

The Challenges and Benefits of XRF Spectrometry at Xstrata Zinc Brunswick Smelter

Denis Foulem

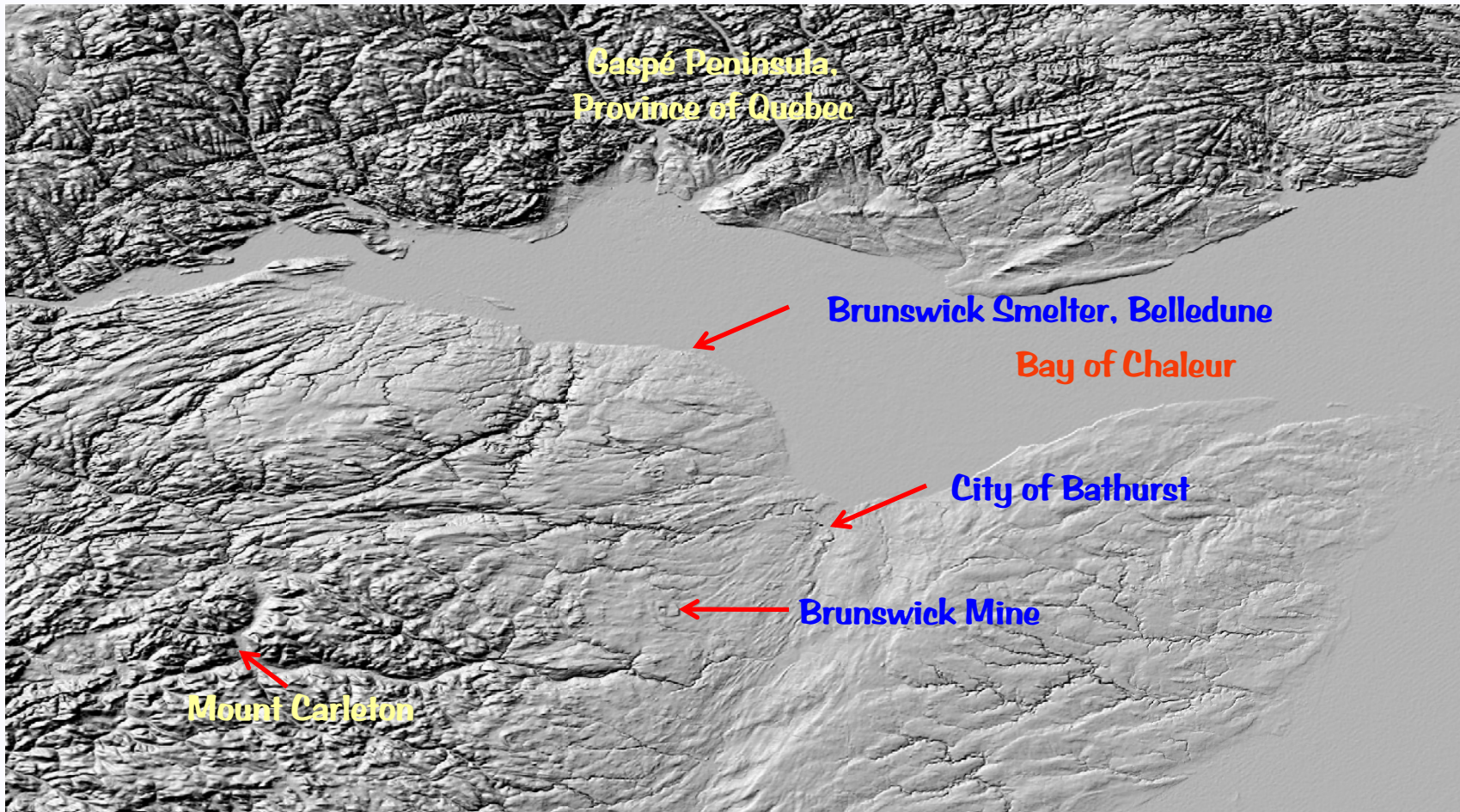
Brunswick Smelter, Belledune, New Brunswick, Canada

**20th Anniversary Symposium for the Short Course of
Modern X-Ray Spectrometry, University of Western Ontario**

Where is Brunswick Smelter?



