



Tracer IIISD/IVSD Calibration Limits

The tables below indicate the calibration limits for the Tracer IIISD and Tracer IVSD. These limits pertain only to the reference standards used, which capture the minimum and maximum concentrations that can be quantified using the factory calibrations included with every Tracer shipment. Software to modify existing calibrations and to develop new ones is included in Tracer support (S1CalProcess). At the end of this document, a brief primer on how calibrations are built can be found.

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Element	Symbol	Minimum	Maximum
Arsenic	As	<1 ppm	69 ppm
Barium	Ba	30 ppm	1.5%
Calcium	Ca	786 ppm	34.66%
Cobalt	Co	1.4 ppm	46.8 ppm
Chrome	Cr	10 ppm	295 ppm
Copper	Cu	5 ppm	429 ppm
Iron	Fe	4267 ppm	6.53%
Manganese	Mn	77 ppm	1239 ppm
Molybedinum	Mo	<1 ppm	166 ppm
Niobium	Nb	2 ppm	16 ppm
Nickel	Ni	14 ppm	302 ppm
Lead	Pb	<1 ppm	28 ppm
Rubidium	Rb	6 ppm	224 ppm
Antimony	Sb	<1 ppm	47.1 ppm
Tin	Sn	<1 ppm	14 ppm
Stronium	Sr	39 ppm	869 ppm
Thorium	Th	2 ppm	14 ppm
Titanium	Ti	479 ppm	5336 ppm
Uranium	U	1 ppm	51 ppm
Yttrium	Y	6 ppm	62 ppm
Zinc	Zn	20 ppm	836 ppm
Zirconium	Zr	17 ppm	338 ppm

Quantification limits for Mudrock Trace calibration (TR2.cfz)

Parameters: 40 keV | 10 - 12 μ A | 1 mil Ti/12 mil Al | Dry Air

Element	Symbol	Minimum	Maximum
Aluminum	Al	9103 ppm	13.07%
Barium	Ba	30 ppm	1.5%
Calcium	Ca	786 ppm	34.66%
Cobalt	Co	1.4 ppm	46.8 ppm
Chrome	Cr	10 ppm	295 ppm
Copper	Cu	5 ppm	429 ppm
Iron	Fe	4267 ppm	6.53%
Potassium	K	1411 ppm	4.3%
Magnesium	Mg	2412 ppm	10.25%
Manganese	Mn	77 ppm	1239 ppm
Sodium	Na	964 ppm	8606 ppm
Nickel	Ni	14 ppm	302 ppm
Phosphorous	P	87 ppm	9819 ppm
Sulfur	S	200 ppm	5.35%
Silica	Si	3.75%	38.2%
Titanium	Ti	479 ppm	5336 ppm
Vanadium	V	22 ppm	1720 ppm
Zinc	Zn	20 ppm	836 ppm

Quantification limits for Mudrock Major calibration in vacuum conditions (MA1.cfz)

Parameters: 15 keV | 25 μ A | N/A | Vacuum <17 Torr

Element	Symbol	Minimum	Maximum
Iron	Fe	3690 ppm	6.85%
Galium	Ga	12 ppm	32 ppm
Manganese	Mn	172 ppm	1775 ppm
Niobium	Nb	2 ppm	640 ppm
Rubidium	Rb	11 ppm	436 ppm
Strontium	Sr	0.2 ppm	291.3 ppm
Thorium	Th	0.8 ppm	82.9 ppm
Yttrium	Y	15 ppm	415 ppm
Zinc	Zn	27 ppm	592 ppm
Zirconium	Zr	60 ppm	3066 ppm

Quantification limits for Obsidian calibration (GL1.cfz)

