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BASE METAL – ZINC CONCENTRATES ANALYSIS BY XRF FUSED BEAD WITH INTERNAL STANDARD

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ABSTRACT

Mining Industries produce Base metal concentrate and world smelters are customer to use concentrate to produce metals. During all the process quantification and exchange for commercial use we require accurate analysis to pay and receive price with actual quantity. Now a days typical wet chemical analysis is very time consuming and expensive method to analyse many elements, so industries are looking for short time analysis with high accuracy. In this scenario, it is required to develop XRF methodology to analyse multiple elements with highest accuracy. Instrument selection is with best setup of crystals detector and collimators to acquire best specific intensity of each element even in very low level.

Keywords **:** XRF methodology ; Base metal concentrate ; Multiple elements ; Concentration calibration overview

INTRODUCTION

Mining Industries produce Base metal concentrate and world smelters are customer to use concentrate to produce metals. During all the process quantification and exchange for commercial use we require accurate analysis to pay and receive price with actual quantity. Now a days typical wet chemical analysis is very time consuming and expensive method to analyse many elements, so industries are looking for short time analysis with high accuracy. In this scenario, it is required to develop XRF methodology to analyse multiple elements with highest accuracy. Instrument selection is with best setup of crystals detector and collimators to acquire best specific intensity of each element even in very low level. During the process , the materials Zinc, Copper, Lead sulphide and oxide concentrates as well as poly-metallic base metal ores are used. The standard covers the determination of following elements:

Main Elements - Zn, Cd, Pb, Cu, Fe , S, As, Ag, Mn,

Oxide Elements – SiO_2, CaO, MgO, Al_2O_3, TiO_2, BaO, K_2O

Minor Elements - Mo, Ni, Co, Sn , Sb, Cr, V, Bi, Se, Te,

Above elements are analysed in non-ferrous concentrates by Lithium Borate fused glass bead and X-ray fluorescence analysis.

The average range of major elements covered are shown below:

Zn - 30 to 65%,

Pb - 0 to 15%,

S - 0 to 35 %,

SiO_2 - 0 to 10 % ,

Fe - 0 to 15 % ,

Other elements - 1 to 10 %

Minor elements - 0 to 1 %.

PRINCIPLE

The sample is melted with flux to form a fused glass bead incorporating an internal standard and x-ray absorber (if required). The glass bead is subjected to measurements by x-ray fluorescence and quantification is by comparison with various matrix-matched prepared standards from pure oxides.

After appropriate preparation and pre-treatment the test sample is dissolved in a mixture of Lithium Tetraborate, Lithium Nitrate and Lithium Bromide containing a known quantity of internal standard (WO_3 or HfO_2).

The mixture is fused in a platinum crucible at high temperature and then cast into a platinum mould to form the glass bead. The glass bead is analysed using a wavelength-dispersive XRF spectrometer, which exposes the glass bead to a high intensity beam of X-rays. Each element in the sample emits characteristic fluorescent X-ray lines. An appropriate line is selected for each element and its intensity is measured. The concentration of each element is calculated by comparison of the X-ray line intensity in the sample disc with X-ray line intensities obtained from measurement of the same lines in calibration standards containing known concentrations of the elements.

INTERFERENCES

Careful selection of X-ray lines and instrument parameters reduces spectral interference to a minimum. The use of specifically prepared matrix-matched calibration standards from oxides and the introduction of a fixed amount of **Hafnium IV oxide (HfO_2) or Tungsten Oxide WO3** as an internal standard into each fused glass disc is used to correct for "matrix effects" and interference.

PRECISION AND REJECTION LEVELS

Analysis is generally conducted in at least duplicate for critical samples for uncertainty value .Calibration performance is derived for each elements range and uncertainty as per calibration and validation program. The calibration is linearized across the quoted range, using an algorithm built into the instrument software. Rejection between replicates is based upon the defined repeatability criteria.

FLUX , SAMPLE AND INTERNAL STANDARD RECIPE

Flux weight and sample weight will decide as per Platinum Crucible, Mould capacity, also as per XRF sample cup holder diameter and required fused bead size. Recommended size is 40 mm diameter and minimum 5 mm thick, stable and clear fused glass bead. (Minimum thickness is required to handle critical depth of x-rays with respect to specific elements).

IDEAL RECIPE IS AS FOLLOWING

Always use pre-dried sample (Dry @ 105 ± 5°C for maximum 2 hour) – check samples for susceptibility to oxidation, decomposition and / or sublimation. Samples reported on "*a dry basis"*

If any delay during weighing, then the samples must be kept in desiccator.

Sample weighed using analytical balance and using proper sample handling tool and brush etc.,

Sample weight 1 gm, Flux weight 10 gm and other as per requirement

REAGENTS REQUIRMENTS :

Fluxes, Internal standards uses and drying-/ calcining temperature

Fluxes	
Lithium Tetraborate (99 %)with Lithium Bromide (1 %)	Use - As it is - Kept moisture free
Lithium Tetraborate (99.5%)with Lithium Bromide (0.5 %)	Use - As it is - Kept moisture free
Lithium Nitrate	Dry @ 220 ± 4°C for 1 hour
Lithium Bromide	Use - As it is - Kept moisture free

Internal Standards Hafnium IV oxide (HfO_2), High Purity, 99.9% min Tungsten (VI) Oxide (WO_3) High Purity 99.9%	Dry @ 105 ± 4°C for 1 hour Calcine at 850^{O}C for 1 hour.

Fluxes –main suppliers globally are from ***Claisse, Fluxana and XRF Scientific***. However, required to use consistent quality and type. If the supplier is changed, then newly prepared standards and a new full calibration will be required.

Fluxes, Internal standards uses and drying-Calcining temperature

High Purity Oxide Vanadium V oxide (V_2O_5), 99.6% min Cobalt II, III oxide (Co_3O_4), 99.99% min Copper II oxide (CuO), 99.99% min Manganese IV oxide (MnO_2), 99.99% min Antimony (III) Oxide (Sb2O3) Lead (II) Oxide (PbO) Calcium Carbonate ($CaCO_3$) Manganese (IV) Oxide (MnO_2) Vanadium V oxide (V2O5) Chromium Oxide (Cr_2O_3) Bismuth Oxide (Bi_2O3) Silver Oxide (Ag2O) Silver Chloride (AgCl) Potassium Nitrate (KNO3) Sodium Chloride (NaCl) Lithium Sulphate (Li2SO4) Arsenic(III) oxide As2O3 Selenium Oxide (SeO2) Tellurium Oxide (TeO2)	Molybdenum Oxide (MoO_3) Barium Nitrate (Ba $(NO_3)_2$) Zinc Oxide (ZnO) Tin Oxide (SnO2) Nickel Oxide (NiO) Cadmium Oxide (CdO) Magnesium II oxide (MgO), 99.99% min Aluminium III oxide (Al_2O_3), 99.99% min Silicon IV oxide (SiO_2), 99.99% min Iron III oxide (Fe_2O_3), 99.99% min Calcium Oxide CaO Titanium Dioxide (TiO2) Zirconium Oxide (ZrO2)
Ammonium dihydrogen phosphate, ($NH_4H_2PO_4$) Sodium carbonate anhydrous (Na_2CO_3)	

Uranium, Thorium and other elements oxide as required.

