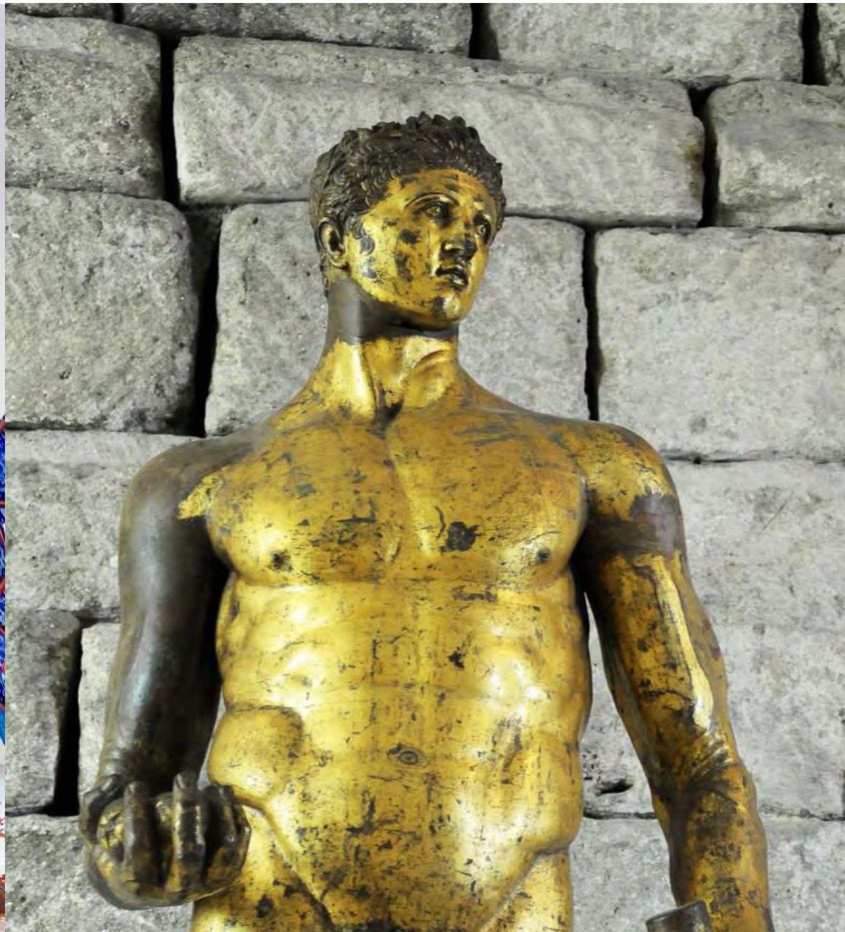
Art & Conservation Series – Part III

TRACER

The benchmark in handheld-XRF for Cultural Heritage





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Applications Specialist
Handheld-Mobile-Portable Products
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Senior Market Application Scientist
Art, Conservation & Archaeology
Bruker Nano Analytics

Supporting Art & Conservation

XRF and Art – a Hand-in-Hand Partnership

- XRF has proven to be a **core analytical technique** in Cultural Heritage studies
- XRF provides key information on objects: **reliable**, **fast**, and **non-invasive**
- **But** the needs are not always the same. They differ in crucial ways with respect to the **what**, the **where**, and the **how.**
- Bruker offers several instruments for one analytical principle

Supporting Art & Conservation
XRF and Art – a Hand-in-Hand Partnership

Bruker's range of instrumentation can address any need

M6 JETSTREAM

M4 TORNADO

CRONO

ELIO

TRACER

Mapping

Spot

Portable

Laboratory based

TRACER
The benchmark in handheld-XRF for Cultural Heritage

Tracer 5 series of handheld-XRF instruments is built on the success of earlier models
- *Tracer III & Tracer IV*



TRACER

The benchmark in handheld-XRF for Cultural Heritage

BRUKER

- The go-to instrument for Art, Conservation and Archaeology applications

  Flexibility of use, in a gallery, laboratory, or in the field

  Customizable options for data measurement, data reduction, and data interpretation

Tracer 5i

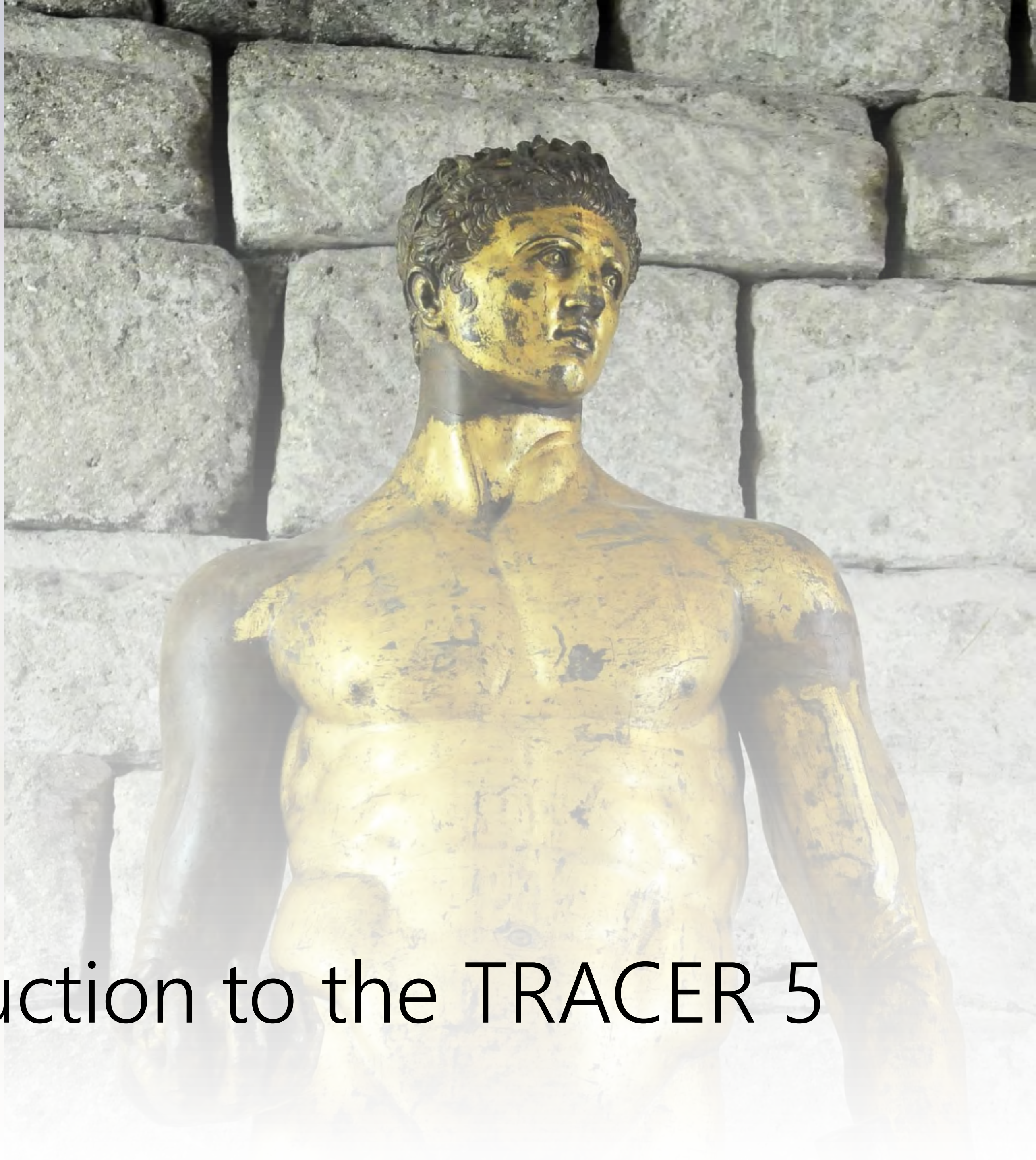
Tracer 5g

Webinar Outline

- TRACER features
- Quantification using calibrations
- A review of Artax – software for handheld-XRF
- TRACER in Cultural Heritage Studies – some examples

  Obsidian Sourcing
  Prof. Robert Tykot
  Dept. of Anthropology, University of South Florida

  French Gilt Bronzes
  Dr. Arlen Heginbotham
  Conservator of Decorative Arts and Sculpture, J. Paul Getty Museum



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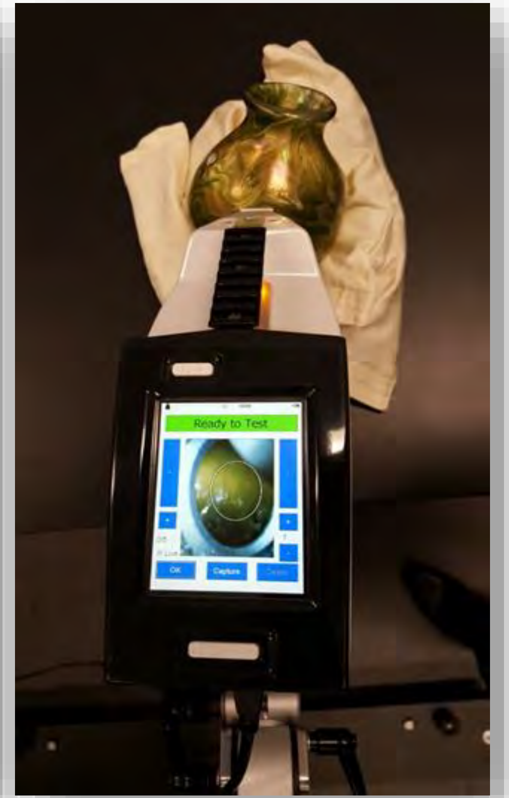
An Introduction to the TRACER 5

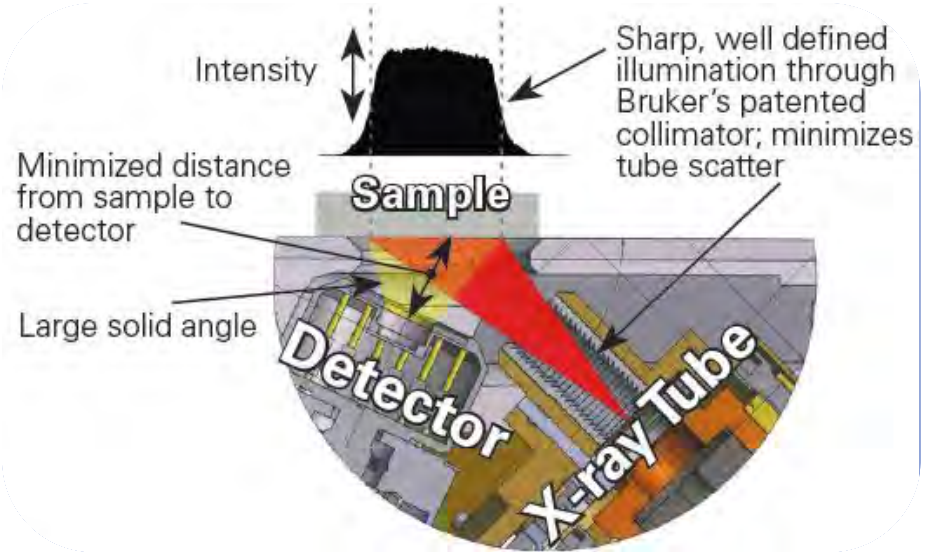
Tracer 5 Family
Instrument Features

- 50 kV 4W Rh X-ray tube
- Large-area SDD detector
- 5i models use Be windows, 5g use graphene
- Tight geometry
- 5-filter wheel **AND** manual filters
- 3 and 8 mm spot options
- VGA camera w/ LED for sample positioning
- Capable of air, vacuum, or Helium atmospheres



Tracer 5 Family – distinctive features
X-ray Geometry

- Compact source + large-area SDD
- Short X-ray path, large solid angle
- SharpBeam™ geometry

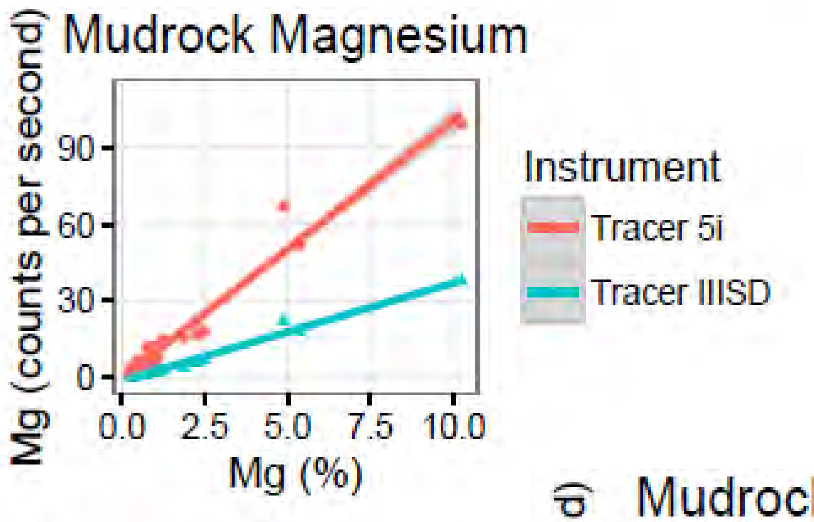


Designed to achieve maximum counts
- Translates to detection of Si and lighter elements

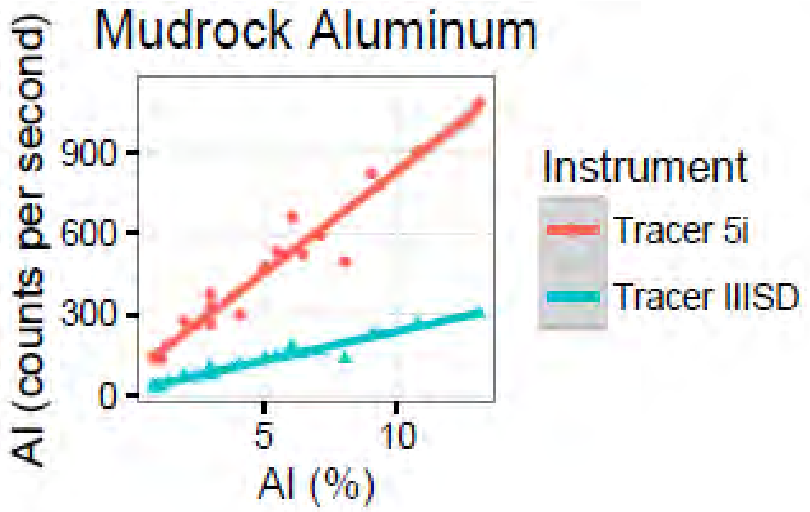
Tracer 5 Family – distinctive features

X-ray Geometry

- Impact of X-ray geometry:
  - Higher count-rate = less measurement time, and/or better precision
  - Improved sensitivity for light elements
  - Tracer 5i: Nominally 3x count rate compared to Tracer III-SD
  - Tracer 5g: Nominally 3x count rate at sodium (Na) compared to Tracer 5i
  - Tracer 5g: capable of measuring fluorine (F)



TRACER 5i - Air
TRACER III-SD - Vacuum



Tracer 5 Family – distinctive features

Tracer 5g performance

- Incorporates new Graphene window detector
- Dramatically improves the low energy sensitivity of the instrument
- Allows detection down to Fluorine, and 3x the sensitivity for Na compared to Tracer 5i
- Why does this matter? Real significance for Art & Conservation
  - Improved detection of Na, Al, Si
  - *Ultramarine (Na-Al-Si-S)*