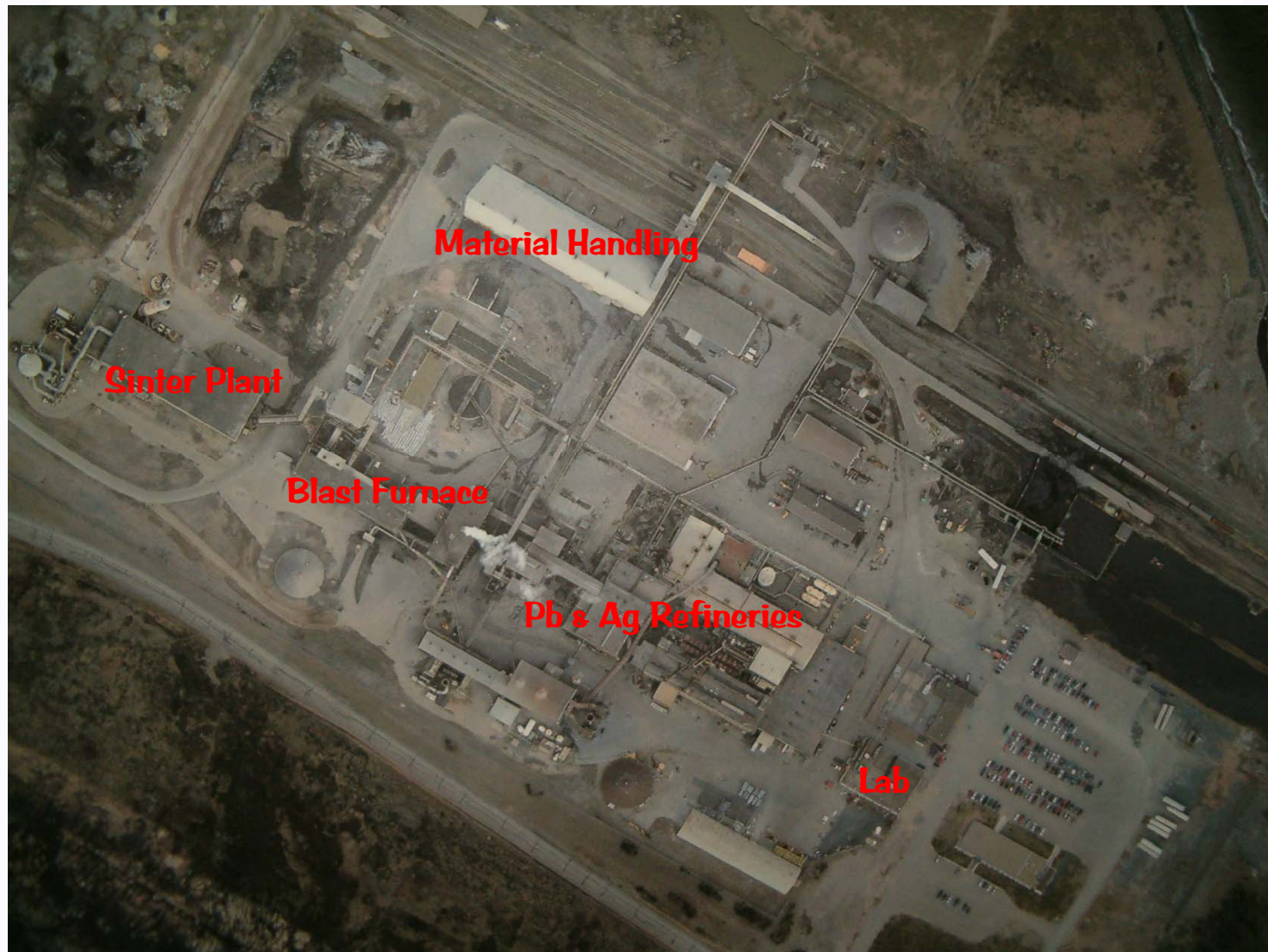
Northern New Brunswick



Brunswick Smelter



Brunswick Smelter Overview



The Lab



Philips PW2400 XRF spectrometers



- Two units, built in 1992, equipped with PW2510 sample changers
- X-Ray Tube rating: 20 to 60 kV, 10 to 125 mA
- Operating medium: vacuum or helium
- Crystals: PX1, PX4, LiF 200, LiF 220, PE 002, Ge 111

Never a dull moment!