Parameters: 40 keV | 30-35 μ A | 6 mil Cu/1 mil Ti/12 mil Al | Dry Air

Element	Symbol	Minimum	Maximum
Aluminum	Al	76.28%	99.85%
Cobalt	Co	<1 ppm	4500 ppm
Chrome	Cr	<1 ppm	2600 ppm
Copper	Cu	<1 ppm	6.48%
Iron	Fe	<1 ppm	1.31%
Manganese	Mn	1120 ppm	1.53%
Nickel	Ni	<1 ppm	2.16%
Lead	Pb	<1 ppm	500 ppm
Tin	Sn	<1 ppm	560 ppm
Strontium	Sr	<1 ppm	330 ppm
Titanium	Ti	<1 ppm	2200 ppm
Vanadium	V	<1 ppm	620 ppm
Zinc	Zn	<1 ppm	6.67%
Zirconium	Zr	<1 ppm	1500 ppm

Quantification limits for aluminum alloys (AL1.cfz)

Parameters: 40 keV | 10 - 12 μ A | 1 mil Ti/12 mil Al | Dry Air

Element	Symbol	Minimum	Maximum
Silver	Ag	<1 ppm	1 ppm
Aluminum	Al	76.28%	99.81%
Cobalt	Co	<1 ppm	4500 ppm
Chrome	Cr	<1 ppm	2600 ppm
Copper	Cu	91 ppm	6.48%
Iron	Fe	710 ppm	1.31%
Magnesium	Mg	<1 ppm	8.11%
Manganese	Mn	77 ppm	1.53%
Nickel	Ni	30 ppm	2.16%
Silicon	Si	670 ppm	17.7%
Titanium	Ti	120 ppm	2200 ppm
Vanadium	V	<1 ppm	620 ppm
Zinc	Zn	88 ppm	6.67%
Zirconium	Zr	<1 ppm	1500 ppm

Quantification limits for aluminum alloys in vacuum set 1 (ALV1.cfz)

Parameters: 15 keV | 25 μ A | N/A | Vacuum <17 Torr

Element	Symbol	Minimum	Maximum
Aluminum	Al	86.67%	99.81%
Cobalt	Co	<1 ppm	4500 ppm
Chrome	Cr	<1 ppm	1981 ppm
Copper	Cu	91 ppm	2788 ppm
Iron	Fe	710 ppm	5900 ppm
Magnesium	Mg	33 ppm	8.11%
Manganese	Mn	77 ppm	1.16%
Nickel	Ni	30 ppm	552 ppm
Silicon	Si	670 ppm	12.4%
Titanium	Ti	120 ppm	1000 ppm
Vanadium	V	<1 ppm	230 ppm
Zinc	Zn	88 ppm	1220 ppm

Quantification limits for aluminum alloys in vacuum set 2 (ALV2.cfz)

Parameters: 15 keV | 25 μ A | N/A | Vacuum <17 Torr

Element	Symbol	Minimum	Maximum
Cobalt	Co	12.95%	99.99%
Chrome	Cr	<1 ppm	30.56%
Iron	Fe	15 ppm	30.1%
Manganese	Mn	<1 ppm	1.89%
Molybdenum	Mo	<1 ppm	7.95%
Niobium	Nb	<1 ppm	3.94%
Nickel	NI	12 ppm	58.33%
Tungsten	W	<1 ppm	15.12%

Quantification limits for cobalt alloys (CO1.cfz)

Parameters: 40 keV | 10 - 12 μ A | 1 mil Ti/12 mil Al | Dry Air

Element	Symbol	Minimum	Maximum
Silver	Ag	<1 ppm	3.03%
Arsenic	As	<1 ppm	2500 ppm
Bismuth	Bi	<1 ppm	4.98%
Cobalt	Co	<1 ppm	3290 ppm
Copper	Cu	58.3%	99.9%
Iron	Fe	<1 ppm	4.48%
Manganese	Mn	<1 ppm	51.8%
Niobium	Nb	<1 ppm	1.22%
Nickel	Ni	<1 ppm	29.6%
Lead	Pb	3 ppm	9.5%
Antimony	Sb	<1 ppm	6060 ppm
Tin	Sn	2 ppm	9.75%
Zinc	Zn	<1 ppm	39.8%
Zirconium	Zr	<1 ppm	4041 ppm

