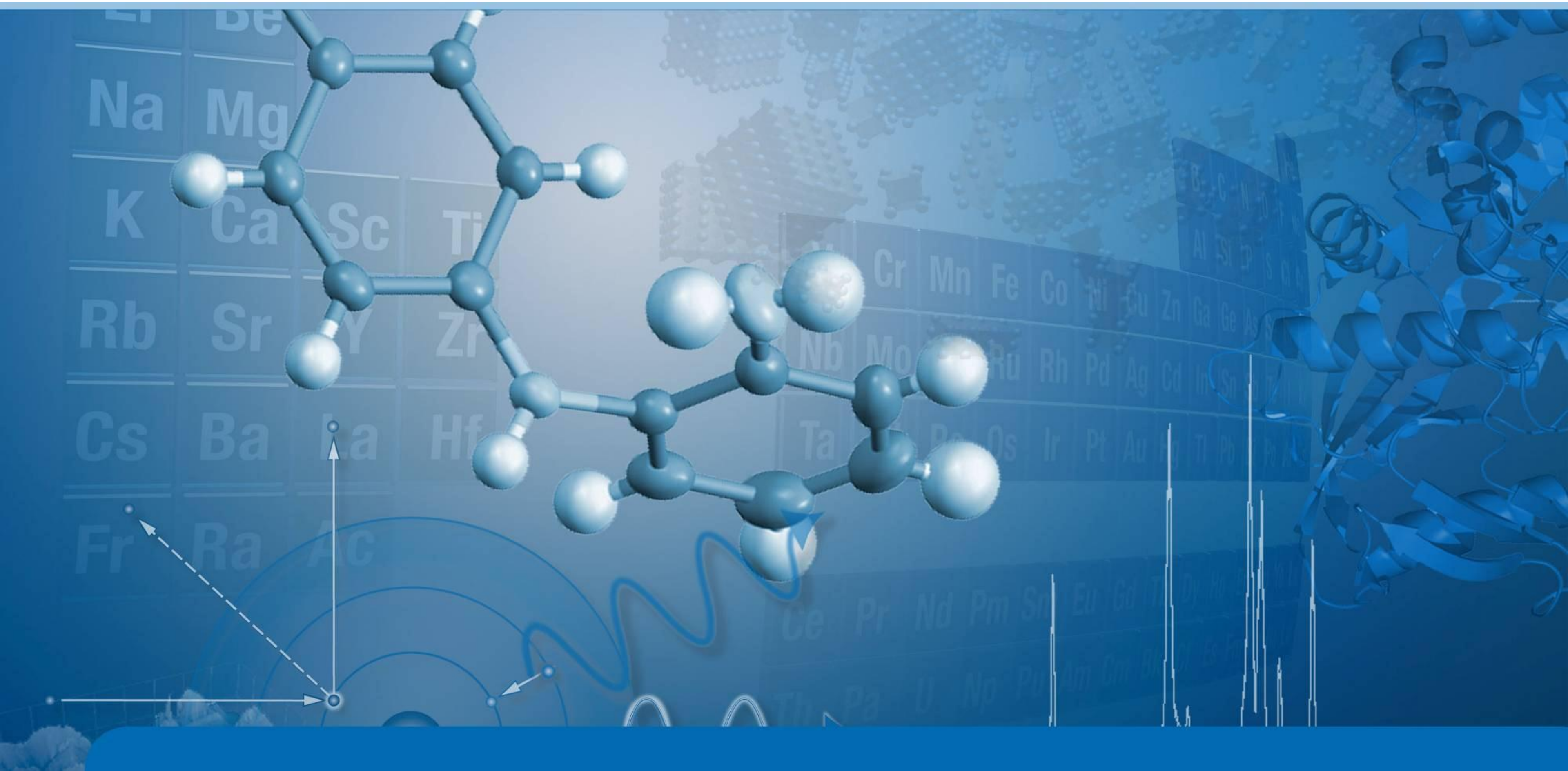
Good Diffraction Practice Webinar Series

LYNXEYE XE - Combining 1D Speed with 0D Background

June 27 2013

www.bruker-webinars.com



Welcome

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Topics

Introduction

- Monochromatization
  - Common artifacts in PSD data
- Silicon strip detectors
  - How do they work
  - Energy resolution
  - Feature comparison LYNXEYE and LYNXEYE XE

Application examples

- Mineral analysis
  - Fluorescence suppression in Iron containing samples
  - Low angle performance in clays
- Steel samples
  - Retained Austenite
  - Corrosion
- Battery materials
  - Fluorescence suppression for Co and Mn