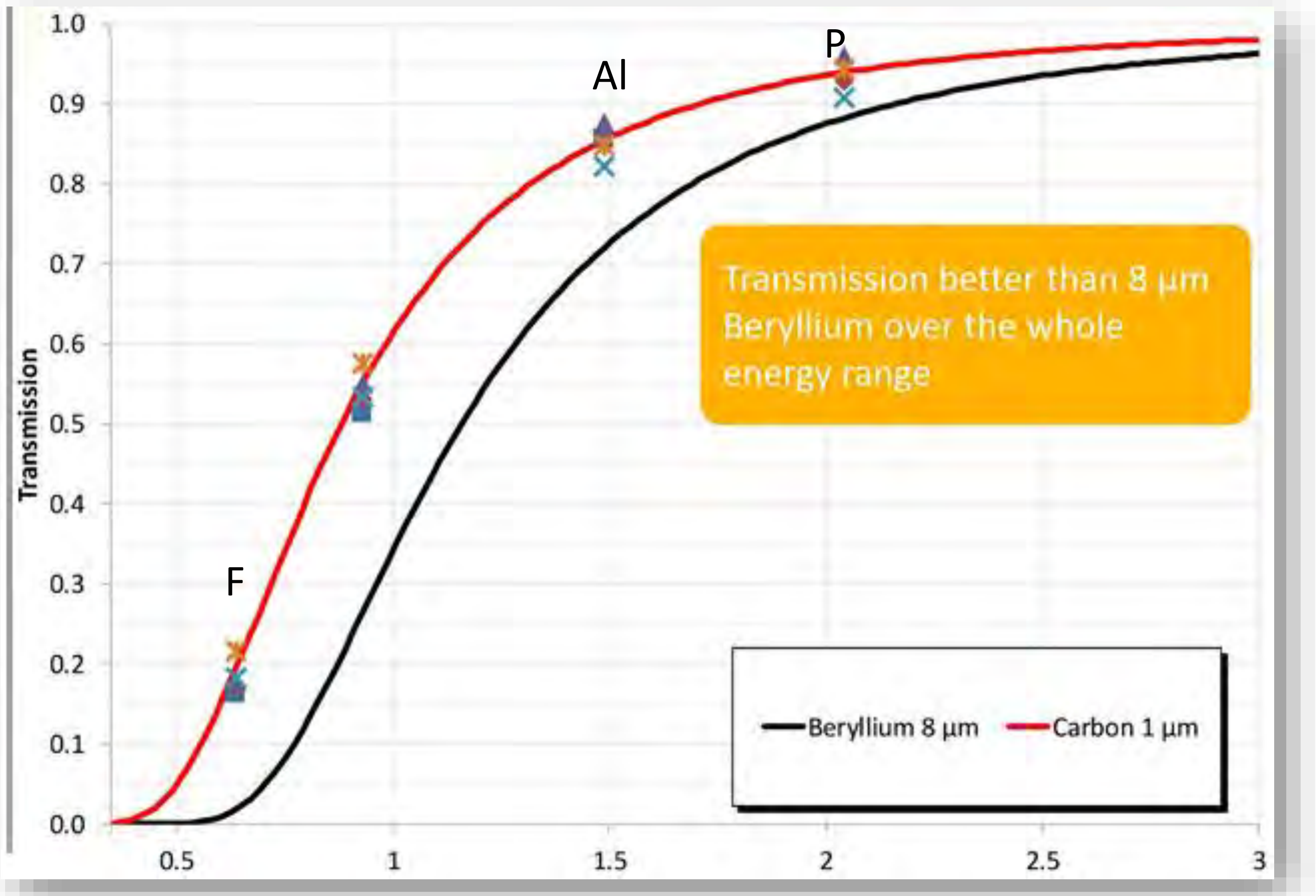


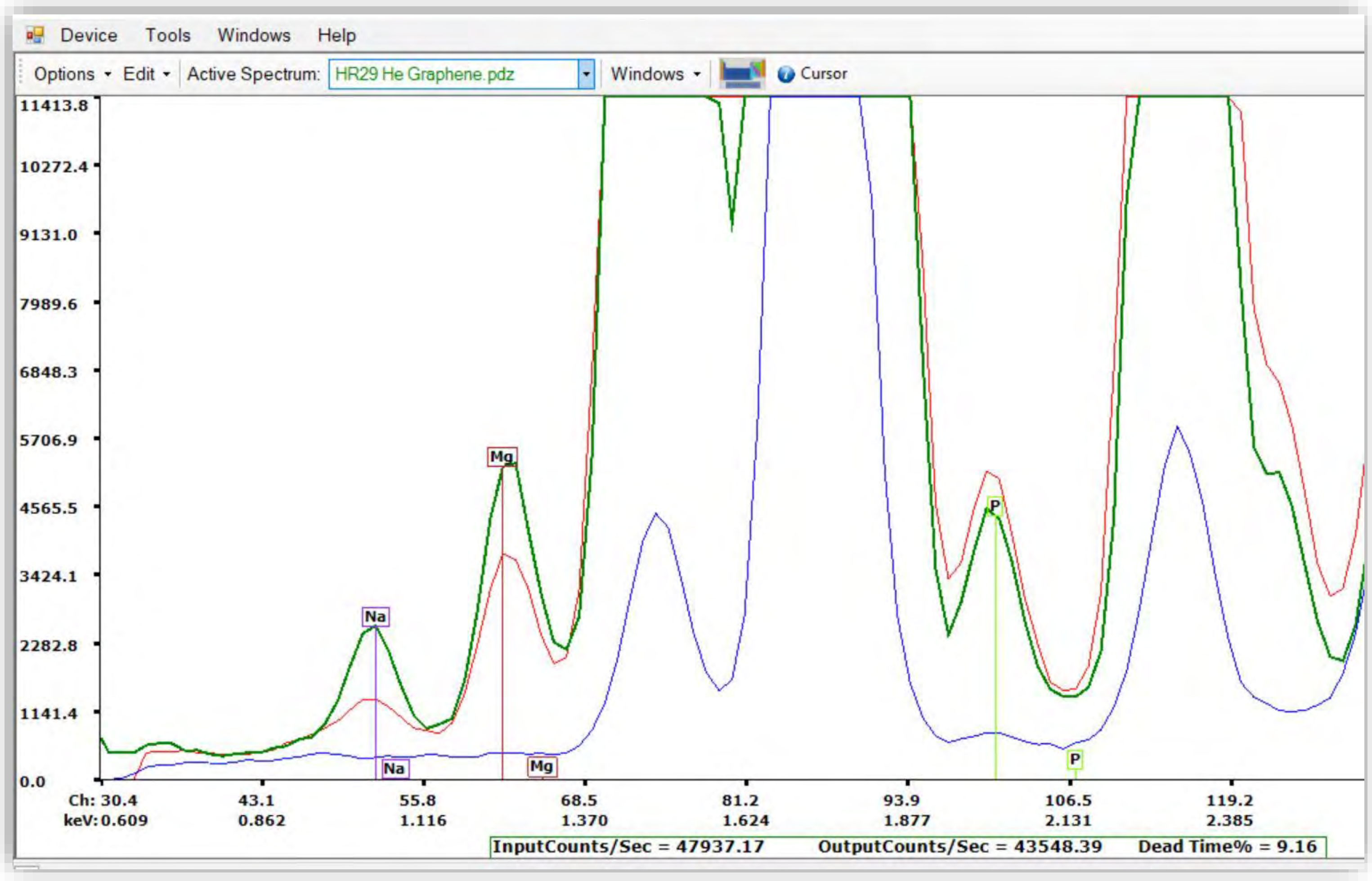
Photo Credit: Key Tech Inc.

Tracer 5 Family – distinctive features
Tracer 5g performance

- Impact of graphene window (Tracer 5g):
  - ➢ 3x sensitivity for Na
  - ➢ 2x sensitivity for Mg
  - ➢ No window, He flush (60s) gives Levels of Detection of:

    Na < 300ppm

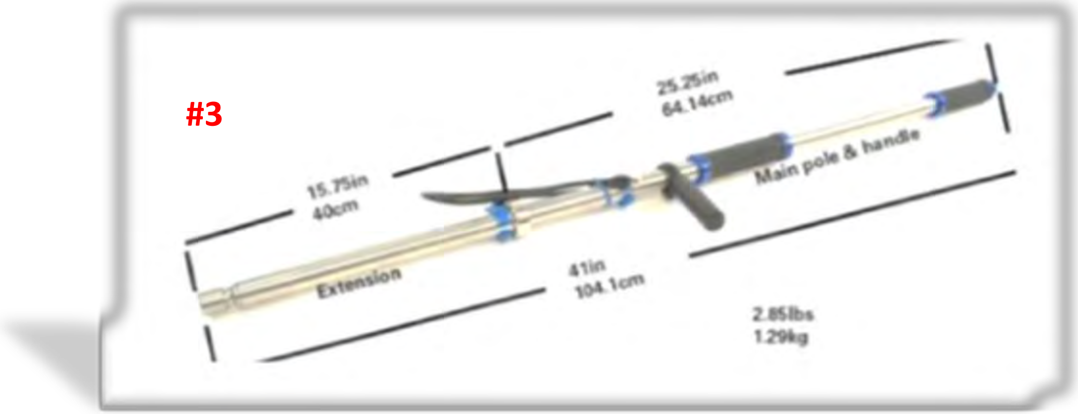
    Mg < 100ppm



Overlay of Tracer III-SD, Tracer 5i, Tracer 5g

Tracer 5 Family – distinctive features
Universal EasyAccess^{TM} Rail

- Full versatility in the field or laboratory:
  1) Desktop stand
  2) Tripod stand
  3) Extension pole



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Tracer 5 Family – distinctive features

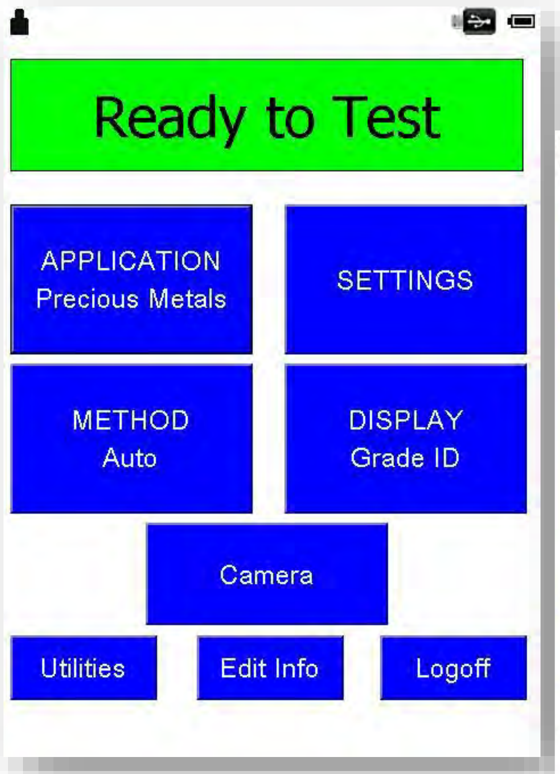
Optional accessories

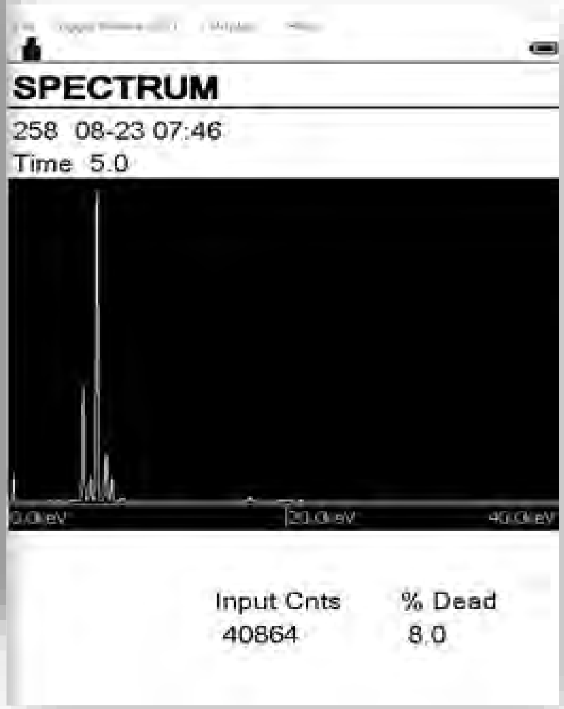
- Enhance Measurements and Collection
  - Portable vacuum (pictured)
  - Specialized filters (pictured)
  - Helium flush kit
  - Bluetooth & Wi-Fi enabled, remote Data Streaming

Full Suite of Software Tools

BRUKER REMOTE

- Allows duplication of instrument screen to laptop for full control of Tracer
- Camera and picture control
- Live spectrum display, with count rate and deadtime

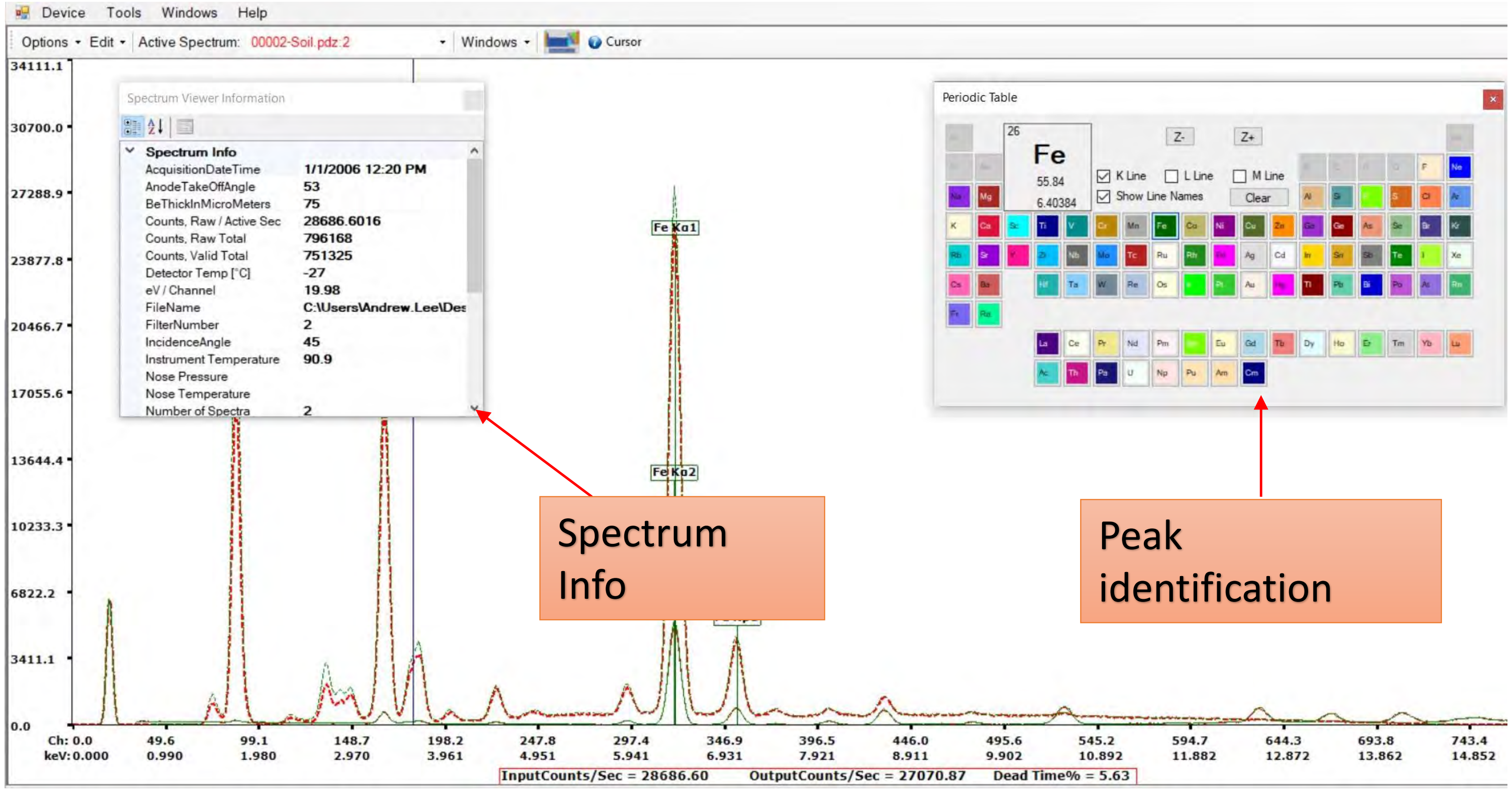




Full Suite of Software Tools

BRUKER INSTRUMENT TOOLS (BIT)

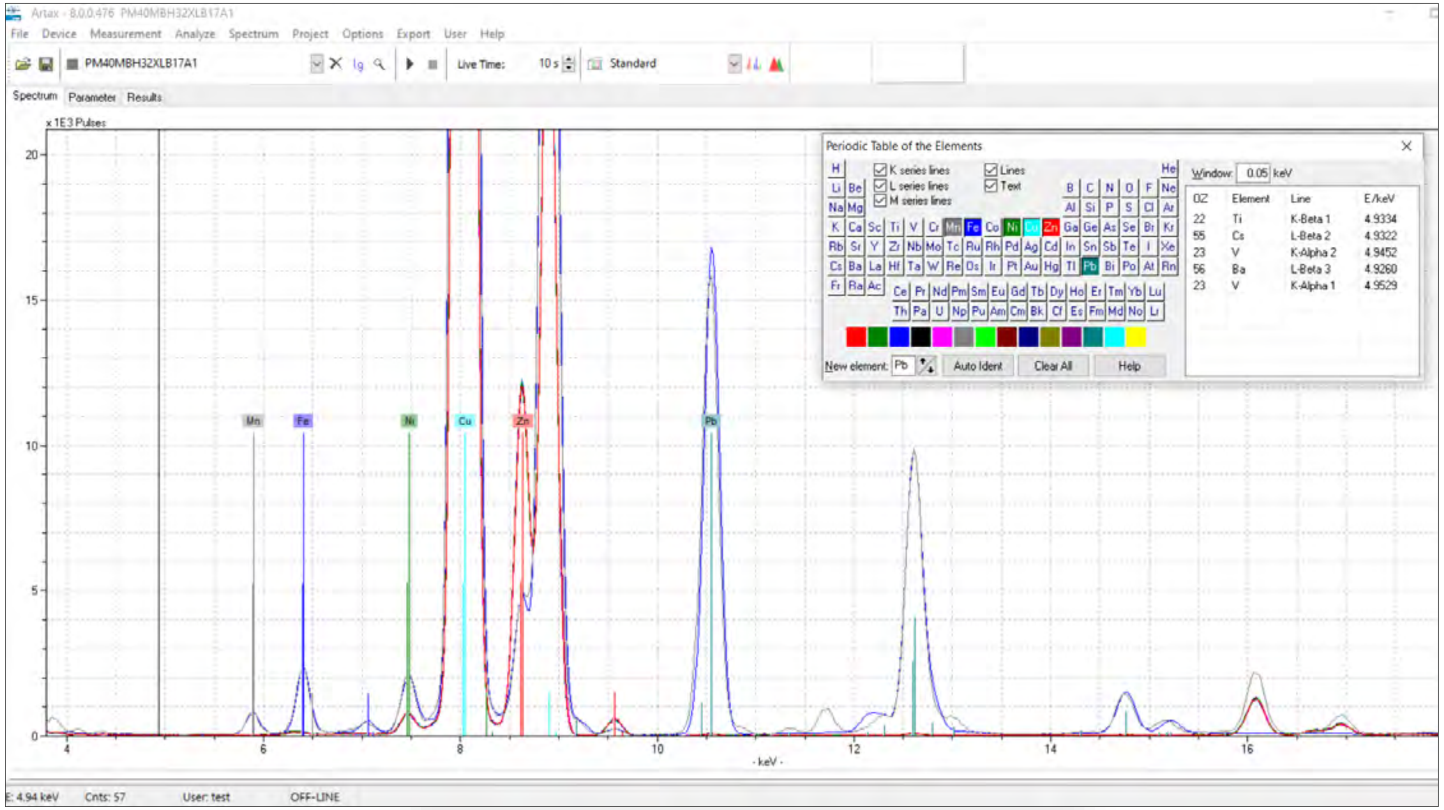
- Full spectrum viewer w/ spectrum info, peak ID
- Data management:
  - Report generator
  - Control library and limits
  - Instrument explorer
  - Calibration and SW installation



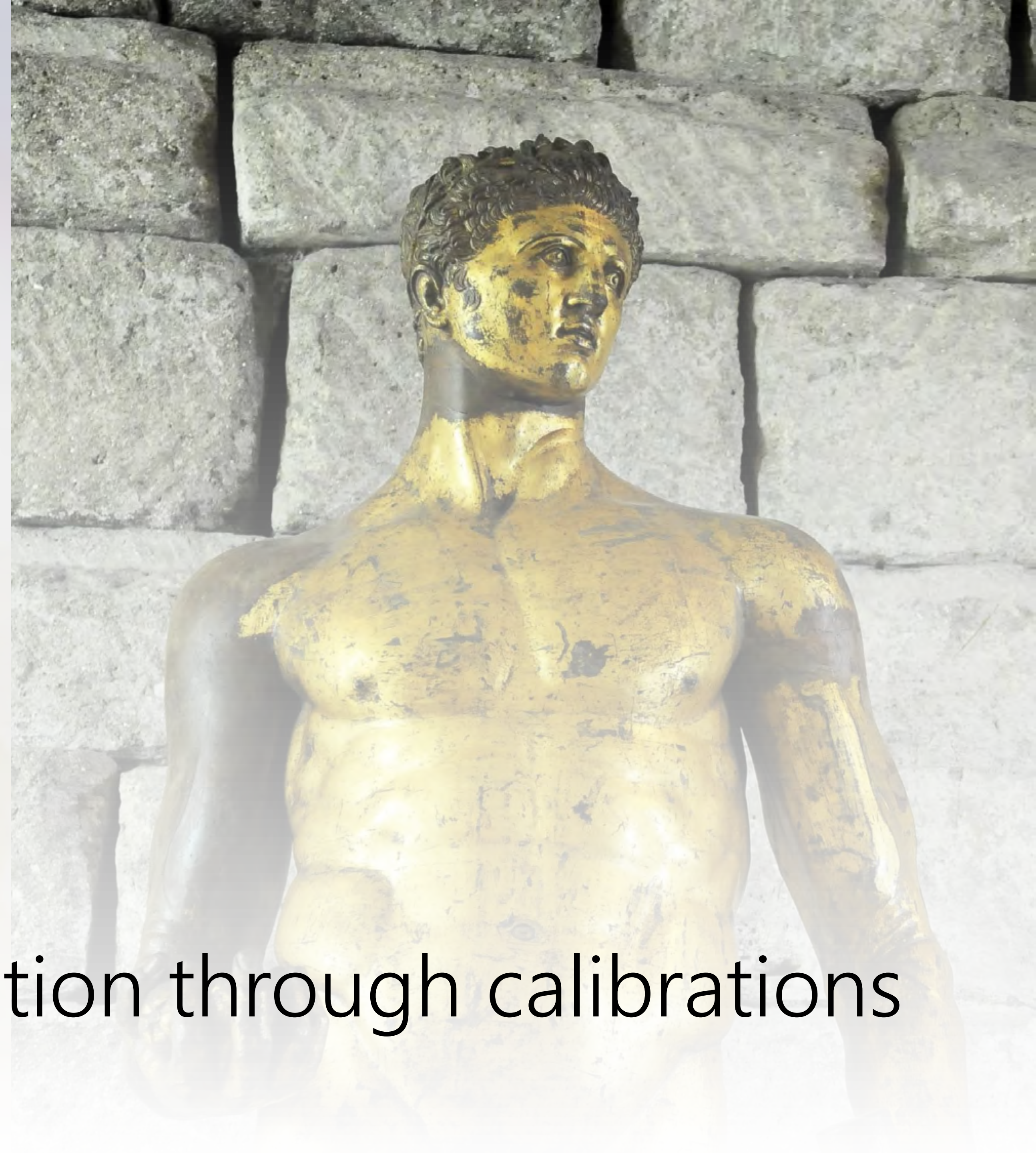
Full Suite of Software Tools

ARTAX Software

- Measurements can be run from Artax
- Interrogating data:
  - Spectrum display, peak ID, overlays, deconvolution, export intensities
  - Project database, can process large number of spectra
  - Outputs data to Excel for further processing
  - Spectral matching/fingerprinting)