Quantification limits for copper alloys first set (CU1.cfz)

Parameters: 40 keV | 10 - 12 μ A | 1 mil Ti/12 mil Al | Dry Air

Element	Symbol	Minimum	Maximum
Silver	Ag	<1 ppm	417 ppm
Arsenic	As	<1 ppm	760 ppm
Bismuth	Bi	<1 ppm	1910 ppm
Copper	Cu	59.99%	95.77%
Iron	Fe	358 ppm	4.48%
Manganese	Mn	17 ppm	5.18%
Nickel	Ni	50 ppm	4.7%
Lead	Pb	86 ppm	9244 ppm
Antimony	Sb	<1 ppm	6060 ppm
Tin	Sn	58 ppm	1.91%
Zinc	Zn	130 ppm	37.49%
Zirconium	Zr	<1 ppm	46 ppm

Quantification limits for copper alloys second set (CU2.cfz)

Parameters: 40 keV | 10 - 12 μ A | 1 mil Ti/12 mil Al | Dry Air

Element	Symbol	Minimum	Maximum
Silver	Ag	<1 ppm	18.2%
Gold	Au	41.69%	91.68%
Copper	Cu	4.91%	40.9%
Indium	In	<1 ppm	1.15%
Nickel	Ni	<1 ppm	14.49%
Zinc	Zn	1600 ppm	11.58%

Quantification limits for gold alloys (AU1.cfz)

Parameters: 40 keV | 10 - 12 μ A | 1 mil Ti/12 mil Al | Dry Air

Element	Symbol	Minimum	Maximum
Cobalt	Co	<1 ppm	18.0%
Chrome	Cr	13 ppm	25.74%
Copper	Cu	<1 ppm	5.56%
Iron	Fe	30.1%	99.94%
Manganese	Mn	10 ppm	19.5%
Molybedinum	Mo	<1 ppm	9.445
Niobium	Nb	20 ppm	6481 ppm
Nickel	Ni	<1 ppm	3.34%
Titanium	Ti	<1 ppm	2.03%
Vanadium	V	<1 ppm	9.5%
Tungsten	W	<1 ppm	18.17%

Quantification limits for iron alloys first set (FE1.cfz)

Parameters: 40 keV | 10 - 12 μ A | 1 mil Ti/12 mil Al | Dry Air

Element	Symbol	Minimum	Maximum
Cobalt	Co	<1 ppm	18.03%
Chrome	Cr	151 ppm	25.74%
Copper	Cu	<1 ppm	5.56%
Iron	Fe	30.1%	97.95%
Manganese	Mn	178 ppm	19.5%
Molybedinum	Mo	100 ppm	6.18%
Niobium	Nb	<1 ppm	6481 ppm
Nickel	Ni	288 ppm	48.2%
Titanium	Ti	10 ppm	2.03%
Vanadium	V	<1 ppm	3875 ppm
Tungsten	W	<1 ppm	3.52%

Quantification limits for iron alloys second set (FE2.cfz)

Parameters: 40 keV | 10 - 12 μ A | 1 mil Ti/12 mil Al | Dry Air

Element	Symbol	Minimum	Maximum
Cobalt	Co	41 ppm	33.46%
Chrome	Cr	80 ppm	29.9%
Copper	Cu	<1 ppm	32.34%
Iron	Fe	<1 ppm	44.9%
Halfnium	Hf	0	1.46%
Manganese	Mn	<1 ppm	1.5%
Molybedinum	Mo	10 ppm	32.44%
Niobium	Nb	<1 ppm	5.18%
Nickel	Ni	32.01%	99.78%
Tantalum	Ta	<1 ppm	3.2%
Titanium	Ti	<1 ppm	5.76%
Vanadium	V	<1 ppm	1.0%
Tungsten	W	<1 ppm	13.45%

