Determination of Accuracy of SPECTRO XEPOS EDXRF Spectrometer

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Abstract

This research work is to determine the accuracy of SPECTRO XEPOS EDXRF Spectrometer at the Experimental Nuclear Physics Lab, Department of Physics, University of Mandalay and it also intends to inform the current performance of this Spectrometer. In order to obtain the reliable accuracy of this Spectrometer, every step of sample preparation, X-Ray measurement and data analysis were thoroughly performed. To determine the accuracy of SPECTRO XEPOS EDXRF Spectrometer IAEA-SL-1 (Lake Sediment), IAEA-SL-3 (Lake Sediment), IAEA-359 (Cabbage), IAEA-413 (Algae), IAEA-A-13 (Animal Blood) and NIST-2710 a (Montana Soil) were used. Two pellets of each Certified Reference Material were prepared for X-Ray measurement. Ten-Time measurements of each pellet were conducted.

Key Words: SPECTRO XEPOS EDXRF Spectrometer, Accuracy of the Spectrometer, Certified Reference Materials

Introduction

SPECTRO XEPOS EDXRF Spectrometer at the Experimental Nuclear Physics Lab, Department of Physics, University of Mandalay can be used to determine the elemental compositions and concentrations of soil, rock, sediment and vegetable samples. Most of the PhD students and staff from Universities of Upper Myanmar used this Spectrometer for their research works especially to determine the elemental compositions and concentration of their interested samples. Every researcher has to know the performance of the Spectrometer and decide which is suitable or not for their research works.

Experimentation

Tube Excitation System for X-Ray Measurement

The SPECTRO XEPOS EDXRF Spectrometer (Kleve, Germany) at the Department of Physics, University of Mandalay was used for this research work. The spectrometer is mainly equipped with Palladium (Pd) anode X-Ray Tube, three secondary targets, silicon drift detector (SDD) detection system and 12-position sample changer as shown in Figure (1). X-Rays emitted from Pd anode X-Ray Tube excite the secondary target. The characteristic fluorescence X-Rays from the secondary target with lowest background intensity is used to excite the samples. Characteristic fluorescence X-Rays emitted from the elements contained in the analyzed sample is detected by silicon drift detector. Instrument specification of SPECTRO XEPOS EDXRF Spectrometer is described in Table (1).

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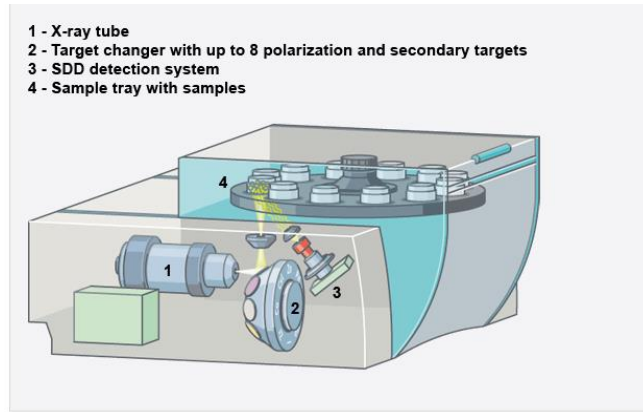[2] MRes Student, Department of Physics, University of Mandalay

Figure (1) SPECTRO XEPOS EDXRF Spectrometer

Table (1) Instrument Specification of SPECTRO XEPOS EDXRF Spectrometer

Excitation	• X-ray tube with Pd anode • Power max 50 W • Voltage max 50 kV • Secondary targets are used (HOPG, Mo, Al_2O_3)
Detection system	• Silicon Drift Detector with Peltier cooling ($\leq$ - 25 ˚C/ - 13 ˚F) • Large active detector area • 8 µm – Be window • Spectral resolution (FWHM) at Mn K$\alpha \leq 160$ eV
Sample handling	• Sample tray with 12 positions for samples with diameters of 32 mm • Analysis in air, He atmosphere or optional in vacuum
Software	• Menu- based X-LAB Pro software for control of spectrometer functions and for evaluation of data

Sample Preparation for XRF Measurement

Certified Reference Materials used in this research work are described in Figure (2). Sample preparation is very important in XRF measurement. In this research work, digital balance (PW 254), Pellet Machine (Retsch PP 25), mould and die set were used. Figure (2) is cleaning the mould and die set before pelletizing. Optimum sample to binder ratio for SPECTRO XEPOS EDXRF spectrometer is 5:1. 5 g of sample and 1 g of binder were weighed for each pellet by using digital balance (PW 254) and mixing them to get homogeneous powder. Figure (4) shows weighing the sample and binder by using digital balance (PW 254).