Element	Line	Energy/ke	Cycl.	Net	Backgr.
Ca	K12	3.692	0	64025	13797
Fe	K12	6.405	0	19033	33837
Ni	K12	7.48	0	3237	29559
Zn	K12	8.637	0	7918	17500
Y	K12	14.958	0	831	69
Y	L1	1.924	0	3043431	64787



Quantification through calibrations

Calibrations

- Bruker handheld instruments use empirical calibrations to quantify elemental (or oxide) weight fractions from spectra
- Calibrations are matrix-specific and instrument-specific
- Sample preparation, homogeneity, and placement are critical factors
- Bruker uses different types of calibrations to best fit the samples and goals, which will be explored in further detail:
    - Standard “factory” cal
    - Pure custom cal
    - Type standardized cal

Calibrations

Bruker Factory Calibrations

- Developed and installed by Bruker
  - ➢ Installation occurs at the factory
- Each instrument must measure the reference materials
  - ➢ *Instrument-specific*
- Uses a comprehensive range of standards
  - ➢ extensively researched
  - ➢ acquired by Bruker applications specialists
- Acquisition parameters are fixed, except for runtimes
- Calibration sheets with performance expectations available

Calibrations

Bruker Factory Calibrations

- Example of abbreviated calibration sheet for Tracer 5 Obsidian cal, showing analyzed elements, calibrated ranges, nominal detection limits

Obsidian calibration: This calibration is intended for elemental analysis of solid silica based volcanic glass samples

Obsidian	Model	Spot	Si	Mn	Fe	Zn	Ga	Rb	Sr	Y	Zr	Nb	Th
Calibration range [ppm]	Tracer 5i/5g	8 mm	93% - 99.5%	170 - 1800	0.37%-6.85%	27 - 600	10 - 30	10 - 440	0 - 290	15 - 420	60 - 3000	0 - 640	0 - 83
LOD in pure SiO2 [ppm]	Tracer 5i		NA	18	11	<5	<5	<5	<5	<5	<5	<5	<5

Parameter	Value
Excitation voltage [kV]	40
Filter	Ti25Al300
Measurement time [sec]	60
Atmosphere	Air
Spot size [mm]	8

- When applying a calibration need to consider:
  - Correct matrix
  - Expected element concentration within the calibrated range
  - Sample preparation is consistent with methods used for the calibration

Calibrations

Bruker Factory Calibrations

- Factory calibrations used for Art, Conservation, and Archaeology:
  - **Ancient Bronze** — Cu alloys calibrated with the CHARM set
  - **Alloys** — modern alloys
  - **Precious Metals** — Au and Ag-rich matrices
  - **Obsidian** — calibrated with MURR standards
  - **Glass** — SiO_2 and PbO glass, reports as oxides
  - **GeoExploration** — Silicate-dominated rock samples, packed powders
  - **Mudrock** — Quartz & clay-rich (Si,Ca) matrix, rock/shale


CHARM set

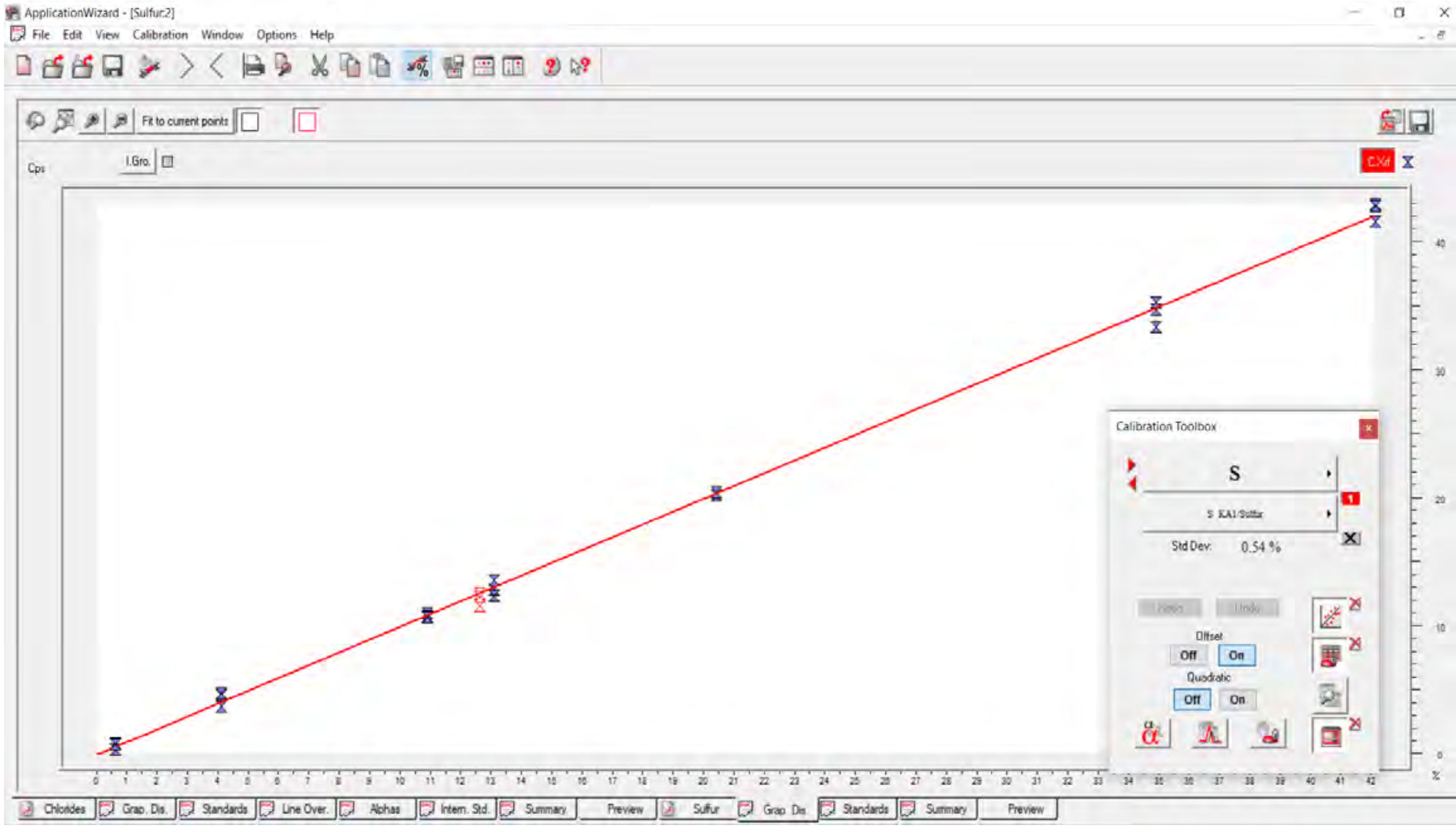
Obsidian

Where standard calibrations do not cover a specific application they can be customized, or new calibrations developed

Calibrations

User developed calibrations - EasyCal

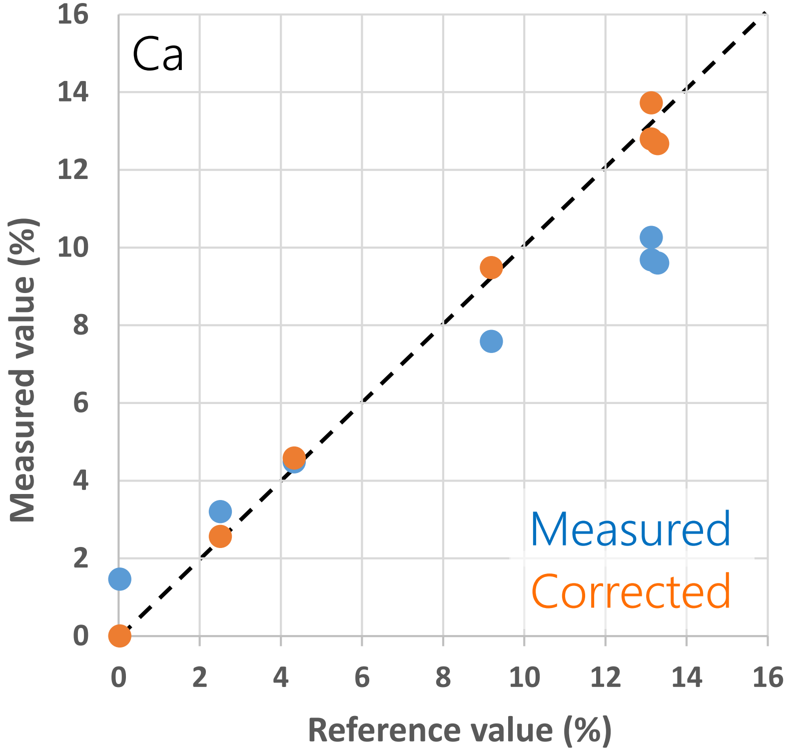
- Users can create their own calibrations
  - ➢ *Bruker's EasyCal Software*
  - ➢ empirical calibration using Lucas-Tooth modeling
  - ➢ graphical interface walks the user through calibration steps
- Requires
  - ➢ reference materials appropriate to the application
  - ➢ knowledge of calibrations and how they work (we can train you!)



Note: these are single-phase custom calibrations

Calibrations
Type Standardization

- Type standardization: additional option to correct for systematic error in results
  - Spreadsheet is used to calculate slope and/or offset corrections
  - For predictable or consistent error (e.g., difference in atmospheric conditions or sample preparation)
  - Corrections can be entered into the instrument and applied to specific calibration

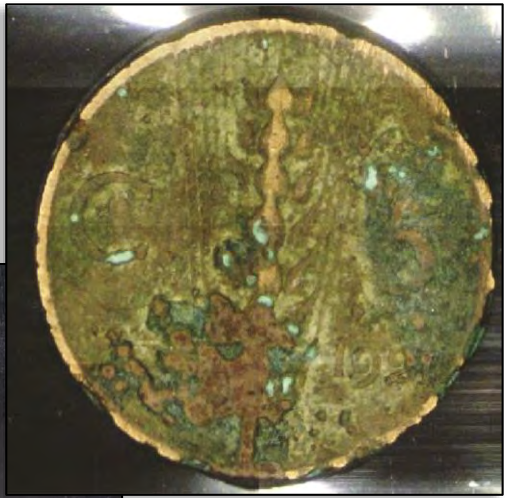


Example of original vs. type standardized results for Ca, correcting for influence a plastic bag

Artax – interactive software for pXRF

Spectrometer Mode
When are calibrations in-appropriate?

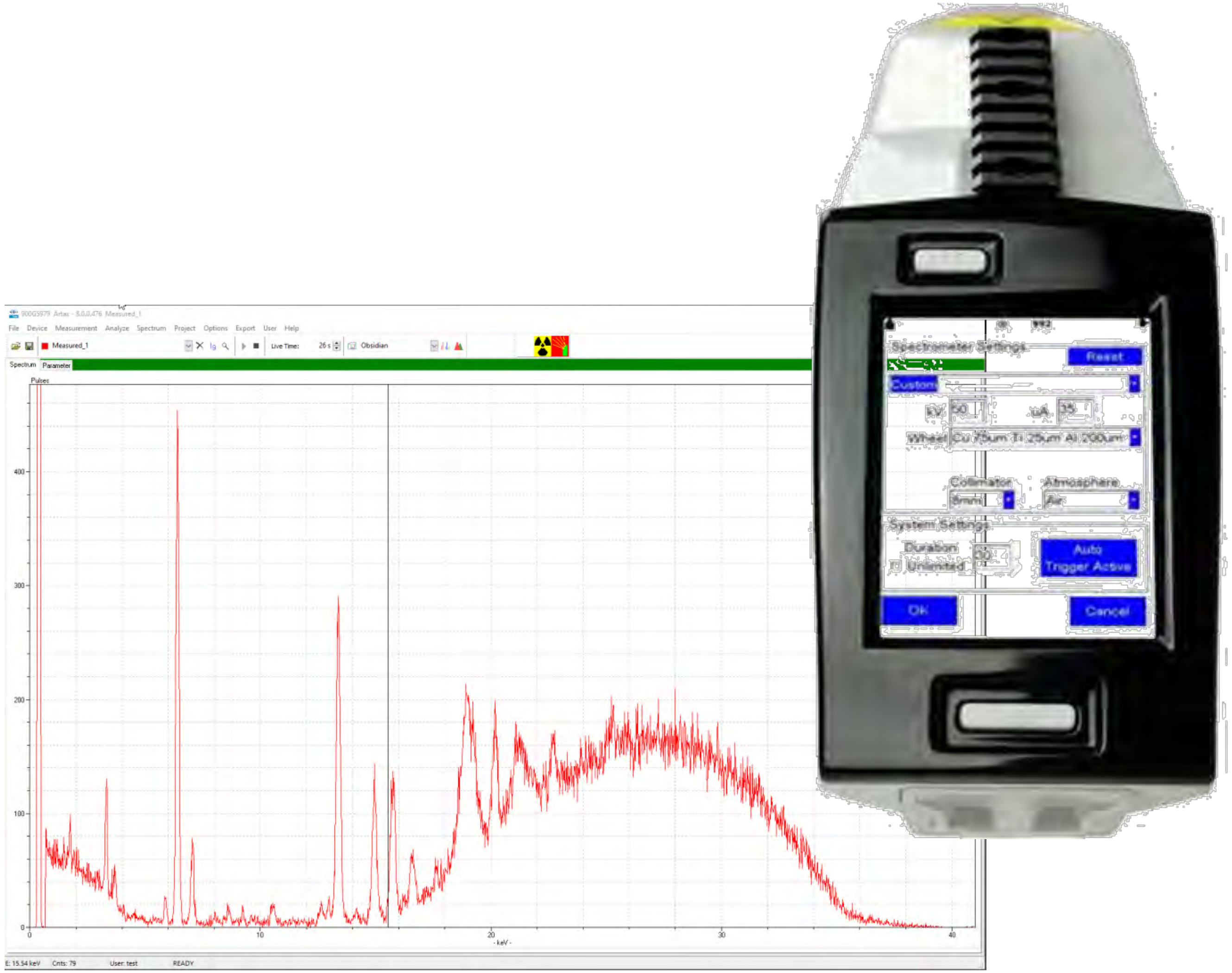
- Where sample conditions do not allow quantification of compositional information, objects should be investigated at the level of the X-ray spectrum
  - ➢ Thin samples (e.g., pigments on paintings; coatings)
  - ➢ Partial or complete covering by patina or other corrosion products
  - ➢ Composition of a material lies outside the range of a calibration
- Investigation of new objects or materials
  - ➢ What elements are present?
  - ➢ What are the most appropriate or optimal conditions for measurements?
- Assessing heterogeneity of a piece, e.g., corrosion, degradation, patina on a metallic object
  - ➢ Assess the extent, type of coating
  - ➢ Finding areas with the least degradation to get closer to true object composition



Spectrometer Mode

When are calibrations in-appropriate?

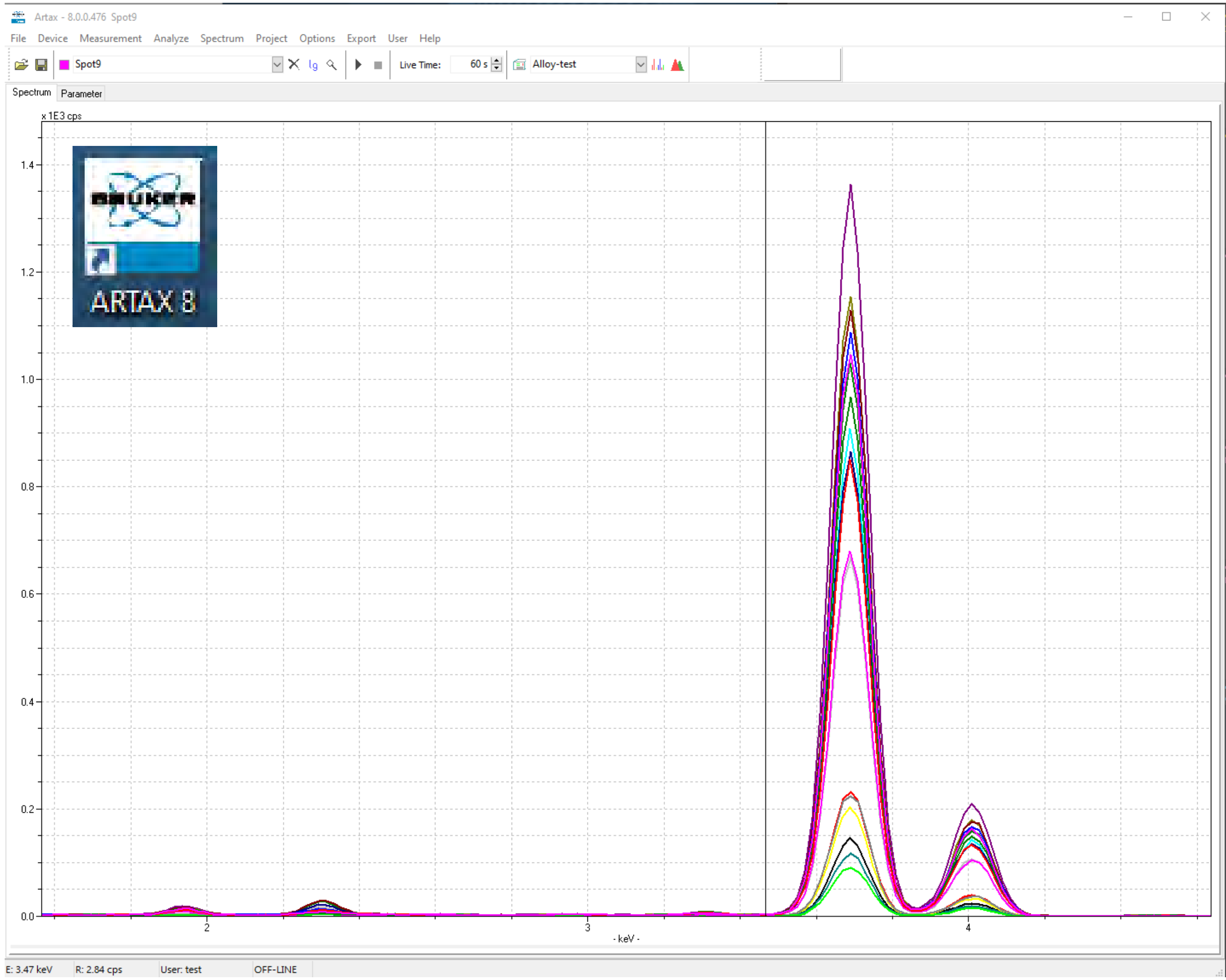
- Spectrometer Mode allows user to select parameters for a single-phase measurement:
  - ➢ Full control over measurement parameters: voltage (kV), current (µA), filters, spot size, atmosphere (Air, He, Vacuum)
  - ➢ Instrument displays live spectrum, count-rate, dead-time %
  - ➢ User can optimize parameters for specific target materials and required outcomes
  - ➢ Measurement may be viewed live on the instrument or in Artax or BIT, and spectrum files interpreted for data reduction and semi-quantitative analysis (i.e., peak intensities)