XRF and the Pb Smelting Business



- **Good for XRF: Pb is present in practically all samples!**

xstrata

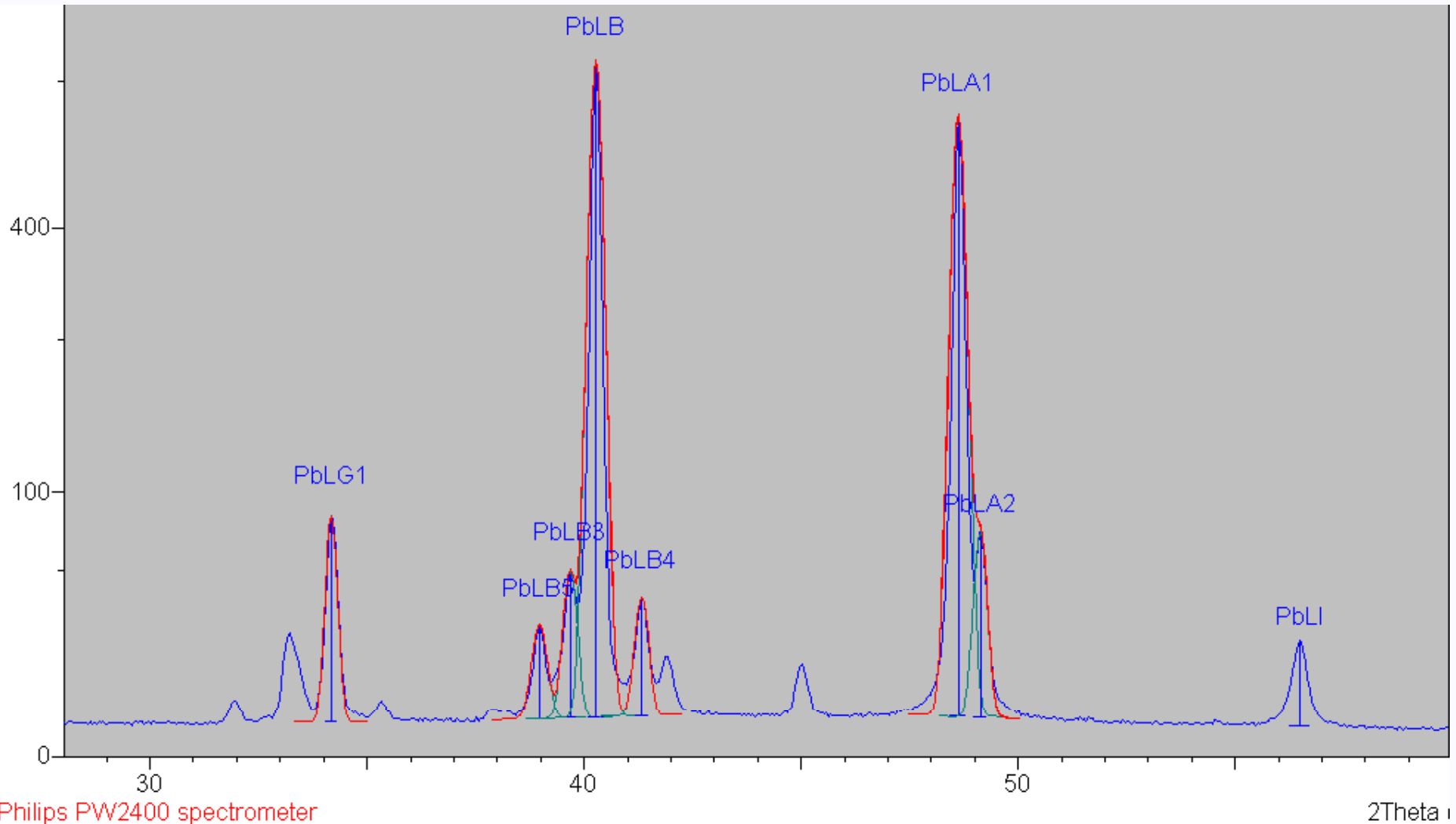