Zinc/Bulk Concentrate - Direct Fusion Classical Calibration Standards

Concentration: %

Standard	Zn	Pb	Cd	Cu	Fe	S	As	Mn	Ag
ZINC-LEAD STD 01	65.0000	4.0000	0.0000	2.0000	4.0000	7.0500	4.0000	2.5000	0.1000
ZINC-LEAD STD 02	62.5000	15.0000	0.0000	0.0000	2.0000	14.2000	0.0000	0.0000	0.0000
ZINC-LEAD STD 03	60.0000	5.0000	0.0000	0.5000	6.4000	20.0000	5.0000	0.0000	0.0500
ZINC-LEAD STD 04	57.5000	10.0000	0.0000	1.0000	13.0000	13.1000	0.0000	0.2500	0.1000
ZINC-LEAD STD 05	55.0000	7.0000	0.1000	2.0000	12.0000	5.3500	0.0000	0.5000	0.2000

ZINC-LEAD STD 06	52.5000	3.0000	0.3000	4.0000	15.8000	5.0000	0.0000	1.0000	0.3000
ZINC-LEAD STD 07	50.0000	2.0000	0.5000	6.0000	5.0500	8.0000	0.0000	2.0000	0.4000
ZINC-LEAD STD 08	47.5000	12.0000	1.0000	0.0000	3.5000	12.0000	0.0000	3.0000	0.5000
ZINC-LEAD STD 09	45.0000	8.5000	1.5000	3.0000	4.0000	10.0000	0.0000	1.0000	0.7500
ZINC-LEAD STD 10	42.5000	2.0000	2.0000	1.0000	9.5000	3.0000	0.0000	5.0000	1.0000
ZINC-LEAD STD 11	40.0000	9.0000	2.0000	5.0000	3.0000	36.0000	1.0000	0.0000	1.0000
ZINC-LEAD STD 12	37.5000	14.0000	0.0000	6.5000	11.0000	29.0000	2.0000	0.0000	0.0000
ZINC-LEAD STD 13	35.0000	13.0000	0.0000	0.0000	10.0000	26.0000	1.0000	0.0000	0.0000
ZINC-LEAD STD 14	32.5000	12.0000	0.0000	8.0000	8.0000	21.5000	0.0000	0.0000	0.0000
ZINC-LEAD STD 15	30.0000	1.0000	0.0000	0.0000	7.0000	34.0000	0.0000	0.0000	0.0000

Standard	CaO	SiO2	Al2O3	MgO	TiO2	BaO	K2O	Ni	Sn
ZINC-LEAD STD 01	1.0000	1.0000	1.0000	4.0000	0.0000	1.0000	0.7500	0.0000	0.0000
ZINC-LEAD STD 02	0.0000	5.0000	0.0000	0.0000	0.0000	0.3000	0.0000	0.0000	0.0000
ZINC-LEAD STD 03	0.5000	0.5000	0.2500	0.5000	0.0000	0.0000	0.7500	0.0500	0.1000
ZINC-LEAD STD 04	1.0000	1.0000	0.5000	1.0000	0.0000	0.1000	0.0000	0.1000	0.2500
ZINC-LEAD STD 05	2.0000	7.0000	4.0000	1.5000	0.1000	0.2500	0.1000	0.2000	0.5000
ZINC-LEAD STD 06	4.0000	4.0000	2.0000	2.0000	0.2500	0.5000	0.2500	0.3000	1.0000
ZINC-LEAD STD 07	6.0000	6.0000	4.0000	1.0000	0.5000	0.7500	0.5000	0.4000	1.5000
ZINC-LEAD STD 08	2.0000	2.0000	1.5000	2.5000	1.0000	1.0000	1.0000	0.5000	2.0000
ZINC-LEAD STD 09	1.0000	1.0000	3.0000	4.0000	1.5000	1.5000	1.5000	0.7500	2.5000
ZINC-LEAD STD 10	2.0000	0.0000	5.0000	5.0000	2.0000	2.0000	2.0000	1.0000	3.0000
ZINC-LEAD STD 11	0.0000	2.0000	1.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
ZINC-LEAD STD 12	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
ZINC-LEAD STD 13	12.0000	3.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
ZINC-LEAD STD 14	0.0000	8.0000	10.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
ZINC-LEAD STD 15	10.0000	10.0000	8.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Standard	Mo	Sb	Cr	V	Co	Bi	Te	Se	Total
ZINC-LEAD STD 01	1.0000	1.0000	0.1000	0.1000	0.1000	0.1000	0.1000	0.1000	100
ZINC-LEAD STD 02	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	100
ZINC-LEAD STD 03	0.1000	0.0000	0.0500	0.0500	0.0500	0.0500	0.0500	0.0500	100
ZINC-LEAD STD 04	0.2500	0.2500	0.1000	0.1000	0.1000	0.1000	0.1000	0.1000	100
ZINC-LEAD STD 05	0.5000	0.5000	0.2000	0.2000	0.2000	0.2000	0.2000	0.2000	100
ZINC-LEAD STD 06	1.0000	1.0000	0.3000	0.3000	0.3000	0.3000	0.3000	0.3000	100
ZINC-LEAD STD 07	1.5000	1.5000	0.4000	0.4000	0.4000	0.4000	0.4000	0.4000	100
ZINC-LEAD STD 08	2.0000	2.0000	0.5000	0.5000	0.5000	0.5000	0.5000	0.5000	100
ZINC-LEAD STD 09	2.5000	2.5000	0.7500	0.7500	0.7500	0.7500	0.7500	0.7500	100
ZINC-LEAD STD 10	3.0000	3.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	100
ZINC-LEAD STD 11	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	100
ZINC-LEAD STD 12	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	100
ZINC-LEAD STD 13	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	100
ZINC-LEAD STD 14	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	100
ZINC-LEAD STD 15	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	100

During preparation of standard Table, if require to add / remove additional element/s, then require to recalculate standard concentration. Required Oxide Weight calculation to be performed as per available mould size and crucible capacity as well as element concentration.

INSTRUMENT CALIBRATION

Preparing Calibration Standards : Mixtures of high purity elemental oxides are accurately weighed and fused according to the procedure to be used for calibrations. The equivalent concentration of each element in the sample is calculated as follows:

Sample equivalent concentration (%) = Weight of pure oxide (g)/ sample weight (g) x 100

Detailed recipes for calibration standards showing equivalent concentrations are in standards list. Calibration standards are measured using the same instrument conditions as unknown assay samples. A calibration slope (E) and intercept (D) are calculated by linear regression of the individual standard measurements, using the XRF spectrometer computer software.

INSTRUMENT APPLICATIONS AND CONDITIONS

Instrument applications and the measurement conditions for each element are detailed below.