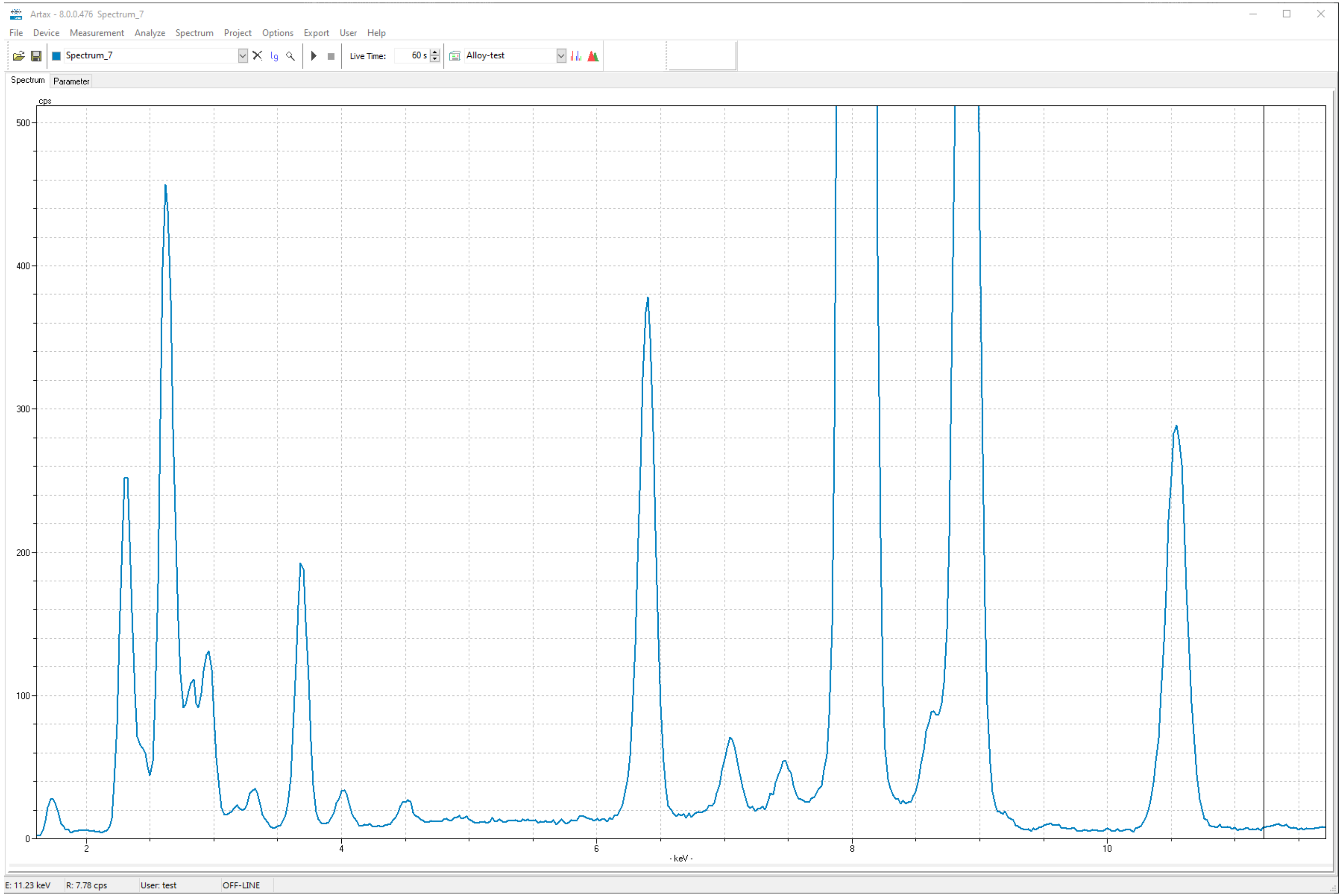
Artax

Fully interactive software for handheld-XRF

- **ARTAX** - enables thorough interrogation of object compositions through flexibility in:
  - ➢ control of the instrument in spectrometer mode
  - ➢ application of customized measurement conditions, and
  - ➢ in-depth interpretation of the resulting data down to the spectrum level



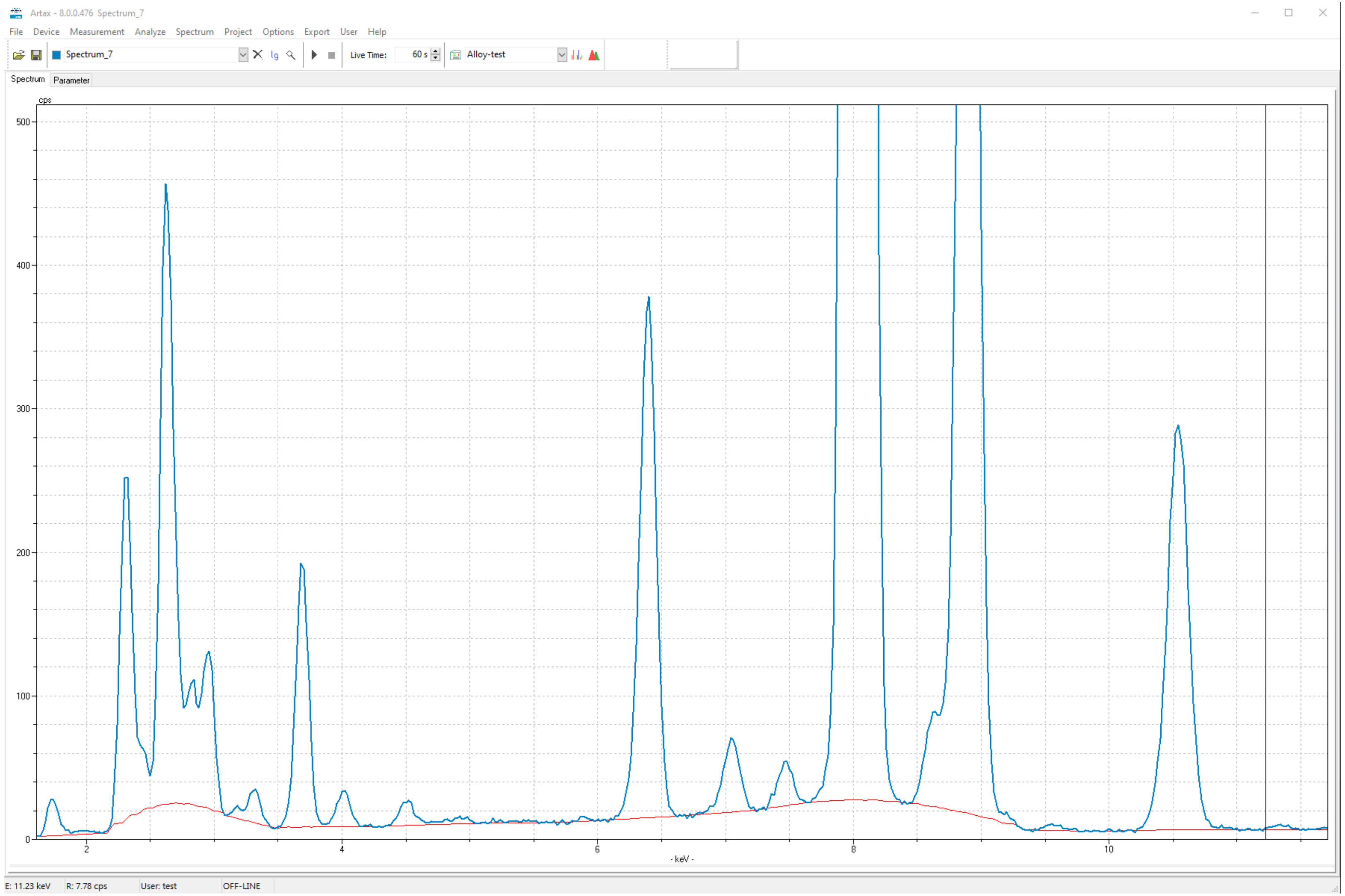
Artax
Data interpretation



Artax

Data interpretation

- Background fitting



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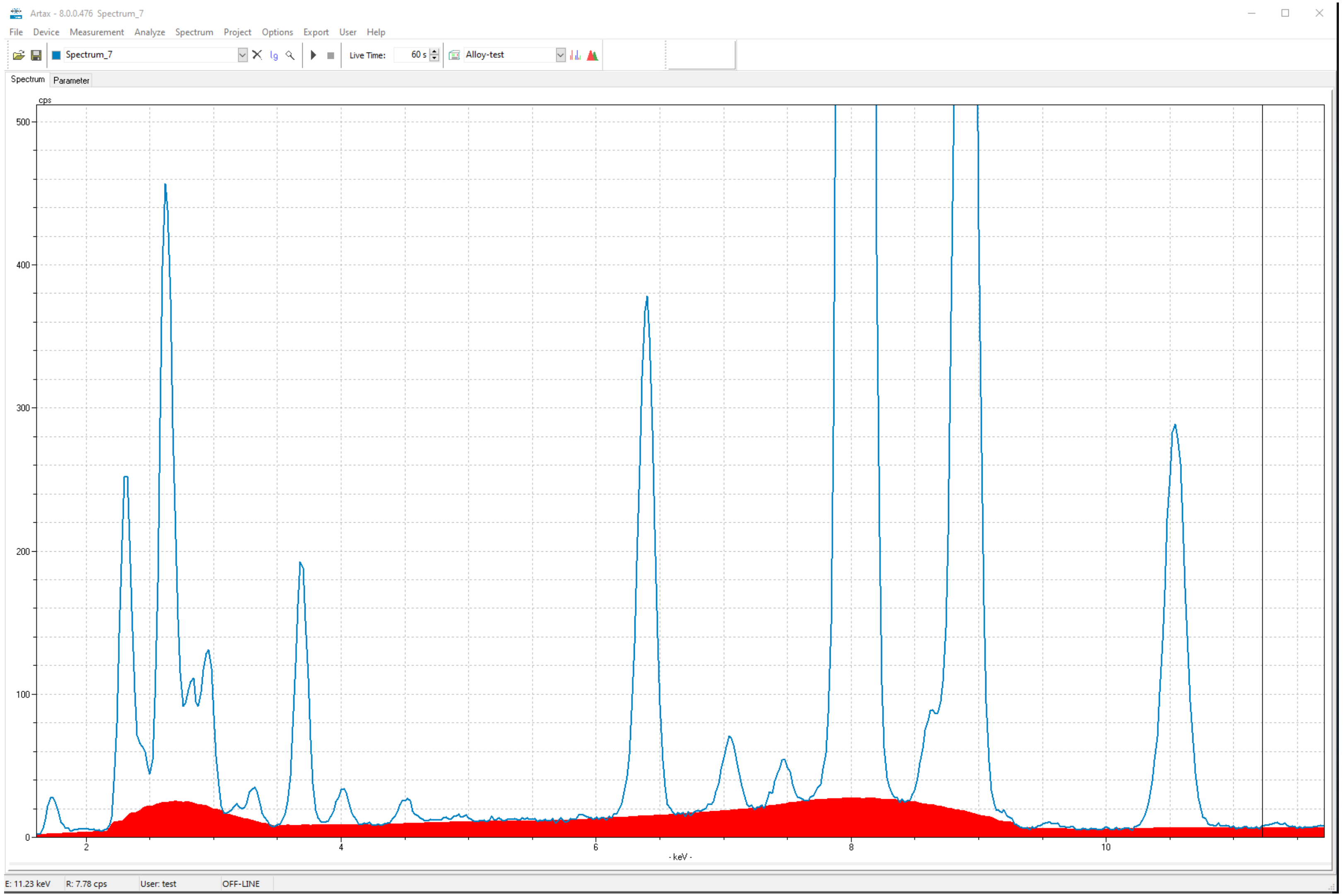
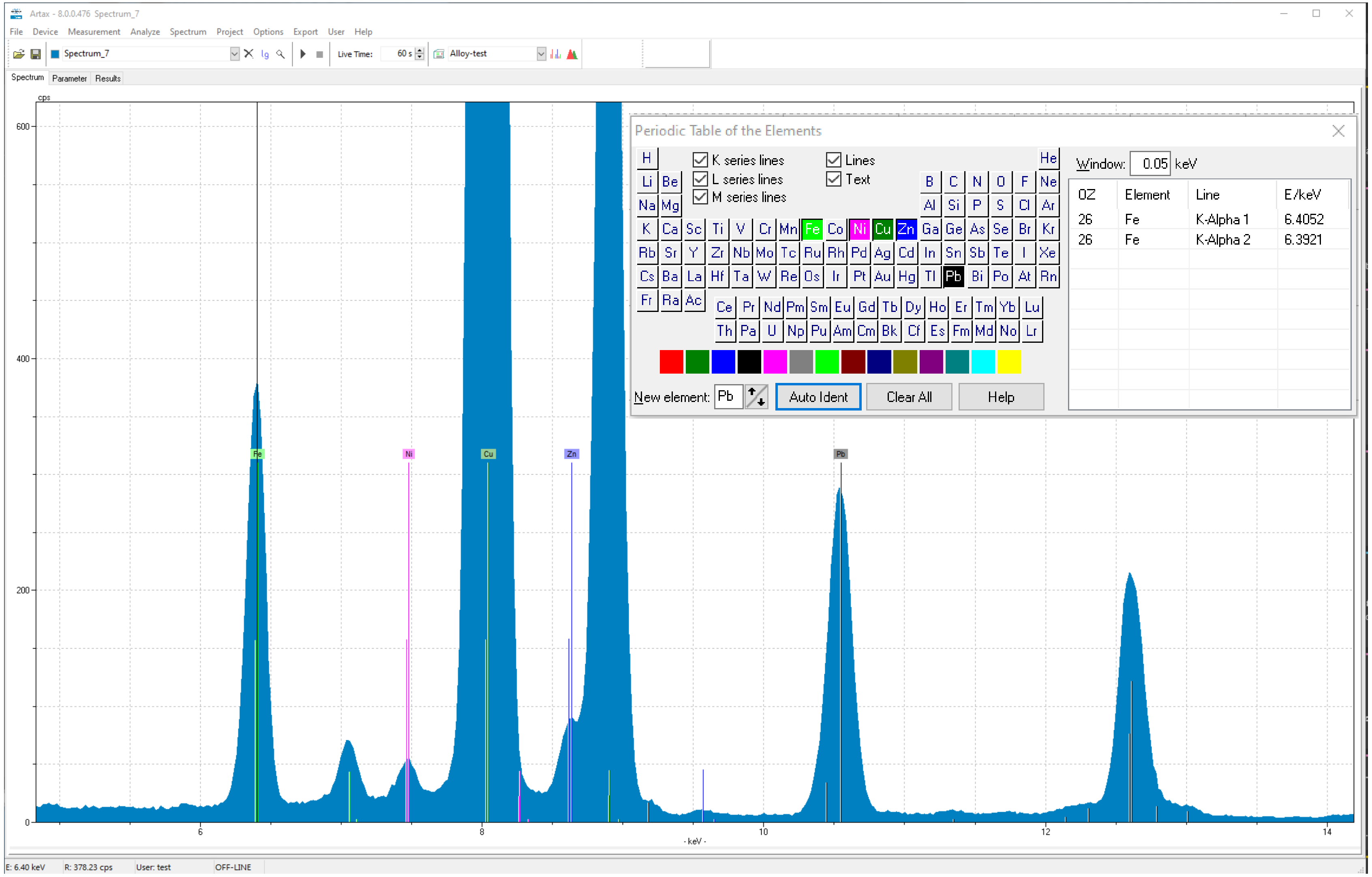


Artax
Data interpretation

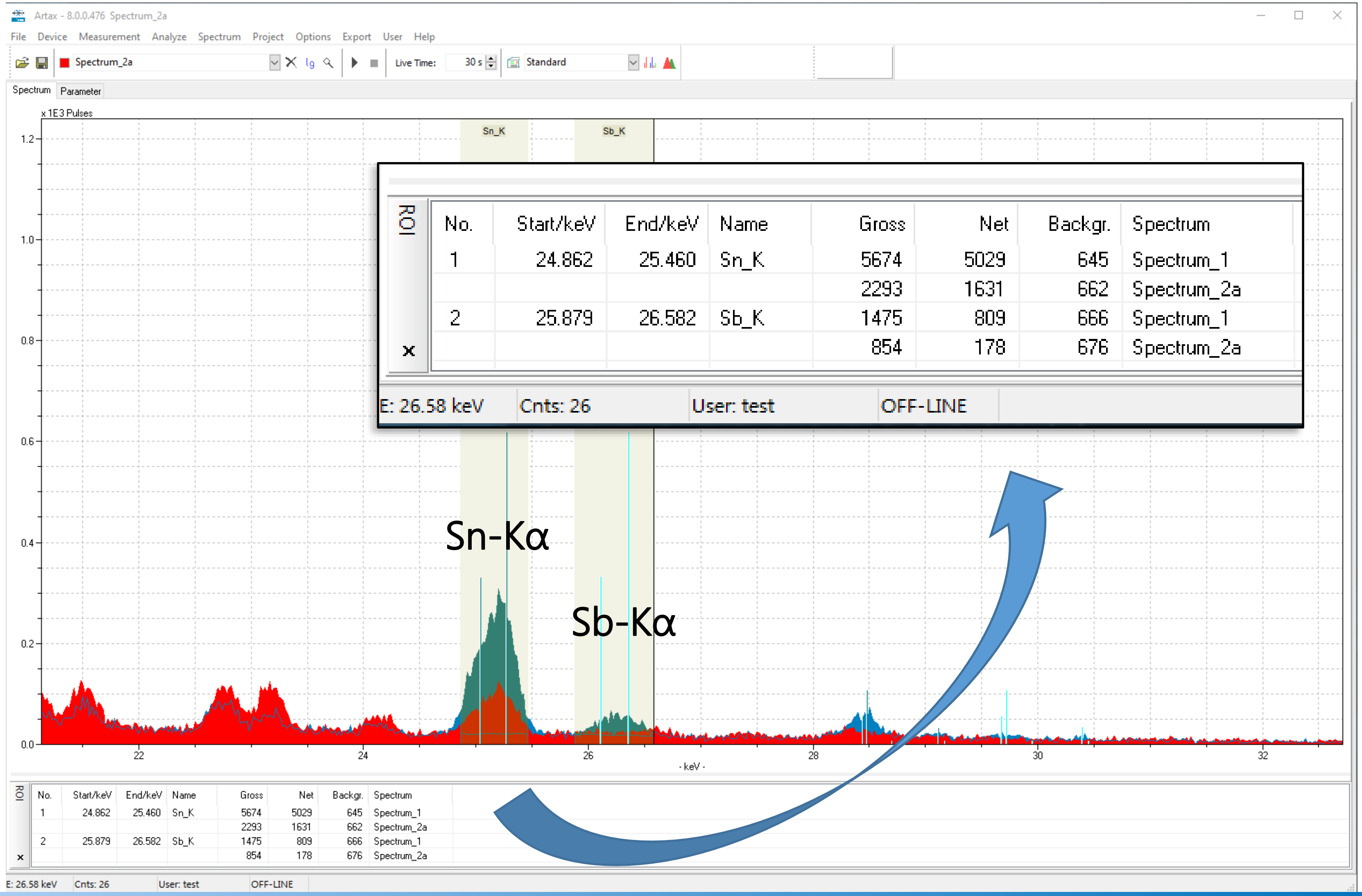
- Background fitting
- Interactive periodic table element finder with X-ray line energies



Artax

Data interpretation

- Background fitting
- Interactive periodic table element finder with X-ray line energies
- X-ray intensities at cursor positions
- Region-of-interest interrogation for spectra comparison