Quantification limits for nickel alloys (NI1.cfz)

Parameters: 40 keV | 10 - 12 μ A | 1 mil Ti/12 mil Al | Dry Air

Element	Symbol	Minimum	Maximum
Chrome	Cr	<1 ppm	12.95%
Copper	Cu	<1 ppm	2.95%
Iron	Fe	100 ppm	1.89%
Manganese	Mn	<1 ppm	4.74%
Molybedinum	Mo	<1 ppm	5.98%
Niobium	Nb	<1 ppm	7.17%
Tin	Sn	<1 ppm	3.98%
Titanium	Ti	68.39%	97.21%
Vanadium	V	<1 ppm	14.91%
Zirconium	Zr	<1 ppm	4.03%

Quantification limits for titanium alloys first set (TI1.cfz)

Parameters: 40 keV | 10 - 12 μ A | 1 mil Ti/12 mil Al | Dry Air

Element	Symbol	Minimum	Maximum
Chrome	Cr	<1 ppm	1.97%
Copper	Cu	<1 ppm	3.84%
Iron	Fe	100 ppm	1.89%
Manganese	Mn	<1 ppm	4.74%
Molybedinum	Mo	21 ppm	15.6%
Niobium	Nb	<1 ppm	1 ppm
Palladium	Pd	<1 ppm	1900 ppm
Tin	Sn	<1 ppm	11.09%
Titanium	Ti	78.9%	99.77%
Vanadium	V	<1 ppm	3.89%

Quantification limits for titanium alloys second set (TI2.cfz)

Parameters: 40 keV | 10 - 12 μ A | 1 mil Ti/12 mil Al | Dry Air

Calibration	File Name	Energy	Current	Filter	Atmosphere
Obsidian	GL1.cfz	40 keV	~30 μ A	12 mil Al/1 mil Ti/6 mil Cu (Green/Filter Position 4)	Dry Air
Mudrock Trace	TR2.cfz	40 keV	~11 μ A	12 mil Al/1 mil Ti (Yellow/Filter Position 1)	Dry Air
Mudrock Major	MA1.cfz	15 keV	~25 μ A	None (Filter Position 2)	Vacuum <17 Torr
Aluminum Alloys	AL1.cfz	40 keV	~11 μ A	12 mil Al/1 mil Ti (Yellow/Filter Position 1)	Dry Air
Aluminum Alloys Vacuum	ALV1.cfz	15 keV	~25 μ A	None (Filter Position 2)	Vacuum <17 Torr
Aluminum Alloys Vacuum	ALV2.cfz	15 keV	~25 μ A	None (Filter Position 2)	Vacuum <17 Torr
Cobalt Alloys	CO1.cfz	40 keV	~11 μ A	12 mil Al/1 mil Ti (Yellow/Filter Position 1)	Dry Air
Copper Alloys	CU1.cfz	40 keV	~11 μ A	12 mil Al/1 mil Ti (Yellow/Filter Position 1)	Dry Air
Copper Alloys	CU2.cfz	40 keV	~11 μ A	12 mil Al/1 mil Ti (Yellow/Filter Position 1)	Dry Air
Gold Alloys	AU1.cfz	40 keV	~11 μ A	12 mil Al/1 mil Ti (Yellow/Filter Position 1)	Dry Air
Iron Alloys	FE1.cfz	40 keV	~11 μ A	12 mil Al/1 mil Ti (Yellow/Filter Position 1)	Dry Air
Iron Alloys	FE2.cfz	40 keV	~11 μ A	12 mil Al/1 mil Ti (Yellow/Filter Position 1)	Dry Air
Nickel Alloys	NI1.cfz	40 keV	~11 μ A	12 mil Al/1 mil Ti (Yellow/Filter Position 1)	Dry Air
Titanium Alloys	TI1.cfz	40 keV	~11 μ A	12 mil Al/1 mil Ti (Yellow/Filter Position 1)	Dry Air
Titanium Alloys	TI2.cfz	40 keV	~11 μ A	12 mil Al/1 mil Ti (Yellow/Filter Position 1)	Dry Air

