Materials Characterisation by XRF & OES

Summer Symposium 2011

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XRF, XRD, OE & Automation Products

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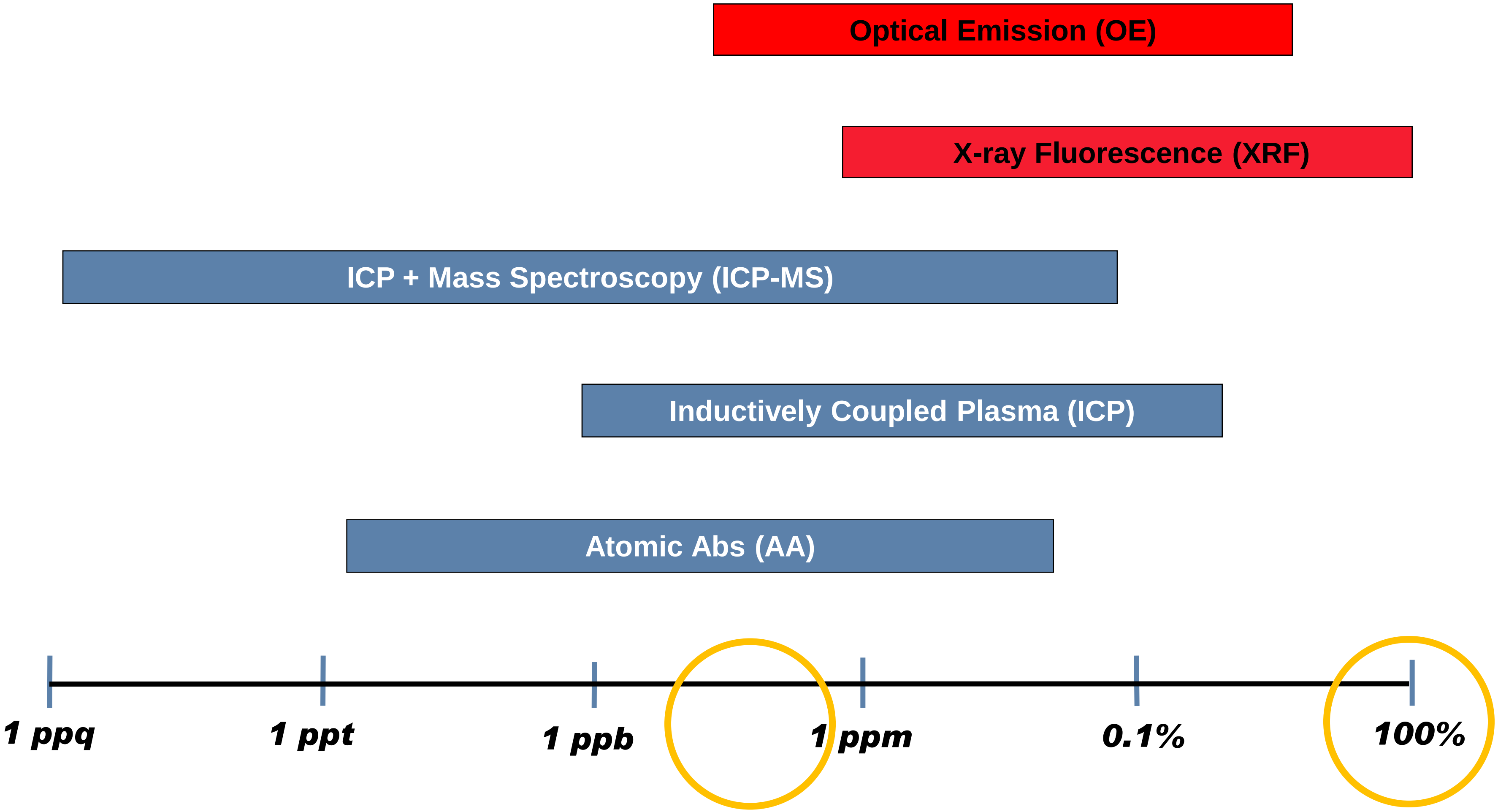
XRF & OES in the Laboratory



Why use Arc/Spark OES?

- Extremely fast and simple analysis of conducting solids
- Can analyze simultaneously > 40 elements from trace (ppm) to major (%) levels in <1 minute
    - ppm or sub-ppm limits of detections
    - Can analyze P, S, C, N, O at low levels
- Sample preparation is fast and simple
- Accurate, precise, stable, reliable
- Cheap analysis: low costs of ownership and maintenance
- High instrument availability
- Long life and robustness
- Well established (exists since 1934)

OES Techniques

TECHNIQUES	
▪ PMT	Uses a photo-multiplier tube, one per element line in conjunction with a mirror and slit
▪ CCD	Uses a solid state detector, covering a range of wavelengths

Todays Product Portfolio - OES

ARL

QuantoDesk

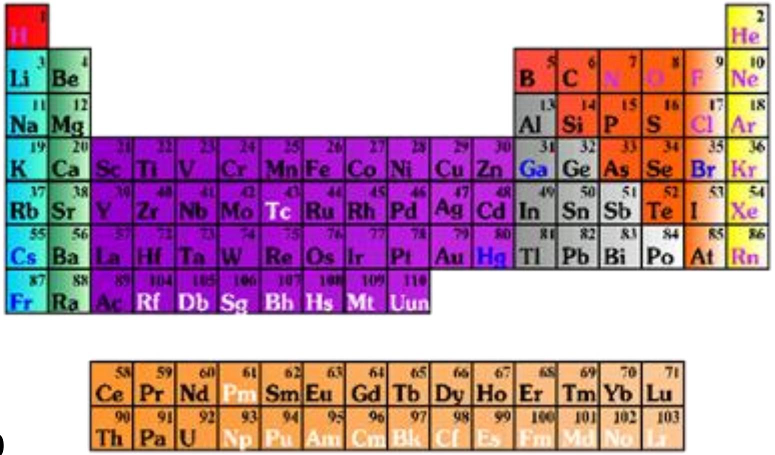
ARL

3460 & 4460

ARL

SMS 2500

What is so great about X-ray Fluorescence ?



1 H																	2 He
3 Li	4 Be											5 B	6 C	7 N	8 O	9 F	10 Ne
11 Na	12 Mg											13 Al	14 Si	15 P	16 S	17 Cl	18 Ar
19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr
37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe
55 Cs	56 Ba	57 La	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn
87 Fr	88 Ra	89 Ac	104 Rf	105 Db	106 Sg	107 Bh	108 Hs	109 Mt	110 Uun								

58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu
90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr

- Multi-element analysis
- Multi-matrix
- Inorganic and Organic materials
- High precision and highly reliable
- Wide dynamic range: sub-ppm to 100%
- Variety of sample types: bulk solids, liquids, loose powders, irregular samples, filters, thin films –
- Analysis of totally unknown samples using Standard-less programs, e.g. UniQuant
  - Physics of XRF and analytical techniques are well established

X-ray Techniques

TECHNIQUES	
XRF	(X-ray fluorescence) is used for chemical characterization (elemental analysis) in solids, powders or liquids, crystalline or not
XRD  	(X-ray diffraction) is used for structural characterization (phase analysis) in crystalline materials - inorganic or organic
	A combination of both provides more complete materials analysis
WDXRF	uses appropriate crystals to separate the emission spectrum into discrete wavelengths before detecting them
EDXRF	uses a detector directly to process the complete spectrum into an energy dispersive scale

Todays Product Portfolio - XRF

ARL QUANT'X

ARL OPTIM'X

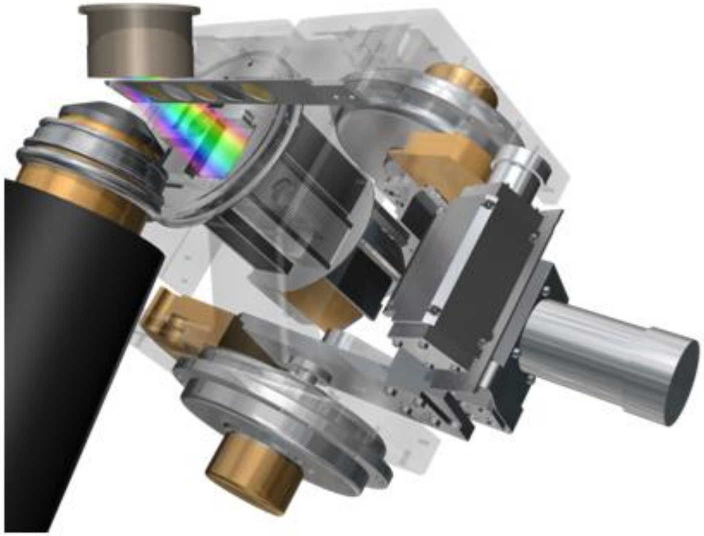
ARL PERFORM'X

ARL 9900

Typical Sample Types for XRF Analysis

Thermo ARL PERFORM'X

- 2.5 kW
  - No external water needed
  - Mapping
  - Small Spot
- 4.2 kW
  - Max sensitivity and resolution
  - Mapping
  - Small Spot



WDXRF Types of Elemental Analysis

Analysis Types

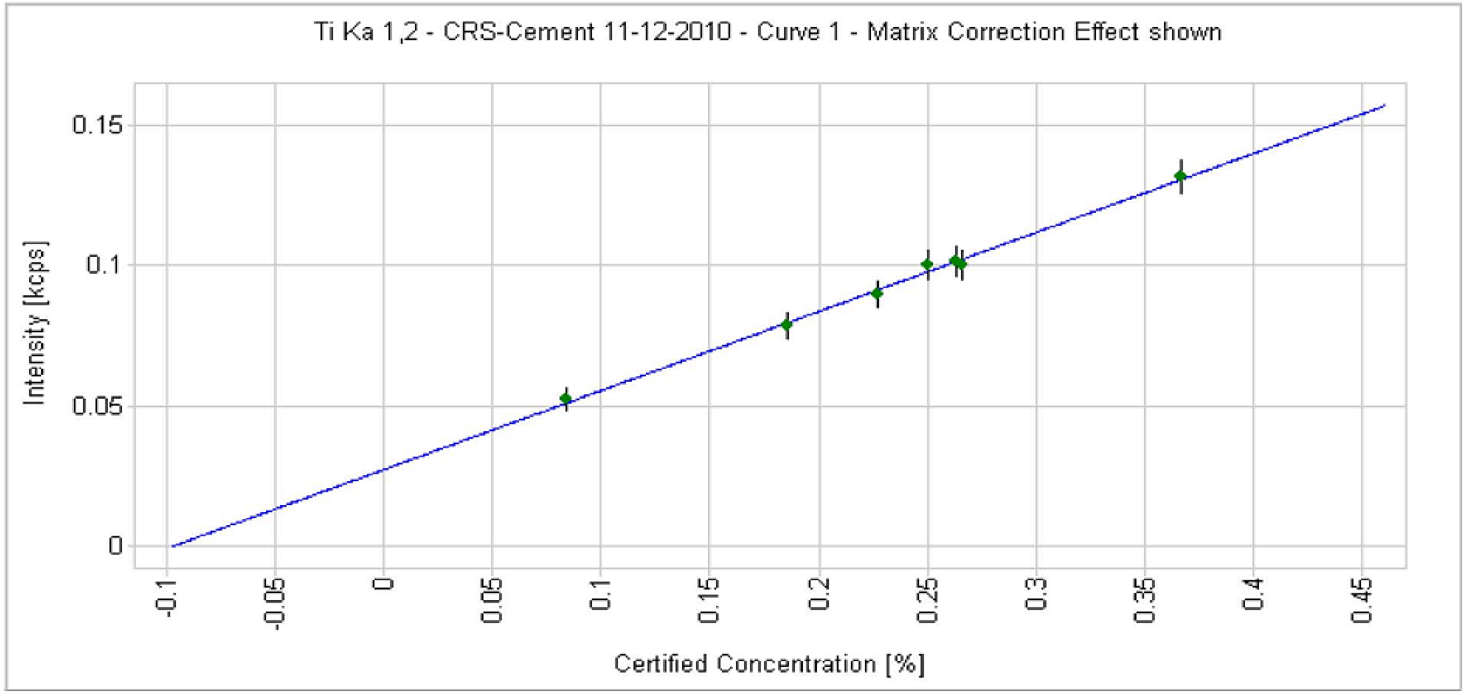
- Standard Linear Regression Analysis
  - Factory calibrations
  - Onsite calibrations
  - Create your own