Zetium -Axios X-Ray Spectrometer,**Rh**target X-Ray Tube.						
Element	Line	Crystal	Collimator	Detector	kV	mA
Zn	Ka	LiF220	150um	Duplex	60	66
W1	La	LiF220	150um	Duplex	60	66
Cd	Ka	LiF200	150um	Scint	60	66
*Rh	Kβ-C	LiF200	150µm	Scint	60	66
S	Ka	Ge	150um	Flow	25	160
Pb	Lβ	LiF220	150um	Scint	60	66
W2	Lβ2	LiF220	150um	Scint	60	66
Cu	Ka	LiF200	150um	Duplex	60	66
Fe	Ka	LiF220	150um	Duplex	60	66
Si	Ka	INSB	700um	Flow	25	160
Ca	Ka	LiF200	150um	Flow	30	133
Mn	Ka	LiF200	150um	Duplex	60	66
Sn	La	LiF200	300um	Flow	25	160
Br	Ka	LiF2201	150um	Scint	60	66

Processing parameters of Compounds

Report Options

Report negative concentrations as:	No
Test when the value is negative.	<0
Report concentrations below threshold as:	No
Test when below threshold level.	trace
Test when below lower limit:	<LL
Test when above upper limit:	>UL
Report after measurement	No

Compounds

Name	Compound	Compound type	Unit	Decimals	Nominal	Lowerlimit	Upper limit	Report	Report sequence
Zn	Zn	Chemical	%	3				Yes	1
Pb	Pb	Chemical	%	3				Yes	2
Cu	Cu	Chemical	%	3				Yes	3
Cd	Cd	Chemical	%	3				Yes	4
Fe	Fe	Chemical	%	3				Yes	5
CaO	CaO	Chemical	%	3				Yes	6
SiO2	SiO_2	Chemical	%	3				Yes	7
Al2O3	Al_2O_3	Chemical	%	3				Yes	8
MgO	MgO	Chemical	%	3				Yes	9
Ba	Ba	Chemical	%	3				Yes	10
Sb	Sb	Chemical	%	3				Yes	11
Bi	Bi	Chemical	%	3				Yes	12
As	As	Chemical	%	3				Yes	13
S	S	Chemical	%	3				Yes	14

Name	Report name	Threshold (ppm)
Zn	Zn	
Pb	Pb	
Cu	Cu	
Cd	Cd	
Fe	Fe	
CaO	CaO	
SiO2	SiO2	
Al2O3	Al2O3	
MgO	MgO	
Ba	Ba	
Sb	Sb	
Bi	Bi	
As	As	
S	S	

CONCENTRATION CALIBRATIONS OVERVIEW

Name	Status	Use	Compound	Unit	D	Lock D	E	Lock E	F
Zn	OK	Yes	Zn	%	0.07640	No	17.38327	No	0.00000
Pb	OK	Yes	Pb	%	-1.52042	No	22.31661	No	0.00000
Cu	OK	Yes	Cu	%	-0.03541	No	11.89624	No	0.00000
Cd	OK	Yes	Cd	%	-0.00990	No	0.18218	No	0.00000
Fe	OK	Yes	Fe	%	0.40353	No	3.08314	No	0.00000
Ca	OK	Yes	CaO	%	-0.05059	No	0.14611	No	0.00000
Si	OK	Yes	SiO2	%	-0.09940	No	0.19564	No	0.00000
Al	OK	Yes	Al2O3	%	-0.93703	No	0.65647	No	0.00000
Mg	OK	Yes	MgO	%	-2.25809	No	0.52497	No	0.00000
Ba	OK	Yes	Ba	%	-0.08401	No	0.25526	No	0.00000
Sb	OK	Yes	Sb	%	0.00663	No	1.00416	No	0.00000
Bi	OK	Yes	Bi	%	-1.83828	No	0.06779	No	0.00000
As	OK	Yes	As	%	-0.27050	No	0.04095	No	0.00000
S	OK	Yes	S	%	-0.26589	No	0.10741	No	0.00000

Name	Lock F	RMS	RE	K	Correlation	Background	Matrixmodel	Regressionmodel	Error weighting
Zn	Yes	0.30249	0.02496	0.07606	0.99993	Yes	None	C=D+ E·R·M	Square root
Pb	Yes	0.14466	0.06191	0.08211	0.99962	Yes	None	C=D+ E·R·M	Square root
Cu	Yes	0.05991	0.05304	0.03750	0.99976	Yes	None	C=D+ E·R·M	Square root
Cd	Yes	0.03181	0.04637	0.03106	0.99898	Yes	None	C=D+ E·R·M	Square root
Fe	Yes	0.30561	0.05600	0.10249	0.99849	Yes	None	C=D+ E·R·M	Square root
Ca	Yes	0.24680	0.08681	0.12315	0.99165	Yes	None	C=D+ E·R·M	Square root
Si	Yes	0.27136	0.05418	0.10547	0.99646	Yes	None	C=D+ E·R·M	Square root
Al	Yes	0.15967	0.47037	0.18852	0.99839	Yes	None	C=D+ E·R·M	Square root
Mg	Yes	0.12724	0.11999	0.08890	0.99766	Yes	None	C=D+ E·R·M	Square root
Ba	Yes	0.05739	0.09528	0.06427	0.99869	Yes	None	C=D+ E·R·M	Square root
Sb	Yes	0.04993	0.07207	0.04732	0.99848	Yes	None	C=D+ E·R·M	Square root
Bi	Yes	0.08816	0.19986	0.12727	0.96564	Yes	None	C=D+ E·R·M	Square root
As	Yes	0.10665	0.25985	0.14776	0.94758	Yes	None	C=D+ E·R·M	Square root
S	Yes	0.51246	0.02088	0.10071	0.99785	Yes	None	C=D+ E·R·M	Square root

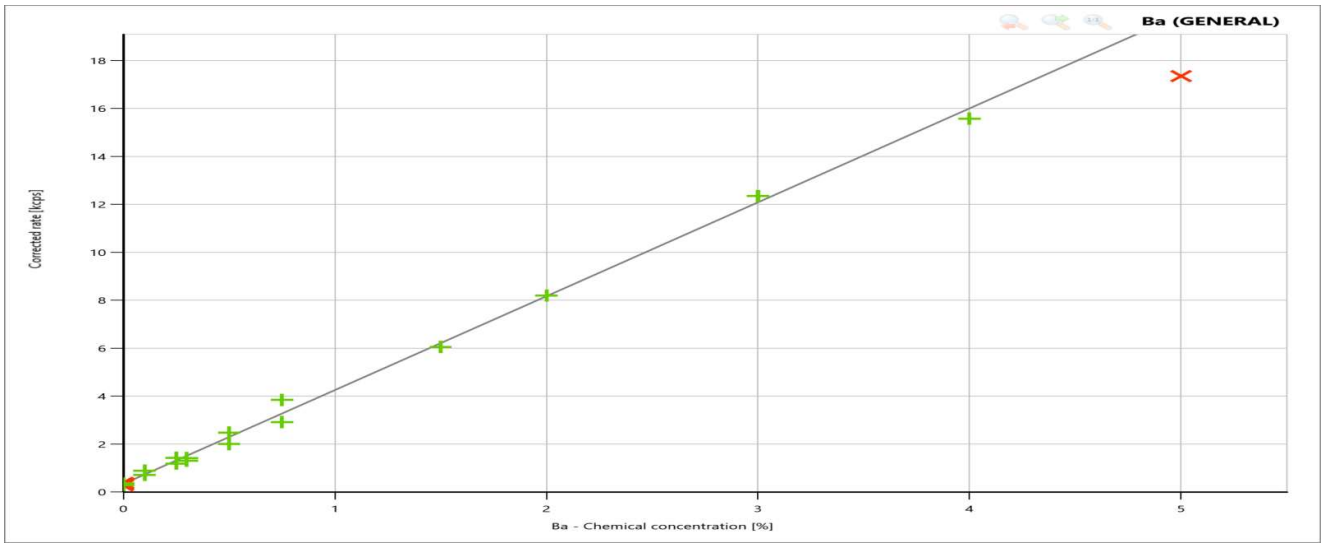
Name	Error weighting constant	Channel	Measuremet Type	Element	Line	Line type	Tags	Detector	Tube filter
Zn	0.10000	Zn	WD-G	Zn	Kα1,2	Fluorescent	GENERAL	Duplex	Al/200
Pb	0.10000	Pb	WD-G	Pb	Lβ1	Fluorescent	GENERAL	Duplex	None
Cu	0.10000	Cu	WD-G	Cu	Kα1,2	Fluorescent	GENERAL	Duplex	None