Artax

Data interpretation

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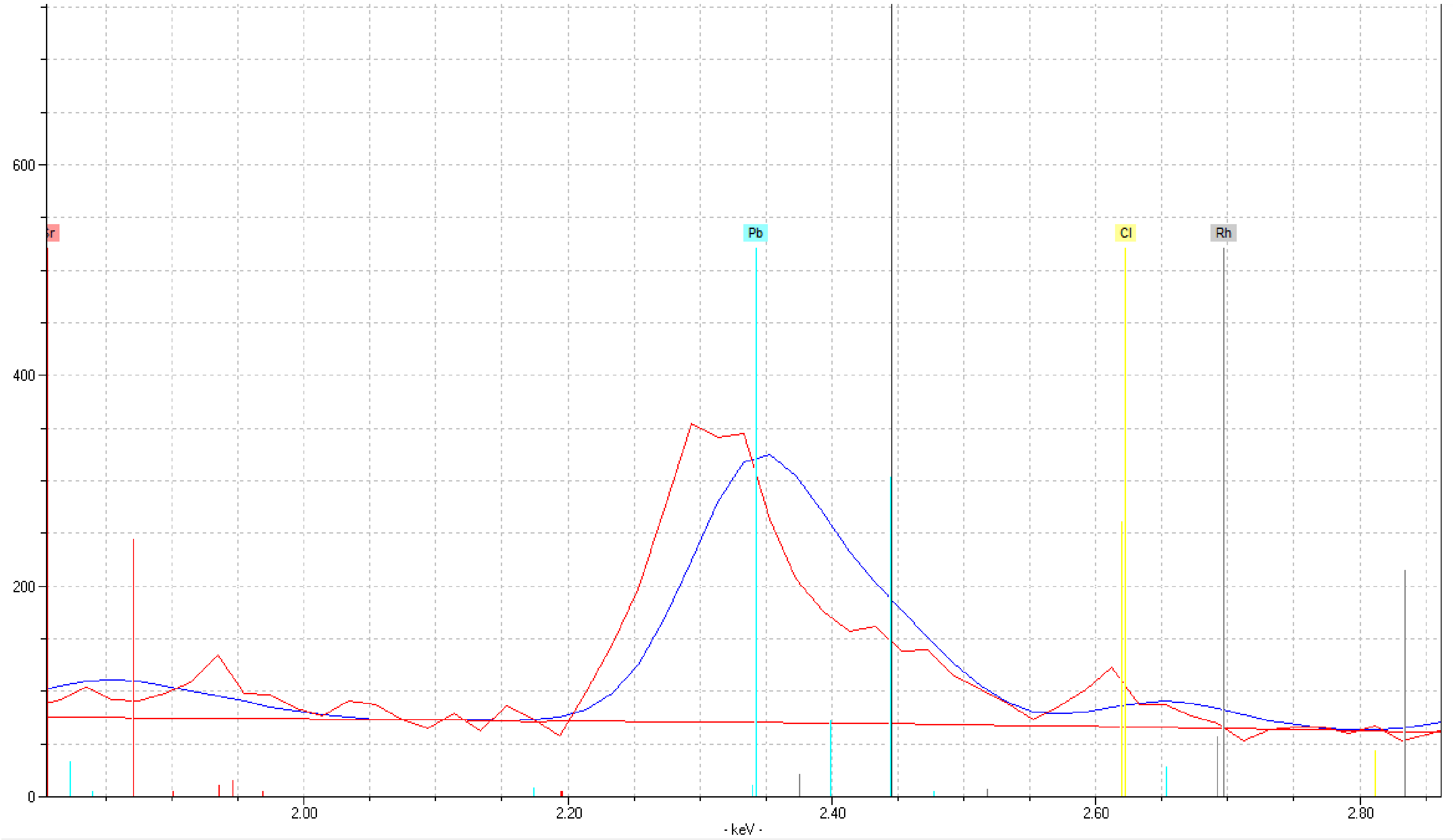
- Curve-fitting for robust peak identification and deconvolution of overlapping peaks



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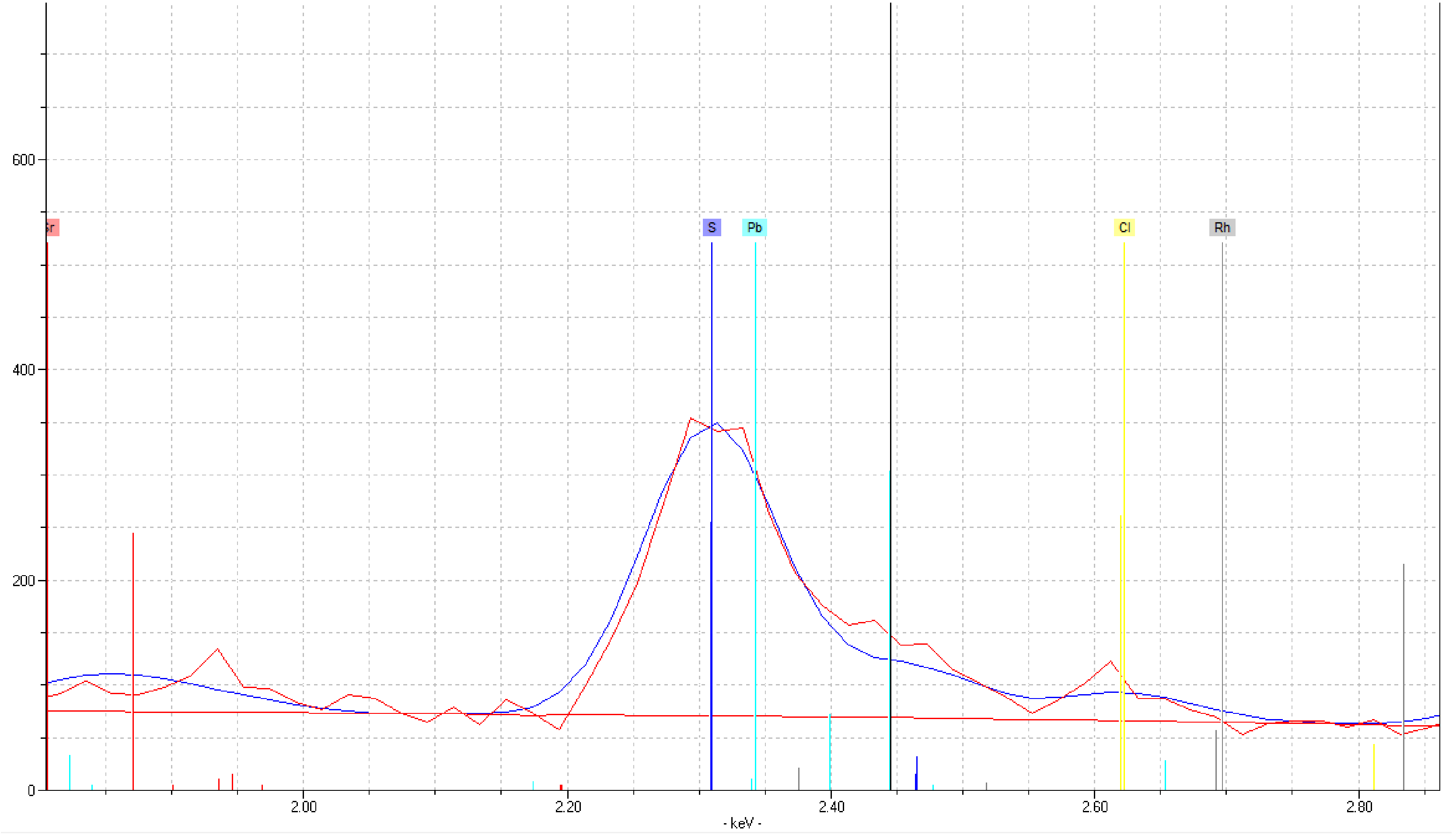

Artax

Data interpretation

- Background fitting
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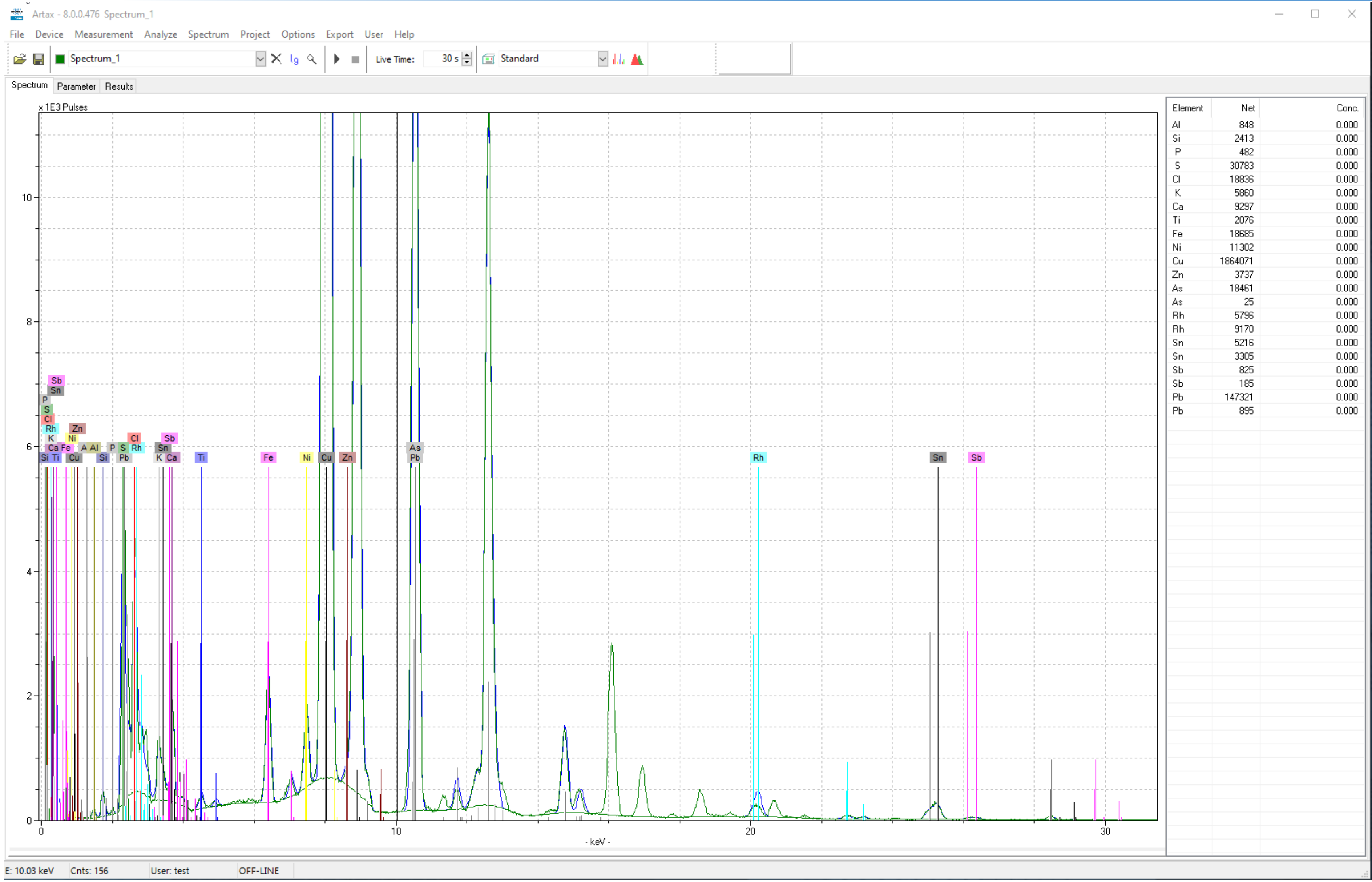
- Full net intensities assessment



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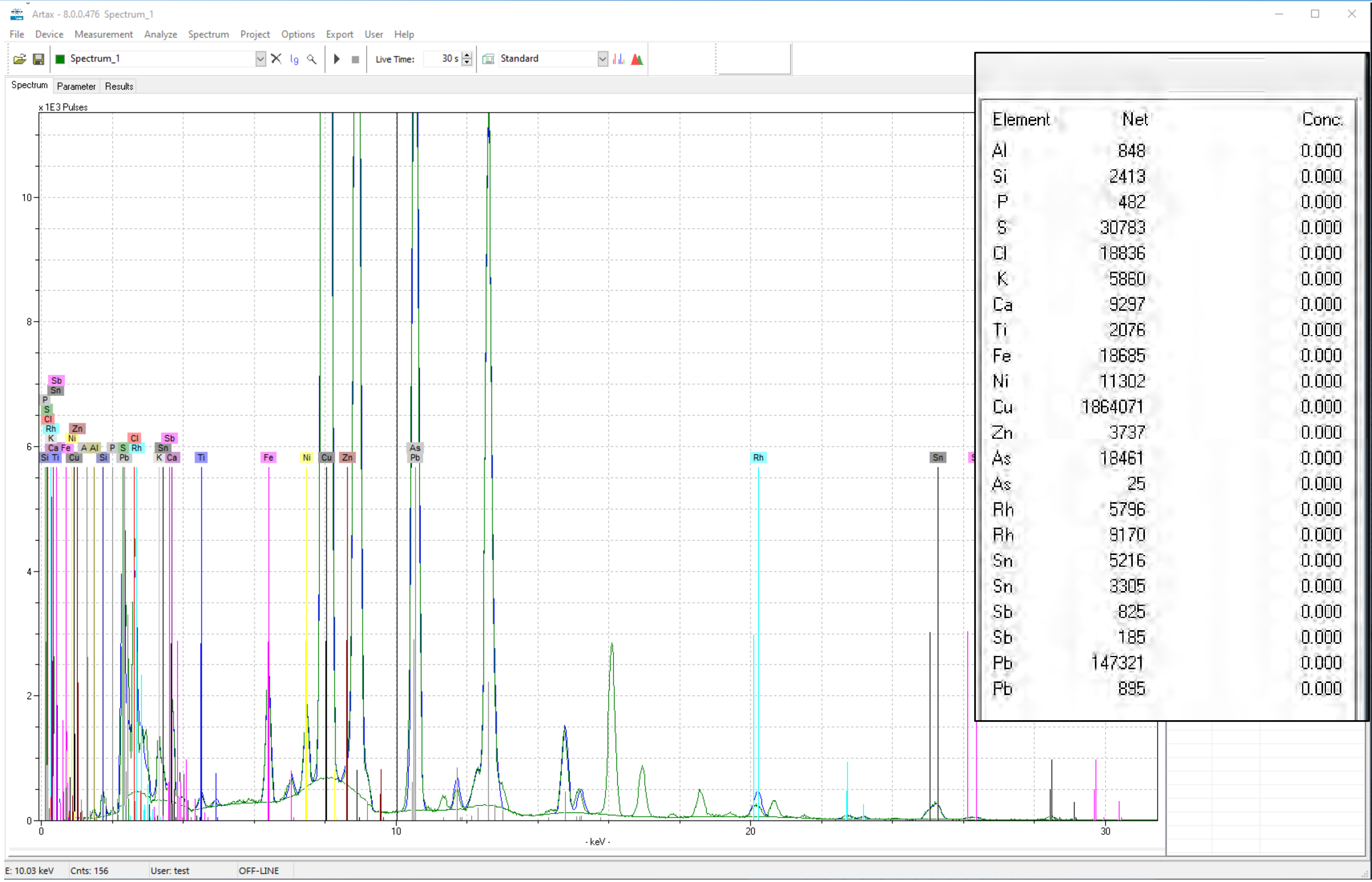
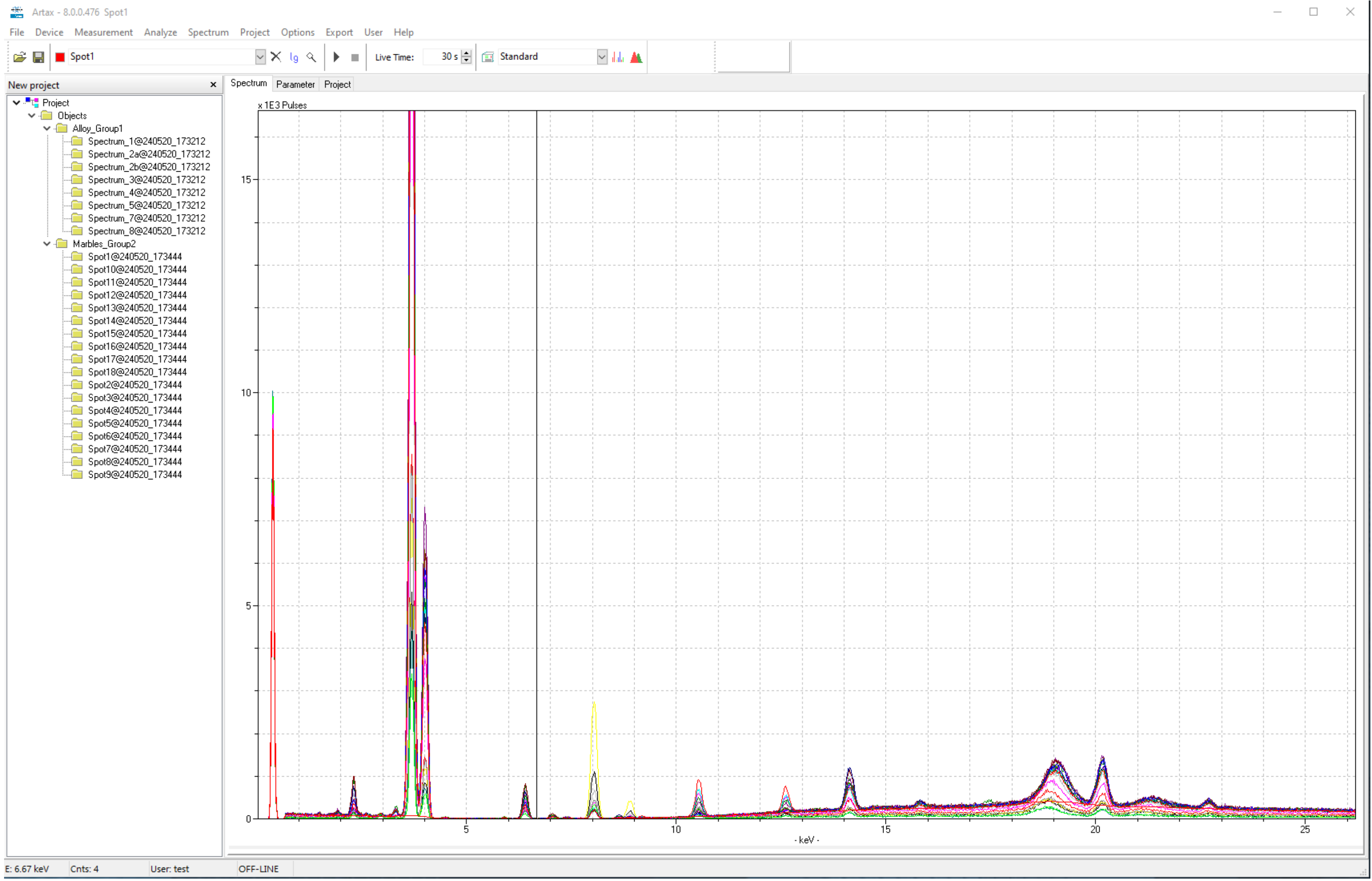


Artax
Project management

- Individual spectra may be collected and grouped to form "Projects"
    - Simple management of large groups of data
    - Allows systematic interpretation through application of consistent data treatment approaches
    - Simplifies comparison of data