- Semi-Quantitative or Standard-less Analysis
  - QuantAS
  - UniQuant

- Qualitative Analysis
  - Scans

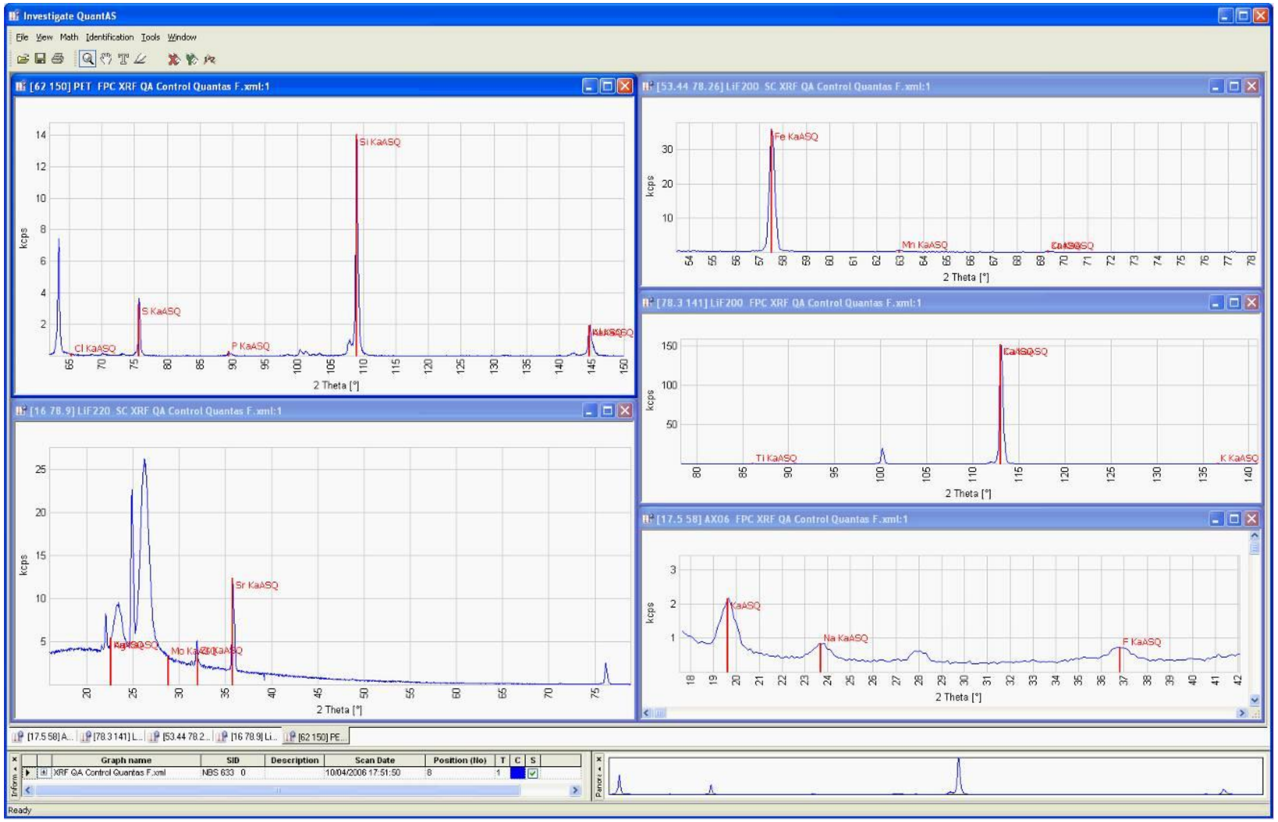
Linear Regression Analysis

- Standard Concentration vs. Intensity
  - Must have standards
  - Calibration is matrix matched
  - Empirical corrections are more accurate than Fundamental Parameters



QuantAS™ – scan-based standard-less software

- The user friendly QuantAS optional package determines quickly concentration levels in unknown liquid or solids samples.
- Full scan covering 70 elements from Fluorine to Uranium can be done in only 3 minutes.



		Graph name	SID	Description	Scan Date	Position (No)	T	C	S
▶	⊞	XRF QA Control Quantas F.xml	NBS 633 0		10/04/2006 17:51:50	8	1	🟦	☑

QuantAS™ – scan-based standard-less software

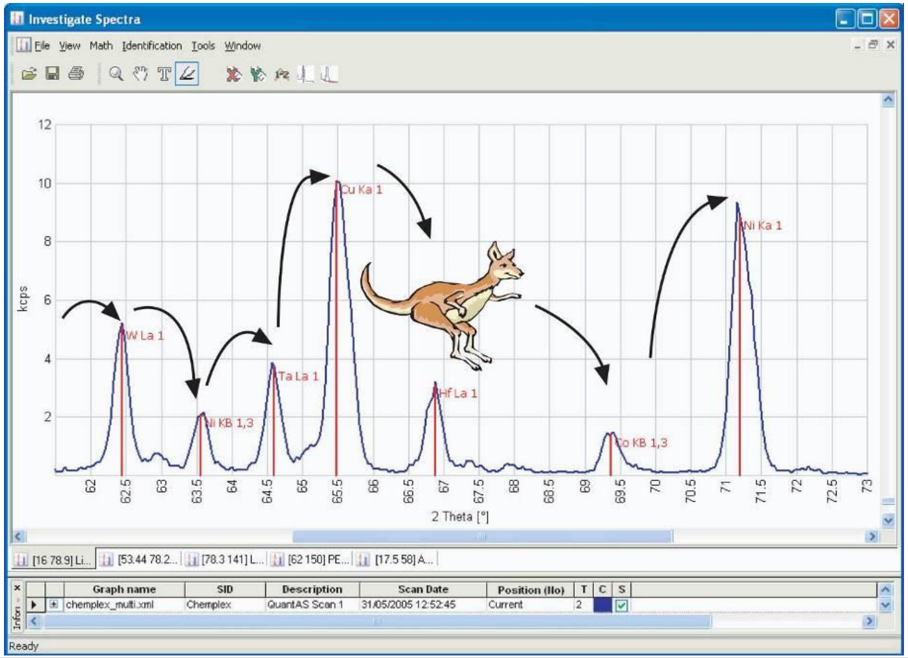
Typical Oxide compound

- Choice of counting time allows fast screening of unknown sample
- Longer counting time provide better limits of detection and determination of lighter elements

	Time Factor Durations of Scan		
	0.2	**0.5**	**1**
Elements	**(2min 40 sec)**	**(6min 16sec)**	**(12min 12sec)**
CaO (%)	42.8	43.1	42.8
SiO2 (%)	31.3	31.7	32.1
Al2O3 (%)	10.2	9.55	9.49
MgO (%)	5.12	5.06	5.10
MnO (%)	2.37	2.39	2.39
SO3 (%)	2.10	2.06	2.11
K2O (%)	1.80	1.63	1.71
Na2O (%)	1.42	1.22	1.26
TiO2 (%)	1.04	0.93	0.88
Fe2O3 (%)	0.96	0.95	0.93
P2O5 (%)	0.62	0.66	0.60
V2O5 (%)	0.21	0.21	0.21
SrO (%)	0.038	0.033	0.044
ZrO2 (%)	0.030	0.022	0.022
La2O3 (%)		0.073	0.051
Y2O3 (%)		0.025	0.025
Cr2O3 (%)			0.014
F (%)			0.092

UniQuant® - industry leading standard-less analyses

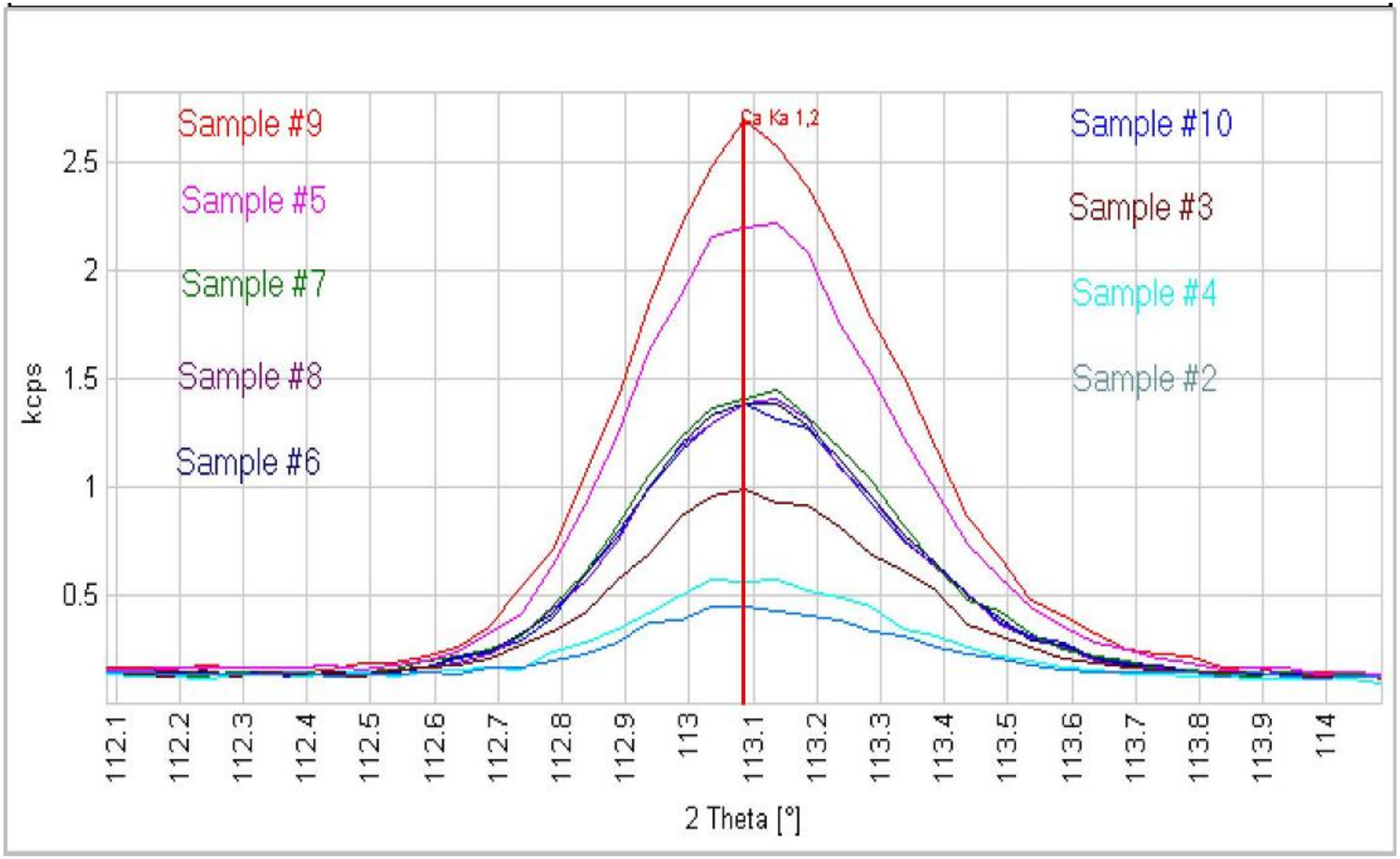
- Most advanced and powerful Fundamental Parameters algorithms
- Ideal for analysis of up to 79 elements in solid and liquids
  - when standard samples are not available
  - when samples can only be obtained in small quantities
  - or as irregular shapes
  - or coatings and layers on a substrate



Graph name	SID	Description	Scan Date	Position (Ilo)	T	C	S
chemplex_multi.xml	Chemplex	QuantAS Scan 1	31/05/2005 12:52:45	Current	2	■	☑

