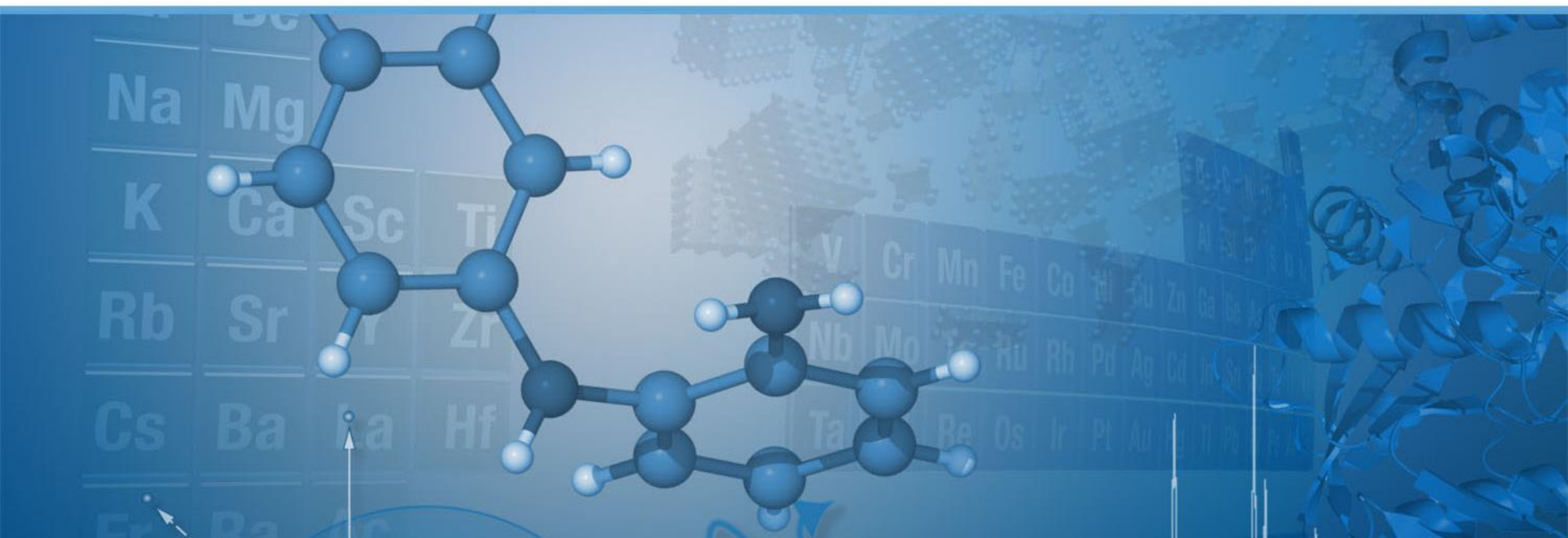
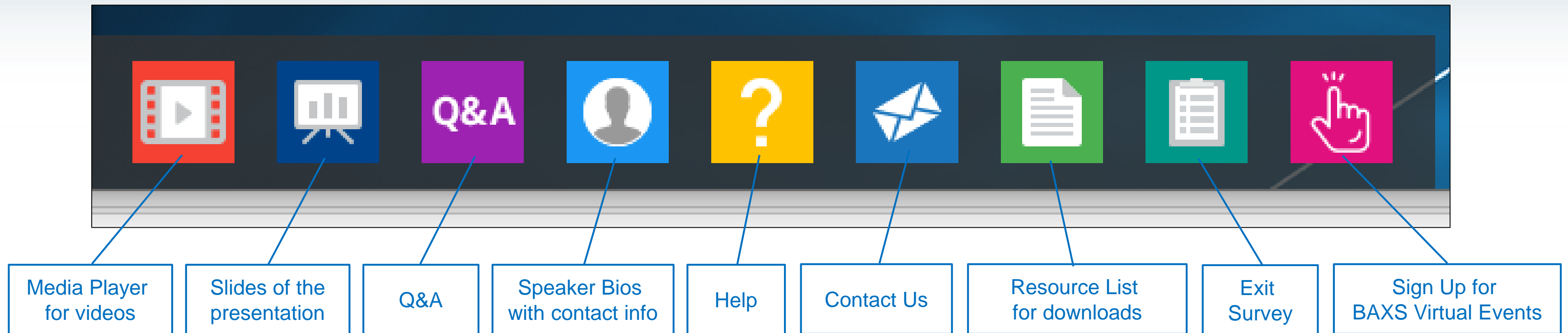


Tips and Tricks for Making Your Own Secondary Standards for XRF



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Speakers



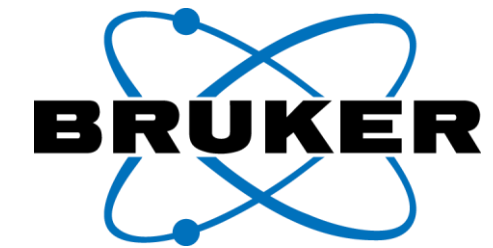
Archibald Harris
XRF Applications Specialist, Bruker AXS GmbH
archibald.harris@bruker.com



Dr. Rainer Schramm
CEO, FLUXANA GmbH & Co KG
rschramm@fluxana.com



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R&D, Bruker AXS GmbH
colin.slater@bruker.com





Dr. Rainer Schramm
CEO, Fluxana GmbH & Co KG



Tips and Tricks for making your own secondary standards for XRF

Dr. Rainer Schramm

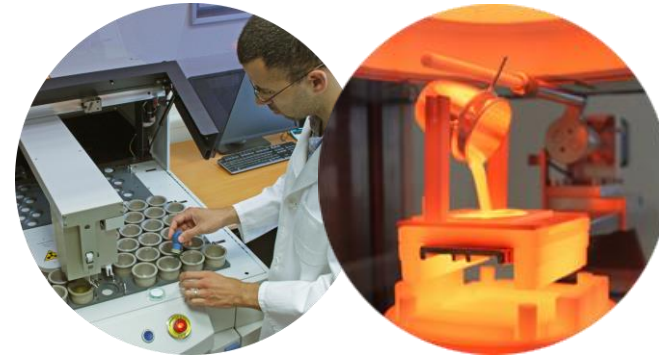
Online Seminar, 28.07.2020

FLUXANA: Introduction

Equipment for Sample Preparation



Commercial Analysis ISO 17025

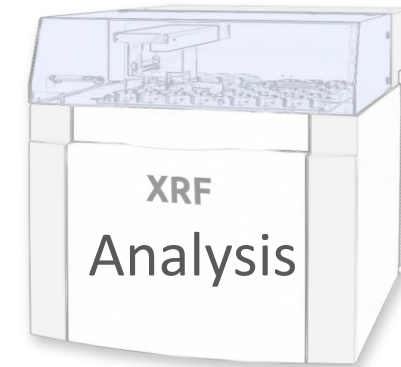


Round Robins

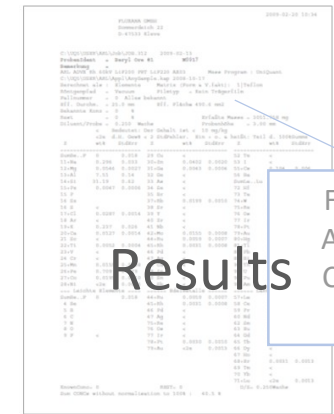


Sample

Sample Preparation



XRF Analysis



Results

Fe: 5,5 %
Au: 0,4 %
Cu: 1,2 %
....



Consumables and Accessories



FLUXaminar
www.fluxaminar.com

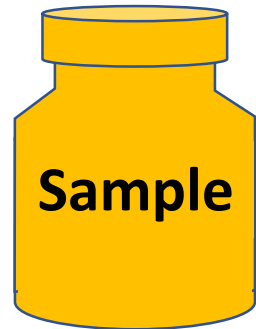
Training and support



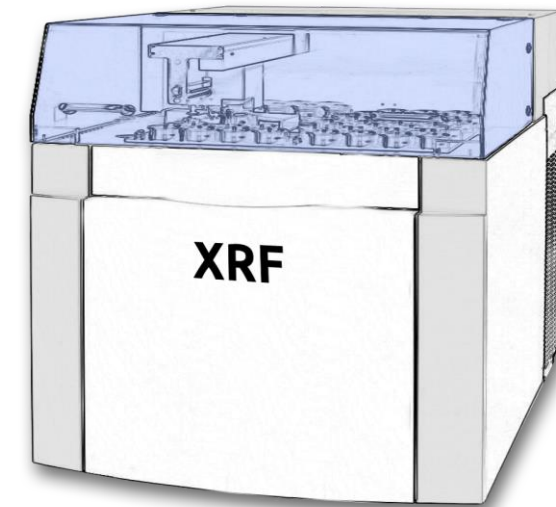
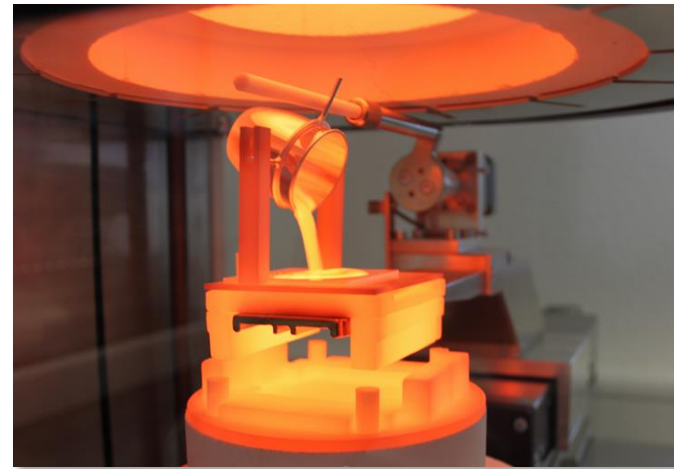
FLUXearch
www.fluxearch.com

Reference Materials
ISO 17034

Sample



Sample Preparation



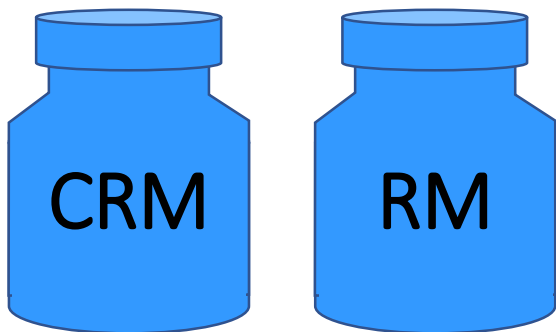
Results

```

FLUXANA OMSU                               2009-02-20 10:34
Gomerdeich 22
D-47533 Kleeve

C:\OQS\USER\ARL\Job\JOB_313 2009-02-13
Probendat = Seryl Ore #1 W5517
Bemerkung =
ARL ADVX 5A 60kV LFP200 FW LFP220 AXES      Mess Program : UniQuant
C:\OQS\USER\ARL\App\AnySample.kap 2008-10-17
Berechnet wie : Elemente Matrix (Form & V.fakt): 1|Teile
Stoßwegprofil = Vacuum Plätttyp = Rein Trägerfile
Pulldiameter = 0 Allein bekannt
Stf. Durchm. = 25.0 mm Stf. Fläche 490.6 mm2
Bekanntes Konz = 0 %
Start = 0 %
Diluent/Probe = 0.250 Wache             Erfasste Masse = 3051.758 mg
                                         Probenhöhe = 5.80 mm
                                         Substrat: Der Gehalt ist < 10 mg/kg
                                         +2w d.S. Gewt = 2 Stührler. Ein = 0.4 heißt: Teil d. 1000Stuche
                                         WtR Stührer Z WtR Stührer Z WtR Stührer Z WtR Stührer Z
-----
Stunde..P 0 0.018 29 Cu < 52 Th <
11-Mn 0.296 0.033 30-Zn 0.040 0.0020 53 I <
12-Mg 0.0548 0.0029 31-Ga 0.0043 0.0004 54-Sr 0.104 0.006
13-Al 7.53 0.14 32-Ge < 56-Ba <
14-Si 31.19 0.62 33-Ar < 58-Ce.Lu 0.056 0.022
15-Pb 0.0047 0.0008 34-Se < 72-Hf <
15-P 35 Sr < 73-Ta <
16-Sa 37-Rb 0.0199 0.0010 74-W 0.149 0.010
18-S < 38-Br < 75-Re <
17-Cd 0.0287 0.0014 39-Y < 76-Os <
18-Ar < 40-Zr < 77-Ir <
19-K 0.237 0.026 41-Nb < 78-Pt 0.0030 0.0010
20-Ca 0.0127 0.0014 42-Mo 0.0155 0.0008 79-Au +2w 0.0013
21-Sc < 44-Ru 0.0059 0.0007 80-Hg <
22-Ti 0.0052 0.0004 45-Rh 0.0031 0.0008 81-Tl +2w 0.0011
23-V < 46-Pd < 82-Pb +2w 0.0014
24-Cr < 47-Ag < 83-Bi <
25-Mn 0.0155 0.0008 48-Cd < 90-Th <
26-Pe 0.709 0.079 49-In < 92-U <
27-Co 0.0196 0.0010 50-Sn < 94-Pu <
28-Ni +2w 0.0004 51-Sb < 95-Am <
-----
Leichte Elemente ----- Edelmetalle ----- Lanthaniden -----
Stunde..P 0 0.018 44-Ru 0.0059 0.0007 57-La 0.0042 0.0010
4-Sr 45-Rh 0.0031 0.0008 58-Ce +2w 0.0024
5-B 46-Pd < 59-Py +2w 0.0026
6-C 47-Ag < 60-Hf <
7-N 72-Os < 62-Sm <
8-O 76-Os < 63-Eu <
9-P < 77-Ir < 64-Gd <
78-Pt 0.0030 0.0010 65-Tb <
79-Au +2w 0.0013 66-Dy <
67-Sm <
68-Er 0.0031 0.0013
69-Tm <
70-Yb <
71-Lu +2w 0.0013
-----
Ergebnis: 0 ERST: 0
Zun CORCw without normalization to 100% : 49.5 %
    
```

Calibration Samples



Example: GEO-QUANT Basic Application

1 H Hydrogen 1.008																	2 He Helium 4.002602				
3 Li Lithium 6.94	4 Be Beryllium 9.0121831															5 B Boron 10.81	6 C Carbon 12.011	7 N Nitrogen 14.007	8 O Oxygen 15.999	9 F Fluorine 18.998403163	10 Ne Neon 20.1797
11 Na Sodium 22.98976928	12 Mg Magnesium 24.305															13 Al Aluminium 26.9815385	14 Si Silicon 28.085	15 P Phosphorus 30.973761998	16 S Sulfur 32.06	17 Cl Chlorine 35.45	18 Ar Argon 39.948
19 K Potassium 39.0983	20 Ca Calcium 40.078	21 Sc Scandium 44.955908	22 Ti Titanium 47.867	23 V Vanadium 50.9415	24 Cr Chromium 51.9961	25 Mn Manganese 54.938044	26 Fe Iron 55.845	27 Co Cobalt 58.933194	28 Ni Nickel 58.6934	29 Cu Copper 63.546	30 Zn Zinc 65.38	31 Ga Gallium 69.723	32 Ge Germanium 72.630	33 As Arsenic 74.921595	34 Se Selenium 78.971	35 Br Bromine 79.904	36 Kr Krypton 83.798				
37 Rb Rubidium 85.4678	38 Sr Strontium 87.62	39 Y Yttrium 88.90584	40 Zr Zirconium 91.224	41 Nb Niobium 92.90637	42 Mo Molybdenum 95.95	43 Tc Technetium (98)	44 Ru Ruthenium 101.07	45 Rh Rhodium 102.90550	46 Pd Palladium 106.42	47 Ag Silver 107.8682	48 Cd Cadmium 112.414	49 In Indium 114.818	50 Sn Tin 118.710	51 Sb Antimony 121.760	52 Te Tellurium 127.60	53 I Iodine 126.90447	54 Xe Xenon 131.293				
55 Cs Caesium 132.90545196	56 Ba Barium 137.327	57 - 71 Lanthanoids	72 Hf Hafnium 178.49	73 Ta Tantalum 180.94788	74 W Tungsten 183.84	75 Re Rhenium 186.207	76 Os Osmium 190.23	77 Ir Iridium 192.217	78 Pt Platinum 195.084	79 Au Gold 196.966569	80 Hg Mercury 200.592	81 Tl Thallium 204.38	82 Pb Lead 207.2	83 Bi Bismuth 208.98040	84 Po Polonium (209)	85 At Astatine (210)	86 Rn Radon (222)				
87 Fr Francium (223)	88 Ra Radium (226)	89 - 103 Actinoids	104 Rf Rutherfordium (267)	105 Db Dubnium (268)	106 Sg Seaborgium (269)	107 Bh Bohrium (270)	108 Hs Hassium (269)	109 Mt Meitnerium (278)	110 Ds Darmstadtium (281)	111 Rg Roentgenium (282)	112 Cn Copernicium (285)	113 Nh Nihonium (286)	114 Fl Flerovium (289)	115 Mc Moscovium (289)	116 Lv Livermorium (293)	117 Ts Tennessine (294)	118 Og Oganesson (294)				

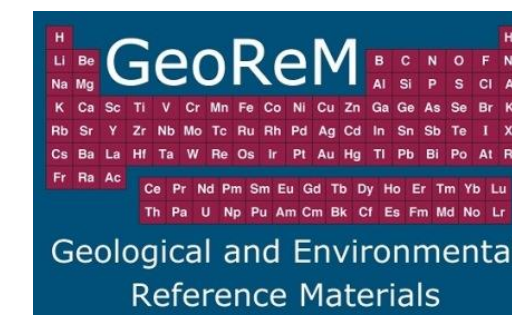
57 La Lanthanum 138.90547	58 Ce Cerium 140.116	59 Pr Praseodymium 140.90766	60 Nd Neodymium 144.242	61 Pm Promethium (145)	62 Sm Samarium 150.36	63 Eu Europium 151.964	64 Gd Gadolinium 157.25	65 Tb Terbium 158.92535	66 Dy Dysprosium 162.500	67 Ho Holmium 164.93033	68 Er Erbium 167.259	69 Tm Thulium 168.93422	70 Yb Ytterbium 173.045	71 Lu Lutetium 174.9668
89 Ac Actinium (227)	90 Th Thorium 232.0377	91 Pa Protactinium 231.03588	92 U Uranium 238.02891	93 Np Neptunium (237)	94 Pu Plutonium (244)	95 Am Americium (243)	96 Cm Curium (247)	97 Bk Berkelium (247)	98 Cf Californium (251)	99 Es Einsteinium (252)	100 Fm Fermium (257)	101 Md Mendelevium (258)	102 No Nobelium (259)	103 Lr Lawrencium (266)