XRF and the Pb Smelting Business



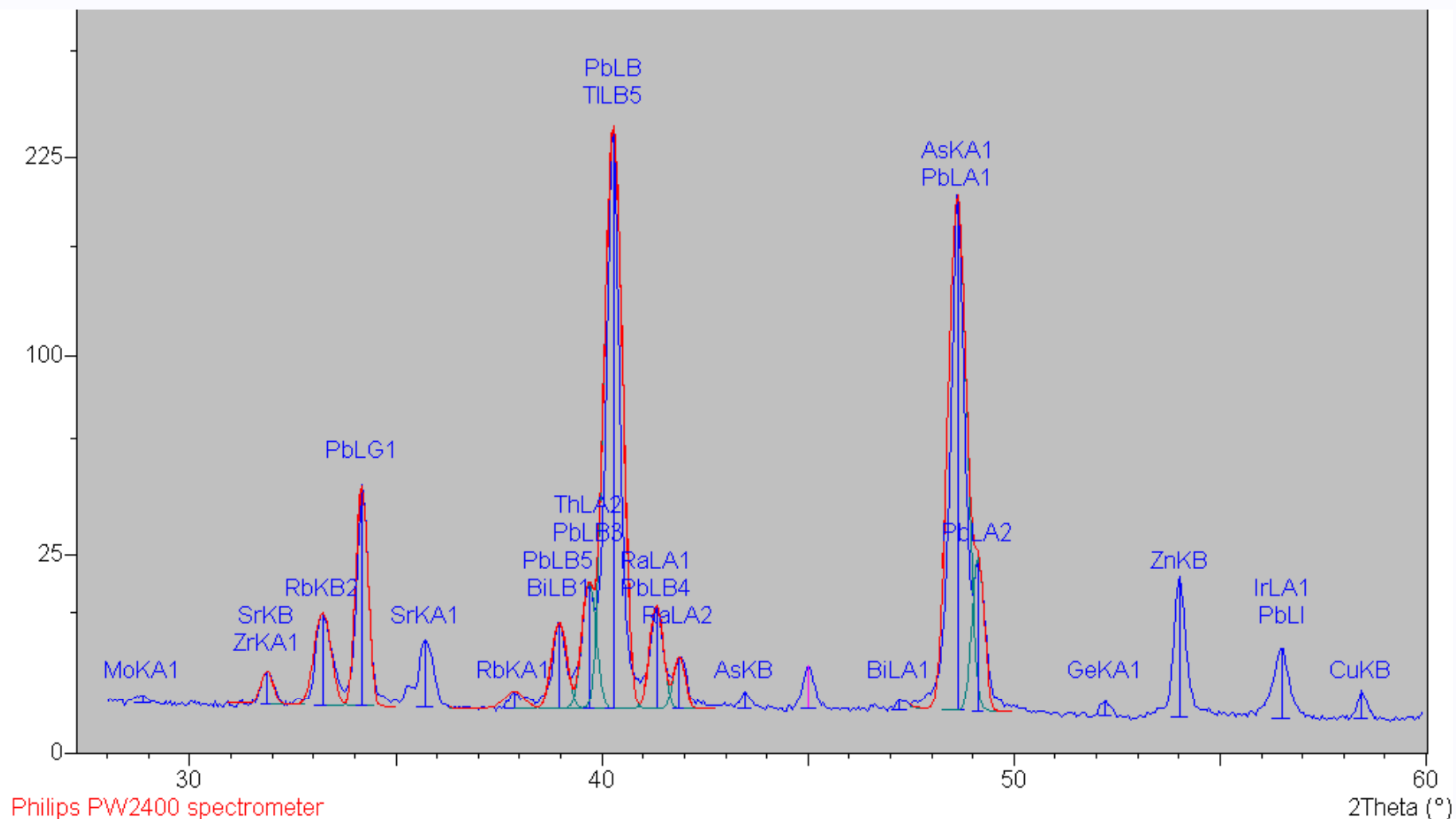
- **Bad for XRF: Pb is present in practically all samples!**