Scan Analysis

- Qualitative peak overlays
  - Quick comparisons of intensities



Elemental Analysis on Metals

The world leader in serving science

Metals Analysis - Copper

Element	Line	Sd (ppm)	LoD (ppm)
Al	Ka	1.36	2.5
Si	Ka	n.d.	2.55
Si	Ka	n.d.	2.24
P	Ka	2.48	4.55
S	Ka	n.d.	0.68
Cr	Ka	0.33	0.94
Mn	Ka	0.21	0.79
Fe	Ka	0.46	1.04
Co	Ka	0.34	0.76
Ni	Ka	0.31	1.49
Zn	Ka	1.45	3.22
Bi	La	0.95	3.29
As	Kb	4.33	10.5
Pb	Lb	1.13	4.38
Ag	Ka	1.69	6.23
Ag	La	5.51	16.6
Cd	Kb	2.85	7.33
Sn	Ka	2.12	5.97
Sb	Ka	1.27	6.3
Te	Ka	1.88	7.09

- Copper Base Analysis

Element	Line	Conc. (ppm)	RSD (ppm)	RSD %
Pb	Ka	421	3.5	0.83
As	Kb	112	4	3.57
Bi	La	336	1.7	0.5
Cd	Ka	105	2.9	2.76
Cr	Ka	156	0.4	0.28
Fe	Ka	505	0.6	0.12
Mn	Ka	594	0.6	0.1
Ni	Ka	145	0.5	0.37
Pb	Lb	462	2	0.43
Sb	Ka	625	3	0.48
Sn	Ka	271	2	0.75
Te	Ka	535	2.8	0.53
Ag	Ka	361	2.3	2.36

- 100 second per element: 12 day stability analysis for %RSD

Metals Analysis – Aluminum

Element	Line	Sd (ppm)	LoD (ppm)
Na	Ka	n.d.	4.75
Mg	Ka	2.58	7.64
Si	Ka	n.d.	1.47
Ti	Ka	0.3	0.43
La	La	0.43	1.36
Ce	La	0.35	0.83
V	Ka	0.14	0.46
Cr	Ka	0.17	0.43
Mn	Ka	0.15	0.53
Fe	Ka	0.17	0.61
Co	Ka	0.15	0.46
Ni	Ka	0.21	0.52
Cu	Ka	0.18	0.50
Zn	Ka	0.13	0.48
Ga	Ka	0.15	0.44
As	Kb	0.34	1.10
Zr	Ka	0.1	0.32
Ag	Lb	0.61	1.92
Cd	Ka	0.46	1.50
In	Ka	0.51	1.78
Sn	Ka	0.62	1.60
Sb	Ka	n.d.	2.06
Hg	La	0.36	1.10
Tl	La	n.d.	0.43
Pb	Lb	0.3	1.00
Bi	La	n.d.	0.57

- Aluminum Base Analysis

Element	Line	Conc (ppm)	RSD (ppm)	RSD %
Cd	Ka	193	0.8	0.4
Cr	Ka	402	0.5	0.13
Cu	Ka	428	0.4	0.08
Fe	Ka	843	0.7	0.9
Ga	Ka	53	0.2	0.35
Mn	Ka	513	0.6	0.11
Ni	Ka	498	0.4	0.09
Sn	Ka	122	0.6	0.51
Sb	Ka	23	1	4.52
Ti	Ka	422	0.7	0.17
Zn	Ka	468	0.4	0.08
Zr	Ka	22	0.1	5.4
Pb	Lb	154	0.4	0.26

- 100 second per element: 12 day stability analysis for %RSD

Metals Analysis – Iron

Element	Line	Sd* (ppm)	LoD (ppm)
Al	Ka	1.59	**4.3**
Si	Ka		**1.9**
P	Ka	0.48	**1.1**
S	Ka		**0.7**
Ti	Ka	0.31	**0.9**
V	Ka	0.29	**0.9**
Cr	Ka	0.3	**1**
Mn	Ka	0.47	**1.6**
Co	Ka	1.45	**4**
Ni	Ka	0.98	**2.4**
Cu	Ka	0.62	**2**
Ta	Lb	2.24	**7.3**
W	La	1.71	**4.9**
As	Kb	2.94	**7.9**
Pb	Lb	1.22	**3.8**
Zr	Ka	0.45	**1.3**
Nb	Ka	0.41	**1.2**
Mo	Ka	0.32	**1.3**
Sn	Ka	2.65	**7.3**
Sb	Ka	2.44	**8.7**

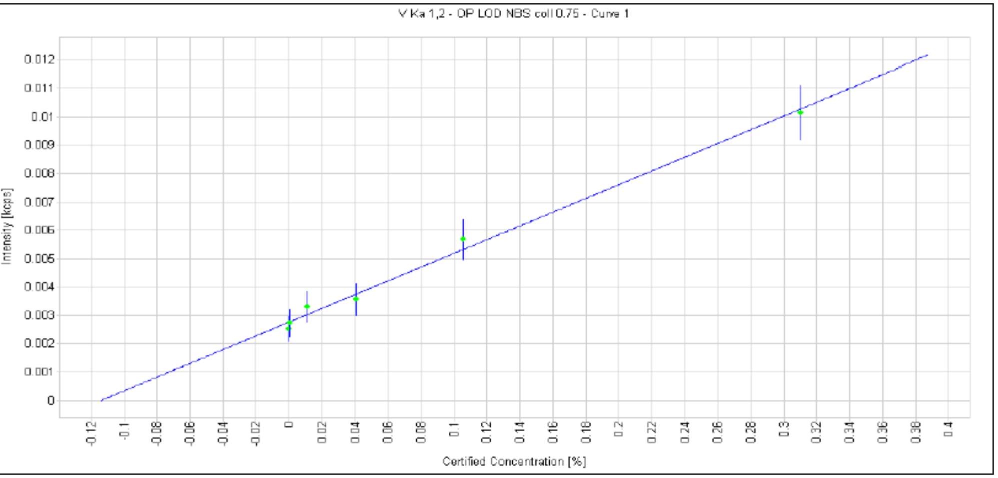
- Iron Base Analysis

Element	Line	Conc (ppm)	RSD (ppm)	RSD %
P	Ka	432	1.1	0.26
S	Ka	340	0.9	0.26
Al	Ka	840	2.6	0.31
Ti	Ka	959	1	0.11
Cr	Ka	3034	1.6	0.05
Ni	Ka	6164	4.1	0.07
Cu	Ka	5243	3.5	0.07
As	Ka	980	4.1	0.42
Nb	Ka	795	1.9	0.06
Mo	Ka	179	1.1	0.13
Sb	Ka	2139	3	1.66
W	La	2139	3.8	0.18
Pb	Lb	55	1.2	2.17
TA	Lb	2207	4.8	0.22

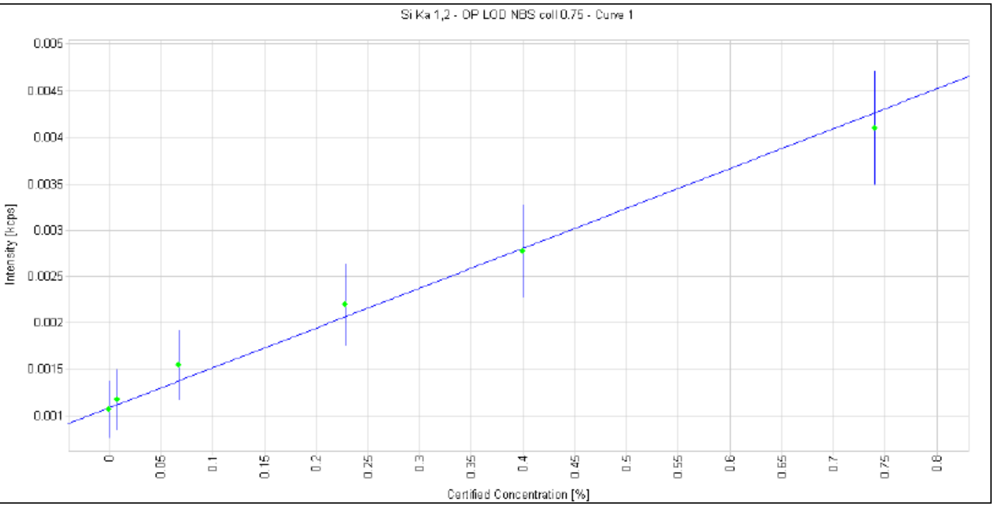
- 100 second per element: 12 day stability analysis for %RSD

Small Spot Calibration 0.5 mm for Ferrous Base

- V Regression



- Si Regression

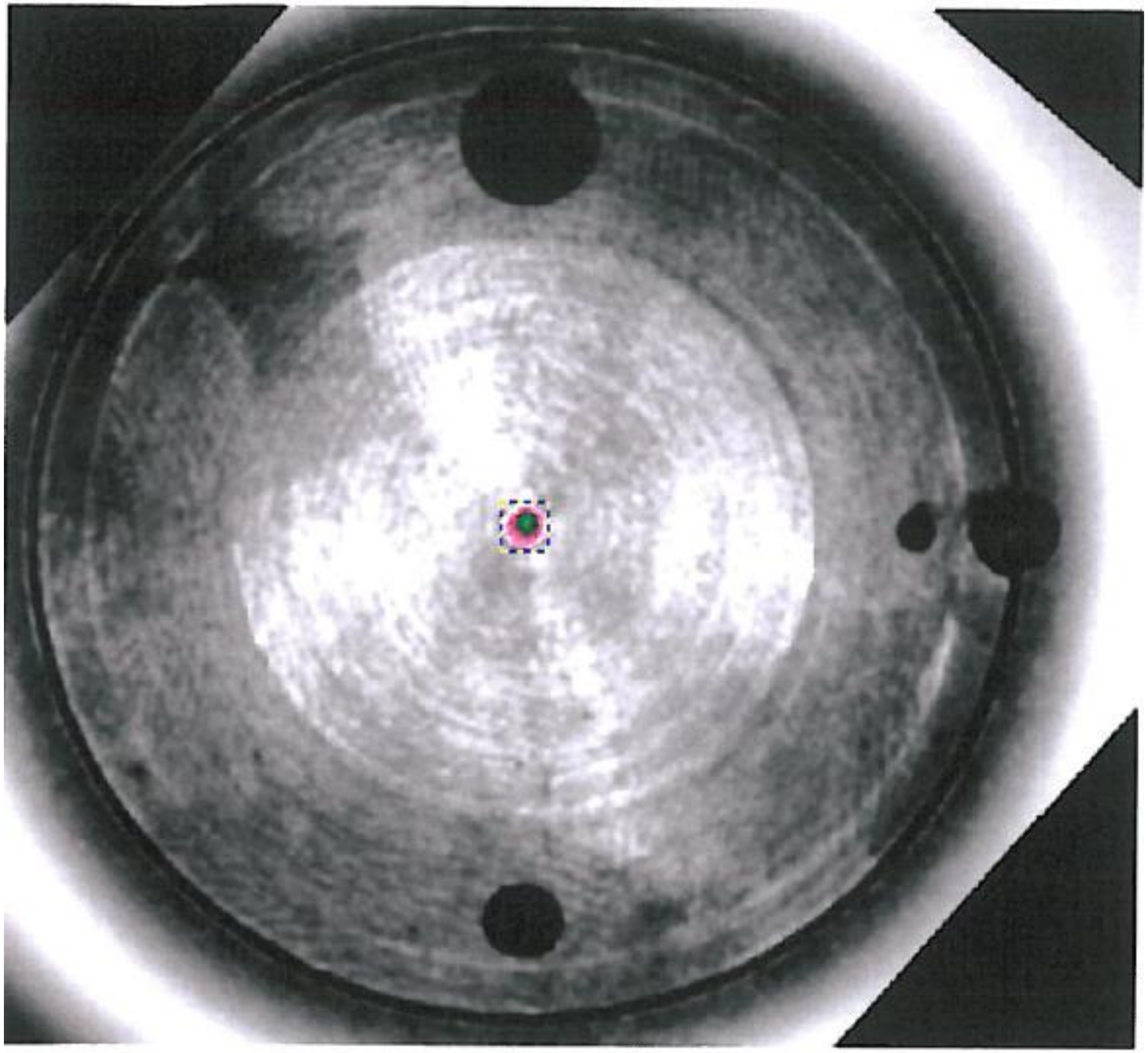


Element	Line	Cert. Conc. (%)	Analyzed Conc. (%)	RSD	RSD %
Al	Ka	0.24	0.21	0.047	22.5
Co	Ka	0.05	NA	0.010	NA
Cr	Ka	1.31	1.32	0.015	1.2
Cu	Ka	0.1	0.10	0.010	9.7
Mn	Ka	1.5	1.47	0.026	1.8
Mo	Ka	0.03	0.03	0.002	7.7
Nb	Ka	0.05	0.06	0.003	4.6
Ni	Ka	0.32	0.32	0.017	5.2
P	Ka	0.03	0.02	0.008	41.7
Si	Ka	0.74	0.63	0.041	6.6
Sn	Ka	0.1	0.10	0.009	8.9
Ta	Lb	N.A.	0.07	0.015	22.2
Ti	Ka	0.05	0.04	0.012	29.7
V	Ka	0.31	0.29	0.012	4.0
Zr	Ka	0.05	0.07	0.003	4.4