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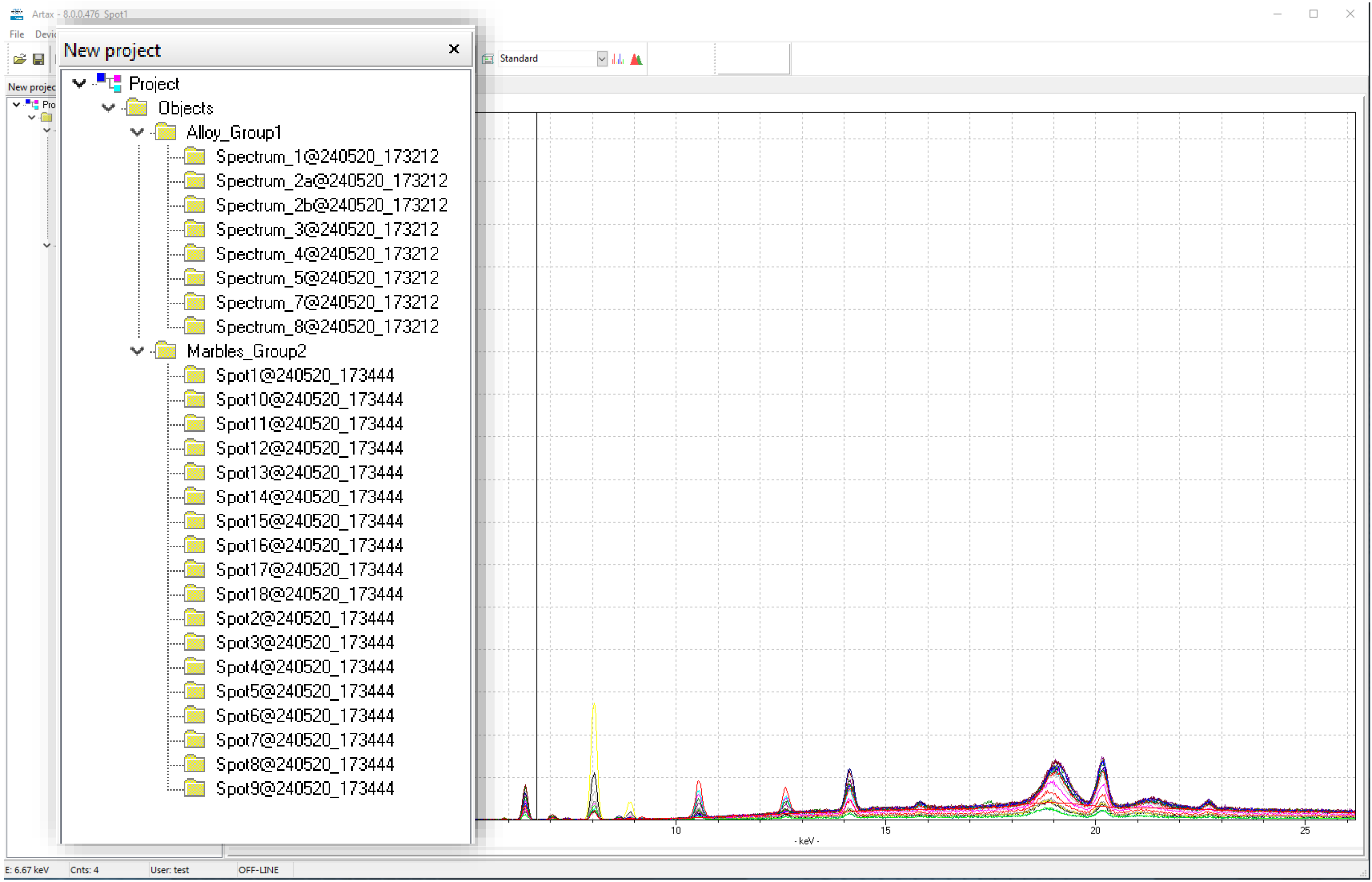
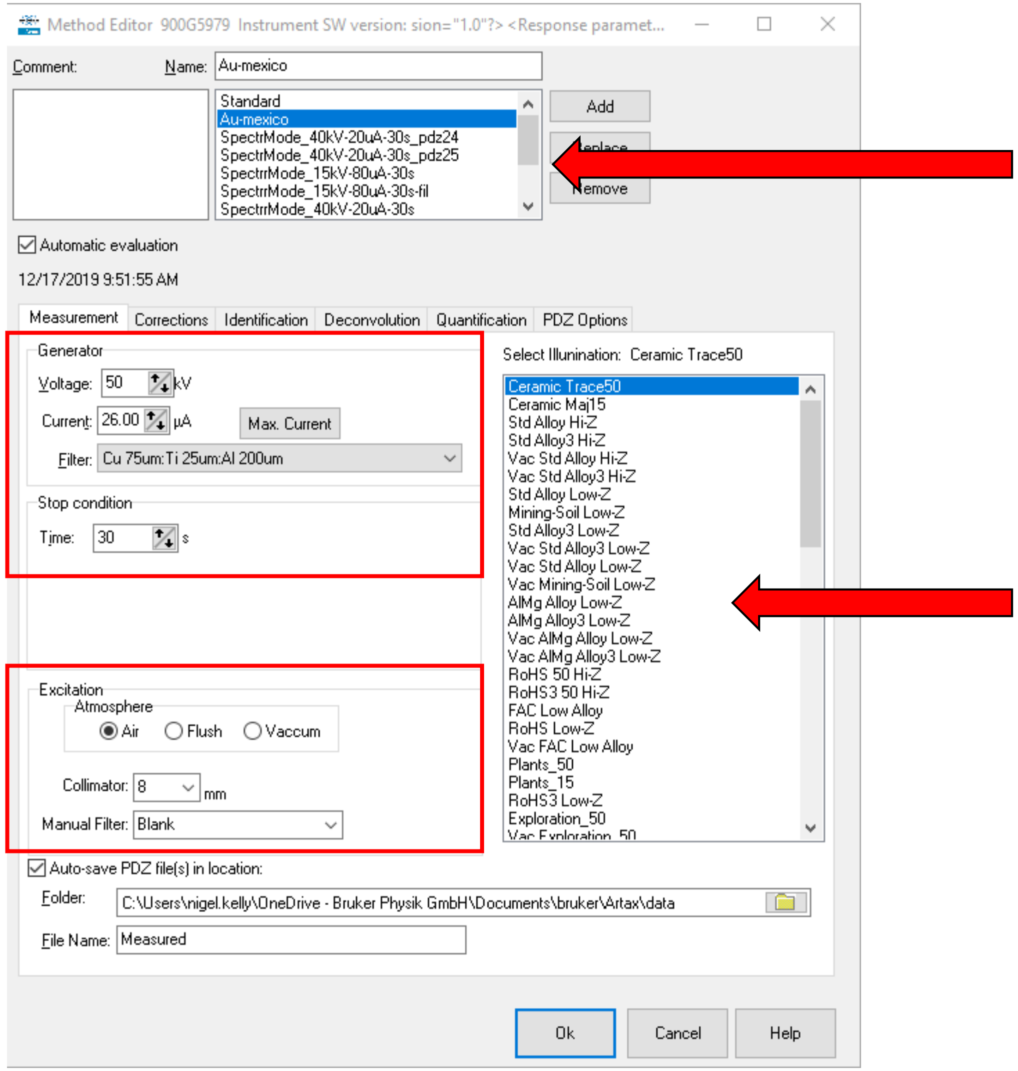


Artax

Methods – customized data collection and data reduction

- Data collection
  - Beam conditions
  - Count times



Saved methods

Measurement conditions to be applied

Methods based on Bruker factory calibrations

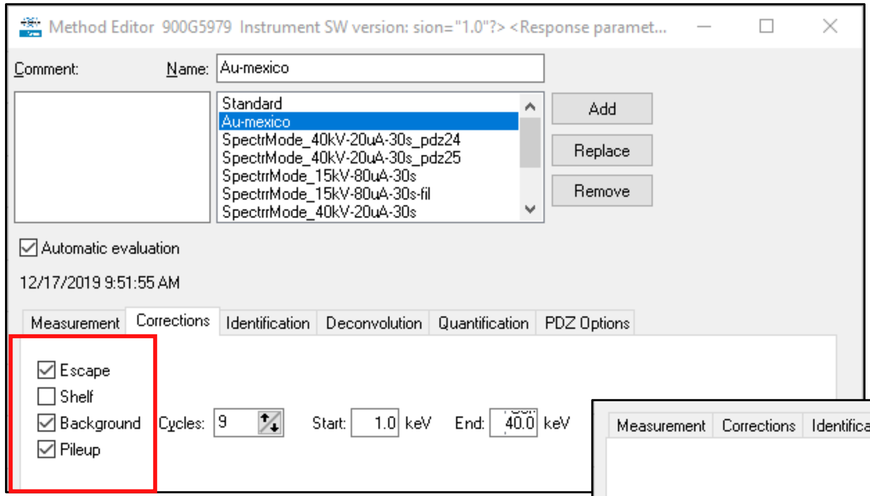
Conditions requiring manual settings on instrument – recorded with data file

Artax

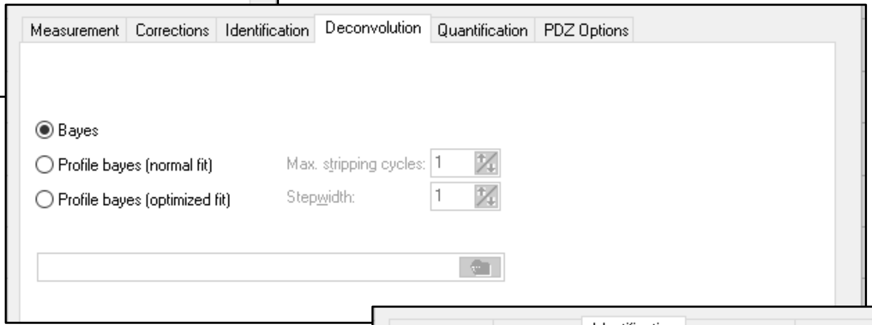
Methods – customized data collection and data reduction

- Data reduction
  - Correction parameters
  - Elements to be included in peak fitting and deconvolution for net intensity calculations
  - Deconvolution settings

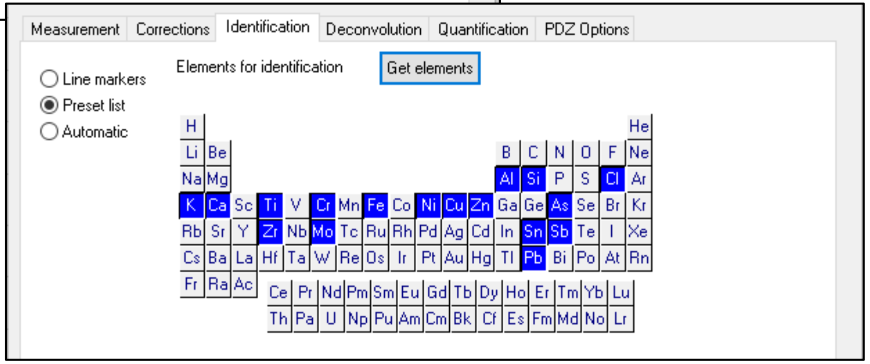
Custom settings may be applied to single or groups of spectra



Apply custom deconvolution settings



Save custom element lists



Artax
Data export

- Individual or groups of spectra
  - Bruker native format (.pdz)
  - Raw text files
- "Results" information for identified peaks
  - peak ID & energy
  - net intensity data
  - background

Raw spectra and metadata can be exported as .csv files using Bruker Cal Toolkit

Raw data and measurement parameters may be stored with object records

Element	Line	Energy/keV	Net	Backgr.	Sigma	Chi
Al	K12	1.486	817	437	41	5.98
Si	K12	1.74	2687	651	63	7.74
Cl	K12	2.622	53958	6179	258	113.24
K	K12	3.314	1837	3229	91	31.57
Ca	K12	3.692	21231	2367	161	45.5
Ti	K12	4.512	2390	3069	92	3.86
Cr	K12	5.415	360	3666	88	1.38
Fe	K12	6.405	53783	5084	253	47.8
Ni	K12	7.48	4449	8358	145	6.29
Cu	K12	8.046	1391363	10292	1188	475.8
Zn	K12	8.637	11356	8943	171	5.32
As	K12	10.543	2589	2903	92	46.47
As	L1	1.282	0	461	30	1.31
Zr	K12	15.775	10596	2033	121	5192.57
Zr	L1	2.044	1	1015	45	0.51
Mo	K12	17.48	3043	1466	77	693.05
Mo	L1	2.292	20924	3479	167	228.19
Sn	K12	25.271	39	373	28	0.82
Sn	L1	3.444	388	2543	74	18.13
Sb	K12	26.359	19	259	23	1.34
Sb	L1	3.604	1	2492	71	34.97
Pb	L1	10.551	54502	2651	245	45.48
Pb	M1	2.342	1847	3976	99	218.25

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TRACER applications in Cultural Heritage

Example: Obsidian sourcing in the Mediterranean

Application study by Prof. Robert Tykot, University of South Florida

What is obsidian?

- Obsidian is volcanic glass formed due to rapid eruption and crystallization of lava
- Homogeneous within a flow, but preserve compositions that are distinctive between locations

Photo credits: R. Tykot

Pantelleria (Italy) obsidian flow (left) and 1 eruption layer (above)

Example: Obsidian sourcing in the Mediterranean

Why is obsidian important?

- Ideal to work into arrow points, spearheads, blades
- Compositions can be traced back to the original source of the obsidian

Photo credits: R. Tykot

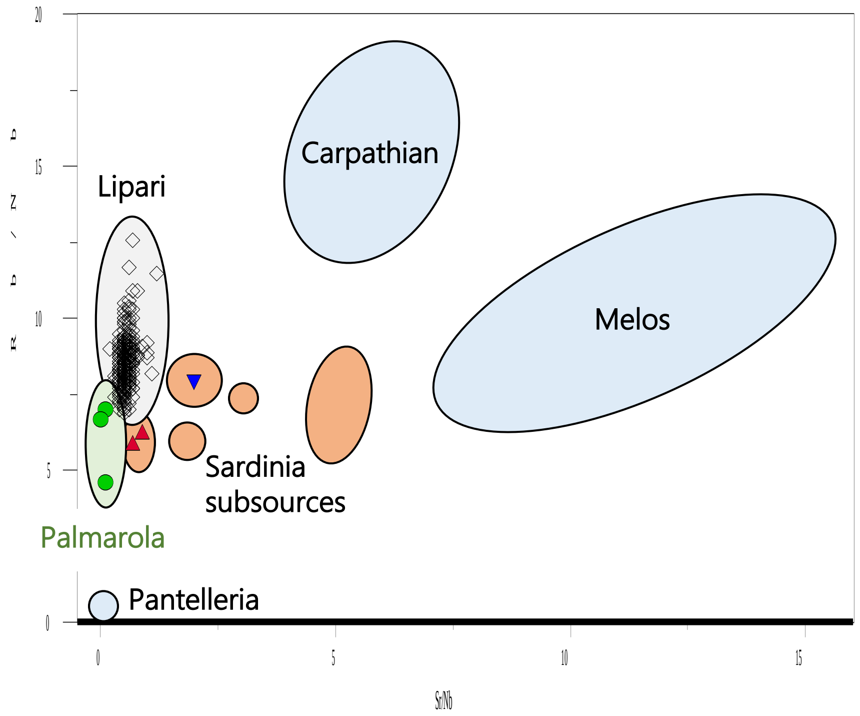
- Placed in a temporal context – when the artifact was used – can help establish trade and other human interconnections within regions

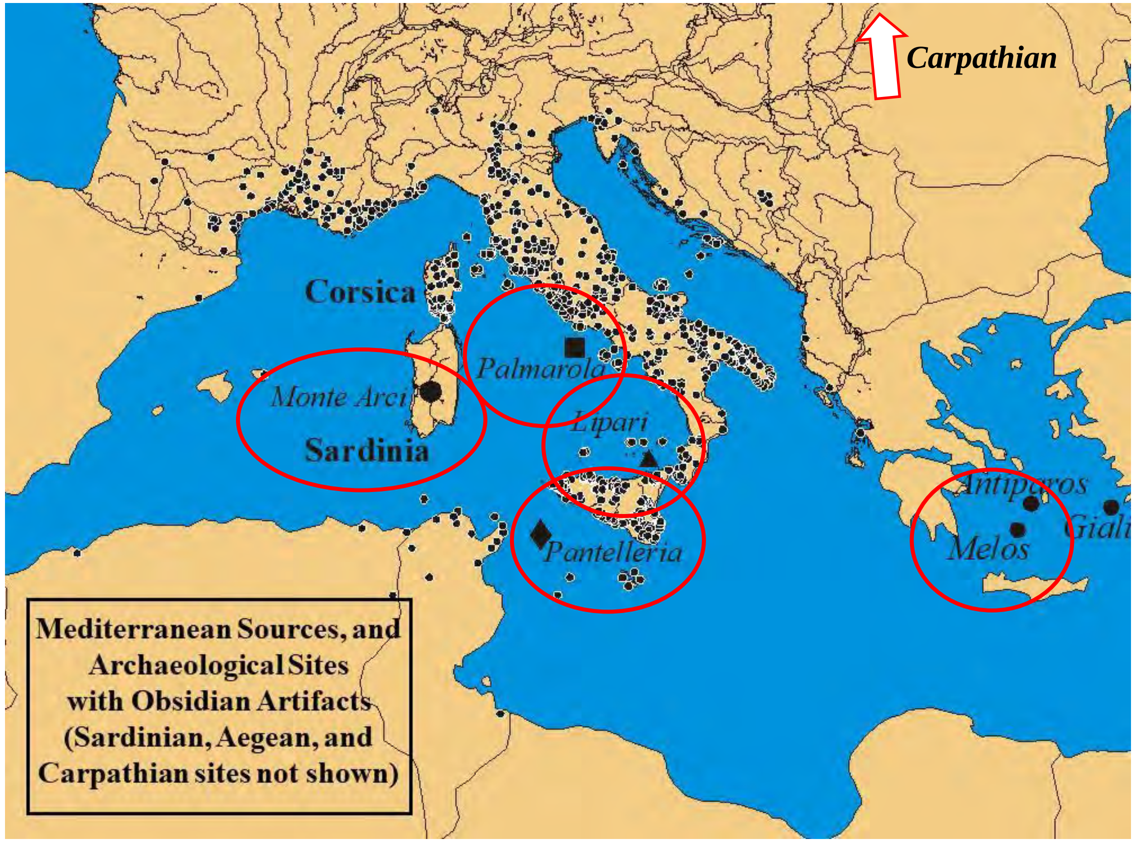
Example: Obsidian sourcing in the Mediterranean

How does sourcing work?

Distinct compositions

- Trace element compositions / ratios are distinct to a location, and sometime a single eruption





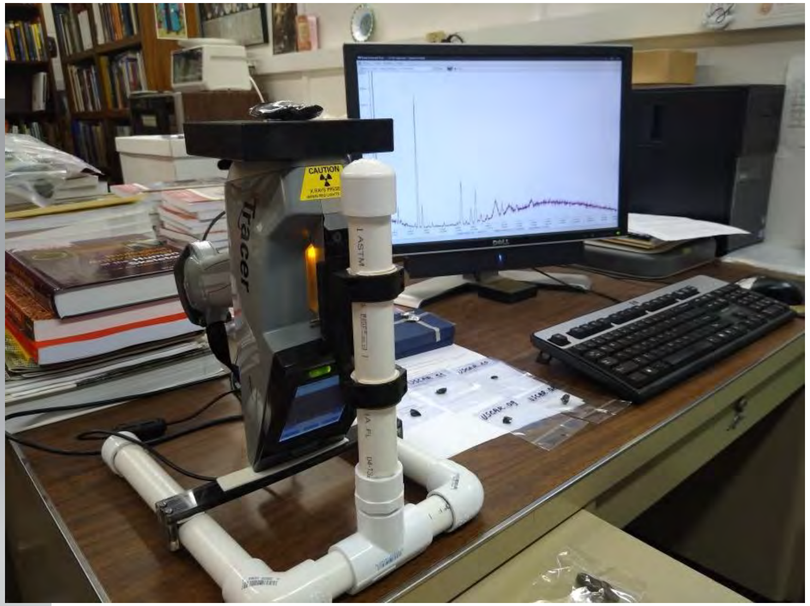
Example: Obsidian sourcing in the Mediterranean

How does sourcing work?