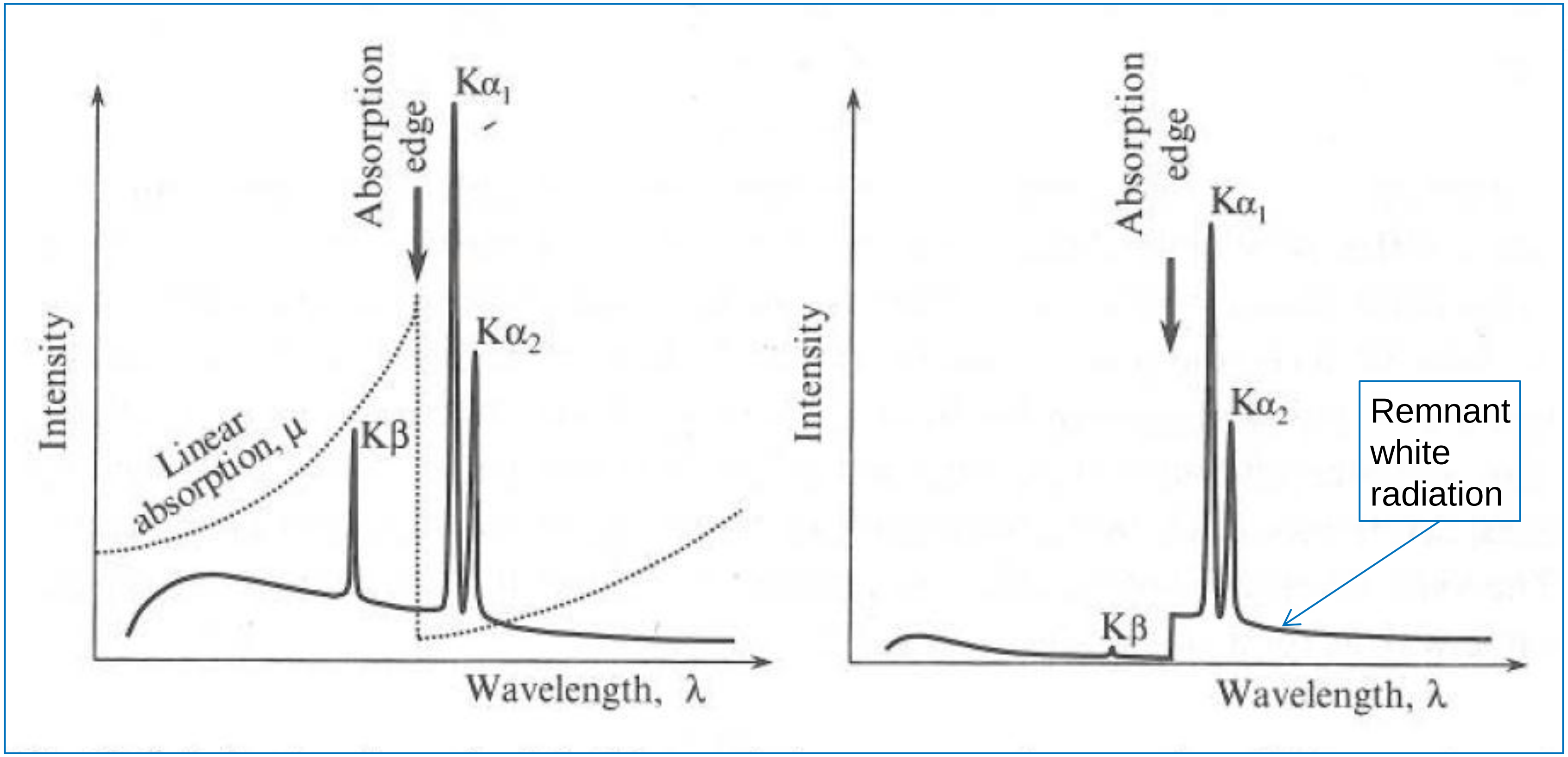
Introduction
Monochromatization

- Before 2000, >90% of all instruments have been equipped with point detectors, > 50% with (secondary) monochromators
  - Intensity loss with monochromator ~70 % with respect to unfiltered radiation

- Today, >90% of all instruments are equipped with a position sensitive detector (PSD), the majority with Kß-Filter
  - Intensity loss ~40-60% with respect to unfiltered radiation
  - Absorption edges, Kß level 0.5-1.5%
  - Poor filtering of fluorescence