Example of Contamination or Inclusion Analysis

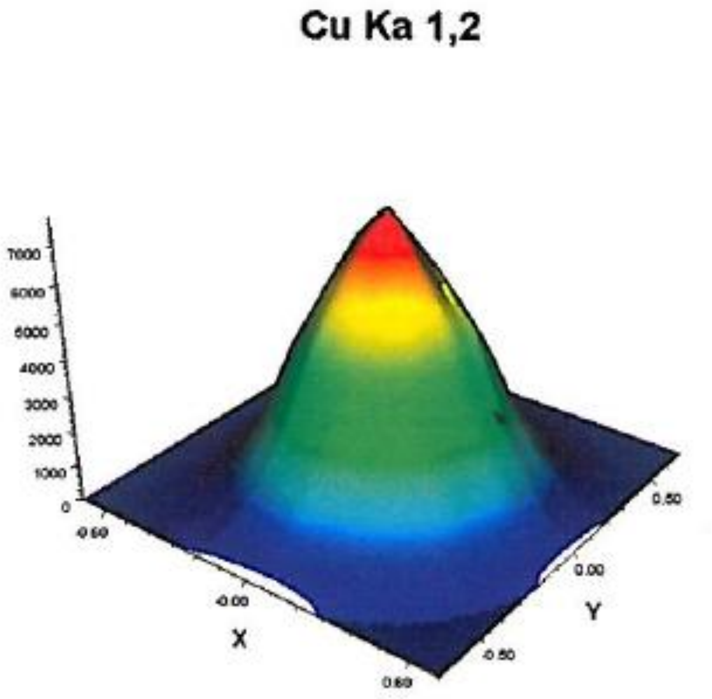
- Small green pink dot is mapped

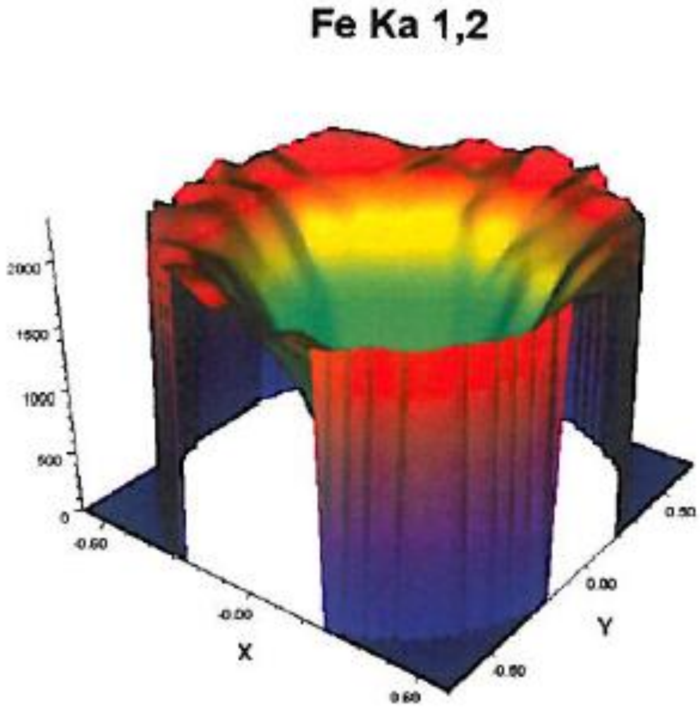
- Two Elements are analyszed: Cu and Fe



Example of mapping:

- 3D visualization in OXSAS

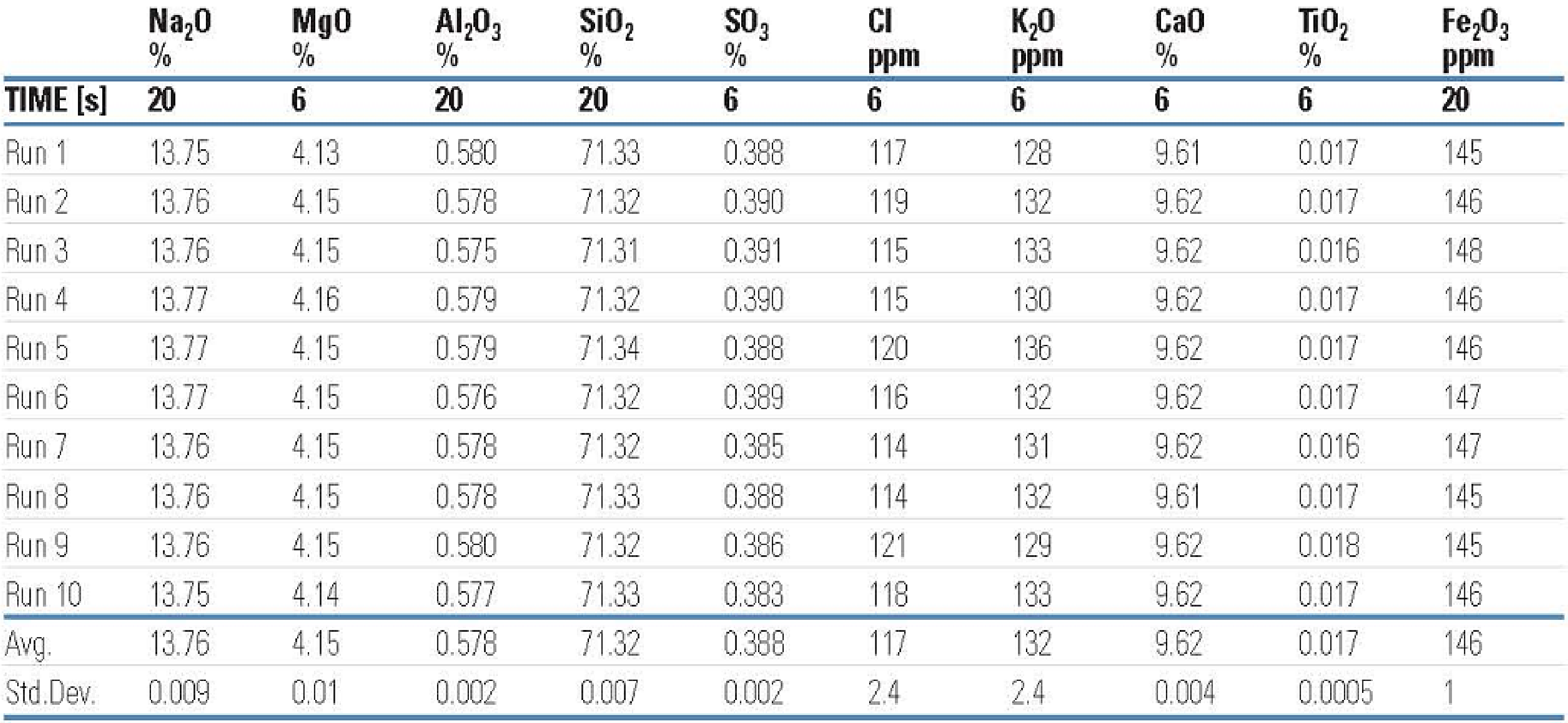

Cu Ka 1,2


Fe Ka 1,2

Elemental Analysis on Glass and Ceramics

Typical Glass Analysis Precision

- Soda-lime glass

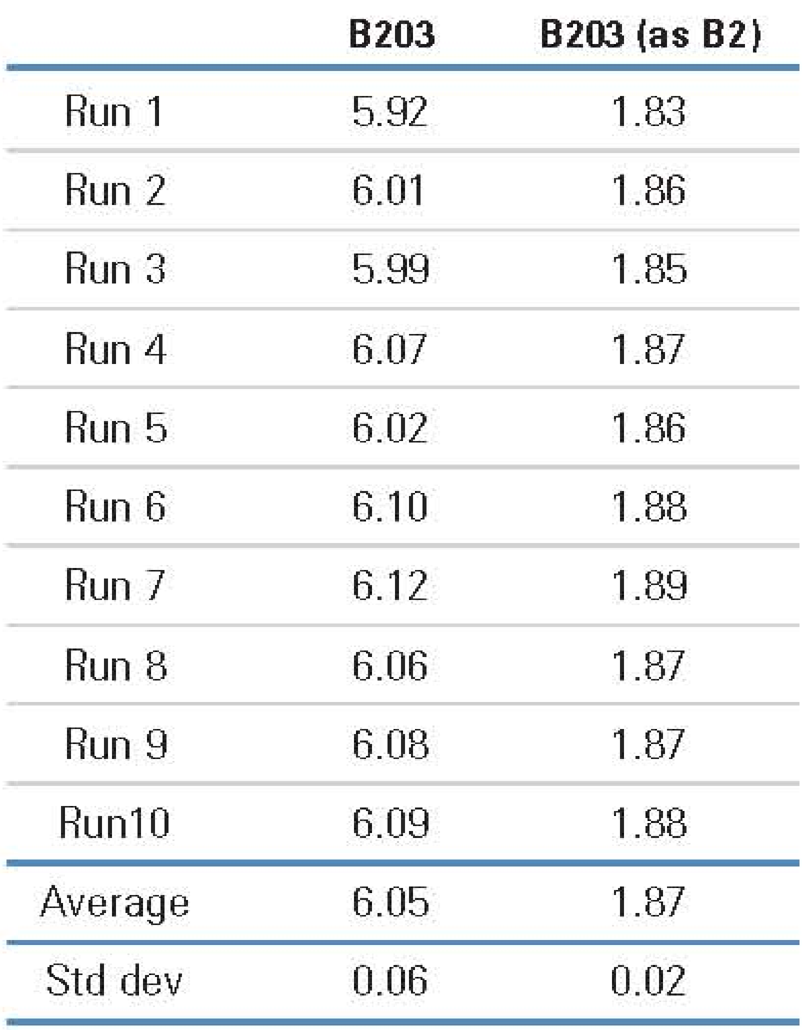


	Na_2O %	MgO %	Al_2O_3 %	SiO_2 %	SO_3 %	Cl ppm	K_2O ppm	CaO %	TiO_2 %	Fe_2O_3 ppm
TIME [s]	**20**	**6**	**20**	**20**	**6**	**6**	**6**	**6**	**6**	**20**
Run 1	13.75	4.13	0.580	71.33	0.388	117	128	9.61	0.017	145
Run 2	13.76	4.15	0.578	71.32	0.390	119	132	9.62	0.017	146
Run 3	13.76	4.15	0.575	71.31	0.391	115	133	9.62	0.016	148
Run 4	13.77	4.16	0.579	71.32	0.390	115	130	9.62	0.017	146
Run 5	13.77	4.15	0.579	71.34	0.388	120	136	9.62	0.017	146
Run 6	13.77	4.15	0.576	71.32	0.389	116	132	9.62	0.017	147
Run 7	13.76	4.15	0.578	71.32	0.385	114	131	9.62	0.016	147
Run 8	13.76	4.15	0.578	71.33	0.388	114	132	9.61	0.017	145
Run 9	13.76	4.15	0.580	71.32	0.386	121	129	9.62	0.018	145
Run 10	13.75	4.14	0.577	71.33	0.383	118	133	9.62	0.017	146
Avg.	13.76	4.15	0.578	71.32	0.388	117	132	9.62	0.017	146
Std.Dev.	0.009	0.01	0.002	0.007	0.002	2.4	2.4	0.004	0.0005	1

