µg/g	389	375 ± 5
Y	µg/g	26	24 ± 2
Zr	µg/g	172	171 ± 5
Nb	µg/g	18	14 ± 4

Table 3: Analytical results for the dunite sample: DTS-2b

Element	Unit	Given	xSORT
Mg	%	29.8	28.3 ± 1.1
Al	%	0.24	< 0.07
Si	%	18.42	18.0 ± 0.1
Ca	%	0.086	0.066 ±0.003
Fe	%	5.43	5.34 ±0.02
V	µg/g	22	34 ± 25
Cr	µg/g	15500	14400 ± 200
Mn	µg/g	830	750 ± 60
Co	µg/g	120	90 ± 50
Ni	µg/g	3780	3980 ± 40
Cu	µg/g	3	< 10
Zn	µg/g	45	55 ± 4
Rb	µg/g	2	2 ± 1

Table 4: Analytical results for the granite sample: GSR-01

Element	Unit	Given	xSORT
Mg	%	0.25	< 0.8
Al	%	7.09	7.5 ± 0.2
Si	%	34.04	35.7 ± 0.1
P	%	0.039	< 0.01
K	%	4.16	4.19 ± 0.02
Ca	%	1.11	1.17 ± 0.01
Ti	%	0.17	0.17 ± 0.01
Fe	%	1.50	1.41 ± 0.01
V	µg/g	24	< 40
Cr	µg/g	5	< 30
Mn	µg/g	465	440 ± 30
Co	µg/g	3.4	< 30
Ni	µg/g	2.3	< 5
Cu	µg/g	3.2	< 5
Zn	µg/g	28	31 ± 2
As	µg/g	2.1	< 2
Rb	µg/g	466	442 ± 4
Sr	µg/g	106	109 ± 3
Y	µg/g	62	62 ± 2
Zr	µg/g	167	143 ± 3
Nb	µg/g	40	31 ± 4
Mo	µg/g	3.5	< 3
Sn	µg/g	12.5	13 ± 2
Ba	µg/g	343	230 ± 50
La	µg/g	54	< 70
Ce	µg/g	108	80 ± 70
Pb	µg/g	31	32 ± 5
Th	µg/g	54	55 ± 4
U	µg/g	19	12 ± 4

Table 5: Analytical results for the soil sample: GSS-08

El.	Unit	Given	Analysis ± ASE (2σ)	Average ± ASD (2σ)
Mg	%	1.44	1.5 ± 0.1	< 0.6
Al	%	6.31	5.2 ± 0.1	5.2 ± 0.1
Si	%	27.39	23.5 ± 0.1	23.5 ± 0.1
K	%	2.01	1.77 ± 0.01	1.78 ± 0.01
Ca	%	5.91	6.18 ± 0.01	6.19 ± 0.02
Ti	%	0.38	0.35 ± 0.01	0.34 ± 0.01
Fe	%	3.13	3.01 ± 0.01	3.00 ± 0.01
V	µg/g	81	80 ± 30	60 ± 40
Cr	µg/g	68	75 ± 20	70 ± 30
Mn	µg/g	620	620 ± 20	600 ± 20
Co	µg/g	12.7	40 ± 30	50 ± 30
Ni	µg/g	31.5	32 ± 3	31 ± 5
Cu	µg/g	24.3	22 ± 3	22 ± 8
Zn	µg/g	68	69 ± 3	70 ± 3
As	µg/g	12.7	12 ± 2	12 ± 2
Rb	µg/g	96	92 ± 2	90 ± 2
Sr	µg/g	236	226 ± 2	224 ± 3
Y	µg/g	26	26 ± 2	24 ± 2
Zr	µg/g	229	228 ± 3	225 ± 4
Nb	µg/g	15	11 ± 2	8 ± 4
Ba	µg/g	480	500 ± 50	523 ± 80
Pb	µg/g	21	20 ± 4	17 ± 3
Th	µg/g	11.8	11 ± 2	12 ± 3

A sample cup with the standard material GSS-8, shown in Table 5, was prepared and measured with a measuring time of 120 s for each measurement as a repeatability check. The analytical standard deviation (ASD; 2σ) given in table 5 is comparable with the absolute statistical error (ASE; 2σ) for the individual analyses. Both ASD and ASE are based on a confidence level of 95%. The results displayed in Table 5 show that for a measuring time of 120 s, the instrument error is negligible compared to the statistical error. Ten sample cups were prepared with different standard material GSD-12 shown in Table 6 and analyzed with a measuring time of 120 s each. In this analytical case, the ASD values for the matrix elements were much higher than the ASE values. This is especially noticeable for the element zirconium.