xstrata

Pb Effects on Full XRF Scan Spectra



Uncorrected Scan Interpretation



Directives for Scan Interpretation



XRF Scan Interpretation - Screen 2

H																			He
Li	Be											B	C	N	O	F		Ne	
Na	Mg											Al	Si	P	S	Cl		Ar	
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br		Kr	
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I		Xe	
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At		Rn	
Fr	Ra	Ac	Rf	Db	Sg	Bh	Hs	Mt	Ds	Rg	Cn								

Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr

- K Lines
- L Lines
- M Lines

Directives

- Pb $L\alpha$ < Pb $L\beta$
- At 45° there is a Pb peak not assigned by the software; ignore it.
- Ignore Zr $K\alpha$ as it occurs at the same location as a Pb line; if Zr $K\beta$ is present use only that line.
- Delete As $K\alpha$ if As $K\beta$ is absent.
- Se $K\beta$ won't appear due to the presence of Pb; ignore it.
- Readjust all peaks associated with the "Pb Hand" located left of center.
- As $K\beta_2$ is not in software; ignore it.
- Br $K\alpha$ is on peak at the far left on the "Pb Hand"; assign it as such only if Br $K\beta$ is present at left of "Pb Hand".
- Bi $L\beta$ is also on far left peak of "Pb Hand"; make sure it isn't overevaluated by checking Bi $L\alpha$ located on left side of Pb peak at right (calculate $L\beta$ peak height based on expected % peak size compared to $L\alpha$).
- Ir L peak is usually spurious, actually a Pb peak; use 'Delete Element' feature to remove it.
- There is no Hg $L\beta$ line.
- Suspect the presence of Ge K lines when analyzing Pb Sulphate samples
- Sr $K\alpha$ near unidentified Pb peak; use only if Sr $K\beta$ is present. Note: Sr $K\beta$ cannot be used as it occurs at the same location as a Pb line, as well as Zr $K\alpha$.
- Ignore Rb $K\alpha$ at 38° unless it is very strong, i.e. higher than the Pb peak at its right.
- Small Pb peak at right of "Pb Hand" (near 42°) is not assigned by the software; ignore it.
- Identify Hg $L\alpha$ only if it's well-defined and its presence is plausible, i.e. the material analyzed isn't a high-temperature residue.
- If the As $K\beta$ peak is especially strong, a secondary As Peak will appear at 43° but is not assigned by the software; ignore it.
- If both Pb and Bi are present, ignore peak at 39° as it corresponds to both Pb $L\beta_5$ and Bi $L\beta_1$ peaks.

Pb Effects on Production Samples



SuperQ / quantitative (system set-up) - [Application - SINTER]

File System Application Monitor Window Help

Application items

General Sample description Quantitative program
 Identification scheme Application compounds Qualitative program
 Conditions Application channels

Application compounds

Normalise Normalise to (%): 100.00 Lock normalise

Compound	Formula	El.	Layer	Source	Unit	Dec.	Minimum	Maximum
OSAg	Calc. Func.		1	Calc. Func.	%	3		
OSAl2O3	Calc. Func.		1	Calc. Func.	%	2		
OSAs	Calc. Func.		1	Calc. Func.	%	2		
OSBi	Calc. Func.		1	Calc. Func.	%	2		
OSCaO	Calc. Func.		1	Calc. Func.	%	2	5.000	15.000
OSCd	Calc. Func.		1	Calc. Func.	%	2		
OSCu	Calc. Func.		1	Calc. Func.	%	2		2.000
OSFeO	Calc. Func.		1	Calc. Func.	%	2	10.000	25.000
OSK	Calc. Func.		1	Calc. Func.	%	2		
OSMgO	Calc. Func.		1	Calc. Func.	%	2		
OSNa	Calc. Func.		1	Calc. Func.	%	2		
OSPb	Calc. Func.		1	Calc. Func.	%	2	30.000	50.000
OSS	Calc. Func.		1	Calc. Func.	%	2		3.500
OSSb	Calc. Func.		1	Calc. Func.	%	2		
OSSI02	Calc. Func.		1	Calc. Func.	%	2	5.000	15.000
OSZn	Calc. Func.		1	Calc. Func.	%	2		5.000
Ag	Ag	Ag	1	XRF	%	3		
Al2O3	Al2O3	Al	1	XRF	%	2		
As	As	As	1	XRF	%	2		
Bi	Bi	Bi	1	XRF	%	2		
CaO	CaO	Ca	1	XRF	%	2		
Cd	Cd	Cd	1	XRF	%	2		
Cu	Cu	Cu	1	XRF	%	2		
FeO	FeO	Fe	1	XRF	%	2		
K	K	K	1	XRF	%	2		
MgO	MgO	Mg	1	XRF	%	2		
Na	Na	Na	1	XRF	%	2		
Pb	Pb	Pb	1	XRF	%	2		