Table 3: Repeatability for the analysis of the main oxides in a flat glass (various counting times as shown, i.e. 116s total counting time)

Borosilicate Glass

- Extreme precision and accuracy on light elements
  - 50 µm Be window tube
  - UCCO (ultra close coupled optics)
  - Very stable analysis environment

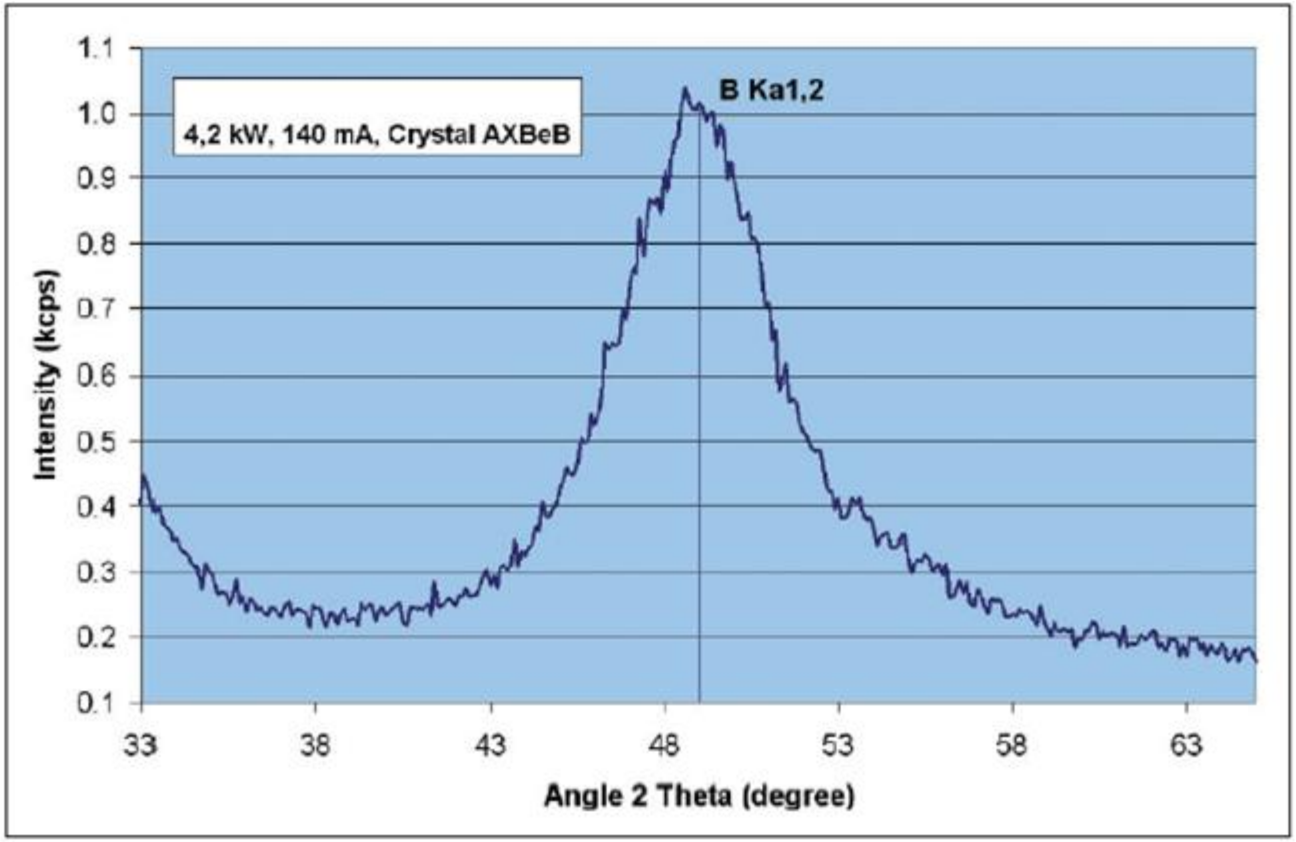


	B203	B203 (as B2)
Run 1	5.92	1.83
Run 2	6.01	1.86
Run 3	5.99	1.85
Run 4	6.07	1.87
Run 5	6.02	1.86
Run 6	6.10	1.88
Run 7	6.12	1.89
Run 8	6.06	1.87
Run 9	6.08	1.87
Run10	6.09	1.88
Average	6.05	1.87
Std dev	0.06	0.02

Table 1: Repeatability for boron analysis in glass (120 s - 30 kV/140 mA)

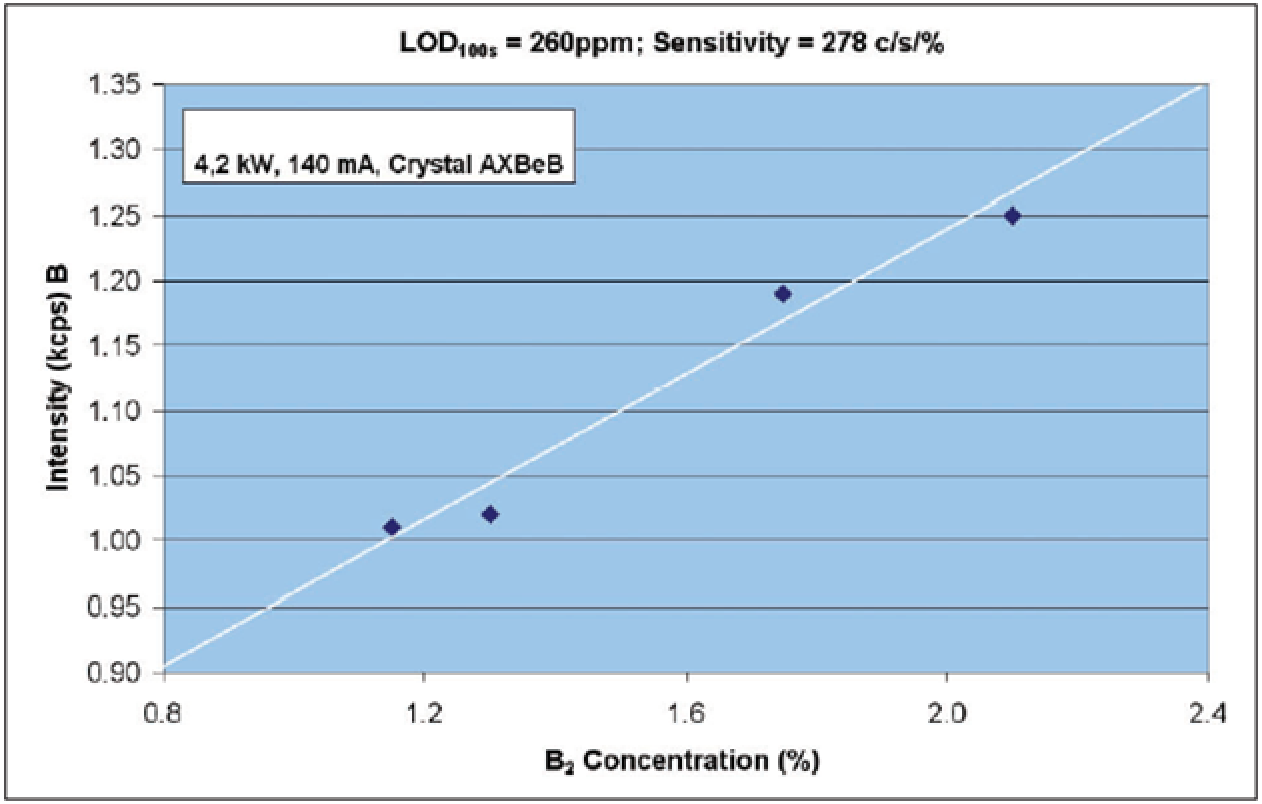
Boron analysis in glass

- AXBeB crystal – 30kV-140mA



Calibration curve for Boron in glass

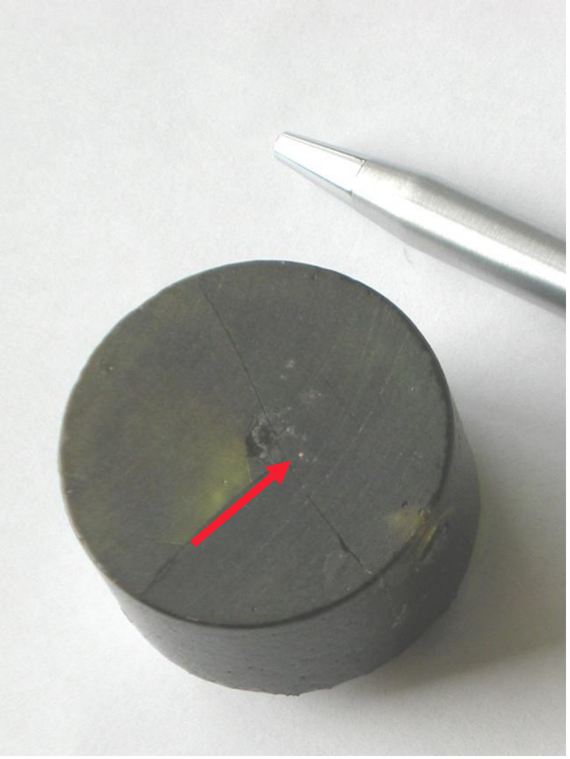
- Limit of detection: 260ppm in 100s



- Note: Analyzed depth of boron in glass is only 0.134 microns

Standard-less Analysis of Contamination

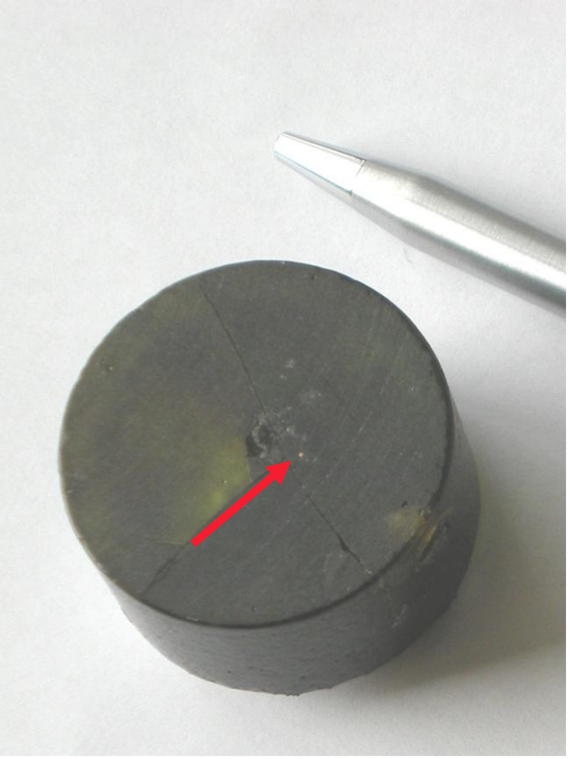
- Glass sample shows a shiny spot
- Traditional UniQuant analysis only provides the global composition over a diameter of 29mm



Oxide/ Element	Conc. %	StdErr %
SiO2	69.77	0.23
Na2O	12.16	0.16
CaO	7.57	0.13
MgO	3.54	0.09
Al2O3	1.09	0.05
K2O	0.330	0.016
SO3	0.215	0.011
Fe2O3	0.200	0.010
TiO2	0.0704	0.0035
BaO	0.0239	0.0052
Cl	0.0228	0.0011
CuO	0.0175	0.0009
MnO	0.0118	0.0007
ZrO2	0.0091	0.0005
P	0.0073	0.0006
Cr2O3	0.0069	0.0006
SrO	0.0066	0.0003
SnO2	0.0018	0.0009
ZnO	0.0016	0.0004
StdErr = error due to counting statistics		