Name	Error weighting constant	Channel	Measuremet type	Element	Line	Line type	Tags	Detector	Tube filter
Cd	0.10000	Cd	WD-G	Cd	Kα1,2	Fluorescent	GENERAL	Scint	Brass/400
Fe	0.10000	Fe	WD-G	Fe	Kα1,2	Fluorescent	GENERAL	Duplex	None
Ca	0.10000	Ca	WD-G	Ca	Kα1,2	Fluorescent	GENERAL	Flow	None
Si	0.10000	Si	WD-G	Si	Kα1,2	Fluorescent	GENERAL	Flow	None
Al	0.10000	Al	WD-G	Al	Kα1,2	Fluorescent	GENERAL	Flow	None
Mg	0.10000	Mg	WD-G	Mg	Kα1,2	Fluorescent	GENERAL	Flow	None
Ba	0.10000	Ba	WD-G	Ba	Lα1,2	Fluorescent	GENERAL	Flow	None
Sb	0.10000	Sb	WD-G	Sb	Lα1,2	Fluorescent	GENERAL	Flow	None
Bi	0.10000	Bi	WD-G	Bi	Lα1,2	Fluorescent	GENERAL	Scint	None
As	0.10000	As	WD-G	As	Kβ1,3	Fluorescent	GENERAL	Scint	None
S	0.10000	S	WD-G	S	Kα1,2	Fluorescent	GENERAL	Flow	None

Name	Medium	Crystal	Collimator	Mask	kV	mA	PHD levels	Ratio channel	Apply finite thickness
Zn	Vacuum	LiF220	150µm	37mm	60	66	12-68	Hf	No
Pb	Vacuum	LiF220	150µm	37mm	60	66	19-64	Hf1	No
Cu	Vacuum	LiF220	150µm	37mm	60	66	13-65	Hf	No
Cd	Vacuum	LiF200	300µm	37mm	60	66	20-65		No
Fe	Vacuum	LiF200	300µm	37mm	60	66	14-70	Hf	No
Ca	Vacuum	LiF200	300µm	37mm	30	133	30-70		No
Si	Vacuum	InSb	700µm	37mm	25	160	28-74		No
Al	Vacuum	PE	300µm	37mm	25	160	21-78		No
Mg	Vacuum	PX1	300µm	37mm	25	160	30-79		No
Ba	Vacuum	LiF200	300µm	37mm	40	100	33-68		No
Sb	Vacuum	LiF200	150µm	37mm	30	133	31-72		No
Bi	Vacuum	LiF220	150µm	37mm	60	66	23-69		No
As	Vacuum	LiF200	150µm	37mm	60	66	19-69		No
S	Vacuum	Ge	300µm	37mm	25	160	29-71		No

Name	Apply FVG	Apply tertiary	Apply multiscatter	Eliminationmethod	Eliminatedcompound
Zn	No	No	No	None	
Pb	No	No	No	None	
Cu	No	No	No	None	
Cd	No	No	No	None	
Fe	No	No	No	None	
Ca	No	No	No	None	
Si	No	No	No	None	
Al	No	No	No	None	
Mg	No	No	No	None	
Ba	No	No	No	None	
Sb	No	No	No	None	
Bi	No	No	No	None	
As	No	No	No	None	
S	No	No	No	None	

CONCENTRATION CALIBRATIONS DETAILS AND COMPOUND CALIBRATION LINE DETAILS



Graph

Selected line

D	-0.08401
E	0.25526
F	0.00000
RMS	0.05739
RE	0.09528
K	0.06427
Correlation	0.99869

General information

Name	Ba
Compound	Ba
Line	Ba-Lα1,2-Fluorescent
Status	OK
Tags	GENERAL
Use	Yes

Regression coefficients

Unit %

	Value	Unit	Locked
D	-0.08401	%	No
E	0.25526	%/kcps	No
F	0.00000	%/kcps²	Yes

Background Yes

Ratio channel

Background	Yes
Matrix model	None
Sample effects:	
Include finite thickness effects	No
Include fluorescence volume geometry(FVG)effects	No
Regression model	C=D+E·R·M
Error weighting	Square root

	Model	Constant	Unit
Error weighting	Square root	0.10000	%

	Value	Unit
RMS	0.05739	%
RE	0.09528	
K	0.06427	√%
Correlation	0.99869	

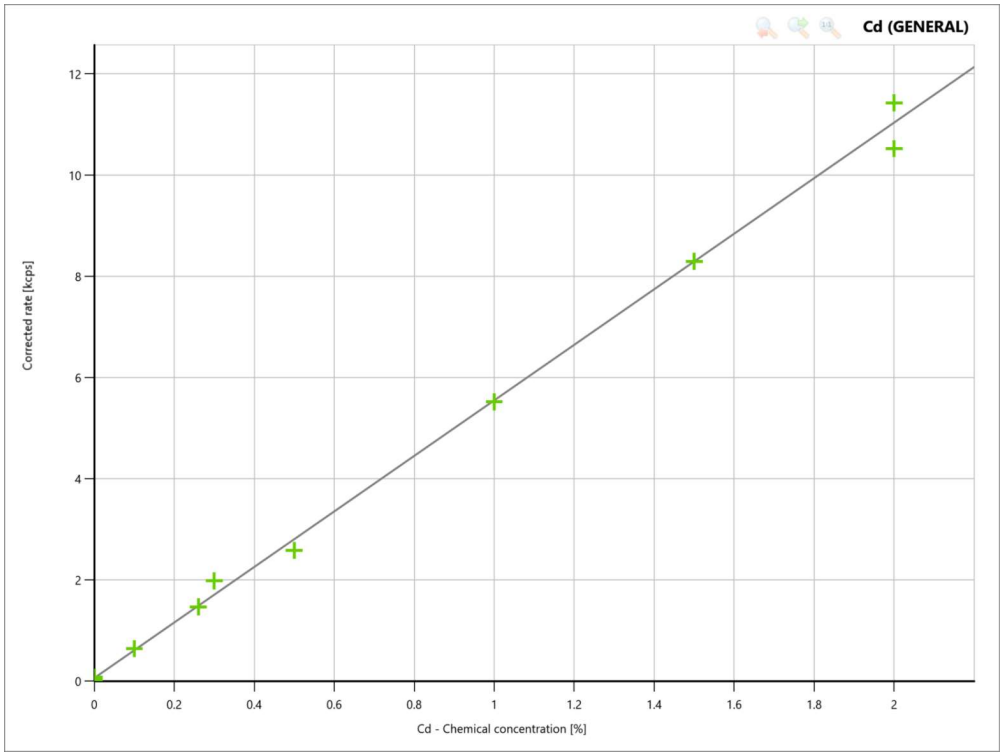
Measurement conditions

Measurement type	WD-G
Detector	Flow
Tube filter	None
Medium	Vacuum
Crystal	LiF200
Collimator	300μm
Mask	37mm
kV	40
mA	100
PHD	33-68