QAQC for XRF



Denis Foulem - LabStats - Explorer

File View Tools Help

New Open Graph Reports Delete Properties Help Log Off

LabStats

- Environmental Lab Method
- Environmental Lab Method Arch
- Environmental Lab Procedure
- Medical Lab
- Spec Lab
 - DES
 - X-Ray 1 vs Wet
 - X-Ray 2 vs Wet
 - XRF 1 - QAQC Check
 - XRF 2 - QAQC Check
 - XRF-1 VIR Check
 - XRF-2 VIR Check
 - XRF1 - 2 Hrs QAQC
 - XRF2 - 2 Hrs QAQC

DES

X-Ray 1 vs Wet

X-Ray 2 vs Wet

XRF 1 - QAQC Check

XRF 2 - QAQC Check

XRF-1 VIR Check

XRF-2 VIR Check

XRF1 - 2 Hrs QAQC

XRF2 - 2 Hrs QAQC

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LabStats

- Environmental Lab Method
- Environmental Lab Method Arch
- Environmental Lab Procedure
- Medical Lab
- Spec Lab
 - DES
 - X-Ray 1 vs Wet
 - OS Fusion - Wet
 - OS Pressed - Wet
 - RS Pressed - Wet
 - SL Fusion - Wet
 - SL Pressed - Wet
 - SL Ratio Fusion
 - SMF Pressed - Wet

A12O3 (1.5%)

CaO (10%)

Cu (0.5%)

FeO (20%)

MgO (0.5%)

Pb (40%)

S (1.5%)

SiO2 (10%)

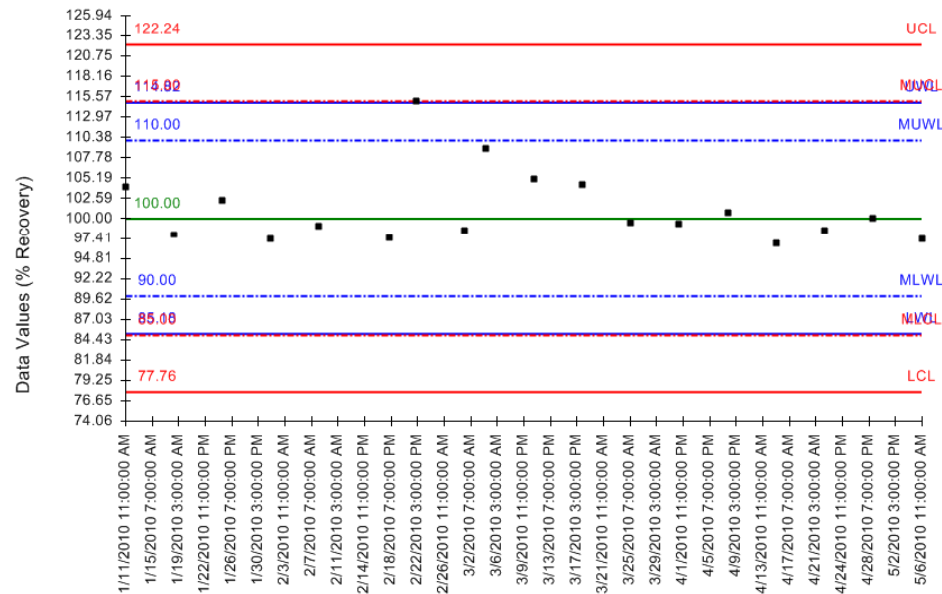
Zn (4%)