Commercially available reference materials

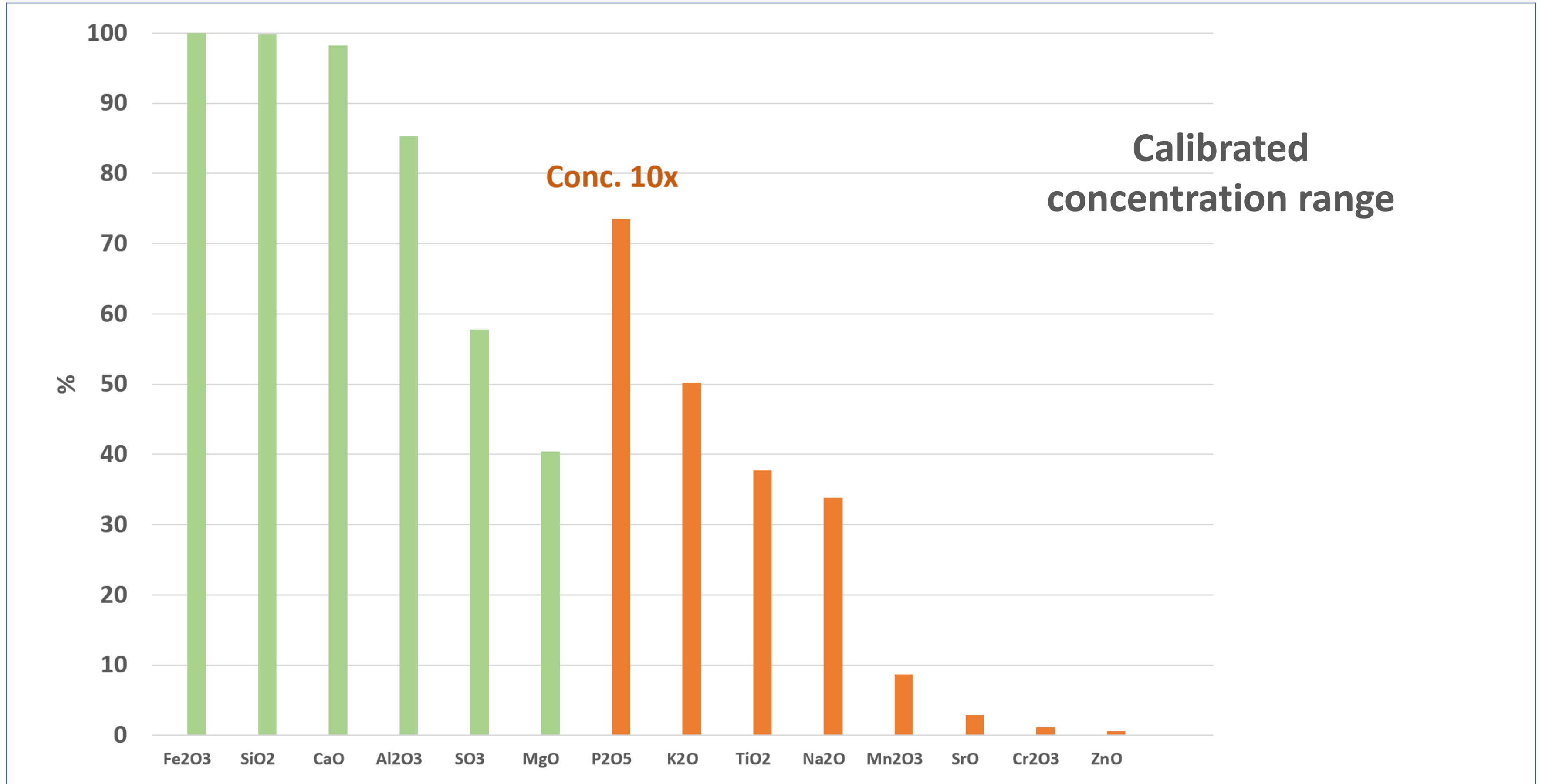


Free accessible databases for the search of reference materials

Database	Reference
FLUXearch	www.fluxearch.com
COMAR	www.comar.bam.de
GeoReM	georem.mpch-mainz.gwdg.de



Example: GEO-QUANT Basic Application

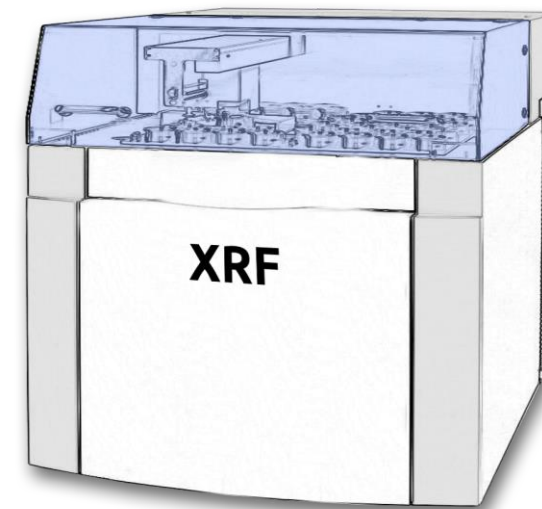
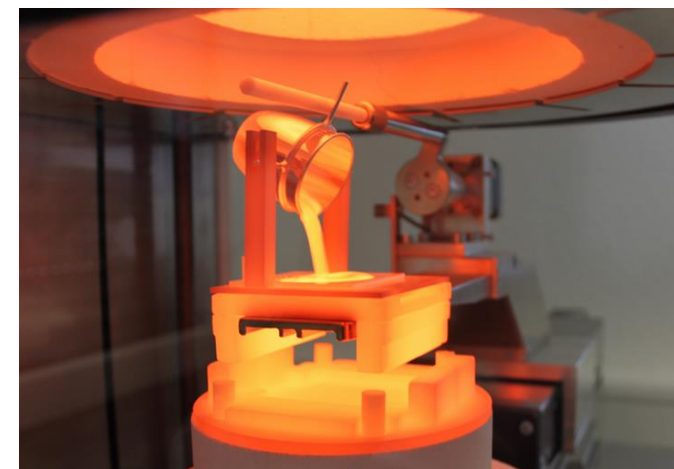


Traceability of Analytical Results

Sample



Sample Preparation



Results

FLUXANA GmbH
Simmending 22
D-47533 Kleeve

2009-02-20 10:34

C:\SQS\USER\ARL\Job\JOB_312 2009-02-13
 Probenname = Beryll Gew #1 M0917
 Bemerkung =
 ARL ADVX 8k 60kV LXP200 IWT LXP220 AXIS Maxx Programm : OutQuest
 C:\SQS\USER\ARL\Appl\AnySample.kap 2009-10-17
 Berechnet als : Elemente Matrix (Form & V.Fakt): 1|TeFlon
 Röntgenrad = Vacuum Filtertyp = Kein Trägerfilm
 Pulldiameter = 0 Alles bekannt
 Riff. Durchs. = 25.0 mm Riff. Fläche 490.6 cm2
 Sekante Konz = 0 %
 Wert = 0 %
 Diluent/Probe = 0.250 Wechs. Brutto Masse = 3051.758 mg
 Probehöhe = 3.00 mm
 = Bedeutung: Der Gehalt ist < 50 mg/kg
 +2s 4.S.D. Gehalt < 2 StdAbw. Min + 0.4 bei 2s Teil d. 1000Summe
 w% StDDev 2 w% StDDev 2 w% StDDev 2

Element	w%	StDDev	Element	w%	StDDev
11-Na	0.296	0.033	39-Zn	0.0482	0.0020
12-Mg	0.0546	0.0027	51-Sb	0.0043	0.0004
13-Al	7.53	0.14	55-Ce	0.104	0.006
14-Si	31.19	0.62	56-Ba	<	<
15-P	0.0047	0.0006	59-Co	0.056	0.003
16-S	<	<	72-Hf	<	<
17-Cl	0.0287	0.0014	73-Ta	<	<
18-Ar	<	<	74-W	0.149	0.010
19-K	0.237	0.026	75-Re	<	<
20-Ca	0.0127	0.0014	76-Os	0.0155	0.0008
21-Zn	<	<	78-Pt	0.0059	0.0007
22-Ti	0.0052	0.0004	80-Hg	0.0059	0.0007
23-V	<	<	81-Tl	0.0031	0.0006
24-Cr	<	<	82-Pb	<	<
25-Mn	0.0155	0.0008	83-Bi	<	<
26-Fe	0.709	0.079	84-Po	<	<
27-Cu	0.0195	0.0010	85-At	<	<
28-Ni	<	<	86-Rn	<	<
29-Co	<	<	87-Fr	<	<
30-Zn	<	<	88-Ra	0.0031	0.0013
31-Ga	<	<	89-Ac	<	<
32-Ge	<	<	90-Th	<	<
33-As	<	<	91-Pa	<	<
34-Se	<	<	92-U	<	<
35-Br	<	<	94-Pu	<	<
36-Kr	<	<	95-Am	<	<
37-Rb	<	<	96-Cm	<	<
38-Sr	<	<	97-Bk	<	<
39-Zn	<	<	98-Cf	<	<
40-Ni	<	<	99-Es	<	<
41-Nb	<	<	100-Fm	<	<
42-Mo	<	<	101-Lr	<	<
43-Tc	<	<			
44-Ru	0.0031	0.0006			
45-Rh	<	<			
46-Pd	<	<			
47-Ag	<	<			
48-Cd	<	<			
49-In	<	<			
50-Sn	<	<			
51-Sb	<	<			
52-Te	<	<			
53-I	<	<			
54-Xe	<	<			
55-Ce	0.0043	0.0004			
56-Ba	<	<			
57-La	<	<			
58-Ce	<	<			
59-Co	0.056	0.003			
60-Ni	<	<			
61-Pb	<	<			
62-Sn	<	<			
63-Bi	<	<			
64-Po	<	<			
65-At	<	<			
66-Rn	<	<			
67-Fr	<	<			
68-Ra	0.0031	0.0013			
69-Ac	<	<			
70-Th	<	<			
71-Pa	<	<			
72-Hf	<	<			
73-Ta	<	<			
74-W	0.149	0.010			
75-Re	<	<			
76-Os	0.0155	0.0008			
77-Ir	<	<			
78-Pt	0.0059	0.0007			
79-Au	0.0059	0.0007			
80-Hg	0.0059	0.0007			
81-Tl	0.0031	0.0006			
82-Pb	<	<			
83-Bi	<	<			
84-Po	<	<			
85-At	<	<			
86-Rn	<	<			
87-Fr	<	<			
88-Ra	0.0031	0.0013			
89-Ac	<	<			
90-Th	<	<			
91-Pa	<	<			
92-U	<	<			
94-Pu	<	<			
95-Am	<	<			
96-Cm	<	<			
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99-Es	<	<			
100-Fm	<	<			
101-Lr	<	<			

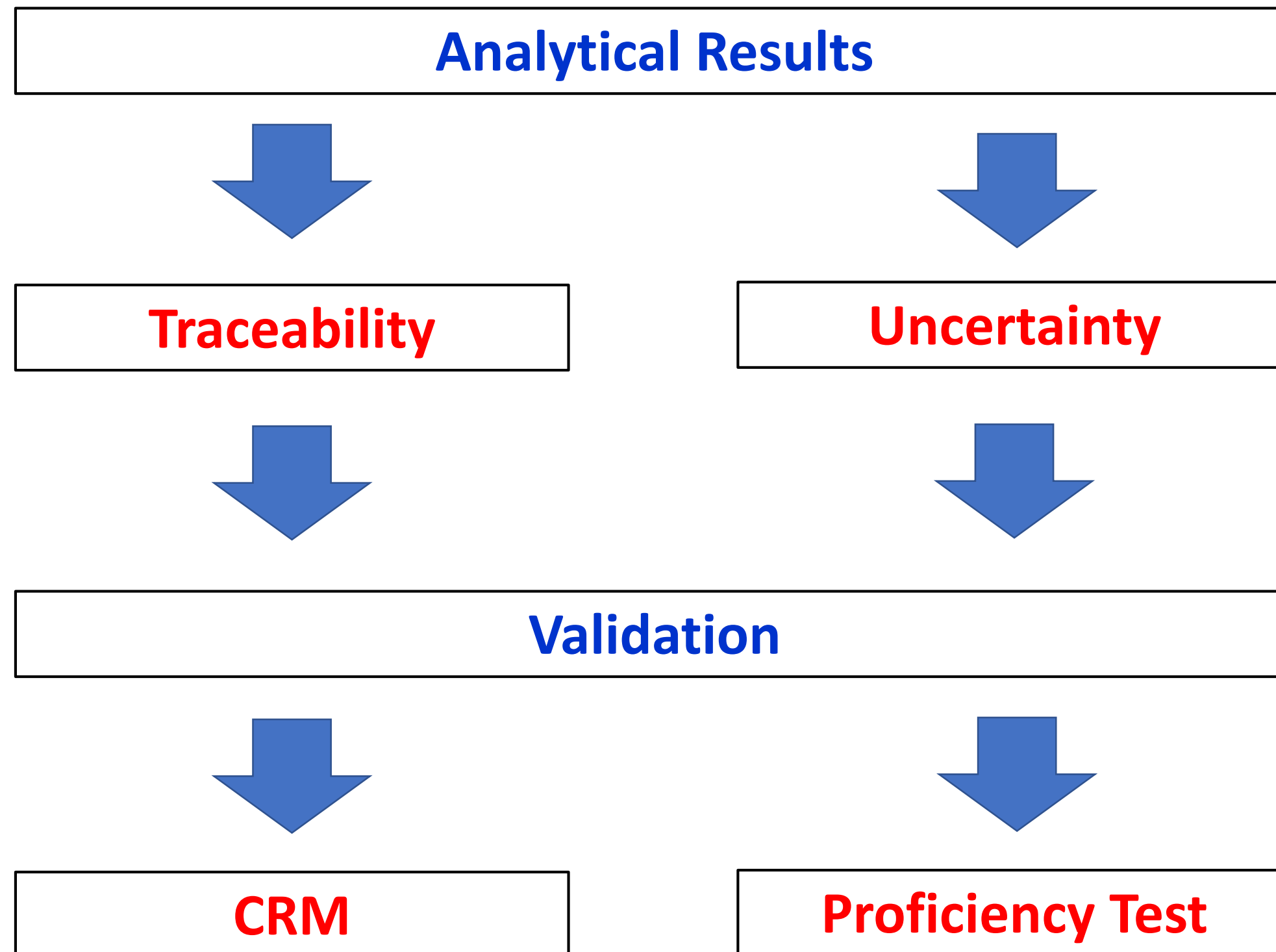
KnownConv: 0 REPT: 0
 Sum CONC without normalization to 100% : 40.5 %

Calibration Samples

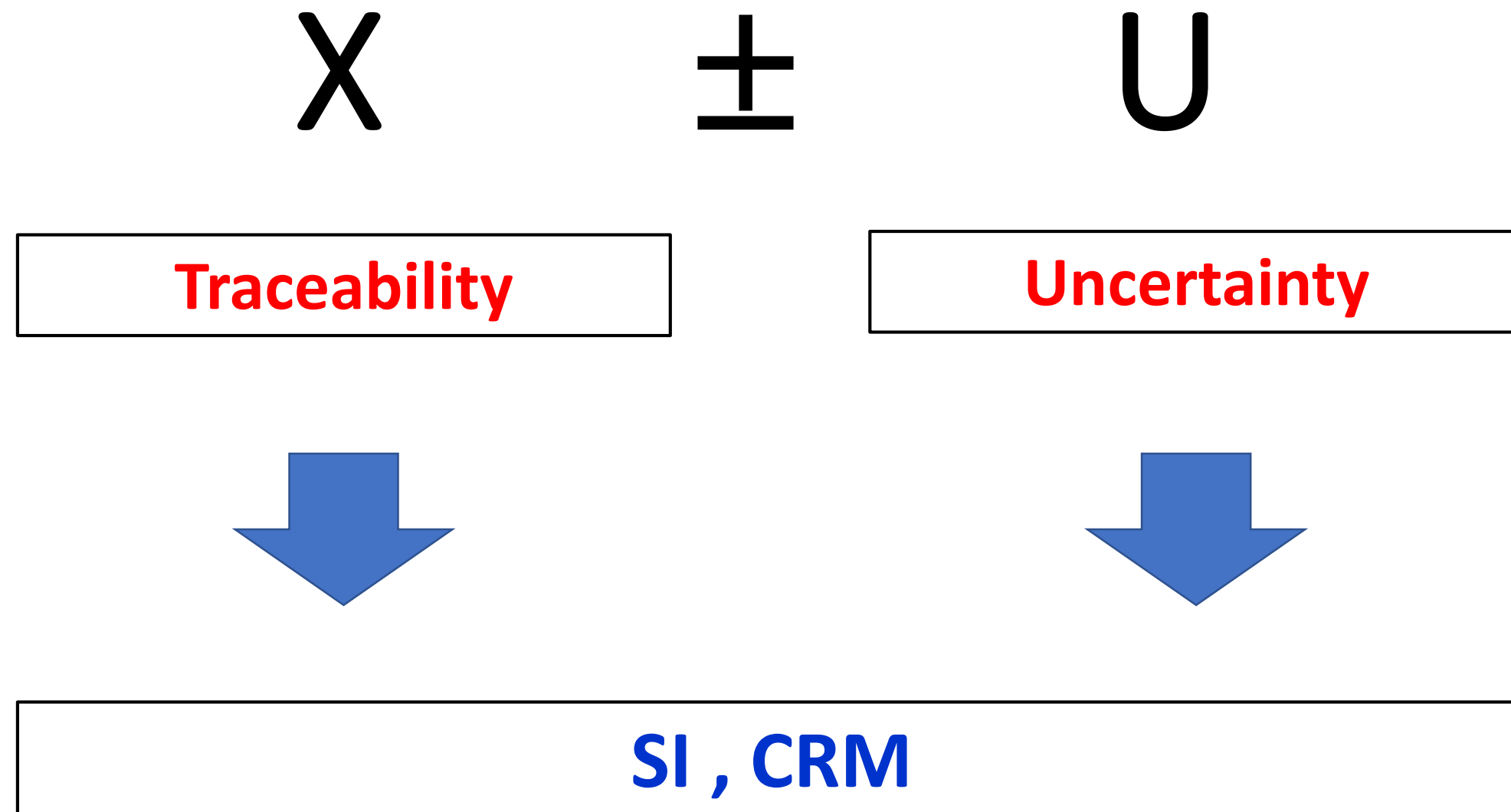


FLUXANA Accreditation ISO 17025, ISO 17034

Traceability of Analytical Results



Traceability of Analytical Results



GEO-QUANT Basic application package

- Certified reference materials
- 10g powders
- Easy to use according customer needs
- Software template for calibration
- Includes drift monitor samples
- Includes validation samples
- Ready to go



Certificate

30:34

Bemerkung =
 ANL ADVX KH 604V LSP200 FWT LSP220 AXIS Messk Programm : DnQuant
 C:\USERS\AM\Appl\AnySample.kap 2008-10-17
 Berechnet als : Elemente Matrix (Pore & V.Fakt): 1Teilern
 Messweg/Mod = Vakuum Filtertyp = Kein Trägerfilm
 Pulvermenge = 0 Allis bekannt
 Ziff. Durchm. = 25.0 mm Ziff. Fläche 490.6 mm2
 Bekannte Konz = 0 %
 Wert = 0 %
 Diluent/Probe = 0.250 Wache Krytische Masse = 3051.758 mg
 Probenhöhe = 3.00 mm
 = Bedeutet: Der Gehalt ist < 10 mg/kg
 +2e S.S. Gew = 2 StDehler, Bin + 2e, % bei St: Teil d. 100Summe
 w% StDerr Z w% StDerr Z

Element	Werte	StDerr	Werte	StDerr
Quelle..F	0	0.018	29 Cu	<
31-Mg	0.296	0.033	30-Zn	0.0482 0.0020 53 I
32-Mg	0.0546	0.0027	31-Ga	0.0043 0.0004 55-Ce
33-Al	7.33	0.14	32-Ge	<
34-Si	31.19	0.42	33-Ar	<
35-P	0.0047	0.0006	34-Sr	<
36-S	<	<	35-Sr	<
37-Mn	<	<	37-Mn	0.0199 0.0010 74-W
38-Zr	<	<	38-Zr	<
39-Ni	0.0287	0.0014	39-F	<
40-Ar	<	<	40-Zr	<
41-K	0.237	0.026	41-Nb	<
42-Mo	0.0127	0.0014	42-Mo	0.0155 0.0008 79-Au
43-Ti	<	<	44-Ru	0.0059 0.0007 80-Mg
44-Ru	0.0052	0.0004	45-Rh	0.0051 0.0006 81-Tl
45-Rh	<	<	46-Pd	<
46-Pd	<	<	47-Ag	<
47-Ag	<	<	48-Cd	<
48-Cd	0.0155	0.0008	48-Cd	<
49-In	0.709	0.079	49-In	<
50-Sn	0.0195	0.0010	50-Sn	<
51-Sb	<	<	51-Sb	<
52-Te	<	<	52-Te	<
53-I	<	<	53-I	<
54-Xe	<	<	54-Xe	<
55-Ce	0.018	0.001	55-Ce	0.0059 0.0007 57-La
56-Ba	<	<	56-Ba	0.0042 0.0010
57-La	<	<	57-La	<
58-Ce	<	<	58-Ce	<
59-Pr	<	<	59-Pr	<
60-Nd	<	<	60-Nd	<
61-Pm	<	<	61-Pm	<
62-Sm	<	<	62-Sm	<
63-Eu	<	<	63-Eu	<
64-Gd	<	<	64-Gd	<
65-Tb	<	<	65-Tb	<
66-Dy	<	<	66-Dy	<
67-Ho	<	<	67-Ho	<
68-Er	<	<	68-Er	0.0021 0.0013
69-Tm	<	<	69-Tm	<
70-Yb	<	<	70-Yb	<
71-Lu	<	<	71-Lu	<