Mixture powder of sample and binder was prepared as pellet by using pellet machine (Retsch PP 25), mould and die set as shown in Figure (5). 10 ton of pressure was used for each pellet. Pellet sample was carefully removed from the mould. Weight of pellet was recorded. Removing the pellet from the mould and recording the pellet weight is presented in Figure (6). Two pellets of each Certified Reference Material, totally twelve pellets, were prepared for X-Ray measurement. Prepared samples are described in Figure (7).

Figure (2) Certified Reference Materials
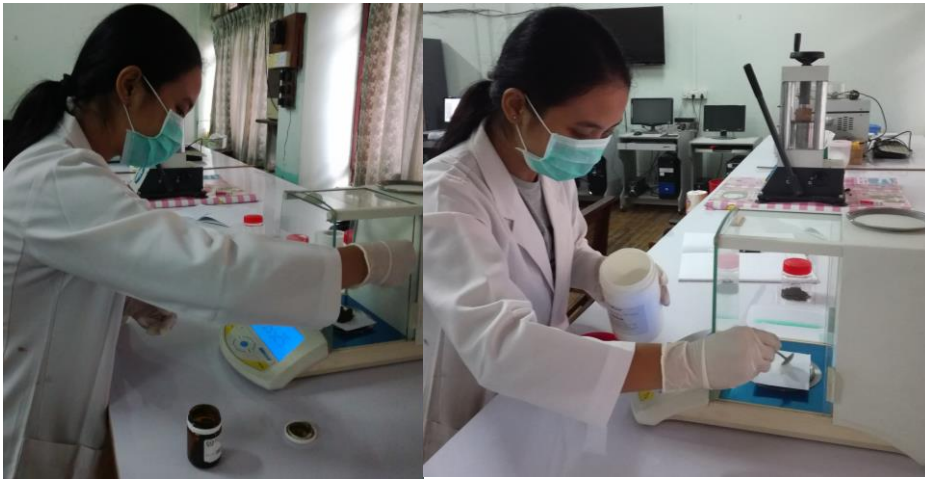

Figure (3) Cleaning the Mould and Die Set

Figure (4) Weighing the Sample and Binder by using
Digital Balance (PW 254)

Figure (5) Pelletizing the Mixture of Sample and Binder by using
Pellet Machine (Retsch PP 25)

Figure (6) Removing the Pellet from the Mould and Recording
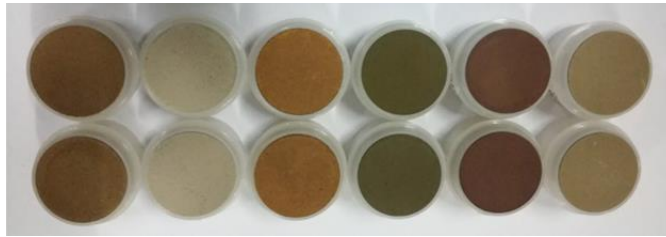the Pellet Weight

Figure (7) Prepared Pellets for XRF Measurement

X-Ray Measurement

Before any measurement, the spectrometer must be kept in standby mode to warm up the X-Ray tube. Measurement can begin after selecting the pellet calibration method and entering the sample identification information. It will take 300 s for each secondary target and 900 s for each sample. During the X-Ray measurement, sample compartment is interlocked. In this research work, ten-time measurement of each pellet for IAEA-SL-1 (Lake Sediment), IAEA-SL-3 (Lake Sediment), IAEA-359 (Cabbage), IAEA-413 (Algae), IAEA-A-13 (Animal Blood) and NIST 2710 a (Montana Soil) was conducted. Maximum analyzed energy is 50 keV. Resolution of detector is 160 eV at 5.894 keV of Mn K$_\alpha$. X-LAB Pro 4.5 software is used to determine the elemental composition and concentration of analyzed sample.

Result and Discussion

Accuracy of SPECTRO XEPOS EDXRF Spectrometer on certified value of IAEA-SL-1 (Lake Sediment) is presented in Table (2). Error difference between two pellets for each element ranges from 0.2 % to 6.32 %. Accuracy of SPECTRO XEPOS EDXRF Spectrometer on certified value of IAEA-SL-1 (Lake Sediment) ranges from 1.88 % to 24.28 %. Accuracy of SPECTRO XEPOS EDXRF Spectrometer on certified value of IAEA-SL-3 (Lake Sediment) is described in Table (3). Error difference between two pellets for each element ranges from 0.51 % to 15.63 %. Accuracy of SPECTRO XEPOS EDXRF Spectrometer on certified value of IAEA-SL-3 (Lake Sediment) ranges from 0.11 % to 37.36 %. Accuracy of SPECTRO XEPOS EDXRF Spectrometer on certified value of IAEA-359 (Cabbage) is shown in Table (4). Error difference between two pellets for each element ranges from 0.08 % to 8.3 %. Accuracy of SPECTRO XEPOS EDXRF Spectrometer on certified value of IAEA-359 (Cabbage) ranges from 0.18 % to 29.36 %.