Measurement parameters for calibrations. 1 mil = 0.0254 mm

Ne (gas)*	Na	Mg	Al	Si	P	S	Cl
0.5%	0.5%	0.05%	150 ppm	100 ppm	100 ppm	100 ppm	75 ppm
K	Ca	Sc	Ti	V	Cr	Mn	Co
75 ppm	75 ppm	75 ppm	60 ppm	30 ppm	15 ppm	15 ppm	8 ppm
Ni	Cu	Zn	As	Se	Sr	Mo	Rh
5 ppm	4 ppm	4 ppm	3 ppm	3 ppm	1 ppm	1 ppm	1 ppm
Ag	Cd	Sn	Sb	Ba	Ta	W	Au
1 ppm	1 ppm	1 ppm	1 ppm	60 ppm	1 ppm	3 ppm	3 ppm
Hg	Tl	Pb	Th	U			
3 ppm	1 ppm	3 ppm	3 ppm	3 ppm			

Detection limits in a pure silicate

Ne (gas)*	Na	Mg	Al	Si	P	S	Cl
0.5%	0.5%	0.05%	150 ppm	100 ppm	100 ppm	100 ppm	75 ppm
K	Ca	Sc	Ti	V	Cr	Mn	Co
75 ppm	75 ppm	75 ppm	75 ppm	75 ppm	75 ppm	60 PPM	60 PPM
Ni	Cu	Zn	As	Se	Sr	Mo	Rh
60 PPM	60 PPM	3 ppm	3 ppm	3 ppm	1 ppm	1 ppm	1 ppm
Ag	Cd	Sn	Sb	Ba	Ta	W	Au
1 ppm	1 ppm	1 ppm	1 ppm	75 ppm	1 ppm	3 ppm	3 ppm
Hg	Tl	Pb	Th	U			
3 ppm	1 ppm	3 ppm	3 ppm	3 ppm			

Detection limits in a normal silicate

How Do You Control Measurement Parameters?

The rules of fluorescence are simple. To see an element, you must send in at least 2 keV more of energy to see that element. This is why the voltage, or energy range, of your analysis is critical. If you select 15 keV, you will be optimized for elements lighter than iron, particularly elements like calcium, potassium, titanium, etc. If you select 40 keV, you will see all elements possible from barium and uranium down to aluminum. However, you won't be optimized for any of these elements - this is a jack of all trades, master of none approach. To get to optimal sensitivity for different materials, you must be able to control the five key parameters:

1. Energy: Allows you to select which elements you see in your spectrum
2. Current: Allows you to dim the light when measuring metals and turn it up when looking for ppm-level concentrations in oxides and organics
3. Filter: Allows you to focus on key elemental ranges to identify elements at detection limits
4. Time of Analysis: This must be controlled for always, particularly when you begin to pursue approaches such as Bayesian deconvolution
5. Atmosphere: Dry air contains nitrogen, oxygen, and argon. This interferes with the measurement of light elements like magnesium and sodium. You can either introduce a vacuum or flow helium through the Tracer to see these light elements

How Deep Can You Measure?

Depth of measurement varies based on two properties: the energy of the element and the density of the matrix. As each photon is emitted as one K or L shell fluorescence, the spectral peaks resulting from such fluorescence can be thought of as atom counts of sorts - though the quantity of photons is determined by both the voltage of the tube and the depth of penetration. The depth of penetration can be calculated by the following equation:

$$I/I_0 = e^{-(\mu/\rho)x}$$

where I is the quantity of photons returning from the sample, I_0 is the quantity of photons entering the sample, μ/ρ represents the mass attenuation coefficient of a given element for a particular matrix, and x represents the density of the object. Assuming a limit of 1% returning photons from a silicate matrix, the depths of analysis of key elements are the following:

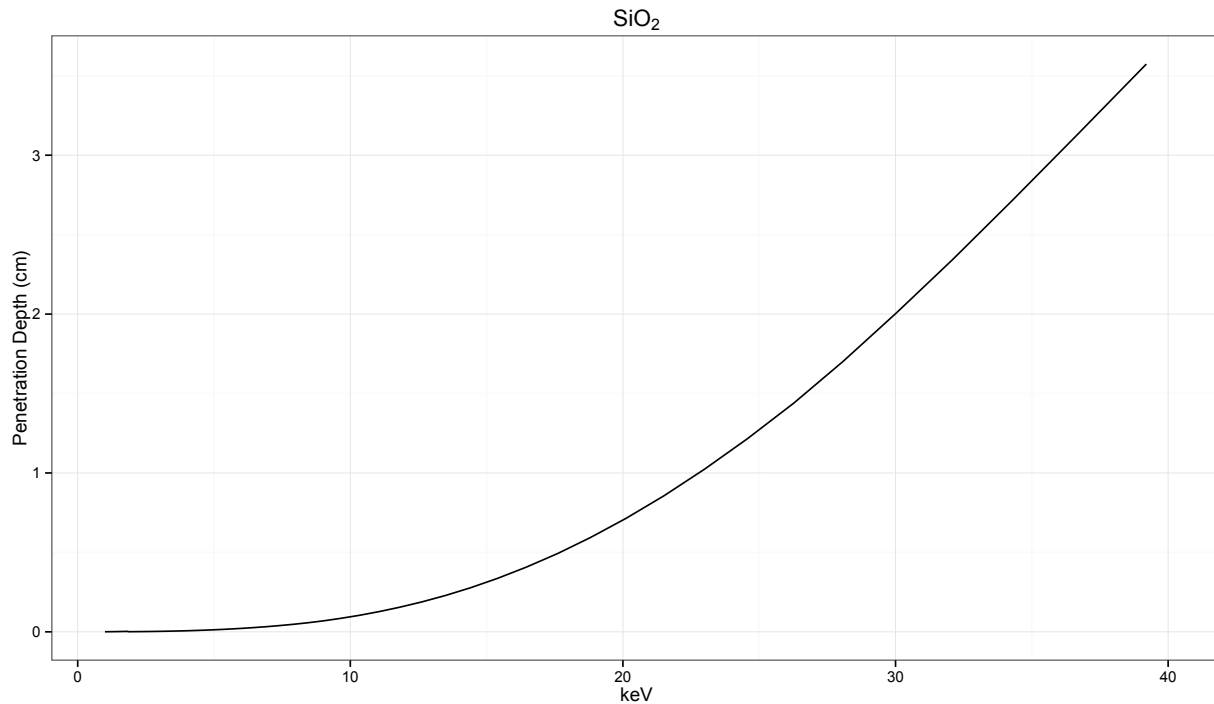


Figure 13: Depth of measurement in a pure silicate

Elements that fluoresce with low energy, such as silica at 1.7 keV, will only return photons from 20 μm deep into a SiO_2 matrix, while elements that fluoresce at higher energies, such as Zirconium at 15.77 keV, will return photons from as deep as 3.4 mm. As a consequence, elements with higher energies of fluorescence will be more easily identified in smaller concentrations. As energy in x ray tubes is non evenly distributed, there is a further discrimination against the fluorescence of elements on the extreme ends of an energy spectrum (near 1 and 40 keV). Furthermore, the fluorescence of one element can influence that of another. A high concentration of Zinc, with a K-alpha fluorescence of 8.78 keV, sits on the absorption edge of Copper, with a K-alpha fluorescence of 8.01 keV. A high concentration of Zinc will distort the quantity of Copper present in the spectra.