Methods for measuring compositions

- ICP-MS, INAA, powder XRF
  - ➢ destructive, require sample processing

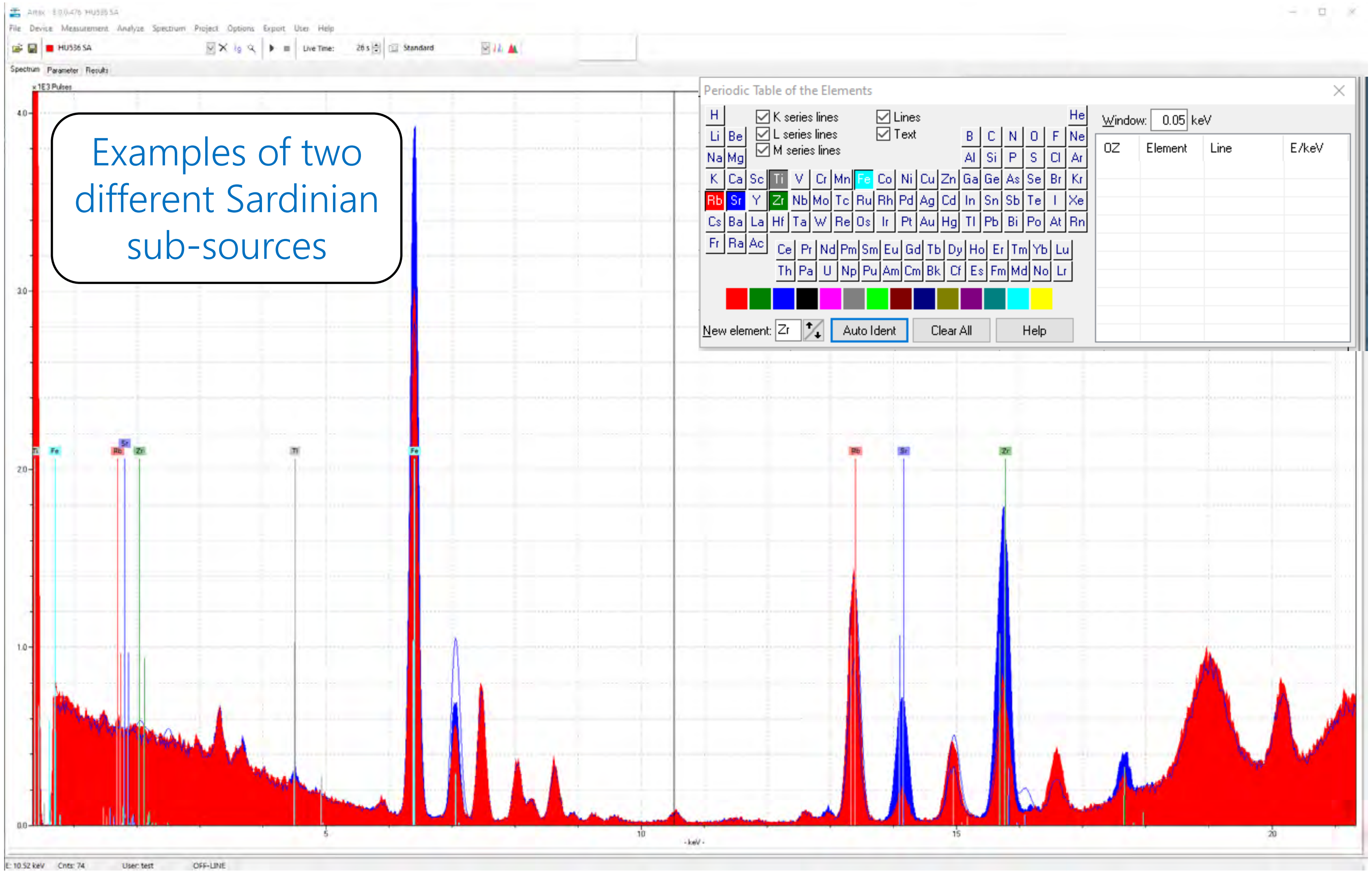
- <u>handheld-XRF</u> (pXRF)
  - ➢ measurements can be conducted in the field, on site
  - ➢ limited sample preparation (cleaning)
  - ➢ measurements can be quantified using custom or Bruker factory obsidian calibration



Data and images courtesy of Prof. Robert Tykot, USF

Example: Obsidian sourcing in the Mediterranean

How does sourcing work?



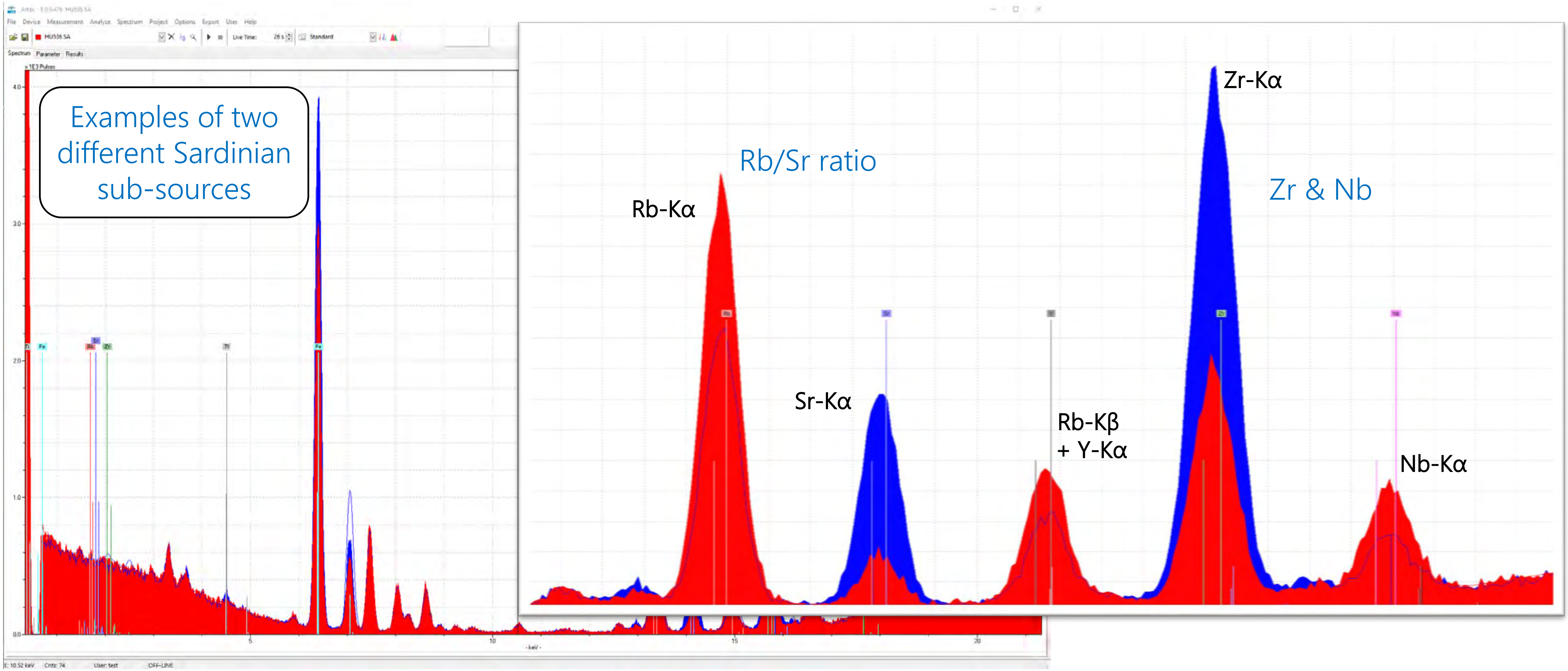
Data being evaluated here using Artax

- Quality of spectra
- Basic differences between objects

Data and images courtesy of Prof. Robert Tykot, USF

Example: Obsidian sourcing in the Mediterranean

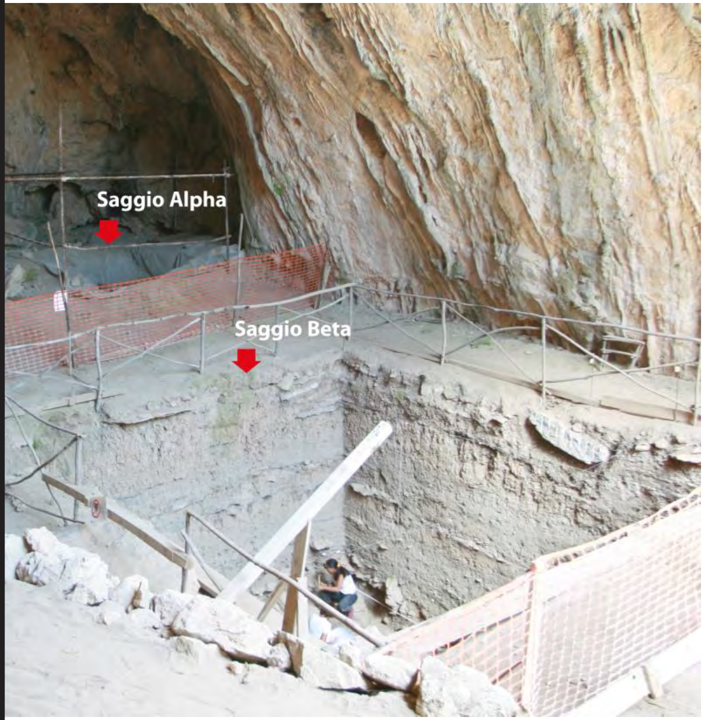
How does sourcing work?

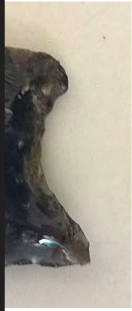


Data and images courtesy of Prof. Robert Tykot, USF

Example: Obsidian sourcing in the Mediterranean

Excavation at Saracena (Grotta di San Michele)



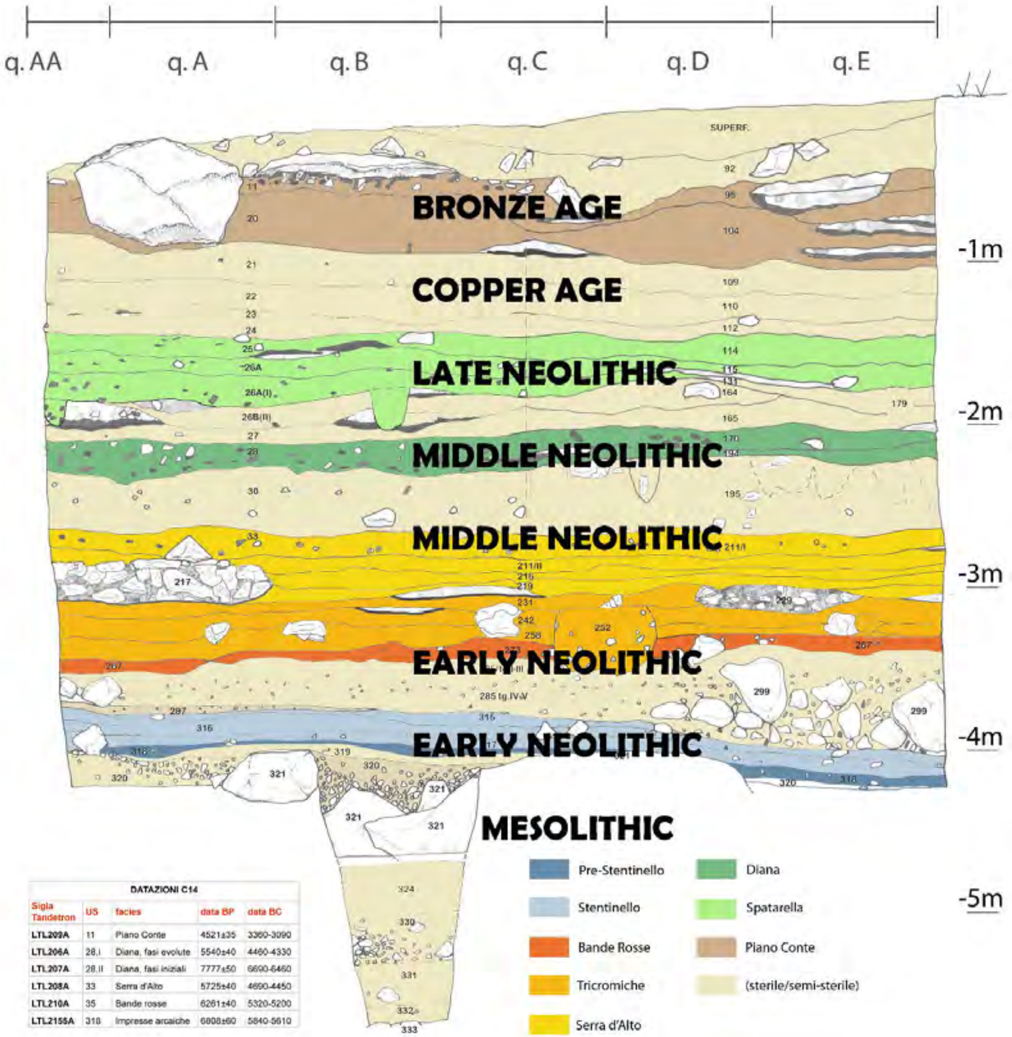


Example: Obsidian sourcing in the Mediterranean

Excavation at Saracena (Grotta di San Michele)

Saracena

- Deep, intact stratigraphy records continuing occupation from
  - *Early Neolithic (ca. 5300 BCE)*
  - *Bronze age*
- Rare preservation
  - *Typically lost due to modern usage and/or erosion*



Cross-section image courtesy of Vincenzo Tinè & Elena Natali

Example: Obsidian sourcing in the Mediterranean
Excavation at Saracena (Grotta di San Michele)

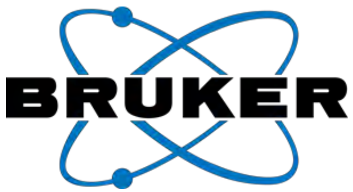
Obsidian Sourcing

- Compositional analysis of 467 objects using Tra
  - quantification using empirical obsidian calibrati
  - access to samples made easier by portable ana approach
  - enabled large number of artifacts to be analyze 'quickly' developing a statistically robust data se
- Compositional data supported by object morphology (techno-typology and use-wear analysis)

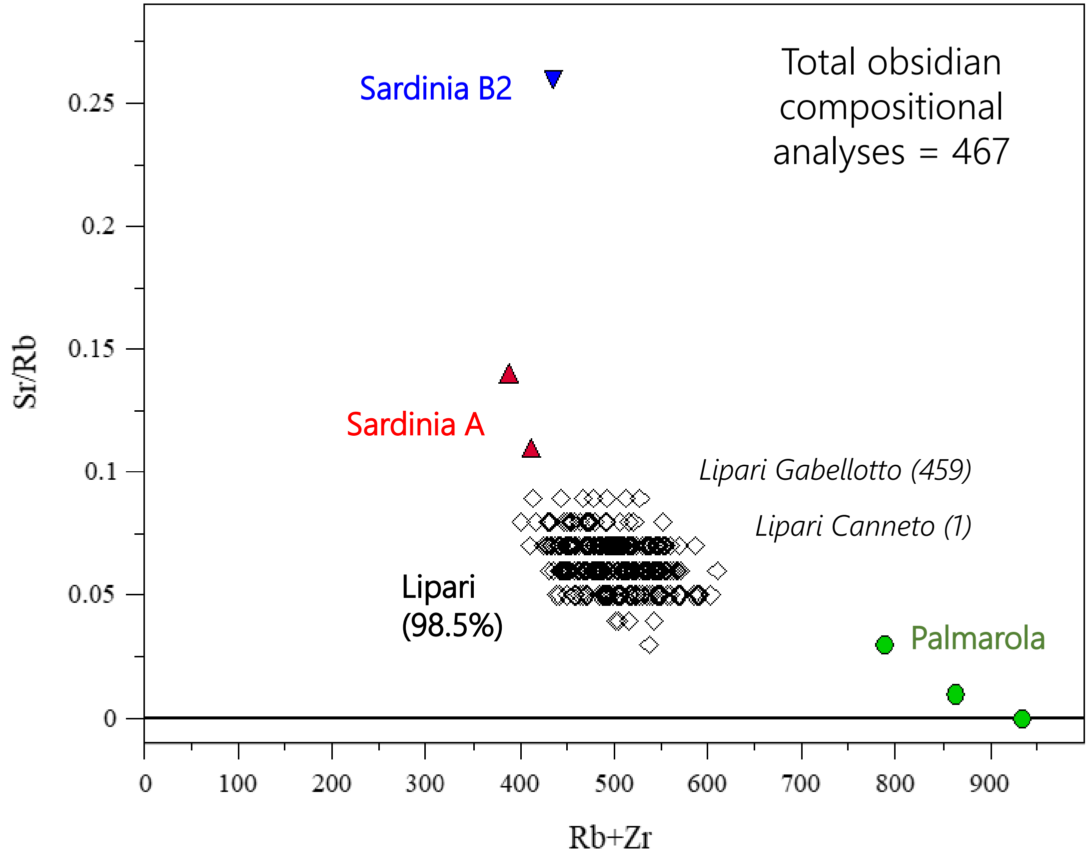
Data and images courtesy of Prof. Robert Tykot, USF

Example: Obsidian sourcing in the Mediterranean

Excavation at Saracena (Grotta di San Michele)



Obsidian Sourcing



Data and images courtesy of Prof. Robert Tykot, USF

Example: Obsidian sourcing in the Mediterranean

Excavation at Saracena (Grotta di San Michele)

Lessons from obsidian source patterns

- Obsidian data suggest maritime trade, including with islands
  - Lack of preservation of boats until much later
  - Supports that maritime trade was limited, hugging islands and coastline
- Data patterns suggest that long-distance distribution was "down-the-line"
  - trade from one place to another
  - numbers decrease along the way

Data and images courtesy of Prof. Robert Tykot, USF

Quantitative analysis of Gilt Bronzes

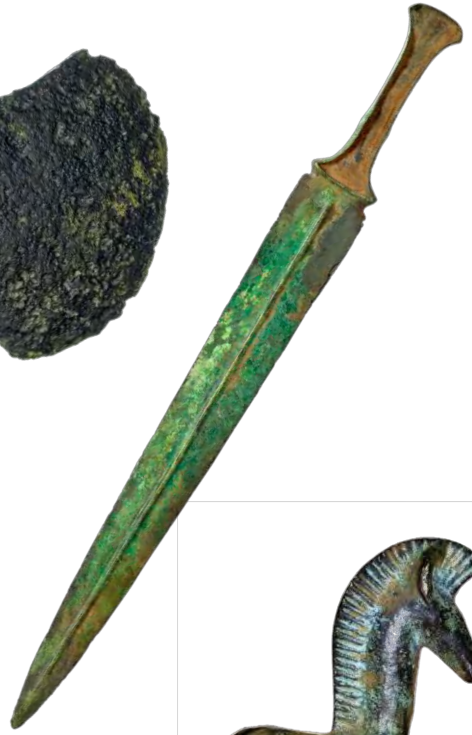
Application example by Dr. Arlen Heginbotham, J. Paul Getty Museum

Example: Quantitative analysis of Gilt Bronzes

Application study by Dr. Arlen Heginbotham, J. Paul Getty Museum

Heritage copper alloys

- Important component to Cultural Heritage collections
- Span from pre-history bronze objects (vessels, blades, axe heads, jewelry), through to modern works of art



Compositional analyses keys to understanding

- Smelting and other metallurgical technologies through time
- Manufacture and production techniques
- Trade (esp. pre-historic artifacts)
- Characterizing genuine vs non-genuine works (reproductions, fakes)

Example: Quantitative analysis of Gilt Bronzes

Establishing Authenticity

What are French Gilt Bronzes

- Actually brass: Cu + Zn alloys
  - Mid-17th to mid-19th century: Zn = 13 – 25%
    Sn & Pb = 0.5 – 2%

Questions about the Gilt Bronzes

- Authentic objects?
- Later copies?
  - Legitimate replacements of broken or lost components
  - Frauds

Example: Quantitative analysis of Gilt Bronzes

Establishing Authenticity

Can you tell the difference?