Accuracy of SPECTRO XEPOS EDXRF Spectrometer on certified value of IAEA-413 (Algae) is presented in Table (5). Error difference between two pellets for each element ranges from 0.04 % to 0.89 %. Accuracy of SPECTRO XEPOS EDXRF Spectrometer on certified value of IAEA-413 (Algae) ranges from 1.09 % to 79.05 %. Accuracy of SPECTRO XEPOS EDXRF Spectrometer on certified value of IAEA-A-13 (Animal Blood) is described in Table (6). Error difference between two pellets for each element ranges from 0 % to 12.5 %. Accuracy of SPECTRO XEPOS EDXRF Spectrometer on certified value of IAEA-A-13 (Animal Blood) ranges from 0.08 % to 17.39 %. Accuracy of SPECTRO XEPOS EDXRF Spectrometer on certified value of NIST 2710 a (Montana Soil) is shown in Table (7). Error

difference between two pellets for each element ranges from 0.06 % to 4.65 %. Accuracy of SPECTRO XEPOS EDXRF Spectrometer on certified value of NIST 2710 a (Montana Soil) ranges from 0.35 % to 37.64 %.

Table (2) Accuracy of SPECTRO XEPOS EDXRF Spectrometer for IAEA-SL-1

Element	Concentration (ppm)			Error (%)	
	Certified Value (IAEA-SL-1, 1999)	Measured Value			
		Pellet (1)	Pellet (2)	Pellet (1)	Pellet (2)
As	27.6	20.94 ± 0.54	21.20 ± 0.35	24.28	23.19
Ba	639	778.10 ± 19.57	782.60 ± 24.60	21.77	22.47
Fe	67400	63725.00 ± 101.13	63219.00 ± 94.80	5.45	6.20
Mn	3460	3395.10 ± 16.69	3333.90 ± 18.77	1.88	3.64
Rb	113	100.19 ± 0.43	98.80 ± 0.43	11.34	12.57
Ti	5170	4562.00 ± 66.24	4551.90 ± 88.63	11.76	11.96
V	170	150.55 ± 8.80	139.80 ± 8.79	11.44	17.76
Zn	223	177.57 ± 1.09	175.70 ± 0.88	20.37	21.21

Table (3) Accuracy of SPECTRO XEPOS EDXRF Spectrometer for IAEA-SL-3

Element	Concentration (ppm)			Error (%)	
	Certified Value (IAEA-SL-3, 1999)	Measured Value			
		Pellet (1)	Pellet (2)	Pellet (1)	Pellet (2)
As	3.2	3.20 ± 0.23	3.70 ± 0.23	6.25	15.63
Hf	9.1	12.50 ± 0.49	12.20 ± 0.68	37.36	34.07
K	8740	8749.30 ± 22.90	8650.40 ± 33.10	0.11	1.03
Rb	38.8	37.40 ± 0.21	37.20 ± 0.14	3.61	4.12
Sr	470	469.80 ± 1.00	465.50 ± 1.10	0.04	0.96

Element	Concentration (ppm)	Error (%)

	Certified Value (IAEA-359, 2000)	Measured Value			
		Pellet (1)	Pellet (2)	Pellet (1)	Pellet (2)
Cu	5.67	5.66 ± 0.25	5.94 ± 0.22	0.18	4.76
Fe	148	104.55 ± 1.29	116.83 ± 1.58	29.36	21.06
K	32500	28082.00 ± 93.07	27849.00 ± 74.90	13.59	14.31
Mn	31.9	39.74 ± 1.11	39.38 ± 0.55	24.58	23.45
Sr	49.2	43.46 ± 0.11	43.50 ± 0.17	11.67	11.59
Zn	38.6	36.30 ± 0.32	36.70 ± 0.32	5.96	4.92