Data Comparison and Graphs



Denis Foulem - LabStats - Response Data										
Instrument	Analyte	Test	Date	Time	Analyst	Measured Value 1	Measured Value 2	Lot Number	Calculated Value	
X-Ray 1 vs Wet	OS Fusion - Wet	Al2O3 (1.5%)	5/6/2010	11:00:00 AM	Foulem, Denis (4...	1.76	1.81		97.24	
X-Ray 1 vs Wet	OS Fusion - Wet	Al2O3 (1.5%)	4/29/2010	11:00:00 AM	Foulem, Denis (4...	1.87	1.87		100.00	
X-Ray 1 vs Wet	OS Fusion - Wet	Al2O3 (1.5%)	4/22/2010	11:00:00 AM	Foulem, Denis (4...	1.84	1.87		98.40	
X-Ray 1 vs Wet	OS Fusion - Wet	Al2O3 (1.5%)	4/15/2010	11:00:00 AM	Foulem, Denis (4...	1.79	1.85		96.76	
X-Ray 1 vs Wet	OS Fusion - Wet	Al2O3 (1.5%)	4/8/2010	11:00:00 AM	Foulem, Denis (4...	1.53	1.52		100.66	

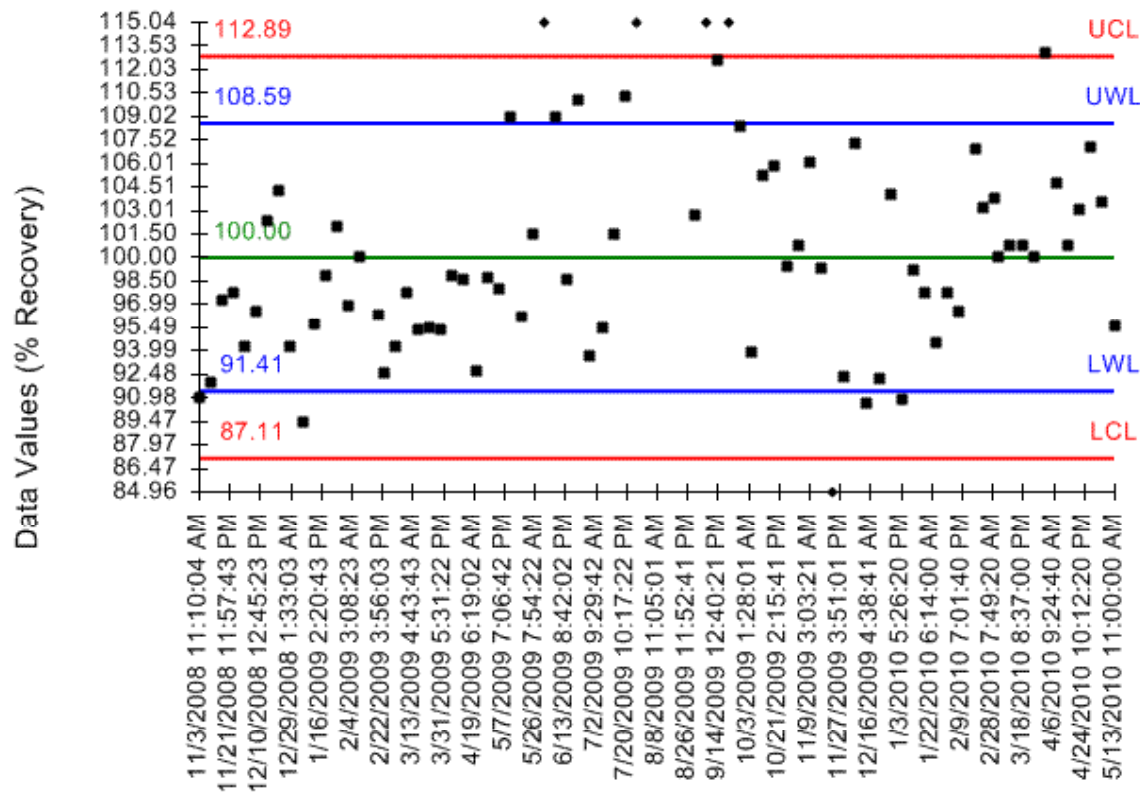
X-Ray 1 vs Wet - OS Fusion - Wet - Al2O3 (1.5%)