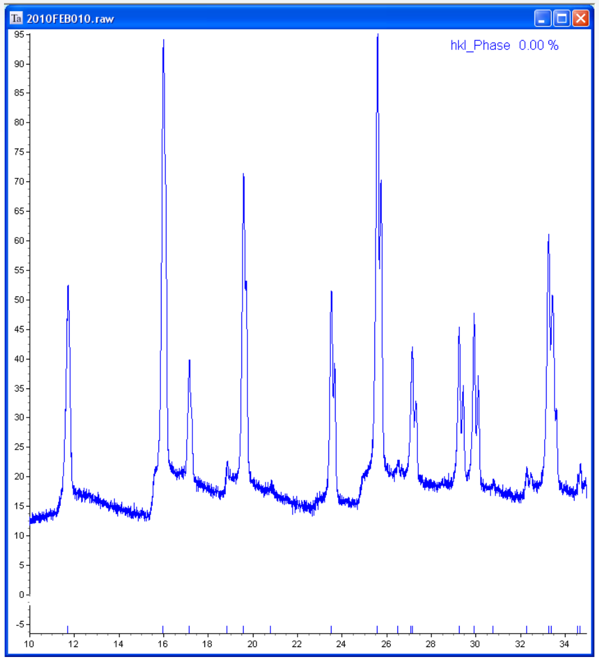
- Depending on the comparison an energy dispersive detector can improve efficiency at least 2 times and reduce errors due to absorption edges and fluorescence

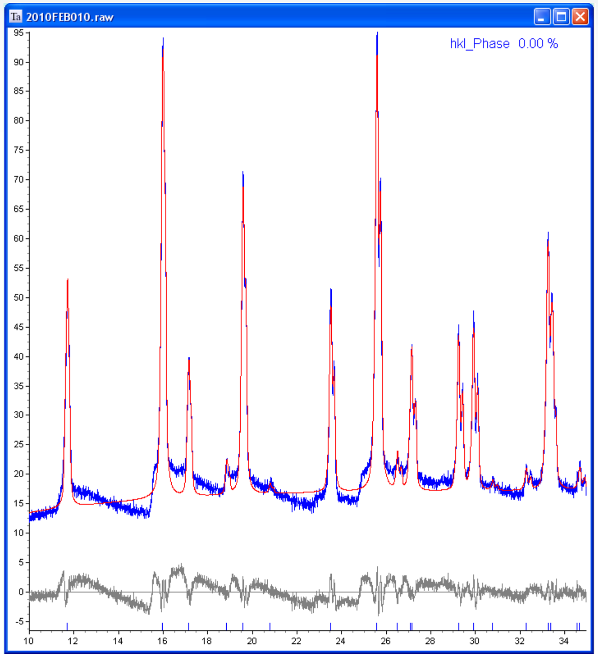
Monochromatization
Effect of Kß-Filter and artifacts



Pecharsky & Zavalij (2009)

Monochromatization
Effect of Kß-Filter





- Kß filters do leave some severe artefacts
  - Absorption edges
  - Kß level 0.5-1.5%
  - Poor filtering of fluorescense
- Severe absorption edge issues with any line profile fitting applications: Goodness-of-fit is poor as background function can’t cope

Introduction
Monochromatization (Cu)

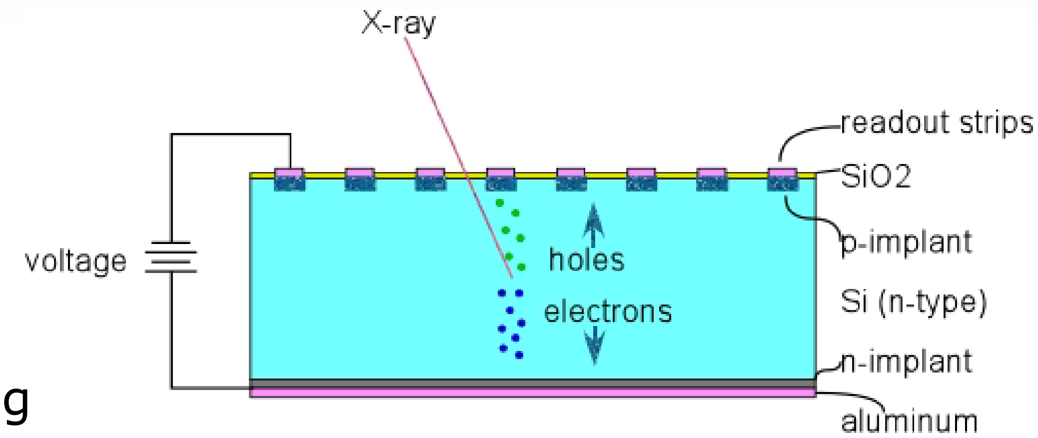
- Kß-Filters
  - Intensity loss ~40-60% with respect to unfiltered radiation
  - Absorption edges, Kß level 0.5-1.5%
  - Poor filtering of fluorescense
- Monochromators
  - Prim. : Intensity loss ~80-90% with respect to unfiltered rad.
  - Sec. 0D : Intensity loss ~70% with respect to unfiltered rad.
  - Sec. 1D : Intensity loss >90% with respect to unfiltered rad.
  - No absorption edges, no Kß
  - Excellent filtering of fluorescense (secondary monochromators)!
- Si(Li) detectors (all wavelengths!)
  - No intensity loss, best peak to background
  - No absorption edges, no Kß
  - Excellent filtering of fluorescense
  - Low linear range, low count rates, life time issues
  - Only 0D!