CONCENTRATION CALIBRATIONS DETAILS AND COMPOUND CALIBRATION LINE DETAILS

Graph

Selected line



D	-0.00990
E	0.18218
F	0.00000
RMS	0.03181
RE	0.04637
K	0.03106
Correlation	0.99898

General information

Name	Cd
Compound	Cd
Line	Cd-Kα1, 2-Fluorescent

Name	Cd
Status	OK
Tags	GENERAL
Use	Yes

Regression coefficients

Unit %

	Value	Unit	Locked
D	-0.00990	%	No
E	0.18218	%/kcps	No
F	0.00000	%/kcps²	Yes

Background	Yes
Ratio channel	
Matrix model	None
Sample effects:	
Include finite thickness effects	No
Include fluorescence volume geometry (FVG) effects	No
Regression model	C=D+E·R·M
Error weighting	Square root

	Model	Constant	Unit
Error weighting	Square root	0.10000	%

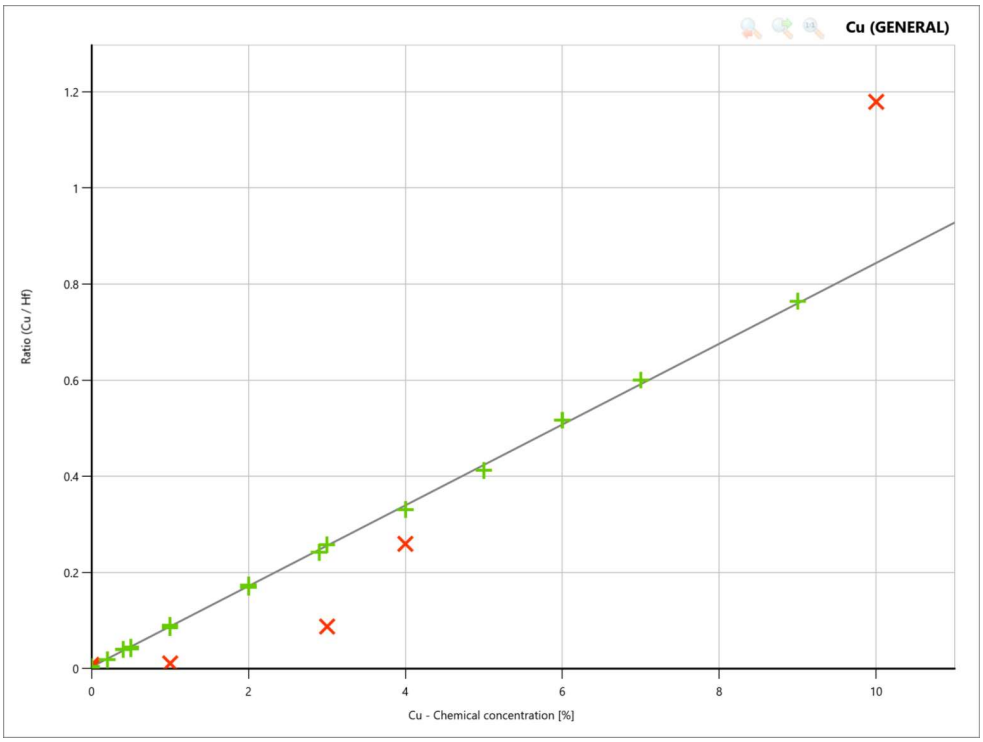
	Value	Unit
RMS	0.03181	%
RE	0.04637	
K	0.03106	√%
Correlation	0.99898	

Measurement conditions

Measurement type	WD-G
Detector	Scint
Tube filter	Brass/400
Medium	Vacuum
Crystal	LiF200
Collimator	300µm
Mask	37mm
kV	60
mA	66
PHD	20-65

CONCENTRATION CALIBRATIONS DETAILS AND COMPOUND CALIBRATION LINE DETAILS

Graph



Selected line

D	-0.03541
E	11.89624
F	0.00000
RMS	0.05991
RE	0.05304
K	0.03750
Correlation	0.99976

General information

Name	Cu
Compound	Cu
Line	Cu-Kα1, 2-Fluorescent
Status	OK
Tags	GENERAL
Use	Yes

Regression coefficients

Unit %

	Value	Unit	Locked
D	-0.03541	%	No
E	11.89624	%/kcps	No
F	0.00000	%/kcps²	Yes

Background	Yes
Ratio channel	Hf

Background	Yes
Matrix model	None
Sample effects:	
Include finite thickness effects	No
Include fluorescence volume geometry (FVG) effects	No
Regression model	C=D+E·R·M
Error weighting	Square root