Scatter effect on Sulfur (Example)



X-Ray 1 vs Wet - OS Fusion - Wet - S (1.5%)



Lab Stats© Statistics on the fly



Laboratory	Instrument	Analyte	Test
Spec Lab	XRF 1 - QAQC Check	HGP Dust	Tl (8.2%)
Spec Lab	XRF 1 - QAQC Check	HGP Dust	Zn (0.34%)
Spec Lab	XRF 1 - QAQC Check	OS Fused	As (0.57%)
Spec Lab	XRF 1 - QAQC Check	OS Fused	Cd (0.18%)
Spec Lab	XRF 1 - QAQC Check	OS Fused	K (0.32%)
Spec Lab	XRF 1 - QAQC Check	OS Fused	Sb (0.20%)
Spec Lab	XRF 1 - QAQC Check	Return Sinter	As (0.72%)
Spec Lab	XRF 1 - QAQC Check	Return Sinter	Cd (0.24%)
Spec Lab	XRF 1 - QAQC Check	Return Sinter	FeO (19.63%)
Spec Lab	XRF 1 - QAQC Check	Return Sinter	MgO (0.69%)
Spec Lab	XRF 1 - QAQC Check	Return Sinter	Pb (37.16%)
Spec Lab	XRF 1 - QAQC Check	Return Sinter	Zn (2.7%)
Spec Lab	XRF 1 - QAQC Check	Slag Fused	As (0.26%)
Spec Lab	XRF 1 - QAQC Check	Slag Fused	Cd (0.01%)
Spec Lab	XRF 1 - QAQC Check	Slag Fused	K (0.68%)
Spec Lab	XRF 1 - QAQC Check	Slag Fused	Na (1.98%)
Spec Lab	XRF 1 - QAQC Check	SMF	CaO (10.15%)
Spec Lab	XRF 1 - QAQC Check	SMF	Cu (0.67%)
Spec Lab	XRF 1 - QAQC Check	SMF	FeO (17.82%)
Spec Lab	XRF 1 - QAQC Check	SMF	Pb (36.03%)
Spec Lab	XRF 1 - QAQC Check	SMF	SiO2 (9.49%)
Spec Lab	XRF 1 - QAQC Check	SMF	Zn (2.43%)
Spec Lab	XRF 2 - QAQC Check	Cu Speiss	As (19%)
Spec Lab	XRF 2 - QAQC Check	Cu Speiss	Fe (0.91%)
Spec Lab	XRF 2 - QAQC Check	Cu Speiss	Pb (15.13%)
Spec Lab	XRF 2 - QAQC Check	Cu Speiss	Sb (1.55%)
Spec Lab	XRF 2 - QAQC Check	HGP Dust	Cd (1.13%)

<u>Statistics</u>	<u>Response Data</u>	<u>Control Data</u>
Average	102.80%	99.99%
Standard Deviation	2.62	2.00
% Relative Standard Deviation:	2.54	2.00

Precision Decision

F Calculated	0.0000
F Lookup Value	3.7870
Degrees of Freedom (Response)	7
Degrees of Freedom (Control)	41
Significant Shift in Precision?	No

Bias Decision

Students t Test for Bias	3.0288
t Lookup Value	2.3646
Degrees of Freedom	7
Significant Bias	Yes
Persuasive Bias	Yes

Conclusion

XRF Spectrometry at Xstrata Zinc Brunswick Smelter:

- **Multitasking**
- **Validation of data by Wet Lab checks and data management**
- **Maintain accuracy of assays**
- **Perform full XRF scans on a regular basis**
- **Troubleshooting**
- **Questions?**