Summe: 0
 Summe ohne Normalisierung zu 100% : 40.5 %

1 H Hydrogen 1.008																	2 He Helium 4.002602
3 Li Lithium 6.94	4 Be Beryllium 9.0121831											5 B Boron 10.81	6 C Carbon 12.011	7 N Nitrogen 14.007	8 O Oxygen 15.999	9 F Fluorine 18.998403163	10 Ne Neon 20.1797
11 Na Sodium 22.98976928	12 Mg Magnesium 24.305											13 Al Aluminium 26.9815385	14 Si Silicon 28.085	15 P Phosphorus 30.973761998	16 S Sulfur 32.06	17 Cl Chlorine 35.45	18 Ar Argon 39.948
19 K Potassium 39.0983	20 Ca Calcium 40.078	21 Sc Scandium 44.955908	22 Ti Titanium 47.867	23 V Vanadium 50.9415	24 Cr Chromium 51.9961	25 Mn Manganese 54.938044	26 Fe Iron 55.845	27 Co Cobalt 58.933194	28 Ni Nickel 58.6934	29 Cu Copper 63.546	30 Zn Zinc 65.38	31 Ga Gallium 69.723	32 Ge Germanium 72.630	33 As Arsenic 74.921595	34 Se Selenium 78.971	35 Br Bromine 79.904	36 Kr Krypton 83.798
37 Rb Rubidium 85.4678	38 Sr Strontium 87.62	39 Y Yttrium 88.90584	40 Zr Zirconium 91.224	41 Nb Niobium 92.90637	42 Mo Molybdenum 95.95	43 Tc Technetium (98)	44 Ru Ruthenium 101.07	45 Rh Rhodium 102.90550	46 Pd Palladium 106.42	47 Ag Silver 107.8682	48 Cd Cadmium 112.414	49 In Indium 114.818	50 Sn Tin 118.710	51 Sb Antimony 121.760	52 Te Tellurium 127.60	53 I Iodine 126.90447	54 Xe Xenon 131.293
55 Cs Caesium 132.90545196	56 Ba Barium 137.327	57 - 71 Lanthanoids	72 Hf Hafnium 178.49	73 Ta Tantalum 180.94788	74 W Tungsten 183.84	75 Re Rhenium 186.207	76 Os Osmium 190.23	77 Ir Iridium 192.217	78 Pt Platinum 195.084	79 Au Gold 196.966569	80 Hg Mercury 200.592	81 Tl Thallium 204.38	82 Pb Lead 207.2	83 Bi Bismuth 208.98040	84 Po Polonium (209)	85 At Astatine (210)	86 Rn Radon (222)
87 Fr Francium (223)	88 Ra Radium (226)	89 - 103 Actinoids	104 Rf Rutherfordium (267)	105 Db Dubnium (268)	106 Sg Seaborgium (269)	107 Bh Bohrium (270)	108 Hs Hassium (269)	109 Mt Meitnerium (278)	110 Ds Darmstadtium (281)	111 Rg Roentgenium (282)	112 Cn Copernicium (285)	113 Nh Nihonium (286)	114 Fl Flerovium (289)	115 Mc Moscovium (289)	116 Lv Livermorium (293)	117 Ts Tennessine (294)	118 Og Oganesson (294)

57 La Lanthanum 138.90547	58 Ce Cerium 140.116	59 Pr Praseodymium 140.90766	60 Nd Neodymium 144.242	61 Pm Promethium (145)	62 Sm Samarium 150.36	63 Eu Europium 151.964	64 Gd Gadolinium 157.25	65 Tb Terbium 158.92535	66 Dy Dysprosium 162.500	67 Ho Holmium 164.93033	68 Er Erbium 167.259	69 Tm Thulium 168.93422	70 Yb Ytterbium 173.045	71 Lu Lutetium 174.9668
89 Ac Actinium (227)	90 Th Thorium 232.0377	91 Pa Protactinium 231.03588	92 U Uranium 238.02891	93 Np Neptunium (237)	94 Pu Plutonium (244)	95 Am Americium (243)	96 Cm Curium (247)	97 Bk Berkelium (247)	98 Cf Californium (251)	99 Es Einsteinium (252)	100 Fm Fermium (257)	101 Md Mendelevium (258)	102 No Nobelium (259)	103 Lr Lawrencium (266)

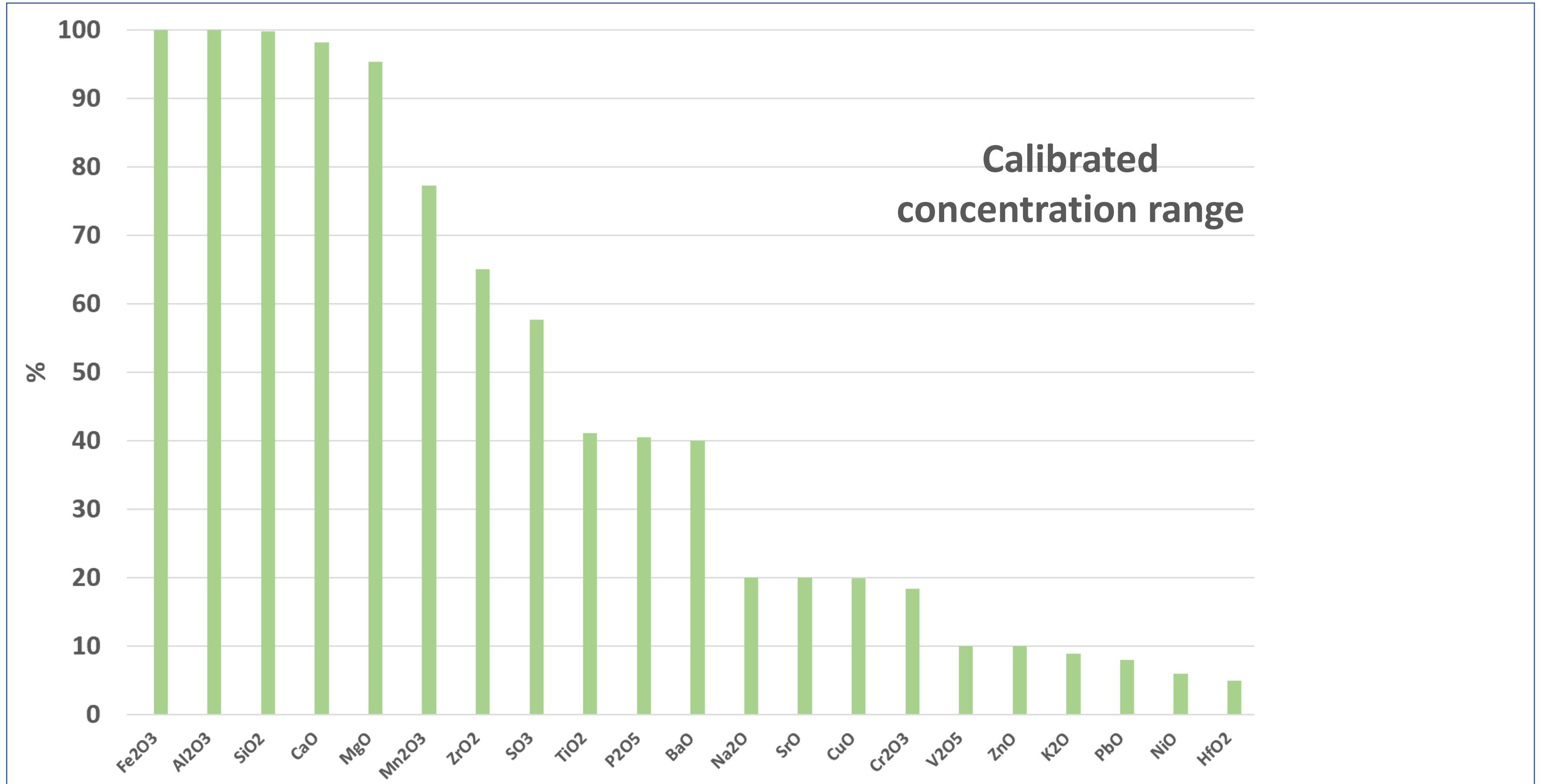
Commercially available
reference materials



Lack of reference materials for
Zr, Hf, Pb, Zn, Ni, Cu



GEO-QUANT Advanced Application



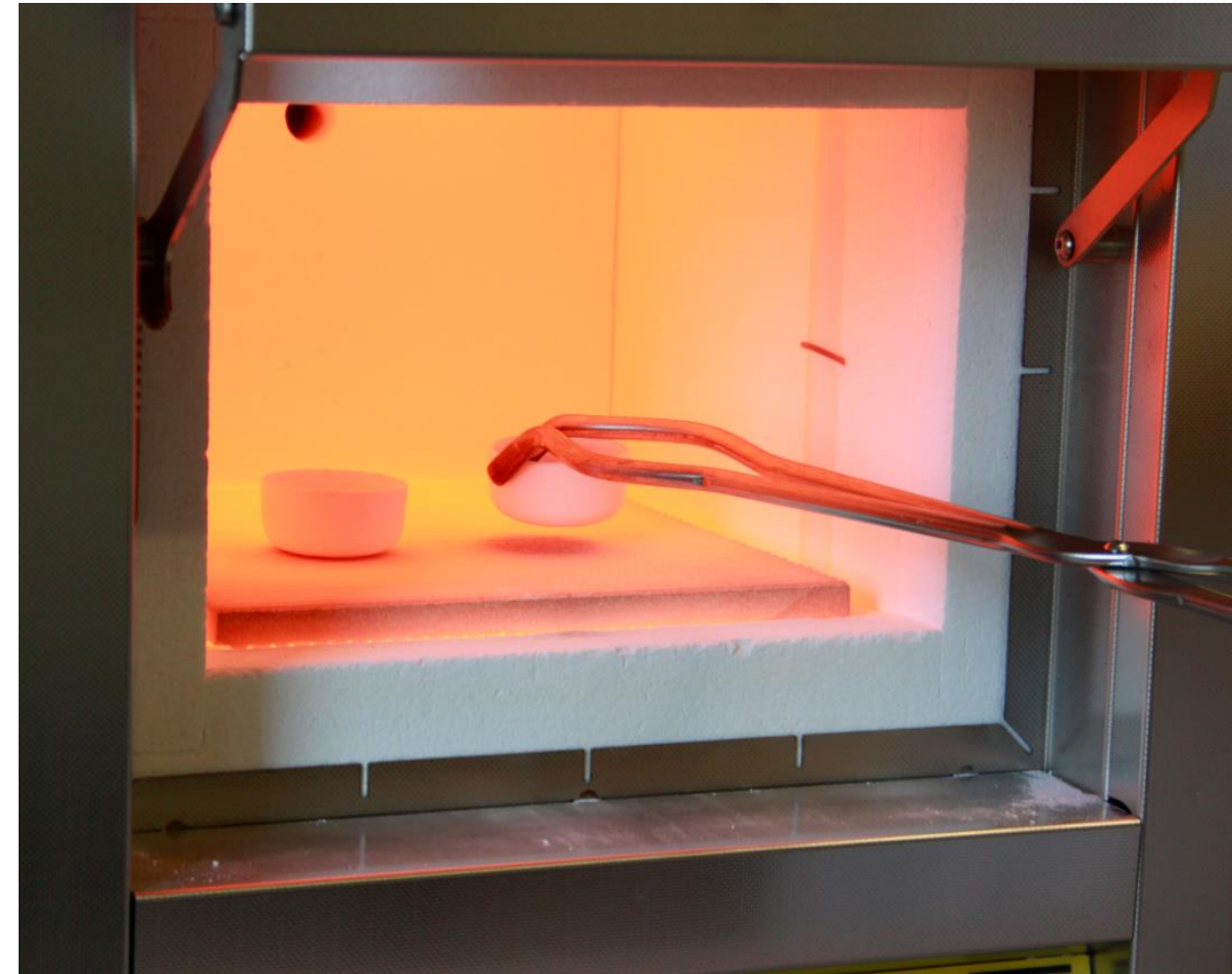
Solution:

Use of pure chemicals like SiO₂, CaCO₃, MgO, Al₂O₃, etc.



1 H Hydrogen 1.008																	2 He Helium 4.002602
3 Li Lithium 6.94	4 Be Beryllium 9.0121831											5 B Boron 10.81	6 C Carbon 12.011	7 N Nitrogen 14.007	8 O Oxygen 15.999	9 F Fluorine 18.998403163	10 Ne Neon 20.1797
11 Na Sodium 22.98976928	12 Mg Magnesium 24.305											13 Al Aluminium 26.9815385	14 Si Silicon 28.085	15 P Phosphorus 30.973761998	16 S Sulfur 32.06	17 Cl Chlorine 35.45	18 Ar Argon 39.948
19 K Potassium 39.0983	20 Ca Calcium 40.078	21 Sc Scandium 44.955908	22 Ti Titanium 47.867	23 V Vanadium 50.9415	24 Cr Chromium 51.9961	25 Mn Manganese 54.938044	26 Fe Iron 55.845	27 Co Cobalt 58.933194	28 Ni Nickel 58.6934	29 Cu Copper 63.546	30 Zn Zinc 65.38	31 Ga Gallium 69.723	32 Ge Germanium 72.630	33 As Arsenic 74.921595	34 Se Selenium 78.971	35 Br Bromine 79.904	36 Kr Krypton 83.798
37 Rb Rubidium 85.4678	38 Sr Strontium 87.62	39 Y Yttrium 88.90584	40 Zr Zirconium 91.224	41 Nb Niobium 92.90637	42 Mo Molybdenum 95.95	43 Tc Technetium (98)	44 Ru Ruthenium 101.07	45 Rh Rhodium 102.90550	46 Pd Palladium 106.42	47 Ag Silver 107.8682	48 Cd Cadmium 112.414	49 In Indium 114.818	50 Sn Tin 118.710	51 Sb Antimony 121.757	52 Te Tellurium 127.60	53 I Iodine 126.90447	54 Xe Xenon 131.293
55 Cs Caesium 132.90545196	56 Ba Barium 137.327	57 - 71 Lanthanoids	72 Hf Hafnium 178.49	73 Ta Tantalum 180.94788	74 W Tungsten 183.84	75 Re Rhenium 186.207	76 Os Osmium 190.23	77 Ir Iridium 192.217	78 Pt Platinum 195.084	79 Au Gold 196.966569	80 Hg Mercury 200.592	81 Tl Thallium 204.38	82 Pb Lead 207.2	83 Bi Bismuth 208.98040	84 Po Polonium (209)	85 At Astatine (210)	86 Rn Radon (222)
87 Fr Francium (223)	88 Ra Radium (226)	89 - 103 Actinoids	104 Rf Rutherfordium (261)	105 Db Dubnium (268)	106 Sg Seaborgium (269)	107 Bh Bohrium (270)	108 Hs Hassium (277)	109 Mt Meitnerium (276)	110 Ds Darmstadtium (281)	111 Rg Roentgenium (282)	112 Cn Copernicium (285)	113 Nh Nihonium (286)	114 Fl Flerovium (289)	115 Mc Moscovium (289)	116 Lv Livermorium (293)	117 Ts Tennessine (294)	118 Og Oganesson (294)

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Pretreatment of primary chemicals for traceability

As powder mixture in a
bottle:

Risk of inhomogeneity



Solution:

As fused glass powder in
a bottle:

Perfectly homogeneous



Preparation of Glass Powder

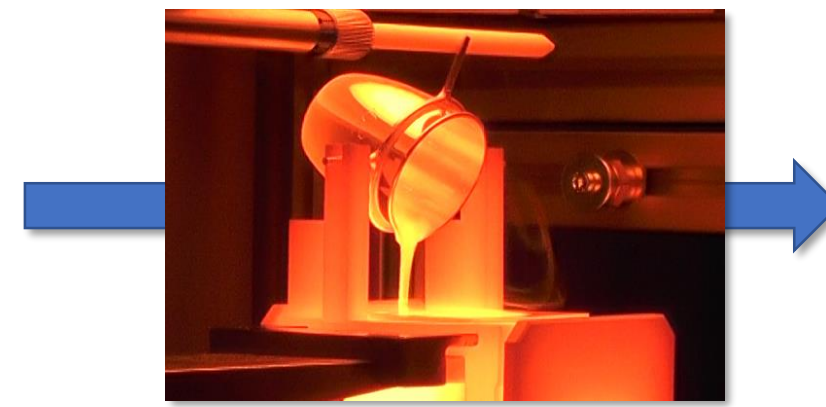


Pure chemicals

+



Lithium borate



Fusion



Glass powder

- homogeneous
- approved quality
- customizable
- independent on fusion process

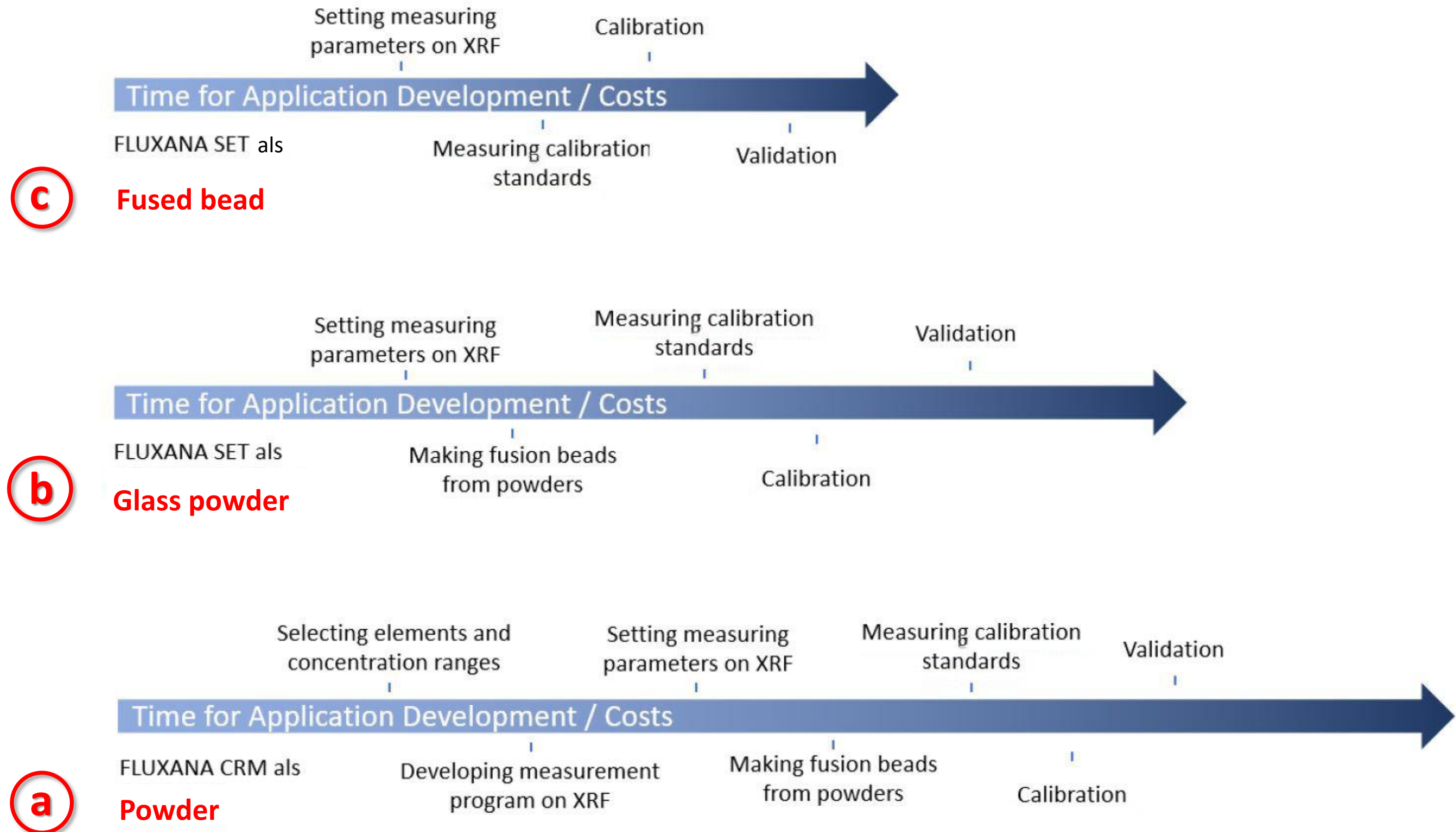
GEO-QUANT Advanced application package

- Glass powder from pure chemicals
- 30g powders including flux
- Just pour into crucible 9 - 10g
- Software template for calibration
- Includes drift monitor samples
- Includes validation samples
- Ready to go