However, density is important too. The same physical relationship is present between elements in a denser matrix, however the total depth is considerably shorter:

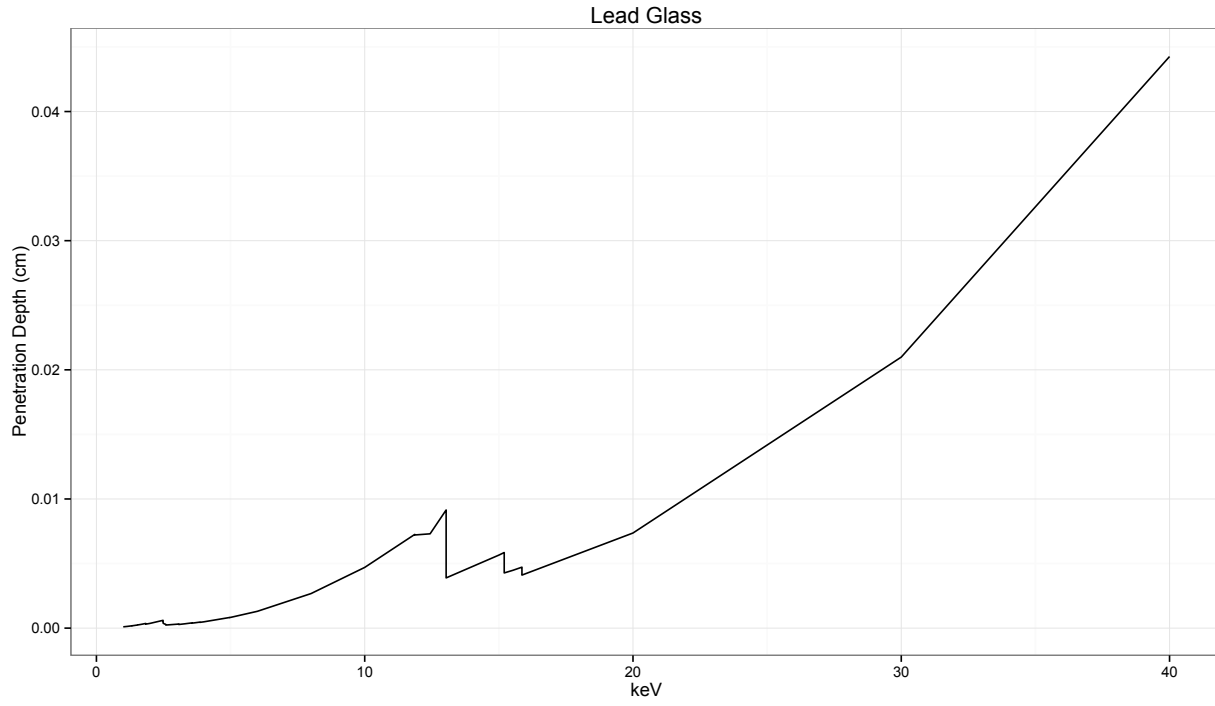


Figure 14: Depth of measurement in a lead glass

How Do Calibrations Work?

The combination of variable depth measurement, uneven voltage distribution, and overlapping elemental peaks precludes simple analysis with X-ray fluorescence data. For these reasons, empirical calibrations are used. Empirical calibrations typically employ a variant of the Lucas-Tooth Empirical Calibration equation:

$$C_i = r_0 + I_i(r_i + \sum r_{in} + I_n)$$

Where C_i represents the concentration of element, r_0 is the intercept/empirical constant for element i , r_i - slope/empirical coefficient for intensity of element i , r_n is the slope/empirical constant for effect of element n on element i , I_i is the net intensity of element i , and I_n is the net intensity of element n . This equation assumes knowledge of the variation of other elements (r_n and I_n in this equation; this is because some elements influence the fluorescence of others. In one critical case for obsidian sourcing, the k-alpha peak for zirconium overlaps with the k-beta peak for strontium. If a quantification procedure does not factor this overlap into its algorithms, then the reported quantity of zirconium will be influenced by the quantity of strontium. A key strength of the Lucas-Tooth algorithm is that it corrects for these effects in producing linear models for the quantification of each element. Empirical calibrations following this algorithm will be accurate within the confines of the regression line (that is to say, minimum and maximum point). Thus, the accuracy of the algorithms is contingent upon the elemental variation captured by the empirical reference set and it's appropriateness to the material being studied.