Contamination in Glass

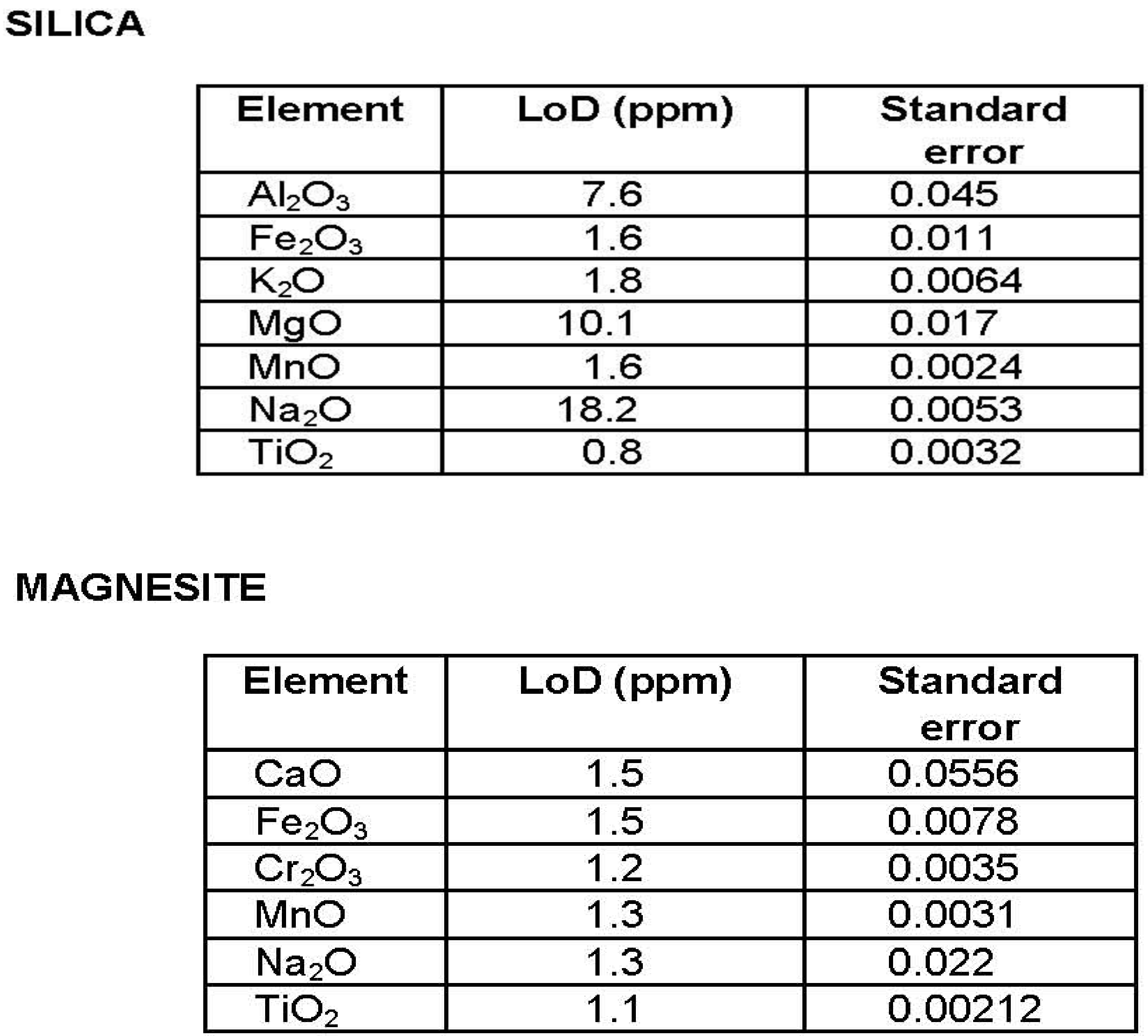
- Glass sample shows a shiny spot
- Analysis of the inclusion tells us it is a copper-tin alloy



Element	Shiny Spot %	Glass Surface %
Al	0.74	0.50
Cu	24.34	0.02
Fe	1.57	0.16
Sn	1.19	0.50
SiO2	34.84	70.46

Spot analyses (all values in %)

Ceramic Analysis



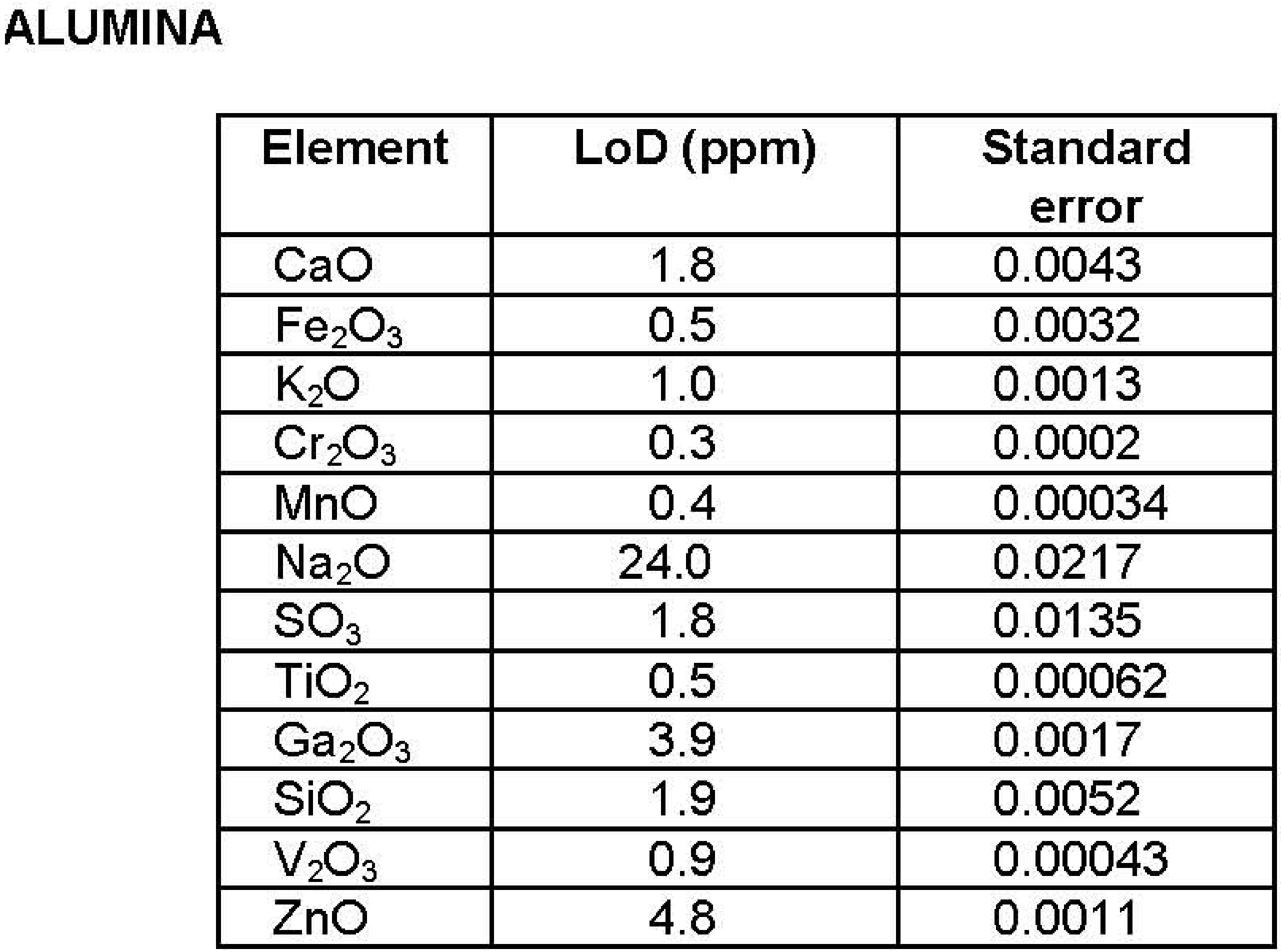
SILICA

Element	LoD (ppm)	Standard error
Al_2O_3	7.6	0.045
Fe_2O_3	1.6	0.011
K_2O	1.8	0.0064
MgO	10.1	0.017
MnO	1.6	0.0024
Na_2O	18.2	0.0053
TiO_2	0.8	0.0032

MAGNESITE

Element	LoD (ppm)	Standard error
CaO	1.5	0.0556
Fe_2O_3	1.5	0.0078
Cr_2O_3	1.2	0.0035
MnO	1.3	0.0031
Na_2O	1.3	0.022
TiO_2	1.1	0.00212

Ceramic Analysis Continued



ALUMINA

Element	LoD (ppm)	Standard error
CaO	1.8	0.0043
Fe_2O_3	0.5	0.0032
K_2O	1.0	0.0013
Cr_2O_3	0.3	0.0002
MnO	0.4	0.00034
Na_2O	24.0	0.0217
SO_3	1.8	0.0135
TiO_2	0.5	0.00062
Ga_2O_3	3.9	0.0017
SiO_2	1.9	0.0052
V_2O_3	0.9	0.00043
ZnO	4.8	0.0011

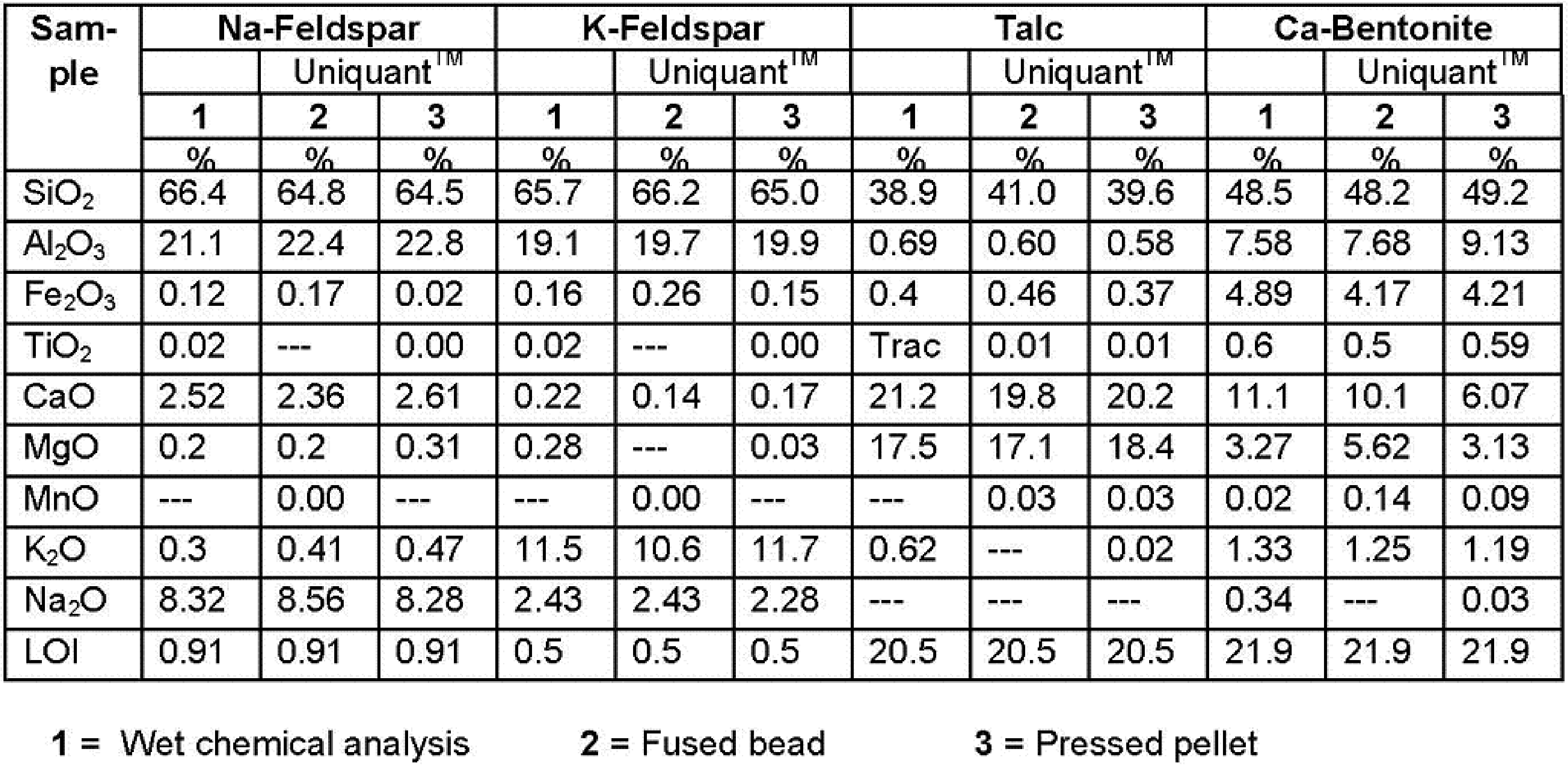
Comparison of UniQuant®/OptiQuant results for glass