Certificate														
Bemerkung: = SDC ADXV 1h 60V LAF200 TWT LAF220 AX13 Max Program : GeoQuant C:\LOCALSERIAL\Appl\AnyTempLap 2008-10-17 Berechnet als : Elemente Matrix (Pore & V.fakt): 1 Yellow Abgleichsfall : Vanadium Filtertyp : Kein Trägerfile Fallnummer : 0 Alle bekannt Ziff. Durchs. : 21.0 mm Ziff. Fläche 490.6 mm ² Substratname Kenn : 0 8 Wert : 0 8 Diluent/Probe : 0.250 Weiche Prüfling Masse = 3051.758 mg Prüflinghöhe = 5.80 mm = bedeutet: Der Gehalt ist < 10 mg/kg =2w = u.U. Gehalt < 2 Stufenher. Min = o. = heißt: Teil d. 1000erwert														
Z	Symbol	StdErr	Z	StdErr	Z	StdErr	Z	StdErr	Z	StdErr	Z	StdErr	Z	StdErr
29	Cu	0.016	29	Cu	<	52	Te	<						
31	Na	0.296	0.033	30	Zn	0.0402	0.0020	53	I	<				
32	Mg	0.0546	0.0027	31	Ge	0.0043	0.0006	55	Ce	0.104	0.006			
33	Al	7.53	0.14	32	Se	<		56	Ba	<				
34	Si	31.39	0.62	33	As	<		58	La	0.051	0.002			
35	P	0.0047	0.0008	34	Se	<		72	Hf	<				
35	P	<		35	Br	<		73	Ta	<				
36	S	<		37	Rb	0.0199	0.0010	74	W	0.149	0.019			
37	Cl	0.0287	0.0014	38	Sr	<		75	Hs	<				
38	Ar	<		40	Zr	<		76	Os	<				
39	K	0.237	0.026	42	Nb	<		78	Pt	0.0030	0.0010			
40	Ca	0.0127	0.0014	42	Mo	0.0155	0.0008	79	Au	<2w	0.0013			
41	Sc	<		44	Ru	0.0059	0.0007	80	Hg	<				
42	Ti	0.0052	0.0004	45	Rh	0.0031	0.0008	81	Tl	<2w	0.0015			
43	V	<		46	Pd	<		82	Pb	<2w	0.0014			
44	Cr	<		47	Ag	<		83	Bi	<				
45	Mn	0.0105	0.0008	48	Cd	<		85	Tb	<				
46	Fe	0.709	0.079	49	In	<		92	U	<				
47	Co	0.0199	0.0010	50	Sn	<		94	Pu	<				
48	Ni	<2w	0.0006	51	Sb	<		95	Am	<				
----- Schwermetalle -----														
----- Leichtmetalle -----														
----- Edelmetalle -----														
----- Lanthaniden -----														
4	Be	0.018	44	Ru	0.0019	0.0007	57	La	0.0042	0.0010				
5	B	<	45	Rh	0.0011	0.0006	58	Ce	<2w	0.0024				
6	C	46	Pd	<	59	Pv	<2w	0.0025						
7	N	47	Ag	<	60	Nd	<							
8	O	75	Hs	<	62	Sm	<							
9	F	76	Os	<	63	Eu	<							
		77	Ir	<	64	Gd	<							
		78	Pt	0.0030	0.0010	65	Tb	<						
		79	Au	<2w	0.0013	0.0013	66	Dy	<					
								67	Ho	<				
								68	Er	0.0031	0.0013			
								69	Tm	<				
								70	Yb	<				
								71	Lu	<2w	0.0013			
KnownComp: 0 REPT: 0 D/G: 0.250Weiche														
Sum CONCw without normalization to 100% : 46.5 %														

Comparison of FLUXANA Calibration Samples



Advantages of ready to go applications:

- Save development time
- Save costs
- Expert advice
- Ready-to-go
- Guarantee on reproducibility
- Calibration with highest quality
- Newcomers become experts quickly
- Guaranteed after sales support
- FLUXANA holds ISO 17025 and 17034 accreditation





Dr. Colin Slater
R&D, Bruker AXS GmbH

So what's the problem here?



So what's the problem here?



MATRIX EFFECTS

**Lack of availability of
CRMs**

So what's the problem here?

The fundamental parameters are:

- Total mass absorbance coefficients
- Mass photoabsorbance coefficients
- Tube spectrum profile and intensity
- Shell fluorescence yields
- Line transition probabilities
- Line Energy
- Concentration of element
- Geometric considerations (instrument parameters)

For the sample as a whole

**For each element
in the sample**

Any method created for XRF (whether standards based or standardless) is only as good as the information given to the model.

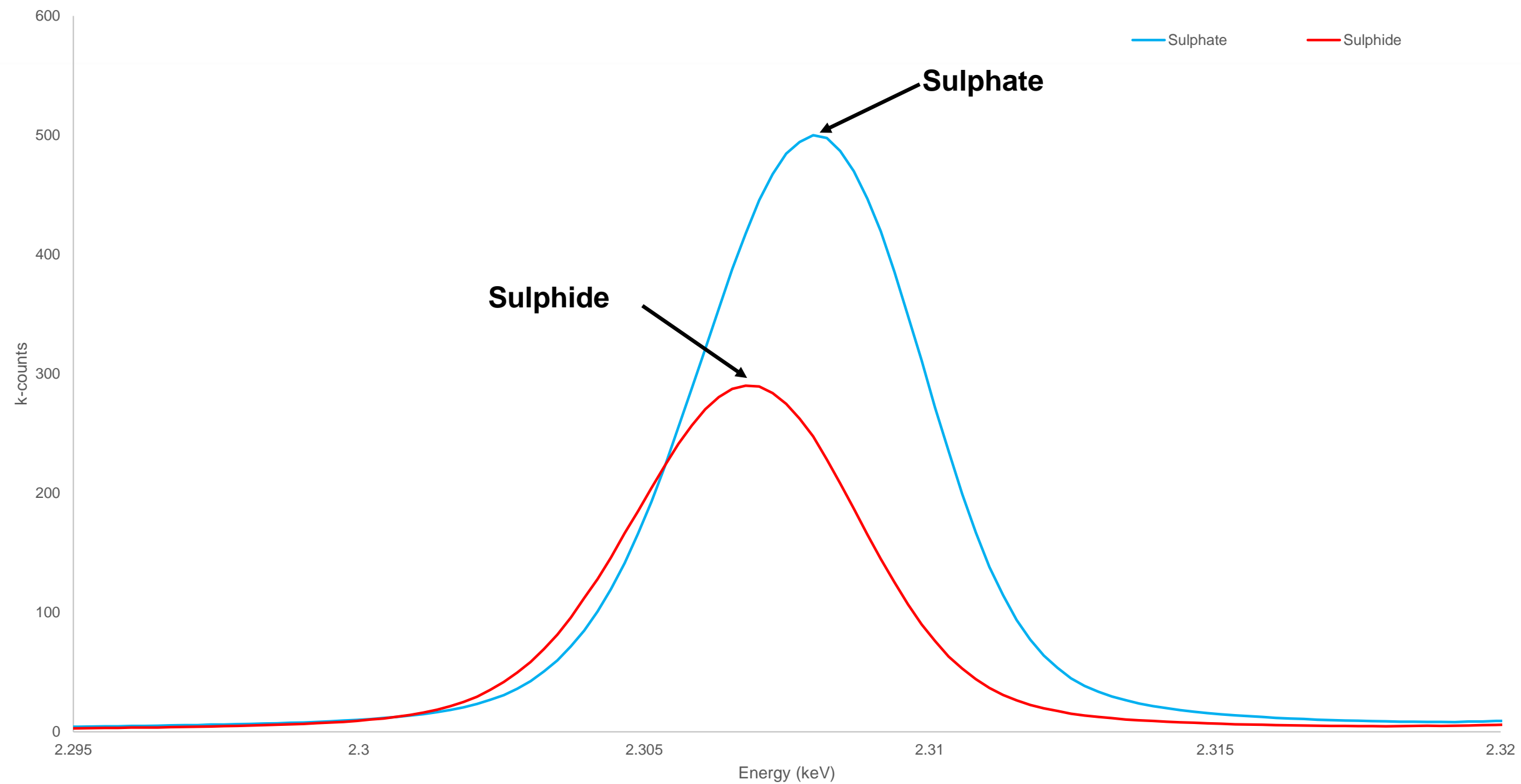
So what's the problem here?

Variable Oxidation State



Shift in peak position.

Variable oxygen content causes changes to mass absorbance.



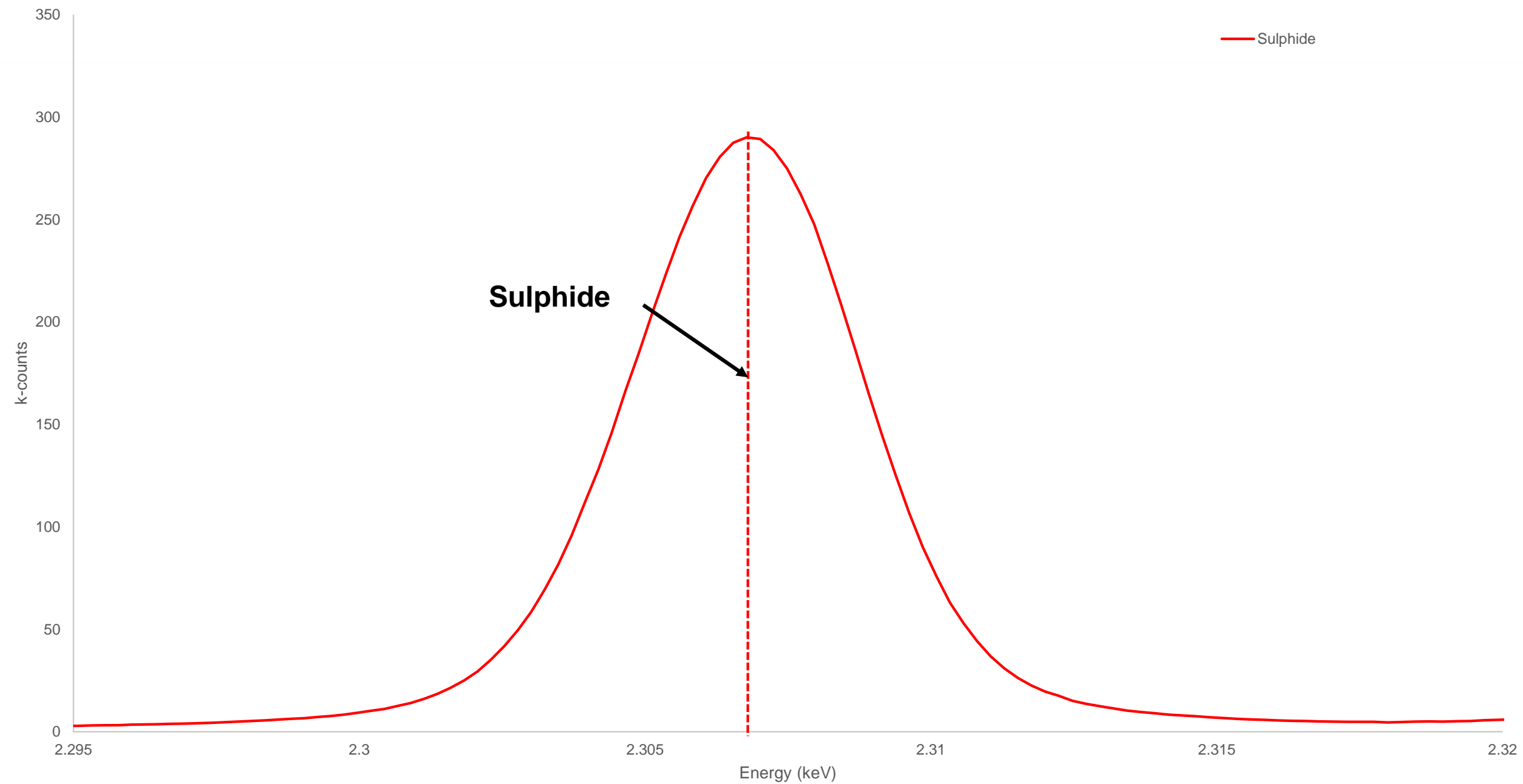
So what's the problem here?

Variable Oxidation State



Shift in peak position.

Variable oxygen content causes changes to mass absorbance.



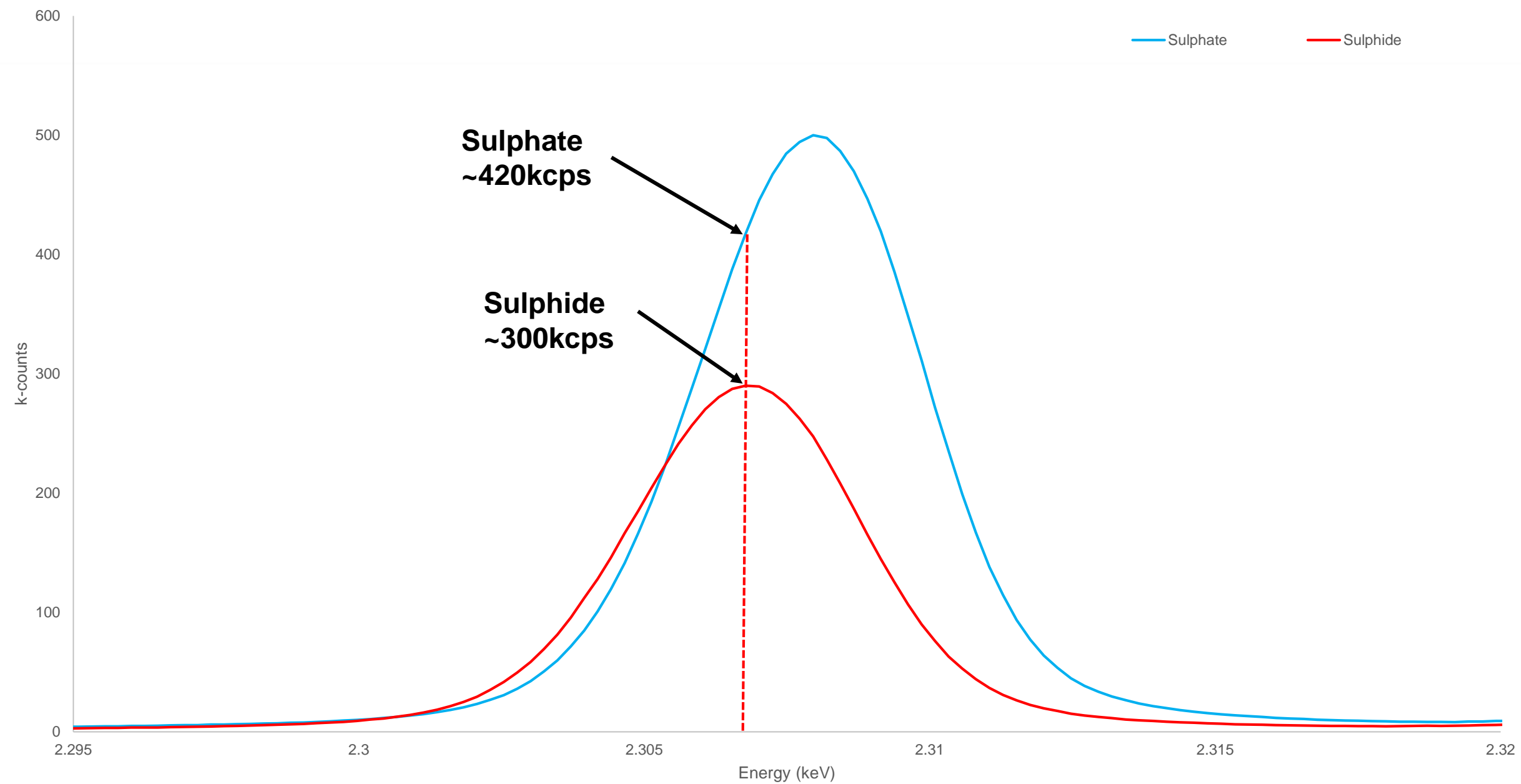
So what's the problem here?

Variable Oxidation State



Shift in peak position.

Variable oxygen content causes changes to mass absorbance.



But there aren't any matrix matched standards for my material!!!



But there aren't any standards for my material... what do I do?

But there aren't any matrix matched standards for my material!!!



But there aren't any standards for my material... what do I do?

What's your sample throughput like?

But there aren't any matrix matched standards for my material!!!



But there aren't any standards for my material... what do I do?

What's your sample throughput like?

For some materials, we need an answer within ~15-20mins of sample arriving.

Other materials are not so time critical.

Quick Review of Sample Preparation Techniques



Fusion Beads

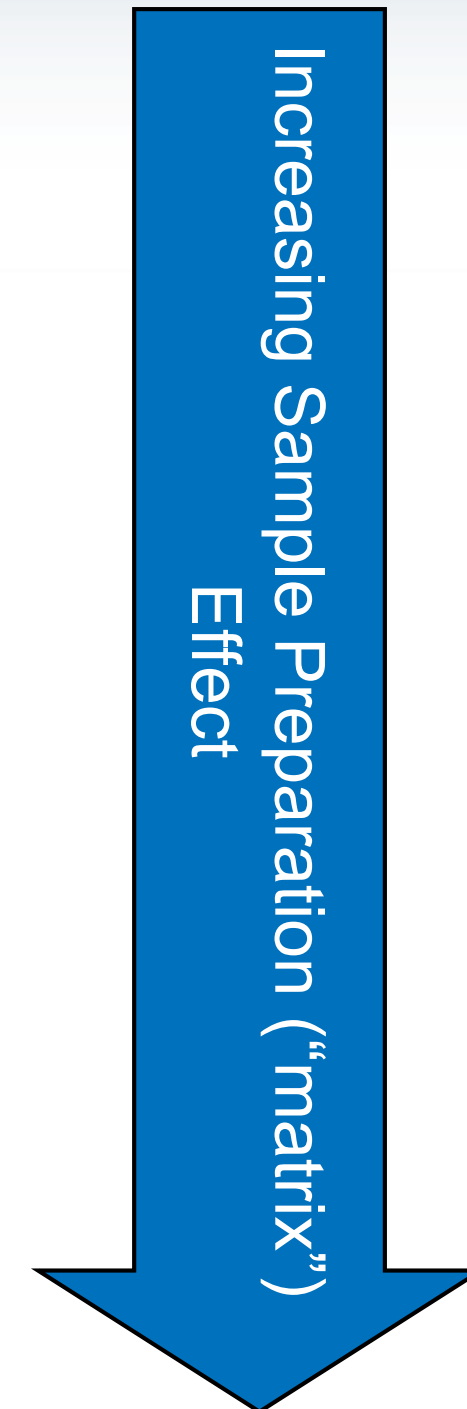
- Ignite sample @ 1050°C (typically 8-12hrs including cooling)
- Weight sample + flux
- Fusion: typically 15-20 mins per cycle, but can produce multiple (up to 6) beads in a cycle
- Cleaning crucibles: 15-20mins in citric acid bath with ultrasound

Pressed Powders

- Grinding of the sample: 15-60s (typically 20-30s for most samples)
- Cleaning of grinding mill: 15-60s (same cycle as sample run) + drying
- Weighing of ground powder
- Weighing of binding agent
- Mixing of sample with binding agent (60-120s)
- Pressing (typically 20-60s)

Loose Powders

- Preparation of liquid cups can be very quick
- Grinding of powders generally quick... typically 15-60 seconds is sufficient for most common sample types
- Weighing of powders very quick



But there aren't any matrix matched standards for my material!!!



But there aren't any standards for my material... what do I do?

What's your sample throughput like?