	Model	Constant	Unit
Error weighting	Square root	0.10000	%

	Value	Unit
RMS	0.05991	%
RE	0.05304	
K	0.03750	√%
Correlation	0.99976	

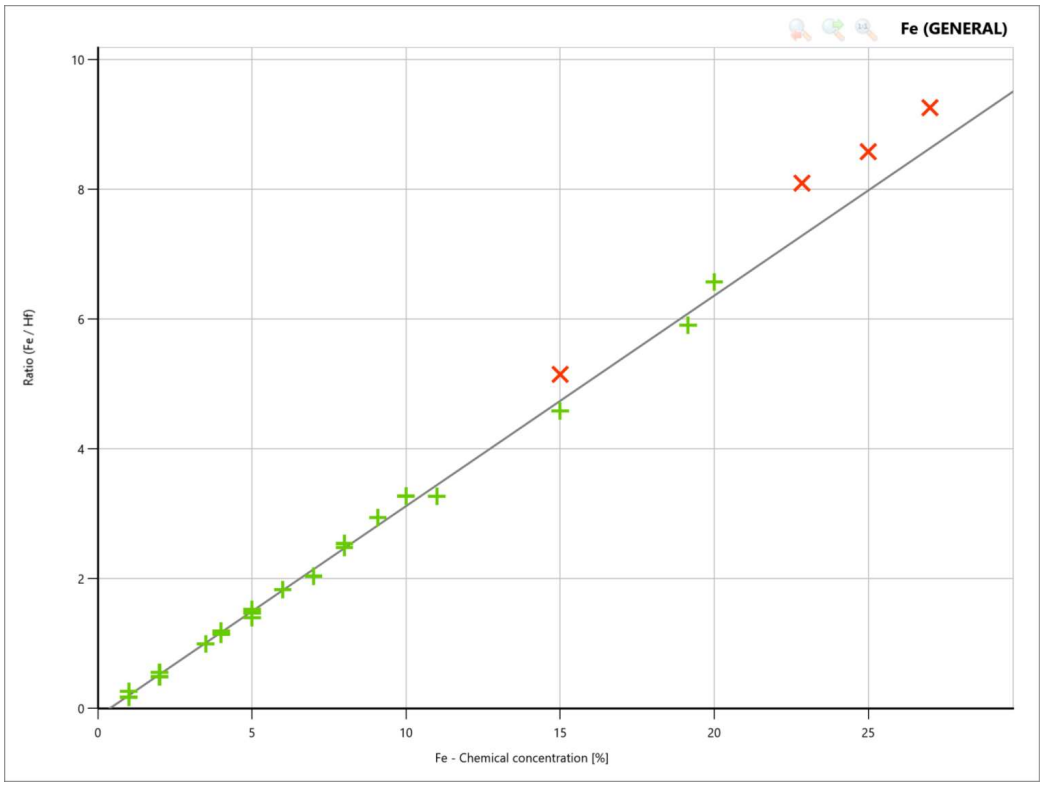
Measurement conditions

Measurement type	WD-G
Detector	Duplex
Tube filter	None
Medium	Vacuum
Crystal	LiF220
Collimator	150µm
Mask	37mm
kV	60
mA	66
PHD	13-65

CONCENTRATION CALIBRATIONS DETAILS AND COMPOUND CALIBRATION LINE DETAILS

Graph

Selected line



	0.40353
D	
E	3.08314
F	0.00000
RMS	0.30561
RE	0.05600
K	0.10249
Correlation	0.99849

General information

Name	Fe
Compound	Fe
Line	Fe-Kα1, 2-Fluorescent
Status	OK
Tags	GENERAL
Use	Yes

Regression coefficients

Unit %

	Value	Unit	Locked
D	0.40353	%	No
E	3.08314	%/kcps	No
F	0.00000	%/kcps²	Yes

Background	Yes
Ratio channel	Hf

Background Yes

Matrixmodel	None
Sample effects:	
Include finite thickness effects	No
Include fluorescence volume geometry (FVG) effects	No
Regression model	C=D+E·R·M
Error weighting	Square root

	Model	Constant	Unit
Error weighting	Square root	0.10000	%

	Value	Unit
RMS	0.30561	%
RE	0.05600	
K	0.10249	√%
Correlation	0.99849	

Measurement conditions

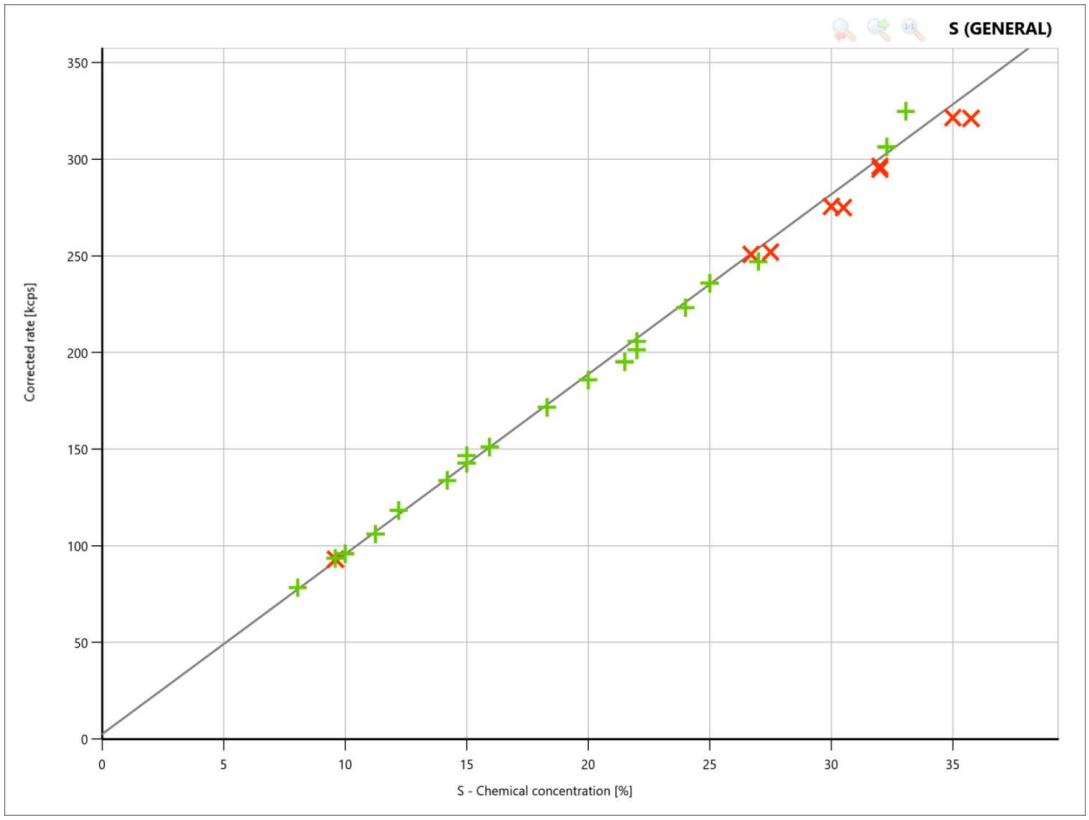
Measurement type	WD-G
Detector	Duplex
Tube filter	None
Medium	Vacuum
Crystal	LiF200
Collimator	300µm
Mask	37mm
kV	60
mA	66
PHD	14-70

CONCENTRATION CALIBRATIONS DETAILS AND COMPOUND CALIBRATION LINE DETAILS

Graph

Selected line

D	-0.26589



E	0.10741
F	0.00000
RMS	0.51246
RE	0.02088
K	0.10071
Correlation	0.99785

General information

Name	S
Compound	S
Line	S-Kα1, 2-Fluorescent
Status	OK
Tags	GENERAL
Use	Yes

Regression coefficients

Unit	%

	Value	Unit	Locked
D	-0.26589	%	No
E	0.10741	%/kcps	No
F	0.00000	%/kcps²	Yes

Background	Yes
Ratio channel	

Background	Yes
Matrix model	None
Sample effects:	
Include finite thickness effects	No
Include fluorescence volume geometry (FVG) effects	No
Regression model	C=D+E·R·M
Error weighting	Square root