Table (4) Accuracy of SPECTRO XEPOS EDXRF Spectrometer for IAEA-359

Table (5) Accuracy of SPECTRO XEPOS EDXRF Spectrometer for IAEA-413

Element	Concentration (ppm)			Error (%)	
	Certified Value (IAEA-413, 2010)	Measured Value		Pellet (1)	Pellet (2)
		Pellet (1)	Pellet (2)		
As	127	86.13 ± 0.58	87.00 ± 0.60	32.18	31.50
Ca	3143	3108.60 ± 33.60	3080.90 ± 31.25	1.09	1.98
Cd	204	106.00 ± 1.90	106.09 ± 1.75	48.04	48.00
Cr	377	304.77 ± 1.28	302.24 ± 1.97	19.16	19.83
Fe	1370	1397.60 ± 6.50	1385.80 ± 7.18	2.01	1.15
K	10740	10529.00 ± 95.04	10523.00 ± 86.16	1.96	2.02
Mn	158	171.58 ± 1.49	170.93 ± 1.91	8.59	8.18
Ni	113	23.72 ± 0.14	23.67 ± 0.08	79.01	79.05
Pb	242	233.05 ± 1.97	231.69 ± 1.47	3.70	4.26
Zn	169	161.10 ± 0.47	160.66 ± 0.86	4.67	4.93

Table (6) Accuracy of SPECTRO XEPOS EDXRF Spectrometer for IAEA-A-13

Element	Concentration (ppm)			Error (%)	
	Certified Value (IAEA-A-13, 2000)	Measured Value		Pellet (1)	Pellet (2)
		Pellet (1)	Pellet (2)		
Br	22	22.40 ± 0.08	22.40 ± 0.63	1.82	1.82
Ca	286	327 ± 9.90	305.68 ± 9.14	14.34	6.88
Cu	4.3	4.90 ± 0.33	4.79 ± 0.41	13.95	11.40
Fe	2400	2313.80 ± 12.	2313.20 ± 18.05	3.59	3.62
K	2500	2097.20 ± 17.70	2083.40 ± 22.13	16.11	16.66
Rb	2.3	1.90 ± 0.07	1.90 ± 0.09	17.39	17.39
Zn	13	12.90 ± 0.13	12.99 ± 0.19	0.77	0.08

Table (7) Accuracy of SPECTRO XEPOS EDXRF Spectrometer for NIST-2710 a

Element	Concentration (%)			Error (%)	
	Certified Value (NIST 2710 a, 2009)	Measured Value		Pellet (1)	Pellet (2)
		Pellet (1)	Pellet (2)		
Al	5.95	5.7321 ± 0.0266	5.6737 ± 0.0293	3.66	4.64
As	0.154	0.1062 ± 0.0002	0.1043 ± 0.0002	31.00	32.27
Ca	0.964	0.9079 ± 0.0034	0.8822 ± 0.0043	5.81	8.49
Cu	0.342	0.3263 ± 0.0009	0.3212 ± 0.0006	4.56	6.06
Fe	4.32	4.3515 ± 0.0067	4.2857 ± 0.0058	0.73	0.79
Pb	0.552	0.4854 ± 0.0005	0.4787 ± 0.0007	12.06	13.28
Mn	0.214	0.2147 ± 0.0006	0.2115 ± 0.0009	0.35	1.13
P	0.105	0.1284 ± 0.0020	0.1268 ± 0.0020	22.29	20.83
K	2.17	2.1868 ± 0.0090	2.1578 ± 0.0080	0.77	0.56
Si	31.1	27.9160 ± 0.0718	27.5330 ± 0.1293	10.24	11.47
Ti	0.311	0.2995 ± 0.0037	0.2986 ± 0.0037	3.69	3.97
Zn	0.418	0.3814 ± 0.0006	0.3786 ± 0.0005	8.74	9.43
Sb	0.00525	0.0040 ± 0.0013	0.0043 ± 0.0002	21.98	17.33
Ba	0.0792	0.0918 ± 0.0019	0.0945 ± 0.0022	16.01	19.32
Cd	0.00123	0.0007 ± 0.0002	0.0008 ± 0.0002	37.64	34.96
Hg	0.000988	0.0008 ± 7.3E-05	0.0008 ± 0.0001	14.84	15.99
Sr	0.0255	0.0241 ± 6E-05	0.0235 ± 5.8E-05	5.24	7.65

Conclusion

Accuracy of SPECTRO XEPOS EDXRF Spectrometer can be determined for Al, As, Ba, Br, Ca, Cd, Cr, Cu, Fe, Hf, Hg, K, Ni, Mn, P, Pb, Rb, Sb, Si, Sr, Ti, V and Zn. Accuracy of SPECTRO XEPOS EDXRF Spectrometer ranges from 0.08 % to 79.05 %. It can be observed that if the concentration of the element is from percent (%) to part per million (ppm) range, accuracy of SPECTRO XEPOS EDXRF Spectrometer is acceptable. If the concentration of the element is a few part per million (ppm) or part per billion (ppb) range, accuracy of SPECTRO XEPOS EDXRF Spectrometer is very large.

Acknowledgements

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