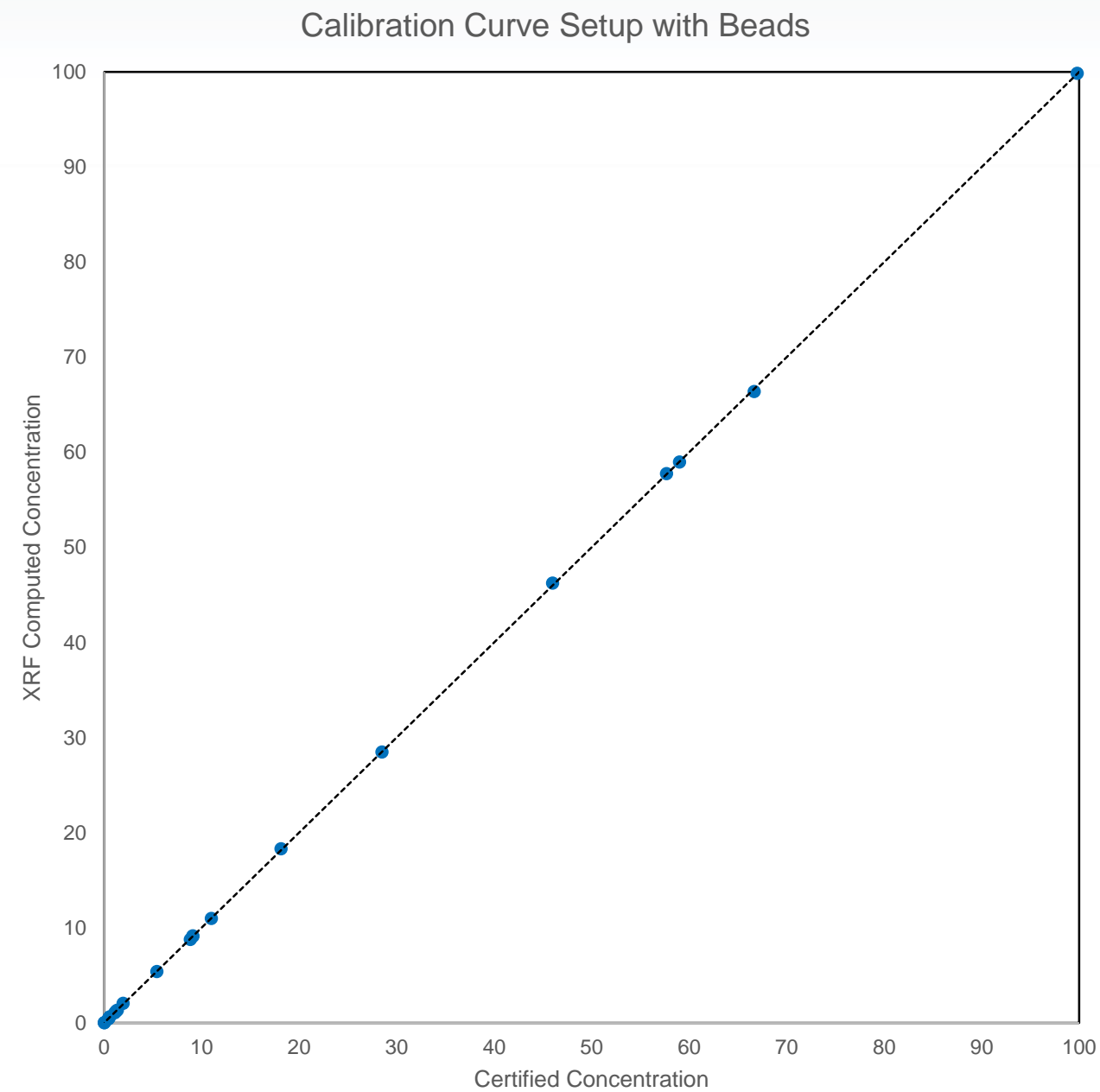
For some materials, we need an answer within ~15-20mins of sample arriving.

Other materials are not so time critical.

Your materials which are not time critical could just be analysed by fusion bead using a wide-oxide calibration.

For the time critical materials, you could make your own secondary standards.

How does a secondary calibration work?



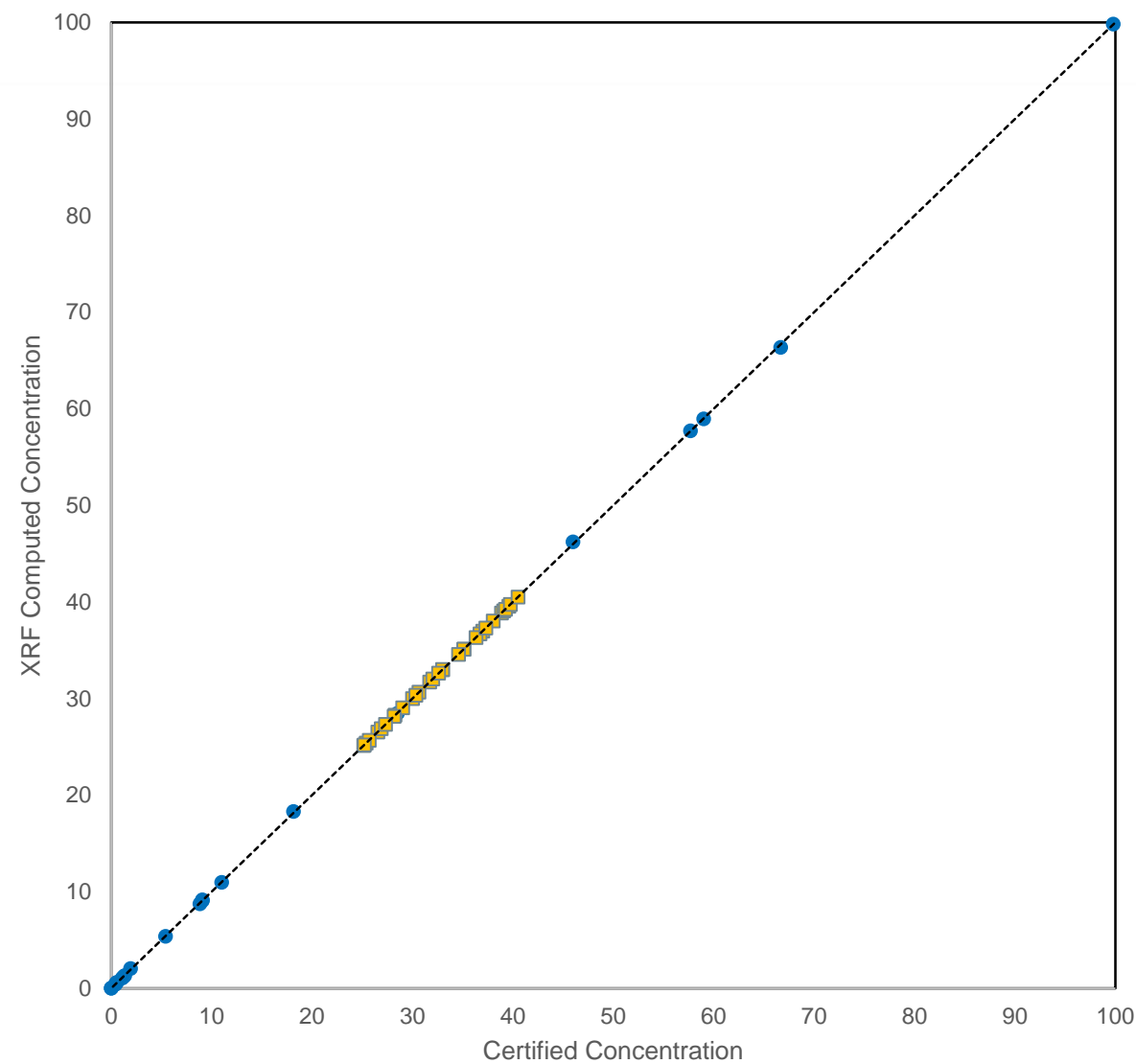
1

Create a calibration using suitable CRMs, prepared as **fusion beads**

How does a secondary calibration work?



Calibration Curve Setup with Beads



2

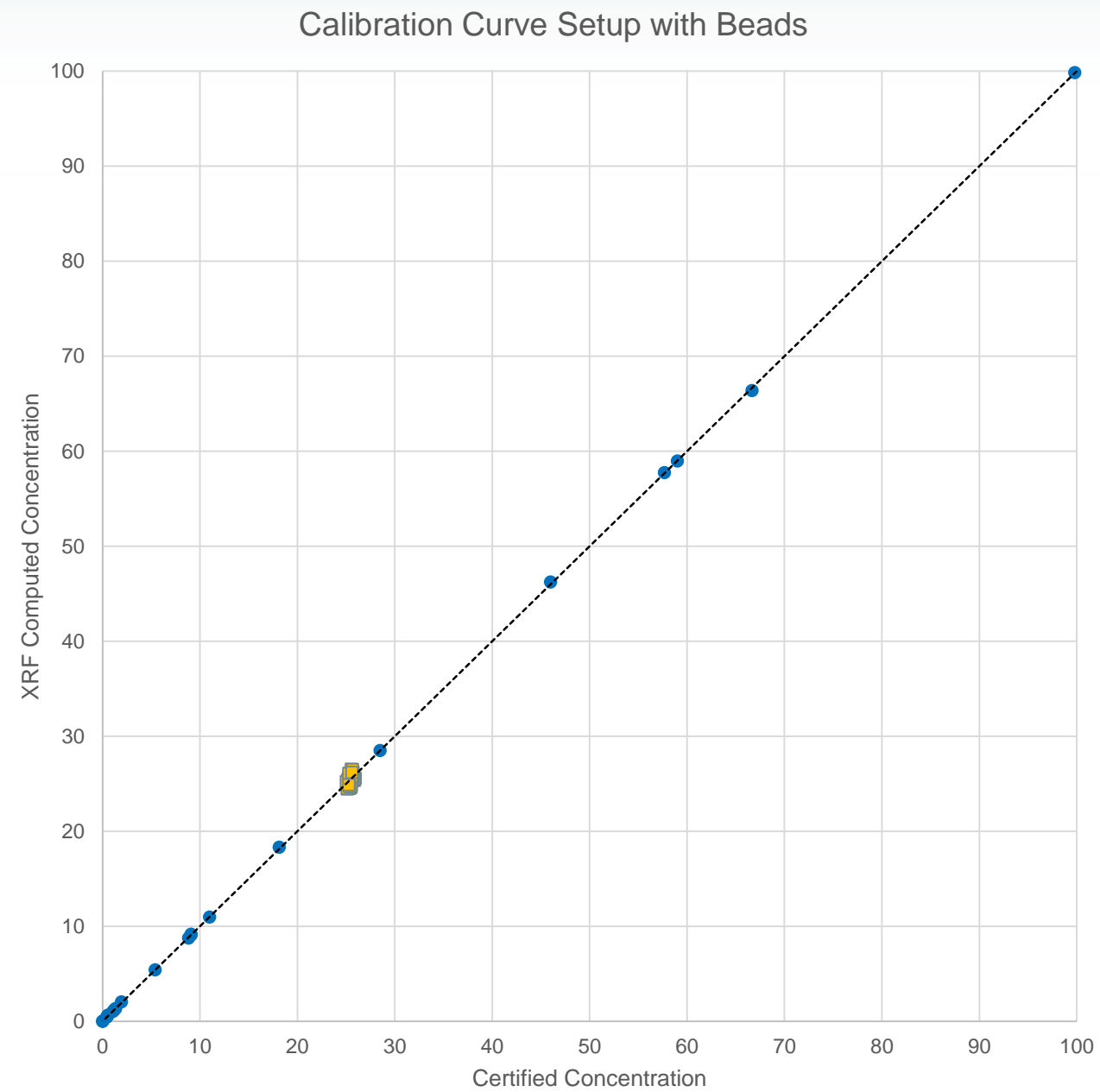
Take a selection of production material, prepare these as fusion beads (same recipe as your calibration) and measure using your fusion bead method.

Top Tip #1



- Production samples don't usually have too wide a spread of concentrations
 - Lots of points on top of each other won't create a good calibration
- Standards Required = $2n + 1$, where n is the number of correction factors applied
- Consider whether you need to test duplicates of each sample to check for homogeneity in the sample you've taken.
- Test more production samples that you think you need.

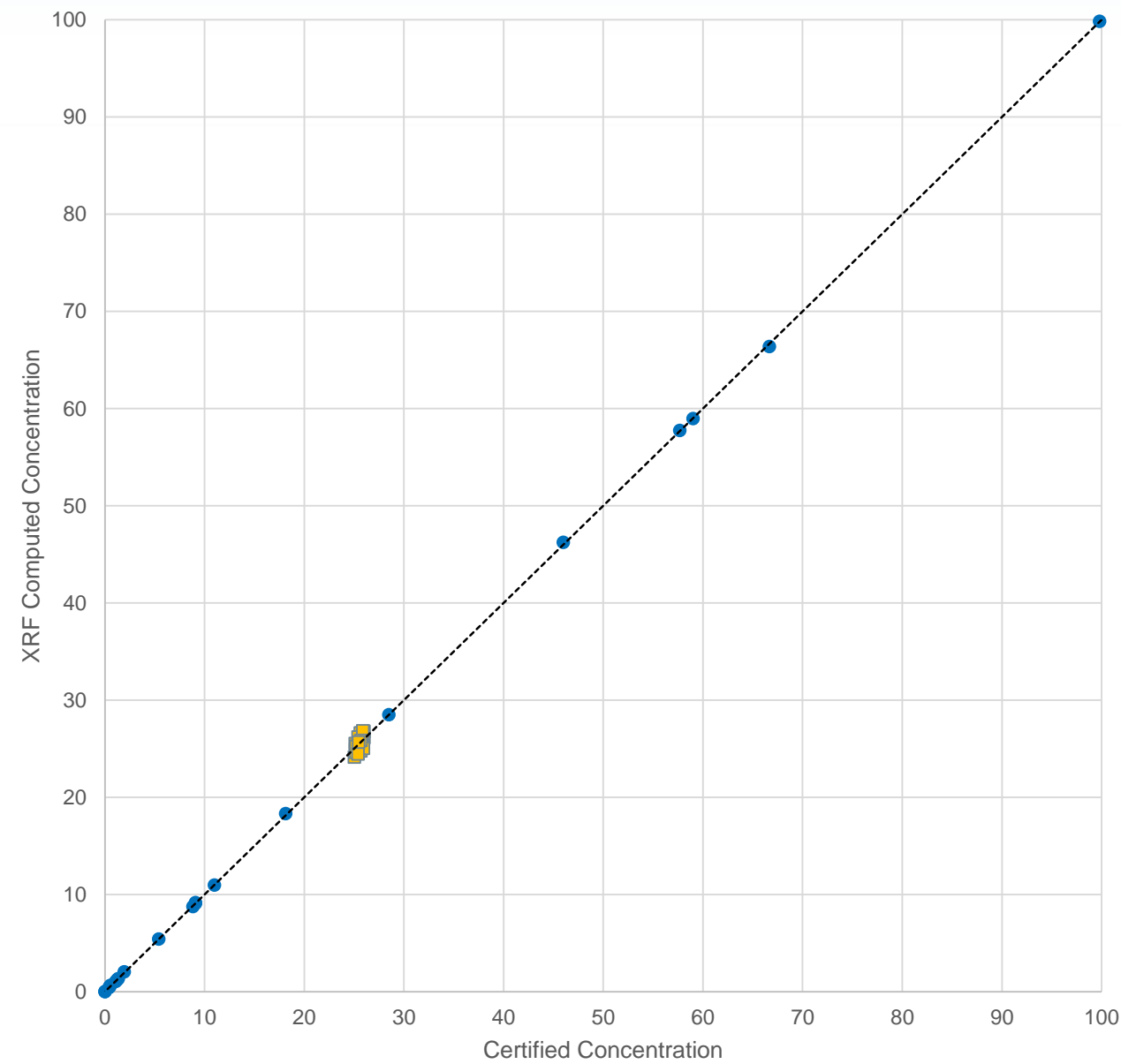
How does a secondary calibration work?



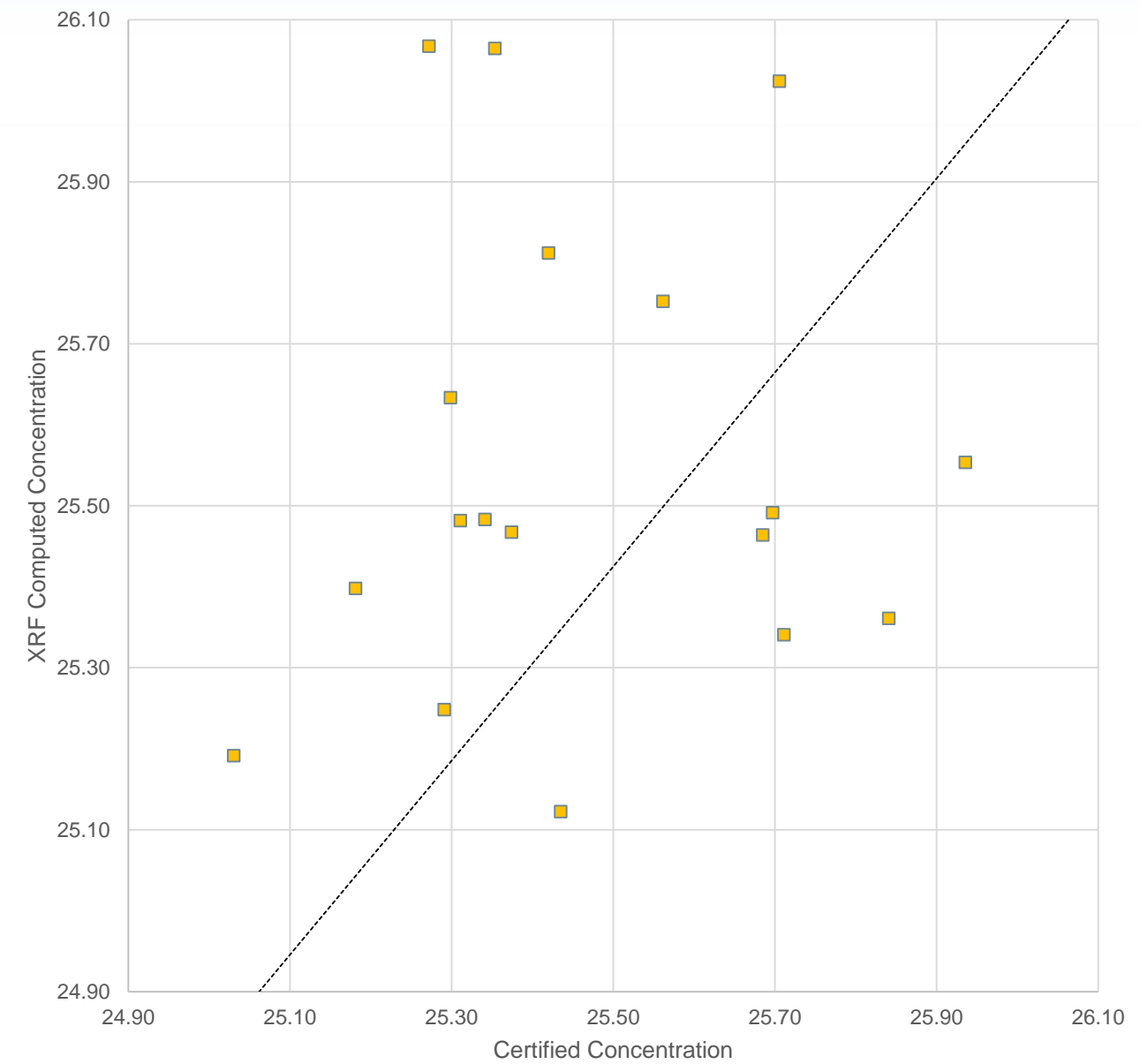
How does a secondary calibration work?