For an empirical calibration to work, a number of assumptions must be met. First, the data must be homogenous or, at the very least, sufficiently well mixed to be practically homogenous. Second, the reference set must consist of the same material. Third, every element present in the sample must also be present in the reference set. Fourth, the reference set must encapsulate the minimum and maximum of every element. In addition to these four principles, the data must be taken with the same parameters. These include the same energy, current, filter, and atmosphere (dry air, vacuum, etc.).

A calibration is necessary to translate luminescence data (e.g. photon quantities/intensities) to quantitative chemical units. A calibration has the secondary effect of reducing instrument-to instrument variation. Every instrument, even those which contain the same components, have slight variations in photon excitation and detection due to manufacturing variation in bulbs and very slight variance in internal geometries (Figure X). These differences manifest themselves in the spectra.

At this point, it is worthwhile pointing out what elements can and can't be identified using XRF as a technique, these are determined by the energy range of photons sent out (in keV) and the resolution of the detector (in eV). Systems with a pin-diode detector (with a XXXeV resolution) can see K-alpha peaks for elements ranging from magnesium to barium and L-alpha peaks from barium to uranium when a range of 40 keV photons are detected in the sample. Systems with a silicon drift detector can include both sodium and neon as those detectors have a resolution of 144 eV at Mn K α 1. There is, however, no ED-XRF system that is able to detect oxygen directly via a K-alpha fluorescence peak. As such, only elements from sodium to uranium are currently quantifiable using XRF. This is important, as the results of XRF analysis are commonly reported in oxide form. While for a piece of obsidian it is undoubtedly likely all elements detected via XRF are bound to oxygen, this is not true for all sedimentary materials, such as rocks or sediments. Many elements have multiple oxidization states. For example, FeO Fe₃O₄, Fe₂O₃ can be present in a sample. Or, hydroxide could be a possibility, Fe(OH)₂. And it isn't the case that one state is predominant enough to justify the assumption that it exists exclusively in a material. FeO forms about 3.8% of oxides in continental composition, while Fe₂O₃ forms 2.5%. An XRF system that interprets an Fe K-alpha peak only as Fe₂O₃ regardless of the sample is potentially inaccurate. XRF cannot determine the molecular structure of a substance, it can only identify elements and their quantities. It is, however, possible to infer molecular structure using XRF data using correlations between K-alpha and L-alpha peaks. For example, a correlation between Ca and S could help identify calcium sulfate (CaSO₄). However, it is important to note that a correlation between elements could also be indicative of a separate chemical process that selected for elements with certain properties.

In some cases, an alternative calibration approach, known as 'Fundamental Parameters' can be employed. This approach typically relies upon iteration of data to converge upon a

chemical concentration consistent with the spectra. In the case of modern metal alloys or other industrial materials this approach can be helpful because of the limited range of options and relatively predictable chemical composition. For archaeological materials that can be much more chemically diverse and variable, this approach is not appropriate. In particular, as the answer to an archaeological material can depend upon a difference of a few parts per million of a trace element (as is the case for obsidian sourcing) empirical calibration is necessary as it can be evaluated externally to determine if the method is appropriate.

While empirical calibrations can provide highly accurate results, a calibration itself adds no new information that was not already present in the X-ray fluorescence spectra - the calibration only translates the variation present in the x-ray spectra into chemical weight percents. As such, semi-quantitative analysis should be possible. One frequently used route is Bayesian Deconvolution, in which known inter-elemental effects are used in multiple resimulations of the data to produce a net photon count for each element. Other, more simple methods can be used, such as normalizing gross photon counts to either the Compton curve or valid count rate.