Example: Quantitative analysis of Gilt Bronzes

Establishing Authenticity

Alternative (complementary) approach - systematic variations in compositions of the alloys

- Ratios of metals change with time…
  - Changing trade patterns: sources of ores and refined metals
  - Advances in metallurgical technology

Example: Quantitative analysis of Gilt Bronzes
Establishing Authenticity

Alternative (complementary) approach - systematic variations in compositions of the alloys

- Ratios of metals change with time…
  - ➢ Changing trade patterns: sources of ores and refined metals
  - ➢ Advances in metallurgical technology

Study dataset – based on Getty collection

- Collection included >250 objects, spanning 1675 through to the present
- Created a reference data base of compositions based on >1300 analyses of objects with ages known with confidence




Heginbotham, 2013

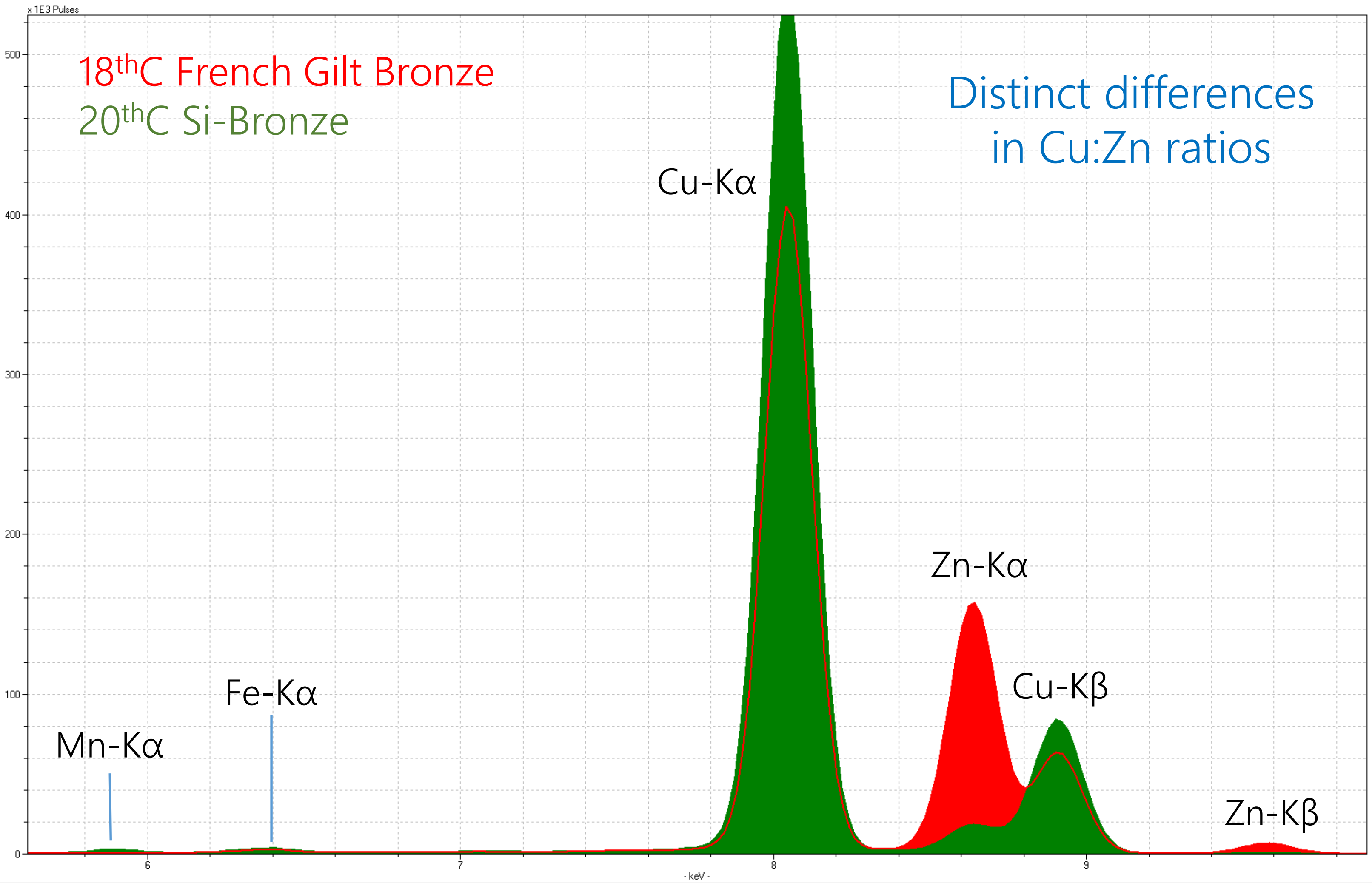
Example: Quantitative analysis of Gilt Bronzes

Compositional analysis

Analyses focused on data for 13 elements

- Major elements
  - Cu, Zn, Pb, Sn (typically >1 wt%)
- Minor elements
  - Mn, Fe, Co, Ni, As, Ag, Cd, Sb, Bi (<1 wt%)
  - Impurities, related to metal refining methods or the ore sources themselves

Raw data quantification based on the CHARM set



Heginbotham, 2013; Heginbotham et al., 2017

Example: Quantitative analysis of Gilt Bronzes
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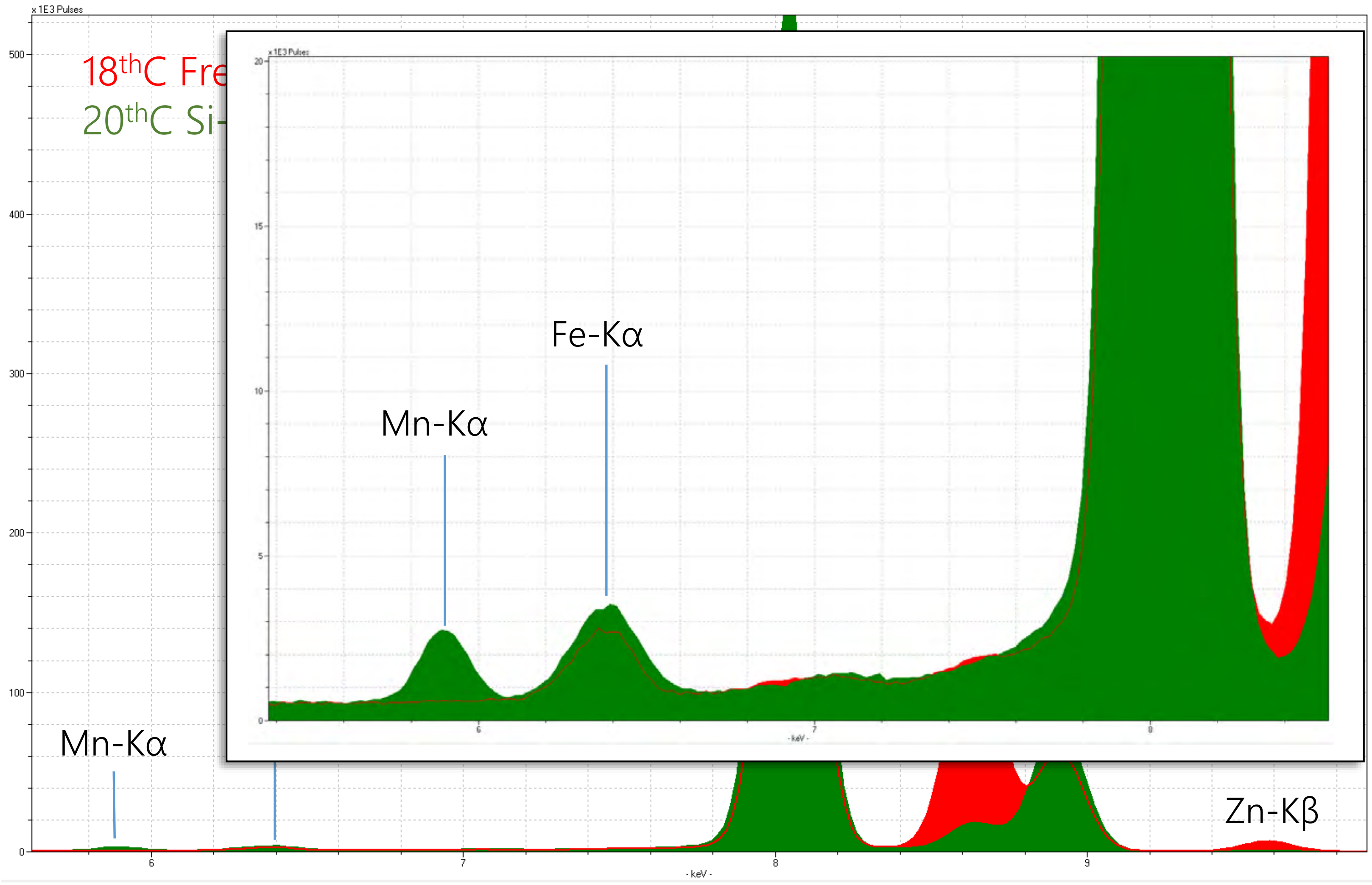
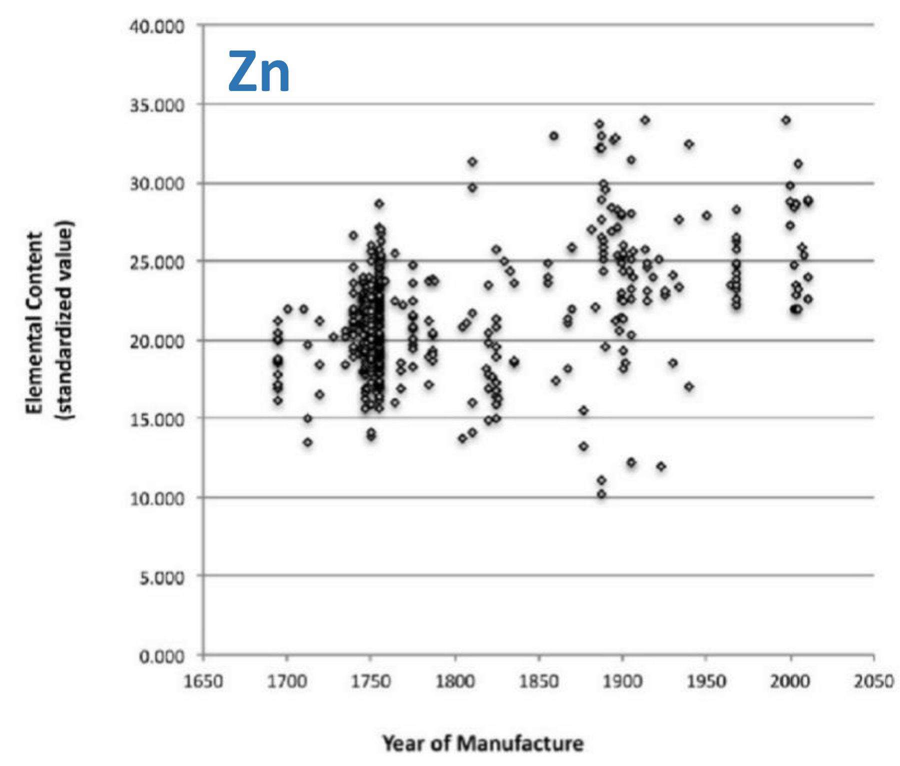


Heginbotham, 2013; Heginbotham et al., 2017

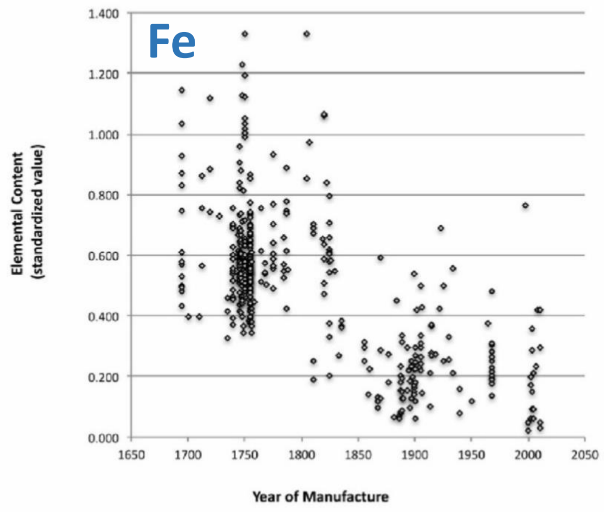
Example: Quantitative analysis of Gilt Bronzes

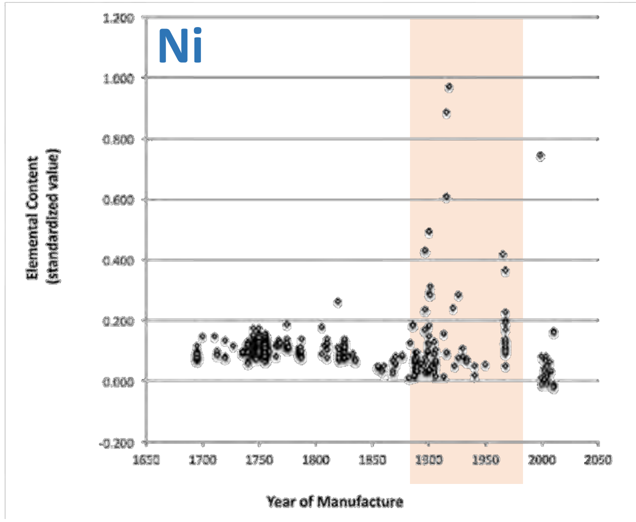
Compositional analysis – key results

Select compositional trends vs year of manufacture



- Zn – increases % with time
  - associated with improvements in brass production technology
- Fe – decreases % with time
  - correlates with transition from cementation brass to spelter brass
- Ni – spike in late 19thC
  - coincides with Cu sourced from Sudbury (elevated Ni content)
  - drop-off coincides with switch to electrolytic refining





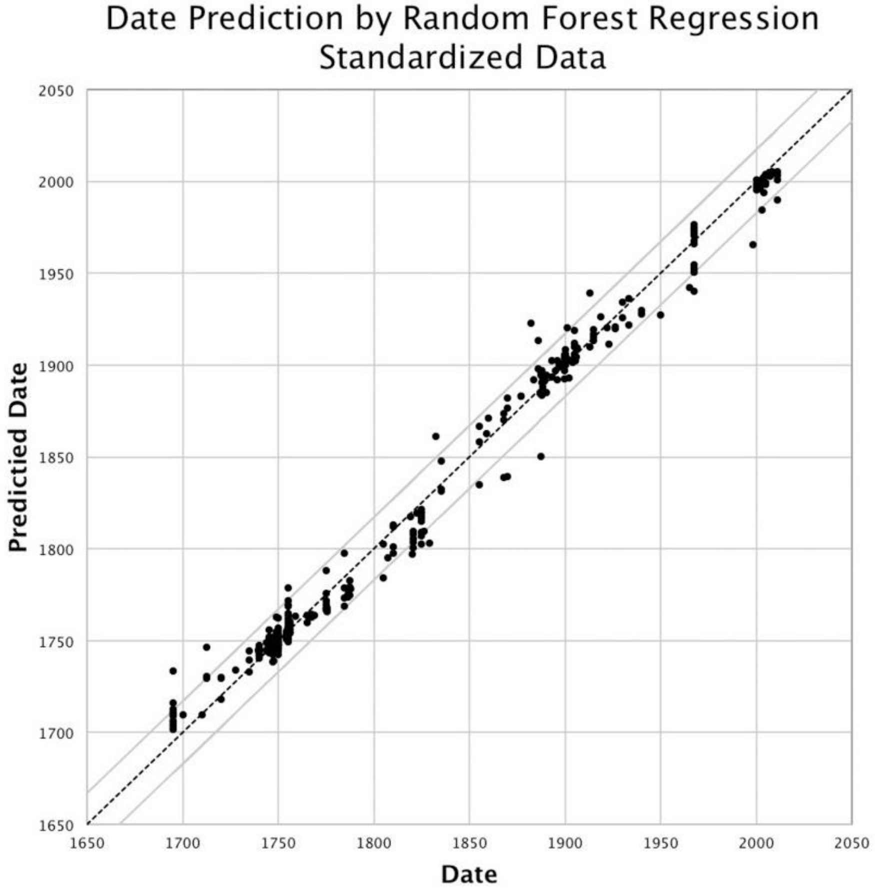
Heginbotham, 2013; Heginbotham et al., 2017

Example: Quantitative analysis of Gilt Bronzes

Taking the data further – predictive dating

Machine learning

- Learning data set of 466 analyses of gilt bronzes
  - Provenance and age well known (±10 yrs)
  - Made in Paris, late 17th to late 20thC
  - Compositions calculated from CHARM-based calibration using PyMCA
  - Cu, Zn, Sn, Pb + Mn Fe, Co, Ni, As, Ag, Cd, Sn, Sb, Pn, Bi
- 2 approaches
  - Support Vector Regression (SVR)
  - Random Forest Regression (RFR)
- *Resulting predictive model: ± 37 yrs*



Heginbotham et al., 2018

Example: Quantitative analysis of Gilt Bronzes

Application study by Dr. Arlen Heginbotham, J. Paul Getty Museum

Some example of dating the bronzes

Bureau Plat by Joseph Baumhauer
1745-49?

1755±37

Original…

Example: Quantitative analysis of Gilt Bronzes

Application study by Dr. Arlen Heginbotham, J. Paul Getty Museum

Some example of dating the bronzes

Pair of wall lights, Paris
1745-49?

1900±37

Copy…

Heginbotham *et al.*, 2018

Example: Quantitative analysis of Gilt Bronzes

Application study by Dr. Arlen Heginbotham, J. Paul Getty Museum

Some example of dating the bronzes

Commode, stamped Delorme
1755?

Purchased by J. Paul Getty 1938

1937±37

<u>Fake!!</u>

Fabricated using pieces of an 18th C commode stamped by Delorme, Bronzes made new in early 20th C

Heginbotham et al., 2018

Summary

- The TRACER represents the benchmark in handheld-XRF for Cultural Heritage studies
- The instrument is reliable, fast, and non-invasive, and produces robust compositional data for a variety of applications
- It is clear from community use of this instrument that data are useful at the level of a raw spectrum, all the way through to more advanced data modeling
- If you have any questions about the TRACER, or any instruments in the Bruker range, don't hesitate to reach out to us or your local Bruker representative

Questions, Thoughts or Comments?

If you have questions please **type your questions**, thoughts, or comments in the **Q&A box** and **press Submit**.

We ask for your understanding if we do not have time to discuss all comments and questions within the session. Any unanswered questions or comments will be answered and discussed by e-mail or in another Webex session.

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