Calibration Curve Setup with Beads



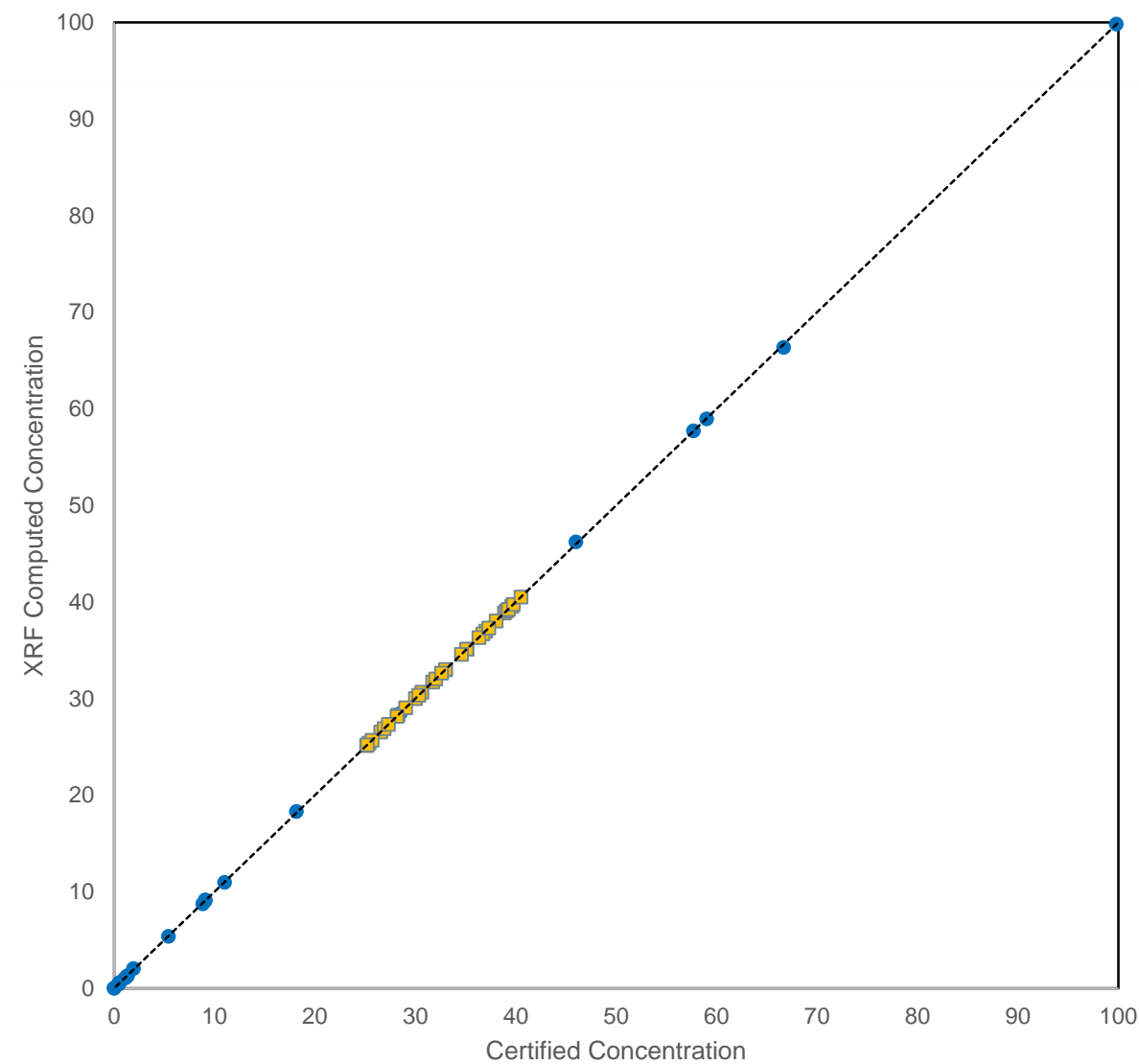
Calibration Curve Setup with Pellets



How does a secondary calibration work?



Calibration Curve Setup with Beads



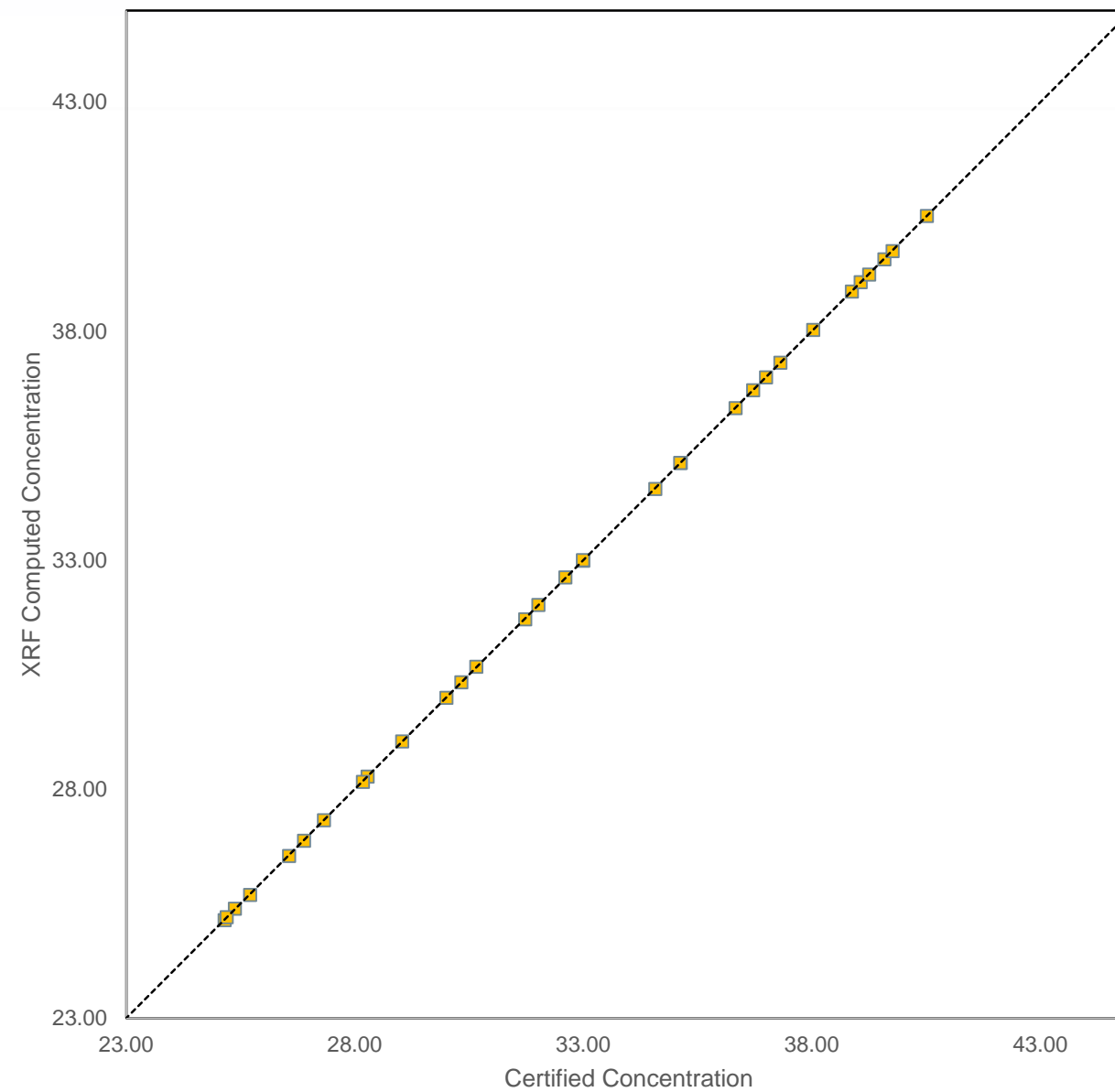
2

Take a selection of production material, prepare these as fusion beads (same recipe as your calibration) and measure using your fusion bead method.

How does a secondary calibration work?



Calibration Curve Setup with Pellets



3

Prepare the same production material as pellets (or which ever preparation your need) and calibrate using the concentrations obtained from your fusion bead measurements.

Fusion Beads do still display matrix effects



- Fusion beads are **NOT** matrix effect free
- The fusion bead technique is an oxidative digestion
 - Your sample is digested and each element converted to a common oxidation state
- These samples are more correctly described as having a *Stabilised Matrix*

Top Tip #2



**Make sure your wide oxide calibration contains standards of similar compositions to those which you wish to analyse.
Pick an appropriate fusion recipe.**

- This will help to make sure that appropriate matrix corrections can be calculated for the right combinations of elements
- Only go to concentrations ranges you're actually going to use
- Not all materials will dissolve properly in all fluxes / flux mixtures

Examples of Primary Calibration Methods

The following methods are available in the Bruker catalogue:

- **CEMENT-QUANT** SiO_2 : 19.5 – 31.2wt%
 - For the analysis of 14 oxides in cements and related materials
- **GEO-QUANT Basic** SiO_2 : 0.51 – 99.85wt%
 - For the analysis of 14 oxides in raw materials and common geological phases
- **GEO-QUANT Advanced**
 - For the analysis of 21 oxides in raw materials and an extended range of geological and industrial phases
- **SLAG-QUANT**
 - For the analysis of slags from the iron and steel making process

Two real life examples

GEO-QUANT Advanced

- Wide Oxide Calibration for 21 elements in Oxide Form
- Fusion Bead Preparation using 'Ready-to-Fuse' Preparation
- Requires ignited sample

SLAG-QUANT

- Wide Oxide Calibration specifically developed for analysis of slag samples
- Sulphur analysis from CGA
- Special fusion method that allows dry sample to be used

GEO-QUANT Advanced



- 32 CRMs for Calibration
- 5 CRMs for QC
- 3 Glasses for DC
- Calibration parameters on a USB Stick

Sample Preparation:

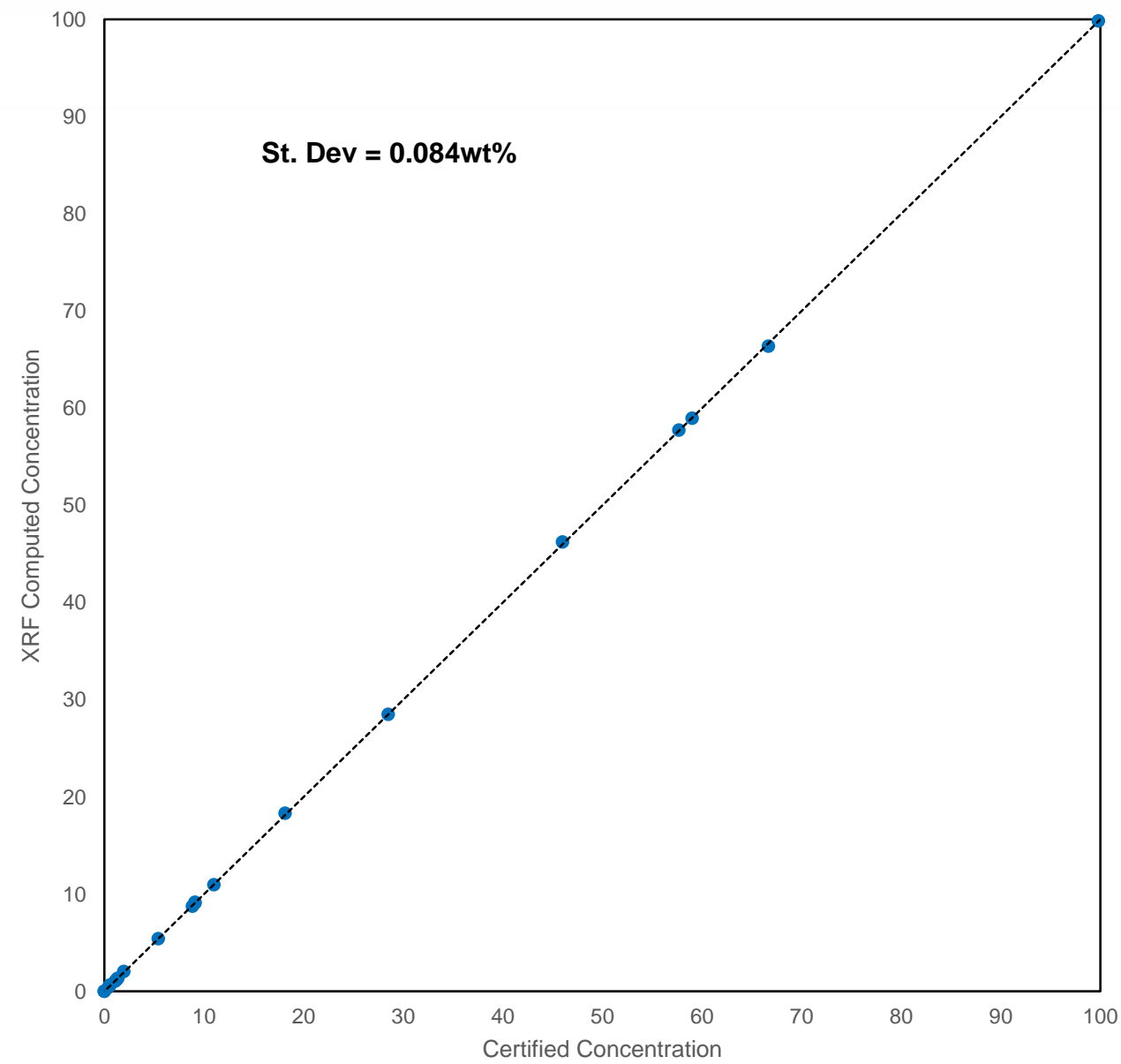
- 0.50g Ignited Sample
- 8.50g 66T:34M Flux

Analyte	Min. (wt%)	Max. (wt%)	St. Dev (1)	St. Dev (2)	LLD (%) ⁽¹⁾
Na ₂ O	0.01	20.06	0.045		0.03
MgO	0.01	95.36	0.1		0.01
Al ₂ O ₃	0.05	100.00	0.084		0.008
SiO ₂	0.40	99.80	0.16		0.009
P ₂ O ₅	0.01	40.57	0.04		0.004
SO ₃	0.01	56.40	0.18		0.004
K ₂ O	0.01	8.96	0.03		0.003
CaO	0.02	97.88	0.17	0.32	0.004
TiO ₂	0.01	40.92	0.15	0.22	0.005
V ₂ O ₅	0.10	10.00	0.029	0.15	0.005
Cr ₂ O ₃	0.01	18.40	0.027	0.22	0.004
Mn ₂ O ₃	0.01	73.26	0.023	0.13	0.004
Fe ₂ O ₃	0.01	100.00	0.078	0.19	0.004
NiO	0.01	6.00	0.009	0.014	0.003
CuO	0.01	20.00	0.062	0.03	0.002
ZnO	0.01	10.00	0.023	0.061	0.002
SrO	0.01	20.00	0.078		0.002
ZrO ₂	0.01	65.00	0.030	0.14	0.01
BaO	0.01	40.00	0.01	0.048	0.01
HfO ₂	0.03	5.00	0.032		0.006
PbO	0.35	8.00	0.081	0.048	0.004