Particle size effects and mineralogical effects lead to increased values for the ASD for these samples. Only by improving the sample preparation this effect, which does not occur for all of the standard samples examined, can be reduced.

Table 6: Repeatability and analytical results for the sediment sample: GSD-12

Ele-ment	Unit	Given	Analysis ± ASE (2σ)	Average ± ASD (2σ)
Mg	%	0.28	< 0.6	< 0.6
Al	%	4.92	4.3 ± 0.1	4.5 ± 0.4
Si	%	36.13	35.8 ± 0.1	35.6 ± 0.4
K	%	2.42	2.25 ± 0.01	2.3 ± 0.05
Ca	%	0.83	0.78 ± 0.01	0.80 ± 0.04
Ti	%	0.15	0.12 ± 0.01	0.12 ± 0.01
Fe	%	3.41	3.16 ± 0.01	3.19 ± 0.05
Cr	µg/g	35	45 ± 20	36 ± 20
Mn	µg/g	1400	1300 ± 30	1300 ± 40
Ni	µg/g	12.8	11 ± 1	9 ± 4
Cu	µg/g	1230	1230 ± 10	1260 ± 40
Zn	µg/g	498	460 ± 6	470 ± 10
As	µg/g	115	106 ± 4	105 ± 9
Rb	µg/g	270	245 ± 2	247 ± 4
Sr	µg/g	24.4	27 ± 2	27 ± 1
Y	µg/g	29.3	27 ± 2	25 ± 4
Zr	µg/g	234	256 ± 3	230 ± 50
Nb	µg/g	15.4	12 ± 2	13 ± 5
Mo	µg/g	8.4	8 ± 2	7 ± 4
Sn	µg/g	54	58 ± 4	55 ± 15
Sb	µg/g	24.3	27 ± 7	21 ± 18
Ba	µg/g	206	130 ± 40	130 ± 30
W	µg/g	37.4	47 ± 11	47 ± 9
Pb	µg/g	285	283 ± 6	285 ± 7
Bi	µg/g	10.9	14 ± 2	15 ± 4
Th	µg/g	21.4	12 ± 4	12 ± 8
U	µg/g	7.8	7 ± 2	7 ± 4

7. Conclusion

For a high number of important elements, using simple sample preparation (loose powder) and a short measuring time, it is possible to conduct laboratory-like analyses for rock and soil samples with the SPECTRO xSORT. The screening analysis for the elements Z > 20 can be concluded after 30 seconds, the risk of sample segregation in the loose samples is negligible. Particle size effects and mineralogical effects can affect the precision and accuracy of the analysis. The short measuring time enables multiple samples to be taken with an average of the analytical results.

www.spectro.com

AMETEK MATERIALS ANALYSIS DIVISION

GERMANY
SPECTRO Analytical Instruments GmbH
Boschstr. 10
47533 Kleve
Tel.: +49.2821.8 92-0
Fax: +49.2821.8 92-22 00
spectro.sales@ametek.com

U.S.A.
SPECTRO Analytical Instruments Inc.
91 McKee Drive
Mahwah, NJ 07430
Tel.: +1.800.548.5809
+1.201.642.3000
Fax: +1.201.642.3091
spectro-usa.sales@ametek.com

HONG KONG (Asia-Pacific)
SPECTRO Analytical Instruments (Asia-Pacific) Ltd.
Unit 1603, 16/F., Tower III
Enterprise Square
No. 9 Sheung Yuet Road
Kowloon Bay, Kowloon
Tel.: +852.2976 9162
Fax: +852.2976 9542
spectro-ap.sales@ametek.com

CHINA: Tel +86.10.8526.2111, Fax + 86.10.8526.2141, spectro-china.info@ametek.com.cn, FRANCE: Tel +33.1.3068.8970, Fax +33.1.3068.8999, spectro-france.sales@ametek.com, GREAT BRITAIN: Tel +44.1162.462.950, Fax +44.1162.740.160, spectro-uk.sales@ametek.com, INDIA: Tel +91.22.61968200, Fax +91.22.28363613, sales.spectroindia@ametek.com, ITALY: Tel +39.02.94693.1, Fax+39.02.94693.650, spectro-italy.sales@ametek.com, JAPAN: Tel +81.3.6809.2405, Fax + 81.3.6809.2410, spectro-japan.info@ametek.co.jp, SOUTH AFRICA: Tel +27.11.979.4241, Fax +27.11.979.3564, spectro-za.sales@ametek.com, SWEDEN: Tel +46.8.5190.6031, Fax + 46.8.5190.6034, spectro-nordic.info@ametek.com.

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