	Model	Constant	Unit
Error weighting	Square root	0.10000	%

	Value	Unit
RMS	0.51246	%
RE	0.02088	
K	0.10071	√%
Correlation	0.99785	

Measurement conditions

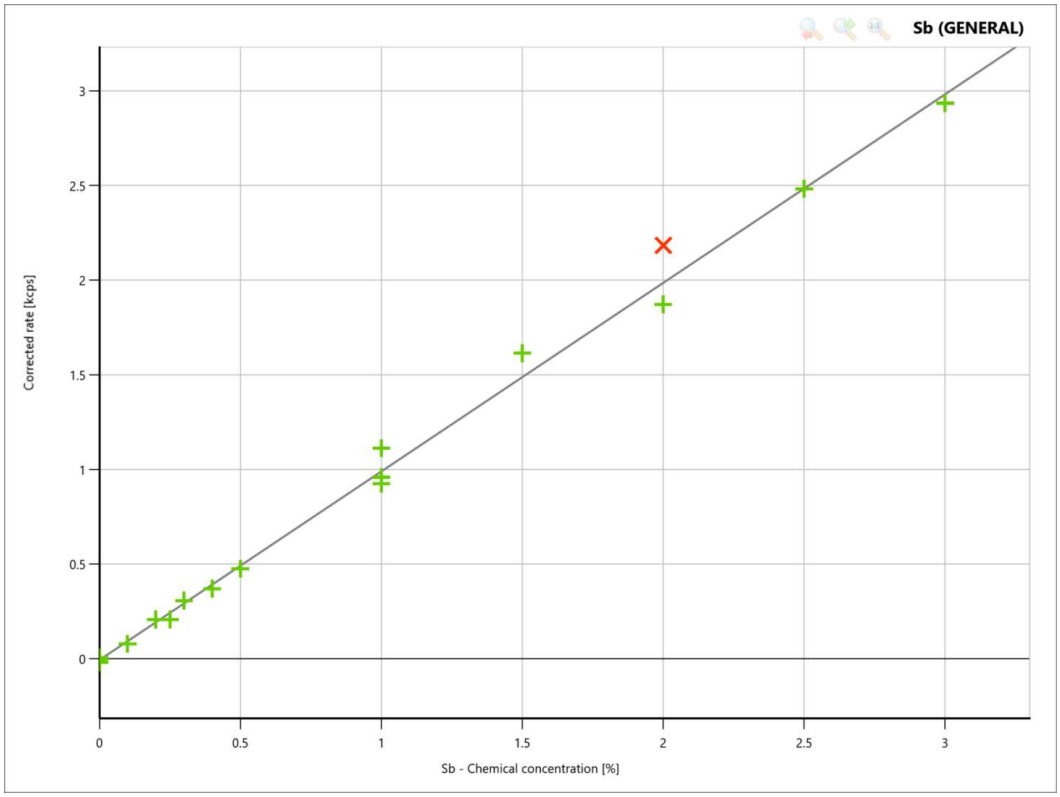
Measurement type	WD-G
Detector	Flow
Tube filter	None
Medium	Vacuum
Crystal	Ge
Collimator	300µm
Mask	37mm
kV	25
mA	160
PHD	29-71

CONCENTRATION CALIBRATIONS DETAILS AND COMPOUND CALIBRATION LINE DETAILS

Graph

Selected line

D	0.00663
E	1.00416



F	0.00000
RMS	0.04993
RE	0.07207
K	0.04732
Correlation	0.99848

General information

Name	Sb
Compound	Sb
Line	Sb-Lα1, 2-Fluorescent
Status	OK
Tags	GENERAL
Use	Yes

Regression coefficients

Unit	%

	Value	Unit	Locked
D	0.00663	%	No
E	1.00416	%/kcps	No
F	0.00000	%/kcps²	Yes

Background	Yes
Ratio channel	

Background	Yes
Matrix model	None
Sample effects:	
Includefinitethicknesseffects	No
Include fluorescence volume geometry (FVG) effects	No
Regression model	C=D+E·R·M
Error weighting	Square root

	Model	Constant	Unit
Error weighting	Square root	0.10000	%

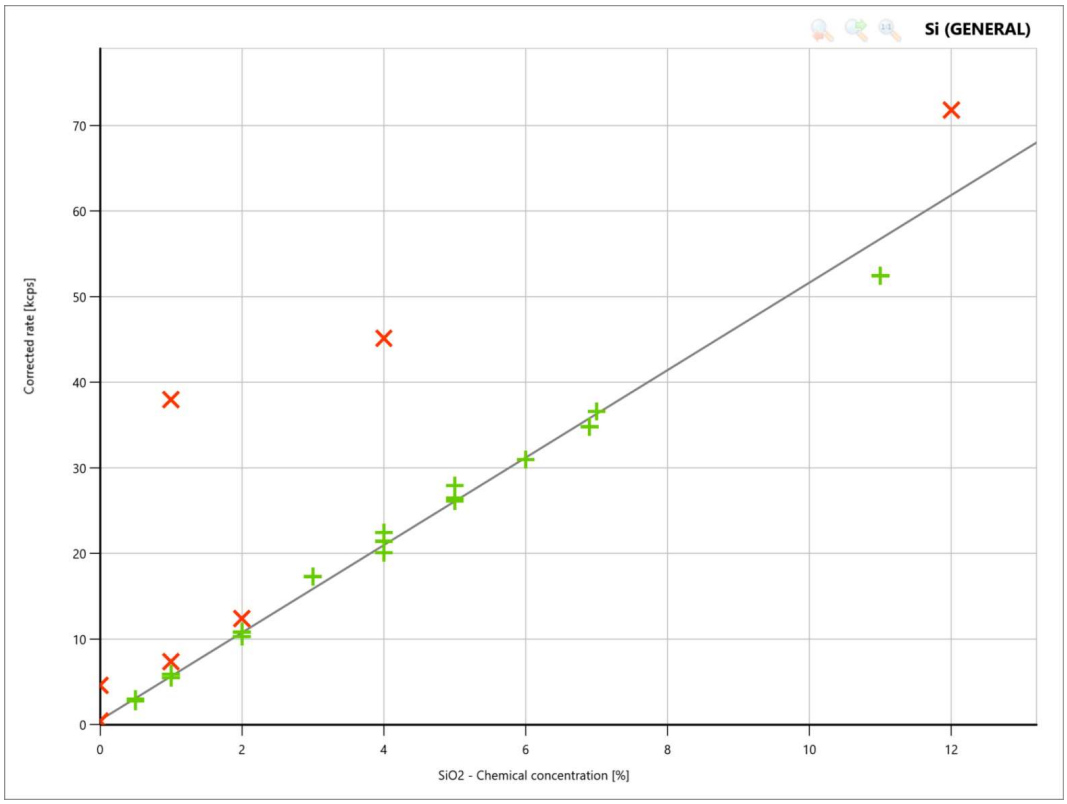
	Value	Unit
RMS	0.04993	%
RE	0.07207	
K	0.04732	√%
Correlation	0.99848	

Measurement conditions

Measurement type	WD-G
Detector	Flow
Tube filter	None
Medium	Vacuum
Crystal	LiF200
Collimator	150µm
Mask	37mm
kV	30
mA	133
PHD	31-72

CONCENTRATION CALIBRATIONS DETAILS AND COMPOUND CALIBRATION LINE DETAILS

Graph



Selected line

D	-0.09940
E	0.19564
F	0.00000
RMS	0.27136
RE	0.05418
K	0.10547
Correlation	0.99646

General information

Name	Si
Compound	SiO2
Line	Si-Kα1, 2-Fluorescent
Status	OK
Tags	GENERAL
Use	Yes

Regression coefficients

Unit %

	Value	Unit	Locked
D	-0.09940	%	No
E	0.19564	%/kcps	No
F	0.00000	%/kcps²	Yes

Background	Yes
Ratio channel	

Background	**Yes**
Matrix model	None
Sample effects:	
Include finite thickness effects	No
Include fluorescence volume geometry (FVG) effects	No
Regression model	C=D+E·R·M
Errorweighting	Square root