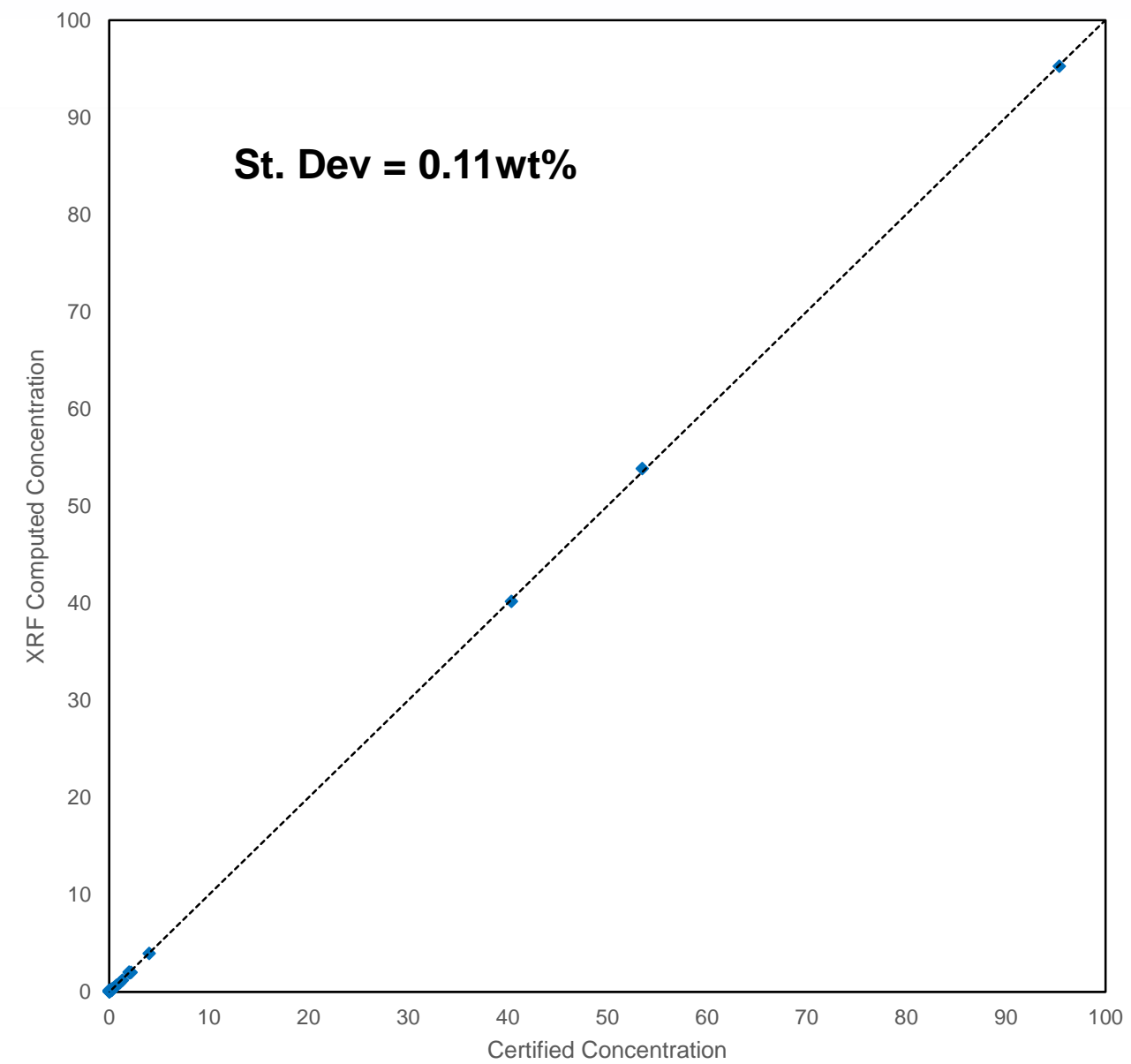
GEO-QUANT Advanced



GEO-QUANT Advanced, Si-KA1



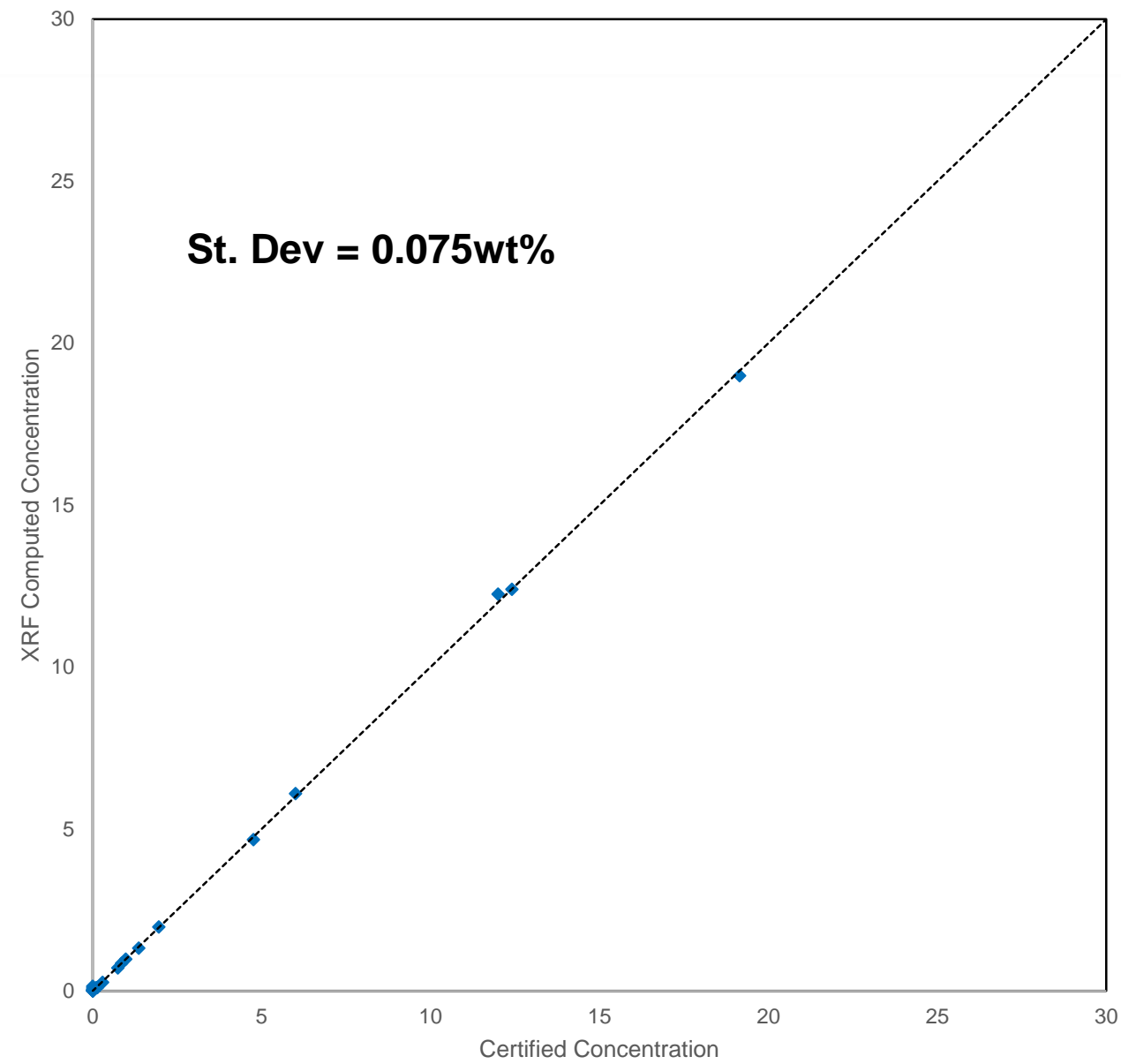
GEO-QUANT Advanced, Mg-KA1



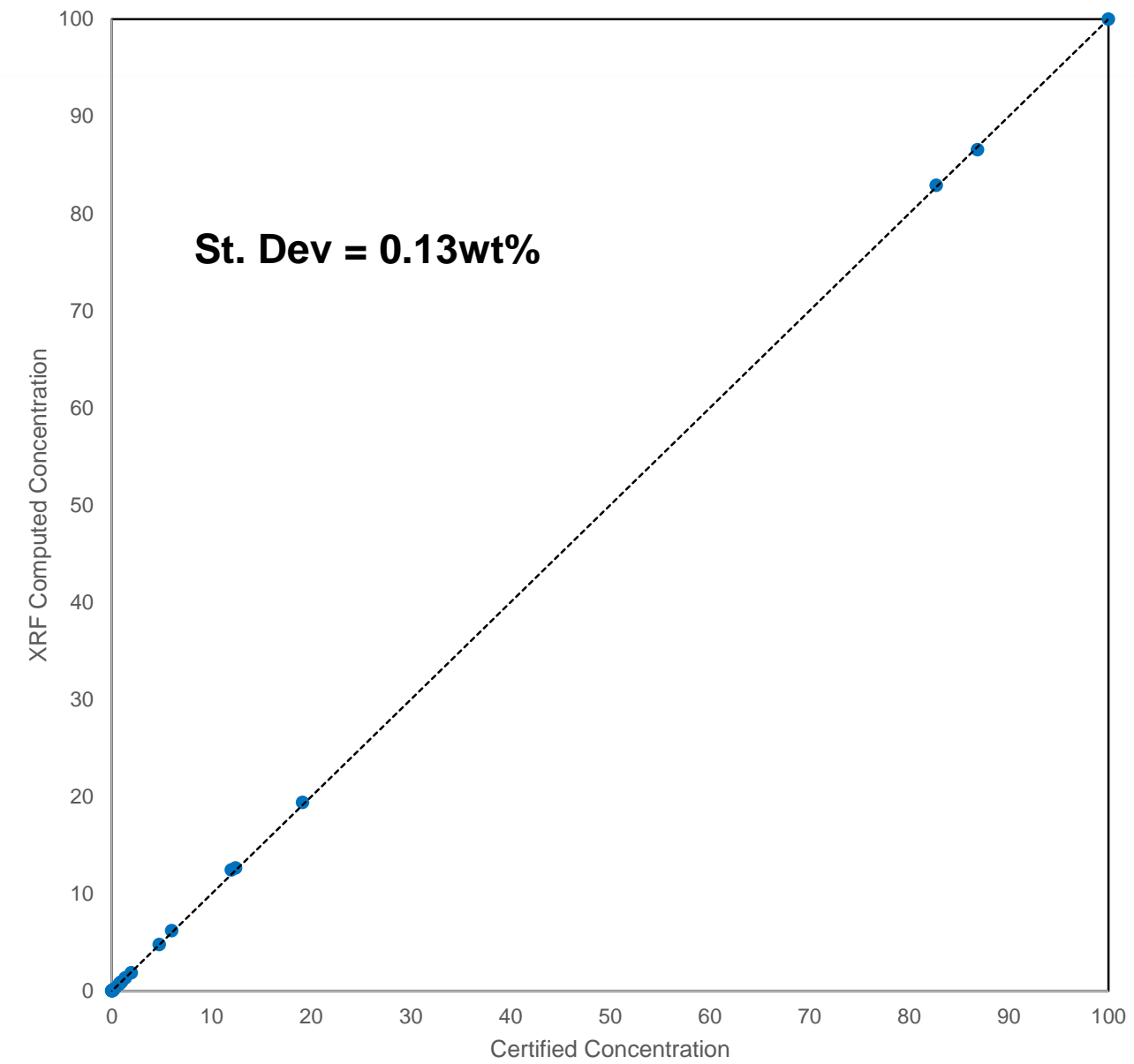
GEO-QUANT Advanced



GEO-QUANT Advanced, Fe-KA1



GEO-QUANT Advanced, Fe-KB1



Top Tip #4



**Don't forget instrumental limitations,
such as linear range and resolution**

- Just because you can make standards from 0 – 100%, doesn't mean you can measure these directly with the same line
- Line switching to alternative lines (KA → KB, or K → L)
 - Take advantage of lower intensities for higher concentrations
 - Avoid spectral overlaps by measuring a line that isn't overlapped

GEO-QUANT Advanced



Slag (BCS-CRM 381)

	GQA	Cert
Na2O (%)	0.100	
MgO (%)	0.941	1.030
Al2O3 (%)	0.829	0.670
SiO2 (%)	8.741	8.780
P2O5 (%)	15.750	15.700
SO3 (%)	0.393	0.474
K2O (%)	0.026	
CaO (%)	49.190	49.000
TiO2 (%)	0.346	0.350
V2O5 (%)	0.950	0.940
Cr2O3 (%)	0.355	0.330
Mn2O3 (%)	3.568	3.516
Fe2O3 (%)	19.140	19.015
NiO (%)	0.001	
CuO (%)	0.000	
ZnO (%)	0.000	
SrO (%)	0.011	
ZrO2 (%)	0.000	
BaO (%)	0.024	
HfO2 (%)	0.470	
PbO (%)	0.000	
Sum (%)	100.835	99.805

Cement (FLX-CRM 101)

	GQA	Cert
Na2O (%)	0.693	0.680
MgO (%)	1.678	1.700
Al2O3 (%)	8.750	8.810
SiO2 (%)	30.190	30.310
P2O5 (%)	0.265	0.191
SO3 (%)	3.029	3.160
K2O (%)	2.112	2.100
CaO (%)	48.380	48.240
TiO2 (%)	0.474	0.469
V2O5 (%)	0.000	
Cr2O3 (%)	0.004	0.010
Mn2O3 (%)	0.120	0.118
Fe2O3 (%)	3.481	3.520
NiO (%)	0.007	
CuO (%)	0.007	
ZnO (%)	0.045	0.044
SrO (%)	0.252	0.248
ZrO2 (%)	0.019	
BaO (%)	0.075	
HfO2 (%)	0.421	
PbO (%)	0.003	
Sum (%)	100.005	99.600

Magnesite (BCS-CRM 319)

	GQA	Cert
Na2O (%)	0.052	
MgO (%)	94.730	95.380
Al2O3 (%)	0.071	0.109
SiO2 (%)	1.040	1.093
P2O5 (%)	0.000	
SO3 (%)	0.000	
K2O (%)	0.009	
CaO (%)	3.133	3.000
TiO2 (%)	0.025	0.007
V2O5 (%)	0.000	
Cr2O3 (%)	0.001	0.004
Mn2O3 (%)	0.130	0.108
Fe2O3 (%)	0.323	0.291
NiO (%)	0.011	
CuO (%)	0.000	
ZnO (%)	0.000	
SrO (%)	0.000	
ZrO2 (%)	0.000	
BaO (%)	0.000	
HfO2 (%)	0.470	
PbO (%)	0.000	
Sum (%)	99.995	99.992

Zircon (SARM 13)

	GQA	Cert
Na2O (%)	0.000	
MgO (%)	0.030	0.044
Al2O3 (%)	0.630	0.611
SiO2 (%)	32.100	32.630
P2O5 (%)	0.339	0.230
SO3 (%)	0.000	
K2O (%)	0.029	
CaO (%)	0.131	0.140
TiO2 (%)	0.420	0.296
V2O5 (%)	0.000	
Cr2O3 (%)	0.630	
Mn2O3 (%)	0.436	
Fe2O3 (%)	0.235	0.187
NiO (%)	0.100	
CuO (%)	0.024	
ZnO (%)	0.002	
SrO (%)	0.000	
ZrO2 (%)	64.000	64.140
BaO (%)	0.005	
HfO2 (%)	1.309	1.290
PbO (%)	0.008	
Sum (%)	100.429	99.568

Limestone (BCS-CRM 513)

	GQA	Cert
Na2O (%)	0.038	
MgO (%)	0.405	0.323
Al2O3 (%)	0.168	0.192
SiO2 (%)	0.448	0.404
P2O5 (%)	0.026	0.008
SO3 (%)	0.031	0.043
K2O (%)	0.013	0.027
CaO (%)	98.740	98.580
TiO2 (%)	0.019	0.008
V2O5 (%)	0.000	
Cr2O3 (%)	0.000	0.002
Mn2O3 (%)	0.008	0.019
Fe2O3 (%)	0.088	0.049
NiO (%)	0.004	
CuO (%)	0.000	
ZnO (%)	0.001	0.003
SrO (%)	0.017	0.031
ZrO2 (%)	0.000	
BaO (%)	0.000	
HfO2 (%)	0.027	
PbO (%)	0.024	
Sum (%)	100.055	99.689


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- Analysis of 16 oxides in slags from the iron and steel making process
- Special sample preparation to avoid the need to work with ignited powders

SLAG-QUANT

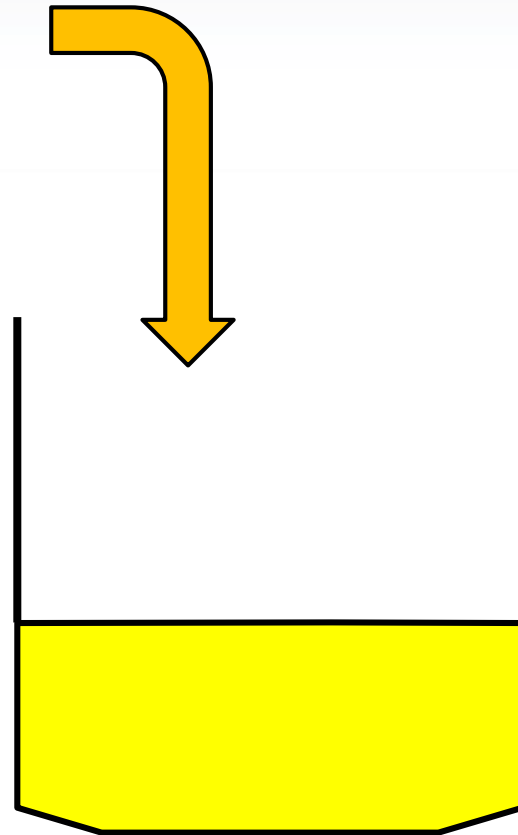


- GEO-QUANT Advanced
 - 0.50g **Ignited** Sample
 - 8.50g 66T:34M Flux
- SLAG-QUANT
 - 0.50g **Dry** Sample
 - 1.00g $\text{Sr}(\text{NO}_3)_2$ 
 - 9.00g 66T:34M Flux

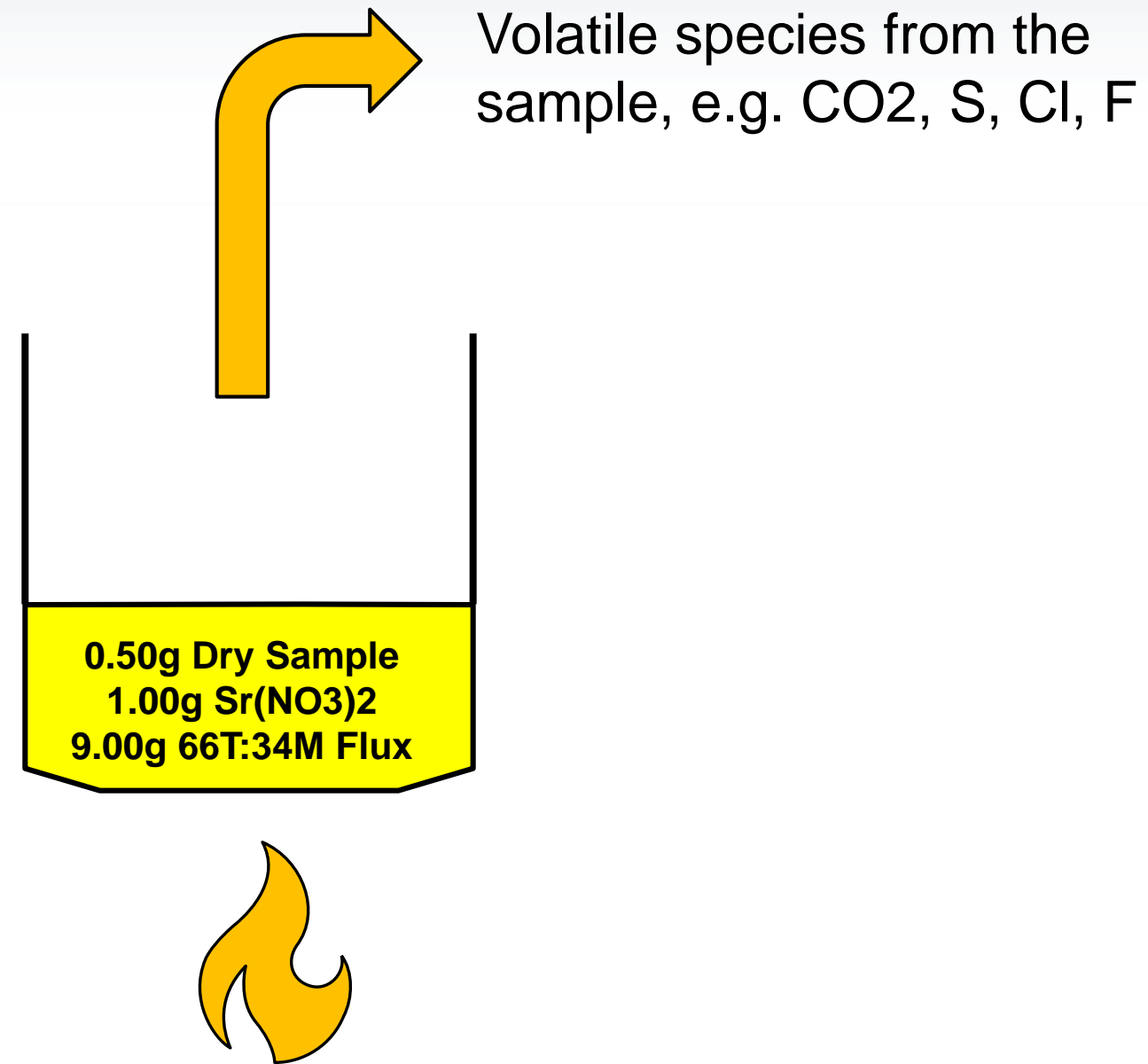
SLAG-QUANT



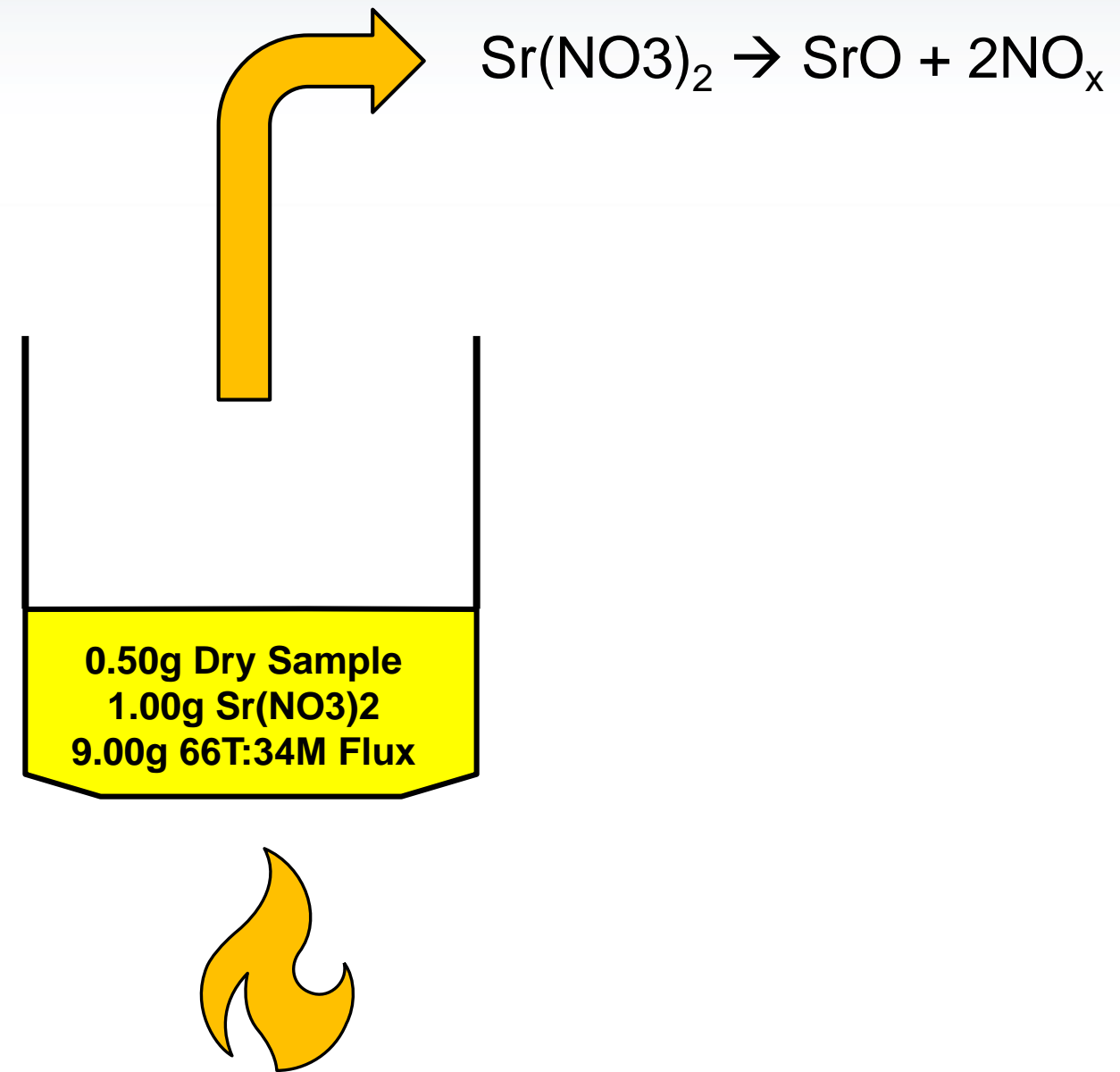
0.50g Dry Sample
1.00g $\text{Sr}(\text{NO}_3)_2$
9.00g 66T:34M Flux