- *Comparison for a glass of*
    - *UniQuant® results on mid power ARL PERFORM'X 2500*
    - *OptiQuant™ results obtained on the ARL OPTIM'X (50W)*
        - *Good agreement for minor and major elements.*

	Glass									
Element	**Na2O**	**MgO**	**Al2O3**	**SiO2**	**SO3**	**K2O**	**CaO**	**TiO2**	**Fe2O3**	**As2O3**
	[%]	**[%]**	**[%]**	**[%]**	**[%]**	**[%]**	**[%]**	**[%]**	**[%]**	**[%]**
Certified	**14.39**	**3.69**	**1.8**	**72.8**	**0.28**	**0.41**	**7.11**	**0.018**	**0.043**	**0.056**
UniQuant	**13.83**	**3.92**	**2.02**	**72.52**	**0.233**	**0.37**	**6.89**	**0.016**	**0.038**	**0.068**
OptiQuant	**13.64**	**3.74**	**1.84**	**73.07**	**0.271**	**0.361**	**6.89**	**0.019**	**0.014**	**0.063**

Sample Preparation effects with UniQuant

Comparison of different sample preparation techniques



Sam-ple	Na-Feldspar			K-Feldspar			Talc			Ca-Bentonite		
		Uniquant^{TM}			Uniquant^{TM}			Uniquant^{TM}			Uniquant^{TM}	
	1	2	3	1	2	3	1	2	3	1	2	3
	%	%	%	%	%	%	%	%	%	%	%	%
SiO_2	66.4	64.8	64.5	65.7	66.2	65.0	38.9	41.0	39.6	48.5	48.2	49.2
Al_2O_3	21.1	22.4	22.8	19.1	19.7	19.9	0.69	0.60	0.58	7.58	7.68	9.13
Fe_2O_3	0.12	0.17	0.02	0.16	0.26	0.15	0.4	0.46	0.37	4.89	4.17	4.21
TiO_2	0.02	---	0.00	0.02	---	0.00	Trac	0.01	0.01	0.6	0.5	0.59
CaO	2.52	2.36	2.61	0.22	0.14	0.17	21.2	19.8	20.2	11.1	10.1	6.07
MgO	0.2	0.2	0.31	0.28	---	0.03	17.5	17.1	18.4	3.27	5.62	3.13
MnO	---	0.00	---	---	0.00	---	---	0.03	0.03	0.02	0.14	0.09
K_2O	0.3	0.41	0.47	11.5	10.6	11.7	0.62	---	0.02	1.33	1.25	1.19
Na_2O	8.32	8.56	8.28	2.43	2.43	2.28	---	---	---	0.34	---	0.03
LOI	0.91	0.91	0.91	0.5	0.5	0.5	20.5	20.5	20.5	21.9	21.9	21.9

1 = Wet chemical analysis **2** = Fused bead **3** = Pressed pellet

UniQuant Examples:
Glass piece in a forensic investigation

Same Glass sample in 3 different forms

	Original broken solid piece	670mg loose powder	1+33LiT Bead	Chemical spec.
MgO	1.6 ± 0.1	1.3	1.4	1.4
Al2O3	12.8 ± 0.3	11.7	12.6	14
SiO2	42.9 ± 0.5	39.9	43.2	43
CaO	34.1 ± 0.06	37.2	34.8	33
Fe2O3	1.8 ± 0.1	1.7	1.6	1.7
ZnO	0.4 ± 0.06	0.5	0.3	0.4
ZrO2	4.3 ± 0.2	4.6	4.5	4.5
BaO	0.3 ± 0.05	0.3	0.3	0.3

Elemental Analysis for Geo-Chemical

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Fused Bead Analysis for Oxides

- Best sample preparation for Geo-Chemical is Fused Beads
  - Minimizes matrix effects
  - Eliminates grains size effects
  - Eliminates mineralogical effects
  - Reduces inter-elemental effects by dilution
  - Easier to locate standards

Element	Line	Conc (ppm)	RSD (ppm)	LOD
Na	Ka	525	10	150
Mg	Ka	144	14	63
Al	Ka	150	13	60
Si	Ka	30	55	50
P	Ka	46	12	15
S	Ka	2712	0	27
K	Ka	23	31	21
Ca	Ka	19	34	18
Ti	Ka	55	5	10
Cr	Ka	15	24	12
Mn	Ka	3	84	10
Fe	Ka	28	12	10

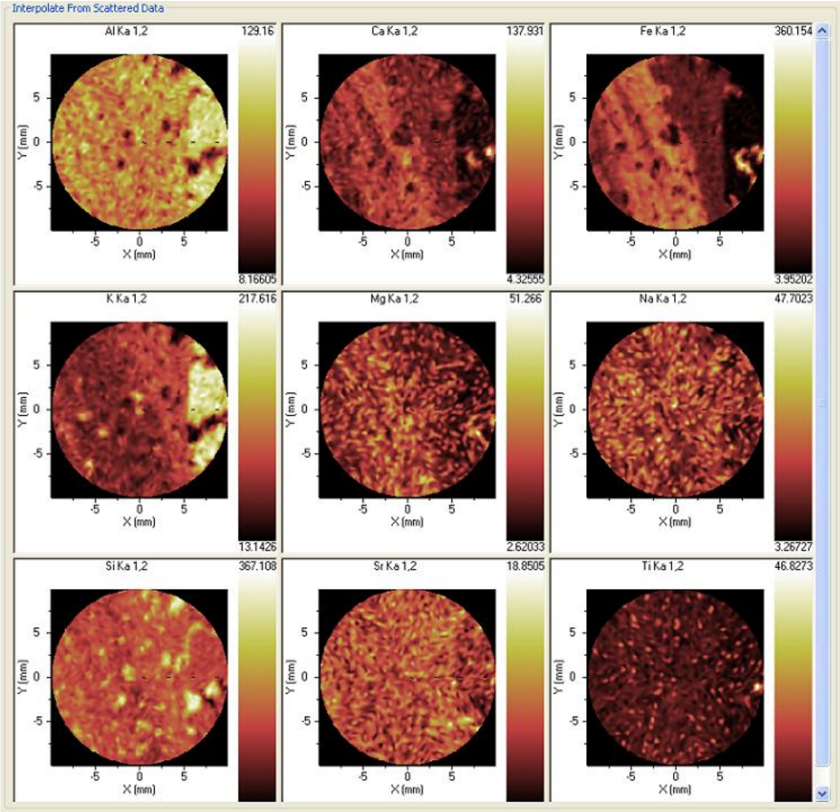
UniQuant Analysis Report
GBW07311, Sediment

Element	Given	UQ
SiO_2	76.3%	76.5%
Al_2O_3	10.4%	13.0%
Fe_2O_3	4.39%	4.15%
K_2O	3.28%	3.55%
MgO	0.620%	0.778%
CaO	0.470%	0.432%
Na_2O	0.460%	0.493%
TiO_2	0.350%	0.335%
MnO	0.321%	0.292%
PbO	685 ppm	668 ppm
Rb_2O	446 ppm	438 ppm
P_2O_5	584 ppm	636 ppm
ZnO	464 ppm	460 ppm
Cl	290 ppm	407 ppm

Element	Given	UQ
SnO_2	420 ppm	371 ppm
BaO	321 ppm	280 ppm
As_2O_3	248 ppm	236 ppm
ZrO_2	207 ppm	198 ppm
WO_3	159 ppm	146 ppm
CuO	98 ppm	98 ppm
V_2O_5	84 ppm	64 ppm
Sb_2O_3	19 ppm	
Y_2O_3	54 ppm	59 ppm
Bi_2O_3	56 ppm	57 ppm
Nb_2O_5	36 ppm	29 ppm
SrO	34 ppm	29 ppm
NiO	18 ppm	16 ppm
Ga_2O_3	25 ppm	21 ppm
ThO_2	27 ppm	

Geo-Chemical Mapping

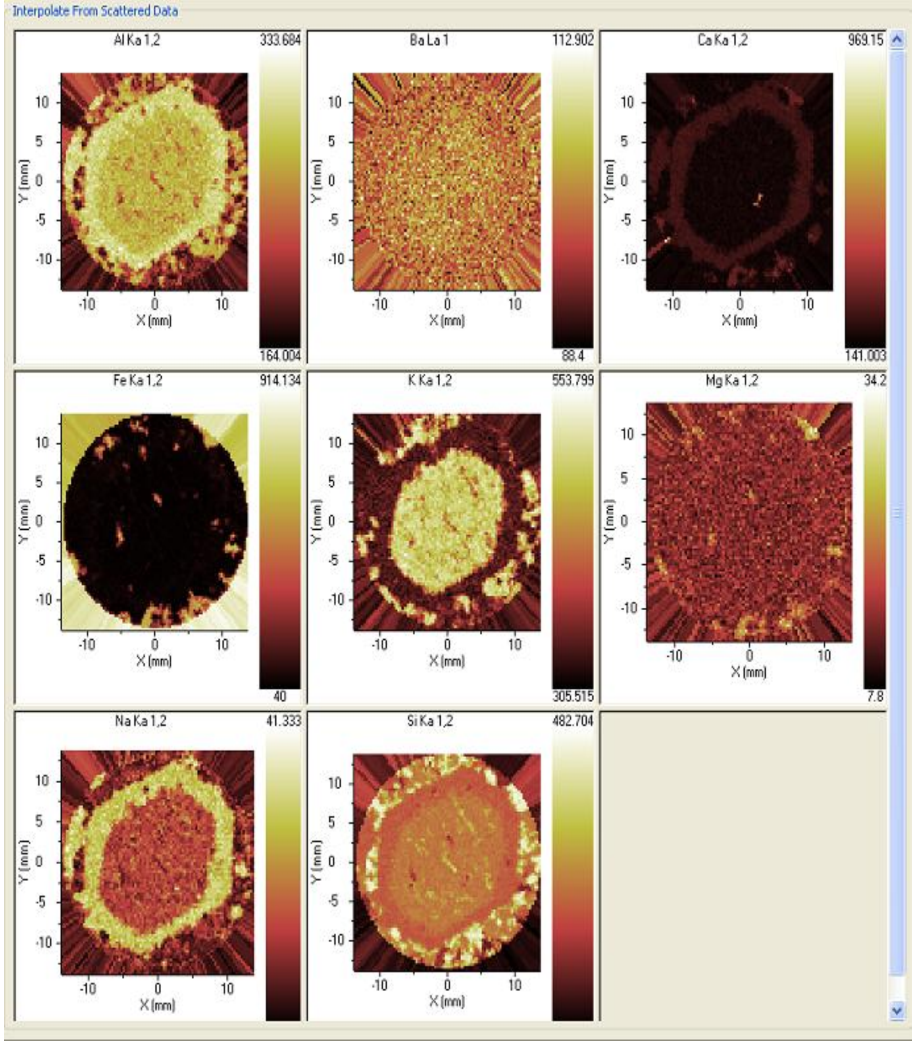
- Pseudotachylite from Sudbury, Ontario
- Mapping shows bands of Fe, and other elements at very sharp boundary with un-sheared rock
- Chemical information upon the genesis of the material