	Model	**Constant**	**Unit**
Error weighting	Square root	0.10000	%

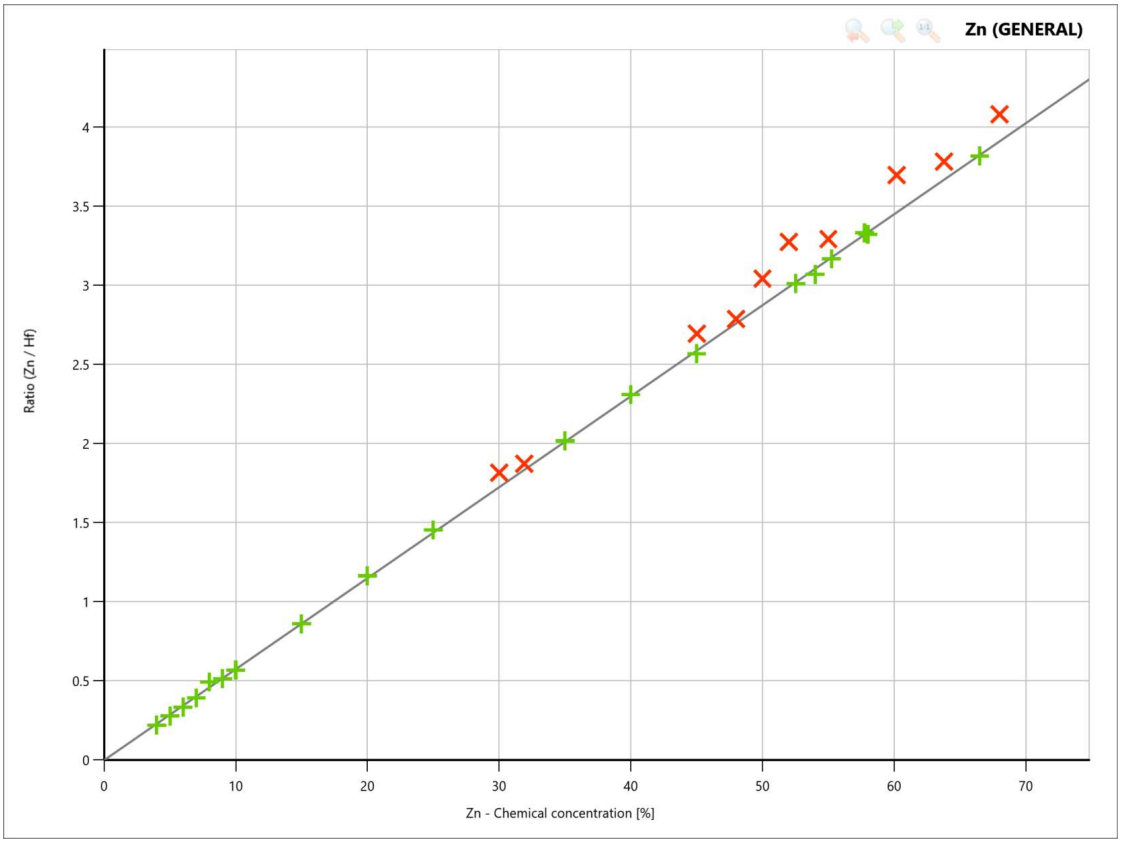
	Value	**Unit**
RMS	0.27136	%
RE	0.05418	
K	0.10547	√%
Correlation	0.99646	

Measurement conditions

Measurement type	WD-G
Detector	Flow
Tube filter	None
Medium	Vacuum
Crystal	InSb
Collimator	700µm
Mask	37mm
kV	25
mA	160
PHD	28-74

CONCENTRATION CALIBRATIONS DETAIL S AND COMPOUND CALIBRATION LINE DETAILS

Graph



Selected line

D	0.07640
E	17.38327
F	0.00000
RMS	0.30249
RE	0.02496
K	0.07606
Correlation	0.99993

	Value	Unit	Locked
D	0.07640	%	No
E	17.38327	%/kcps	No
F	0.00000	%/kcps²	Yes

General information

Name	Zn
Compound	Zn
Line	Zn-Kα1,2-Fluorescent
Status	OK
Tags	GENERAL
Use	Yes

Regression coefficients

Unit	%
Background	Yes
Ratio channel	Hf

Background — Yes

Matrix model	None
Sample effects:	
Include finite thickness effects	No
Include fluorescence volume geometry (FVG) effects	No
Regression model	C=D+E·R·M
Error weighting	Square root

	Model	Constant	Unit
Error weighting	Square root	0.10000	%

	Value	Unit
RMS	0.30249	%
RE	0.02496	
K	0.07606	√%
Correlation	0.99993	

Measurement conditions

Measurement type	WD-G
Detector	Duplex
Tube filter	Al/200
Medium	Vacuum
Crystal	LiF220
Collimator	150µm
Mask	37mm
kV	60
mA	66
PHD	12-68

CONCENTRATION CALIBRATIONS DETAILS ZN OVERLAP CORRECTIONS

Correction type	Using	Interfering compound/channel	Correction factor	Lock factor
Line overlap on C	Concentration of	Cu	0.019920	No
Line overlap on C	Concentration of	Fe	-0.00017079	No
Line overlap on C	Concentration of	Pb	0.0053223	No

DISCUSSION

Mining Industries produce Base metal concentrate and world smelters are customer to use concentrate to produce metals. During all the process quantification and exchange for commercial use , we require accurate analysis to pay and receive price with actual quantity. Now a days typical wet chemical analysis is very time consuming and expensive method to analyse many elements, so industries are looking for short time analysis with high accuracy. In this scenario, it is required to develop XRF methodology to analyse multiple elements with highest accuracy. Instrument selection is with best setup of crystals detector and collimators to acquire best specific intensity of each element even in very low level. The present study is an attempt to present the details of XRF methodology to analyse multiple elements with highest accuracy. Various parameters including principle, interferences, precision /rejection levels, flux/ sample/internal standards, reagents requirements, instrument calibration etc for the above method have been discussed. Concentration calibration overview of important compounds/elements namely Zn, Pb, Cu, Cd, Fe, CaO, SiO_2 ,Al_2O_3 ,MgO , Ba, Sb, Bi, As, S using above technique has been presented .

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[5] ISO/WD 21511 Copper, lead, zinc and nickel ores and their related products — Borate Fusion XRF analysis.

[6] ASTM E-1621 X-Ray Emission Spectrometric Analysis

[7] ASTM - E 1622 - Correction of Spectral Line Overlap in Wavelength-Dispersive X-Ray Spectrometry

[8] ASTM – E 1361 - Correction of Inter-element Effects in X-Ray Spectrometric Analysis

[9] ASTM – E 1172 - Describing and Specifying a Wavelength-Dispersive X-Ray Spectrometer

[10] ISO 9599, Copper, lead and zinc sulfide concentrates — Determination of hygroscopic moisture in the analysis sample — Gravimetric method