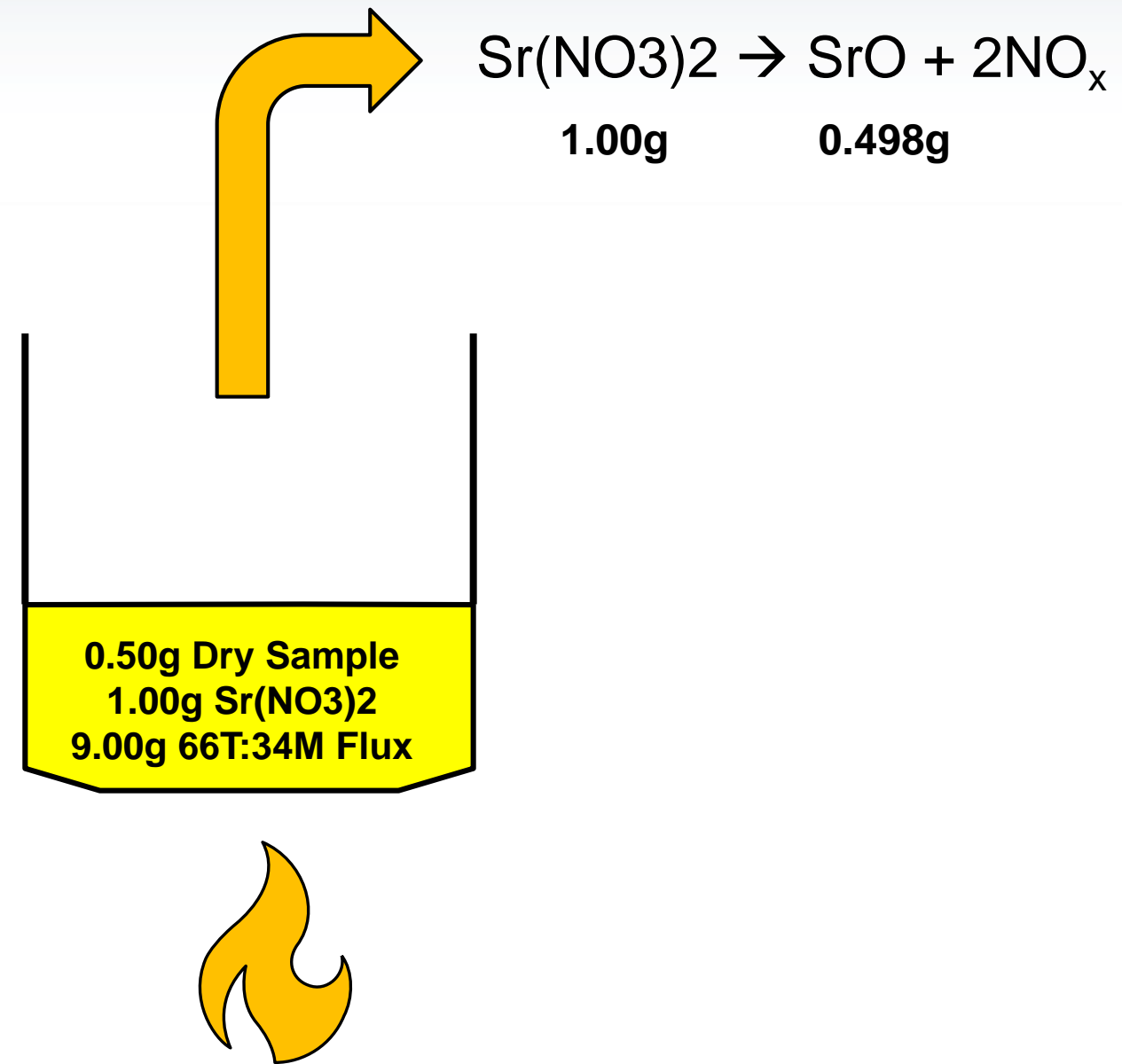
SLAG-QUANT



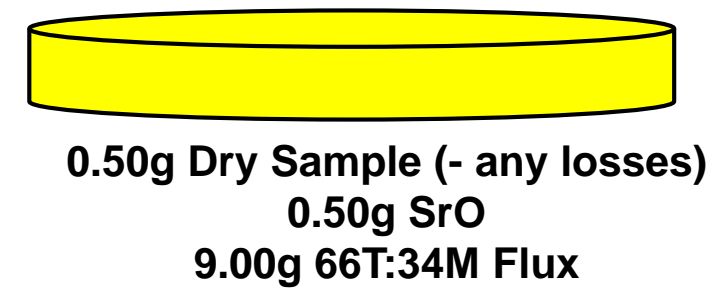
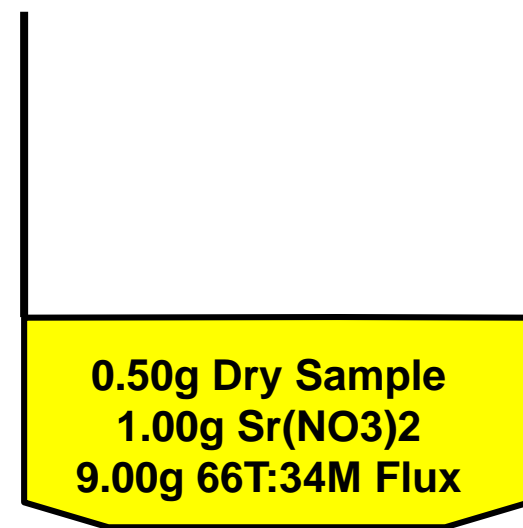
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SLAG-QUANT



SLAG-QUANT



SLAG-QUANT

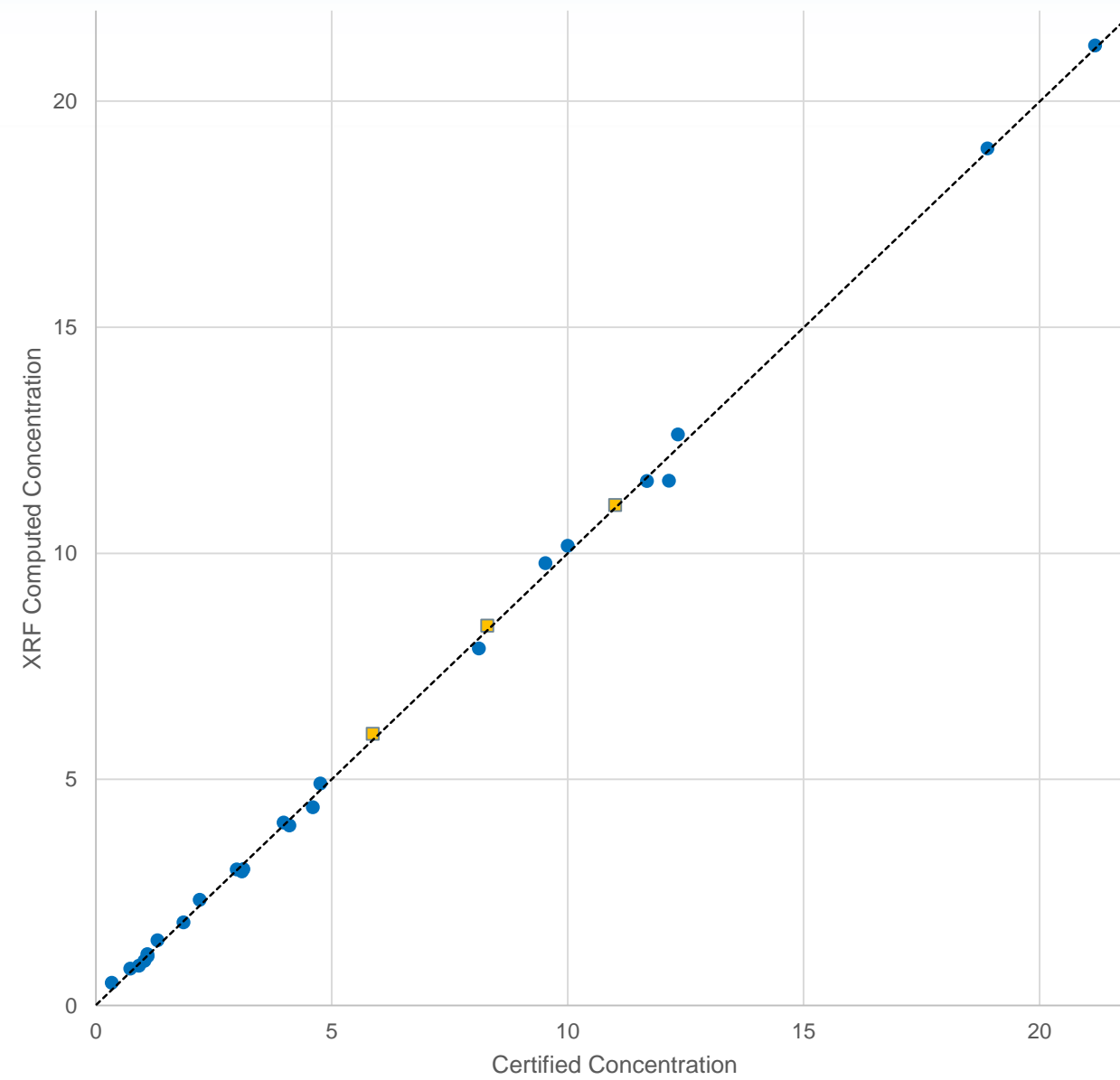


- A series of blast furnace slags from a steel producer were fused and analysed by SLAG-QUANT
- The samples were also analysed for their sulphur content using a G4 ICARUS combustion gas analyser
- These results were used to create a pressed pellet based calibration for rapid production control of blast furnace slag

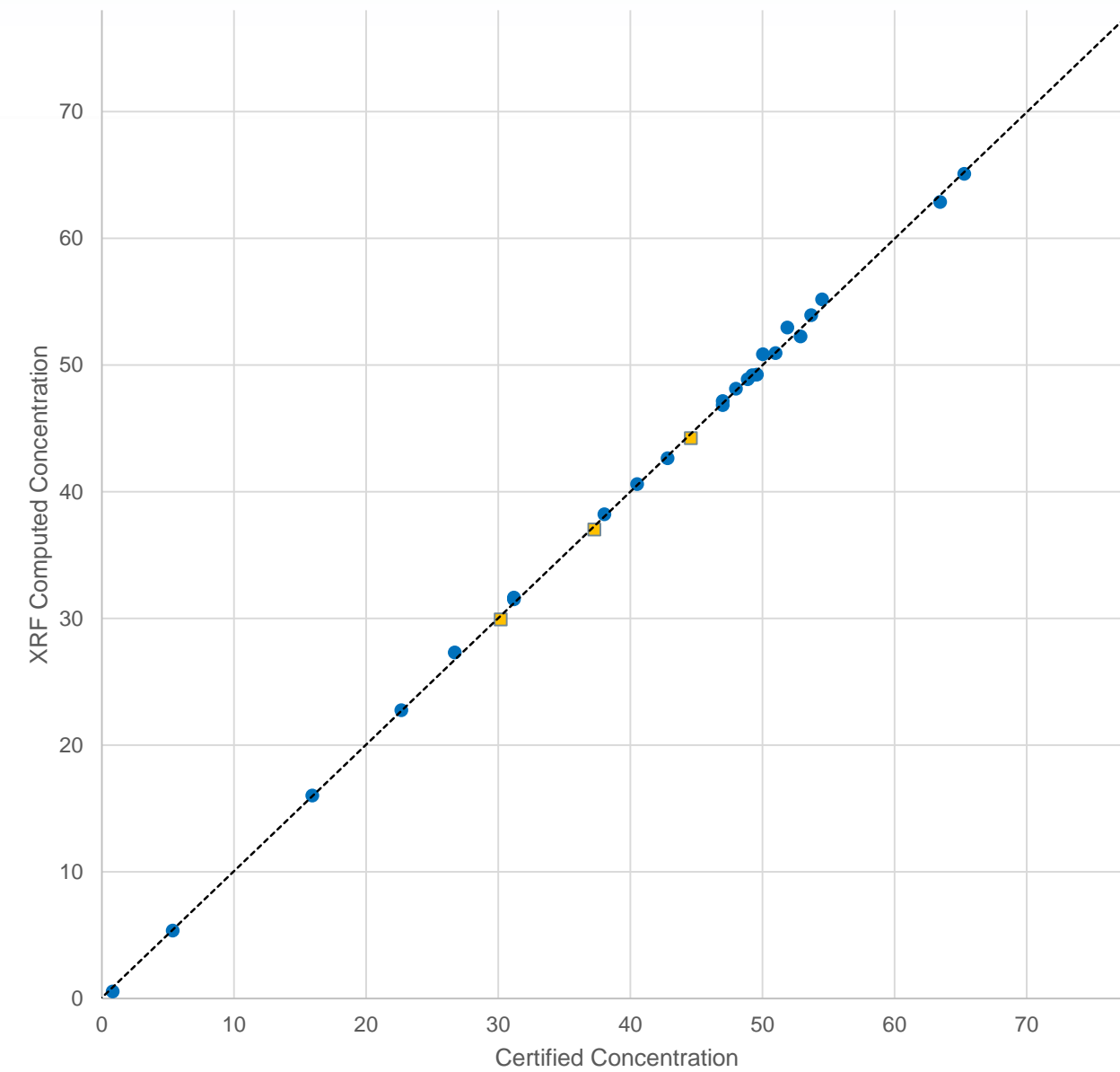
SLAG-QUANT



SLAG-QUANT, Mg-KA1 (St.Dev=0.18wt%)



SLAG-QUANT, Ca-KA1 (St.Dev=0.44wt%)



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Sample Name	Na2O(%)	MgO(%)	Al2O3(%)	SiO2(%)	P2O5(%)	SO3(%)	K2O(%)	CaO(%)	TiO2(%)	V2O5(%)	Cr2O3(%)	Mn2O3(%)	Fe2O3(%)	NiO(%)	CuO(%)	ZnO(%)	PbO(%)
SLAG 01	0.277	6.940	11.441	34.129	0.011	0.896	0.470	39.820	0.614	0.000	0.014	0.492	0.155	0.03109	0.00555	0.01350	0.00209
SLAG 02	0.315	6.525	11.472	33.425	0.011	0.841	0.454	38.885	0.646	0.000	0.015	0.625	0.186	0.03102	0.00579	0.01309	0.00000
SLAG 03	0.315	6.789	11.636	35.830	0.015	0.855	0.556	39.107	0.641	0.000	0.016	0.509	0.580	0.03102	0.00565	0.01343	0.00210
SLAG 04	0.230	7.053	12.017	34.972	0.014	0.926	0.408	39.348	0.598	0.000	0.018	0.462	0.212	0.03130	0.00564	0.01188	0.00000
SLAG 05	0.299	6.892	12.748	34.632	0.006	0.964	0.422	39.764	0.551	0.000	0.017	0.369	0.101	0.03104	0.00567	0.01240	0.00000
SLAG 06	0.237	6.896	11.380	34.143	0.010	0.949	0.447	39.064	0.601	0.000	0.014	0.444	0.138	0.03136	0.00571	0.01300	0.00225
SLAG 07	0.273	6.867	12.363	34.262	0.009	0.926	0.387	39.306	0.584	0.000	0.021	0.433	0.106	0.03112	0.00565	0.01334	0.00208
SLAG 08	0.248	6.830	12.179	33.681	0.005	1.042	0.336	39.217	0.543	0.000	0.019	0.357	0.160	0.03152	0.00561	0.01379	0.00254
SLAG 09	0.237	7.322	11.458	34.177	0.006	0.924	0.383	40.015	0.595	0.000	0.017	0.461	0.118	0.03105	0.00556	0.01335	0.00239
SLAG 10	0.256	7.065	11.324	33.493	0.017	0.899	0.458	39.476	0.619	0.002	0.016	0.578	0.186	0.03131	0.00553	0.01305	0.00497
SLAG 11	0.425	7.062	12.671	35.745	0.040	0.735	0.652	39.763	0.700	0.011	0.018	0.884	0.417	0.03120	0.00579	0.01240	0.00000
SLAG 12	0.298	7.421	11.887	36.052	0.017	0.834	0.501	41.364	0.651	0.000	0.014	0.590	0.264	0.03082	0.00560	0.01193	0.00000
SLAG 13	0.435	6.539	11.689	35.775	0.023	0.721	0.806	39.616	0.706	0.007	0.014	0.710	0.384	0.03091	0.00551	0.01189	0.00000
SLAG 14	0.277	6.873	13.562	34.169	0.014	0.974	0.360	39.222	0.591	0.000	0.019	0.541	0.147	0.03097	0.00572	0.01288	0.00000
SLAG 15	0.350	6.989	12.654	33.884	0.016	0.901	0.405	38.848	0.567	0.000	0.018	0.498	0.137	0.03148	0.00560	0.01260	0.00000
SLAG 16	0.279	6.870	10.945	33.005	0.015	0.869	0.457	40.090	0.599	0.000	0.016	0.519	0.160	0.03117	0.00555	0.01224	0.00000
SLAG 17	0.271	6.941	11.421	33.984	0.012	0.904	0.428	40.231	0.586	0.000	0.014	0.434	0.168	0.03126	0.00562	0.01241	0.00000
SLAG 18	0.289	7.153	11.312	35.050	0.018	0.790	0.488	41.177	0.632	0.000	0.016	0.590	0.128	0.03099	0.00557	0.01261	0.00000
SLAG 19	0.257	6.878	11.101	32.864	0.015	0.918	0.404	40.131	0.564	0.000	0.014	0.438	0.128	0.03145	0.00560	0.01258	0.00000
SLAG 20	0.348	6.856	12.527	36.065	0.013	0.803	0.501	39.960	0.708	0.000	0.019	0.711	0.192	0.03106	0.00553	0.01151	0.00000
MIN	0.230	6.525	10.945	32.864	0.005	0.721	0.336	38.848	0.543	0.000	0.014	0.357	0.101	0.031	0.006	0.012	0.000
MAX	0.435	7.421	13.562	36.065	0.040	1.042	0.806	41.364	0.708	0.011	0.021	0.884	0.580	0.032	0.006	0.014	0.005

SLAG-QUANT



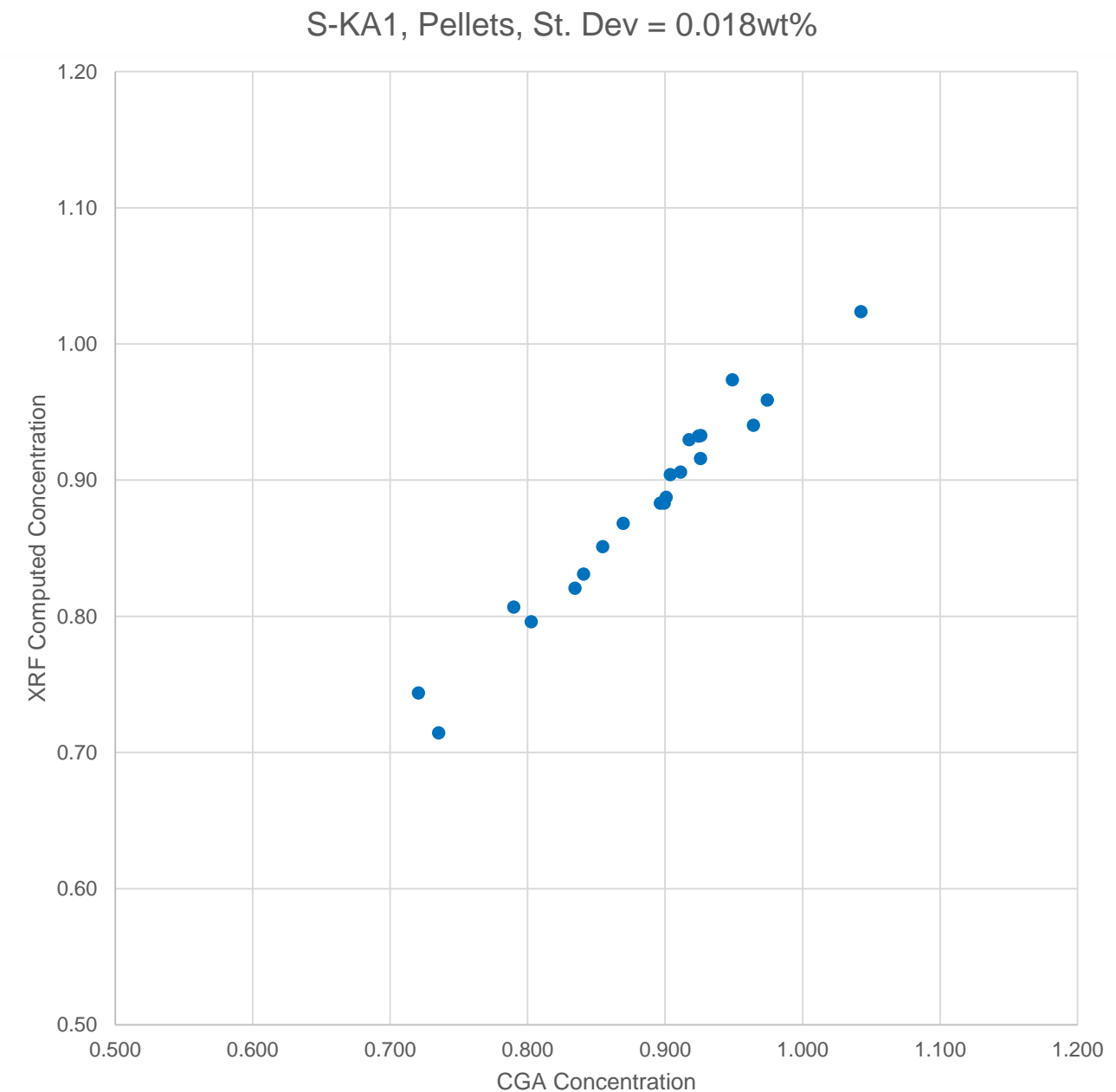
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**Sulphur analysis in fusion beads is challenging → volatile
So what do we do to get good sulphur values?**

SLAG-QUANT



- Sulphur analysis by Combustion Gas Analysis
- G4 ICARUS CS Analyser



Q & A



Please type any questions you may have in the [Q&A tool](#) and click Submit.



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Dr. Colin Slater
R&D, Bruker AXS GmbH
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Thank you for attending!



- We will answer **remaining questions** individually via email
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