Elements analyzed: Al, Ca, Fe, K, Mg, Na, Si, Sr, and Ti

Feldspar Mapping

- Alkali feldspar from Finland
- Elemental mapping helps determine how the plagioclase comes to mantle the earlier alkali feldspar



Elements Analyzed: Al, Ba, Ca, Fe, K, Mg, Na and Sr

Elemental Analysis for Environmental

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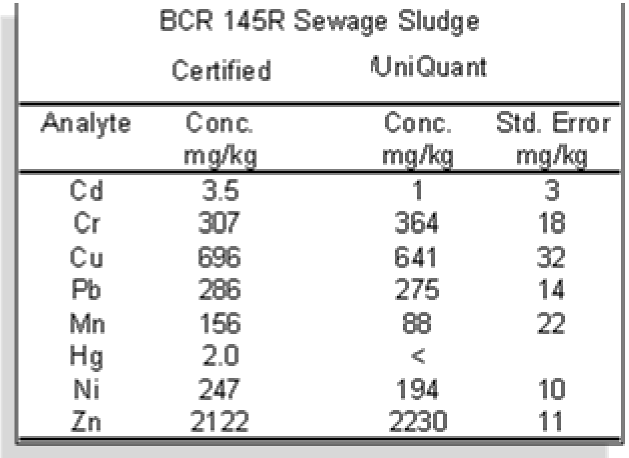
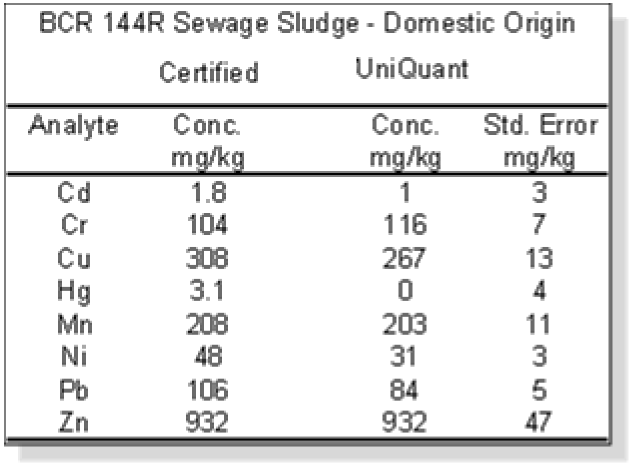
Sewage Sludge: UniQuant FP Analysis in Practice

- Analysis of 3 certified reference materials
- Total measurement time: 10 min



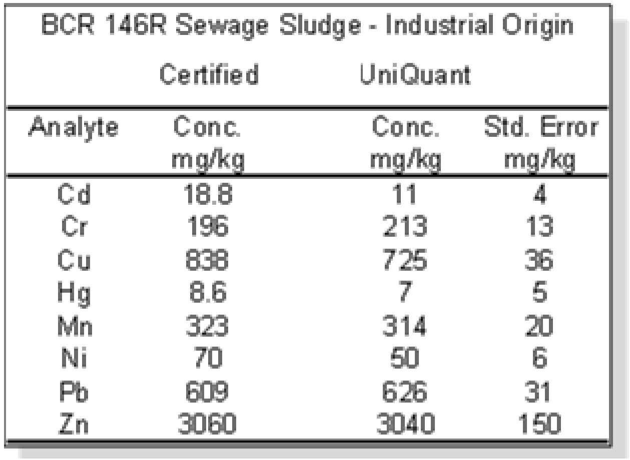
BCR 145R Sewage Sludge

	Certified	UniQuant	
Analyte	Conc. mg/kg	Conc. mg/kg	Std. Error mg/kg
Cd	3.5	1	3
Cr	307	364	18
Cu	696	641	32
Pb	286	275	14
Mn	156	88	22
Hg	2.0	<	
Ni	247	194	10
Zn	2122	2230	11



BCR 144R Sewage Sludge - Domestic Origin

	Certified	UniQuant	
Analyte	Conc. mg/kg	Conc. mg/kg	Std. Error mg/kg
Cd	1.8	1	3
Cr	104	116	7
Cu	308	267	13
Hg	3.1	0	4
Mn	208	203	11
Ni	48	31	3
Pb	106	84	5
Zn	932	932	47



BCR 146R Sewage Sludge - Industrial Origin

	Certified	UniQuant	
Analyte	Conc. mg/kg	Conc. mg/kg	Std. Error mg/kg
Cd	18.8	11	4
Cr	196	213	13
Cu	838	725	36
Hg	8.6	7	5
Mn	323	314	20
Ni	70	50	6
Pb	609	626	31
Zn	3060	3040	150

Analysis of waste solvants

- Current analysis is done by ion chromatography after digestion with Mahler bomb
  - Takes at least one hour for digestion and analysis
  - 0.5g of sample digested - a few microliters are injected in chromatograph
    - Representativity of samples is questionable

Waste solvants preparation for XRF analysis

- Use of a coagulant powder to turn liquid into paste

Analysis of waste solvants

Sample	CIR			
Element	liquid	**Paste**	**Mahler bomb + ion chromatography**	**Comments**
Element	OptiQuant	OptiQuant		
CH2	Rest	Rest		
H2O	53.2	53.2		
S	1540	**1490**	**1520**	Good correlation (XRF to IC)
Si	216	**115**		Si content well determined with XRF technique
Na	4400	**5832**	**6732**	Good correlation (XRF to IC)
K	428	**400**	**534**	Good correlation (XRF to IC)
P	4470	**1430**	**<100**	Higher P content in the liquid phase
Cl	6840	***1.14%***	***1.80%***	Good information of Cl level
Ca	4170	**294**		Higher Ca level in the liquid phase
Br	1670	**1450**		High Br content well determined with XRF technique
Fe	88	**37**		
Mg	188	**87**		
Zn	16	**10**	**12.1**	
Ti	275	**127**		
Cu	45	**44**	**5.5**	

Ion chromatography seems to undervalue the P content

Elemental Analysis for Petro-Chemical

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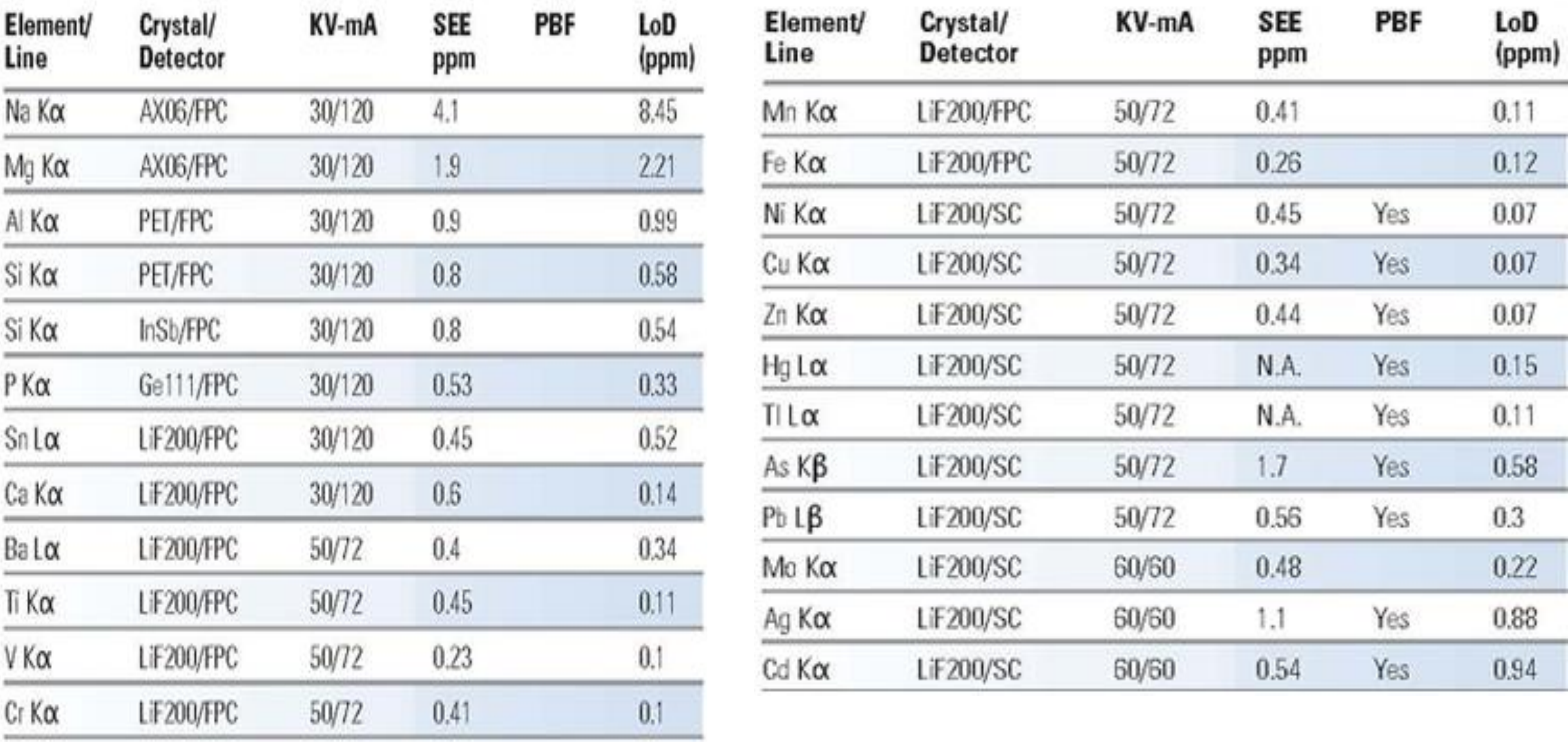
Oil Analysis

- Typical LoD in Oil Analysis



Element/ Line	Crystal/ Detector	KV-mA	SEE ppm	PBF	LoD (ppm)
Na Kα	AX06/FPC	30/120	4.1		8.45
Mg Kα	AX06/FPC	30/120	1.9		2.21
Al Kα	PET/FPC	30/120	0.9		0.99
Si Kα	PET/FPC	30/120	0.8		0.58
Si Kα	InSb/FPC	30/120	0.8		0.54
P Kα	Ge111/FPC	30/120	0.53		0.33
Sn Lα	LiF200/FPC	30/120	0.45		0.52
Ca Kα	LiF200/FPC	30/120	0.6		0.14
Ba Lα	LiF200/FPC	50/72	0.4		0.34
Ti Kα	LiF200/FPC	50/72	0.45		0.11
V Kα	LiF200/FPC	50/72	0.23		0.1
Cr Kα	LiF200/FPC	50/72	0.41		0.1

Element/ Line	Crystal/ Detector	KV-mA	SEE ppm	PBF	LoD (ppm)
Mn Kα	LiF200/FPC	50/72	0.41		0.11
Fe Kα	LiF200/FPC	50/72	0.26		0.12
Ni Kα	LiF200/SC	50/72	0.45	Yes	0.07
Cu Kα	LiF200/SC	50/72	0.34	Yes	0.07
Zn Kα	LiF200/SC	50/72	0.44	Yes	0.07
Hg Lα	LiF200/SC	50/72	N.A.	Yes	0.15
Tl Lα	LiF200/SC	50/72	N.A.	Yes	0.11
As Kβ	LiF200/SC	50/72	1.7	Yes	0.58
Pb Lβ	LiF200/SC	50/72	0.56	Yes	0.3
Mo Kα	LiF200/SC	60/60	0.48		0.22
Ag Kα	LiF200/SC	60/60	1.1	Yes	0.88
Cd Kα	LiF200/SC	60/60	0.54	Yes	0.94

PetroilQuant™

- Factory calibrated package for oils analysis
  - Up to 30 elements
  - Calibration maintained using solid drift standards
  - Good for fuels, lubricants, and oils

Questions & Answers

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www.thermo.com/xray

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