Introduction
Monochromatization

- ***Energy resolution*** is the ability of a detector to resolve two photons of differing energy
- The ***proportionality*** of a detector determines how the size of the generated voltage pulse is related to the energy of the absorbed X-ray photons
- Electronic methods (so-called pulse-height selection or pulse height discrimination) can be used to discriminate between different energies. Signals corresponding to photons with too high or to low energies are discarded.
- A high proportionality thus allows to apply ***energy discrimination*** as a form of monochromatization, where the energy is filtered by the detector rather than by X-ray optics
- Energy resolution is frequently defined as an energy window determined by the full width at half maximum (FWHM) of the so-called detector efficiency curve, specified for a specific wavelength

Introduction
Silicon Strip Sensor Principle

- Electron-hole pairs created in depleted silicon by X-ray photoionization
  - Charge carriers drift to readout strips
- Key advantages High counting rate
  - Typically of order 10^6 counts/strip-sec
- Good spatial resolution
- Good energy resolution compared to other detectors
  - Requires optimized readout



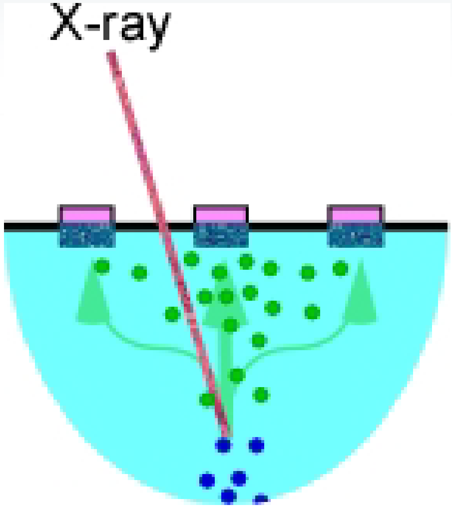
Introduction
Detector efficiency

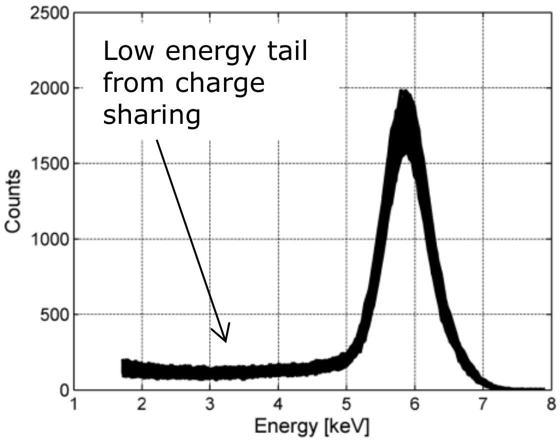
Wavelength	Linear Absorption coefficient for Si (cm^{-1})	Efficiency 300µm sensor	Efficiency 500µm sensor
Cr	439.3	>99%	>99%
Co	216.4	>99%	>99%
Cu	139.4	>98%	>99%
Mo	14.25	~35%	~50%
Ag	7.09	~20%	~30%

LYNXEYE detectors with higher sensor thickness are available for better efficiencies with harder radiation sources (Mo and Ag)

Introduction
Limitations to resolution due to charge sharing effects

- ***Energy resolution*** is accomplished by counting the electron in a strip or pixel
- $\Delta E \geq 2.35\sqrt{\frac{F}{N}}$
- F=Fano factor, N=# of electron-hole pairs
- Problem, ***not all electrons are collected in a given strip (or pixel)***, in general some electrons will diffuse to neighboring strips or pixels: “charge sharing”
- Fall below discriminator level and are thus “lost”
- Lost electrons cause poor energy resolution





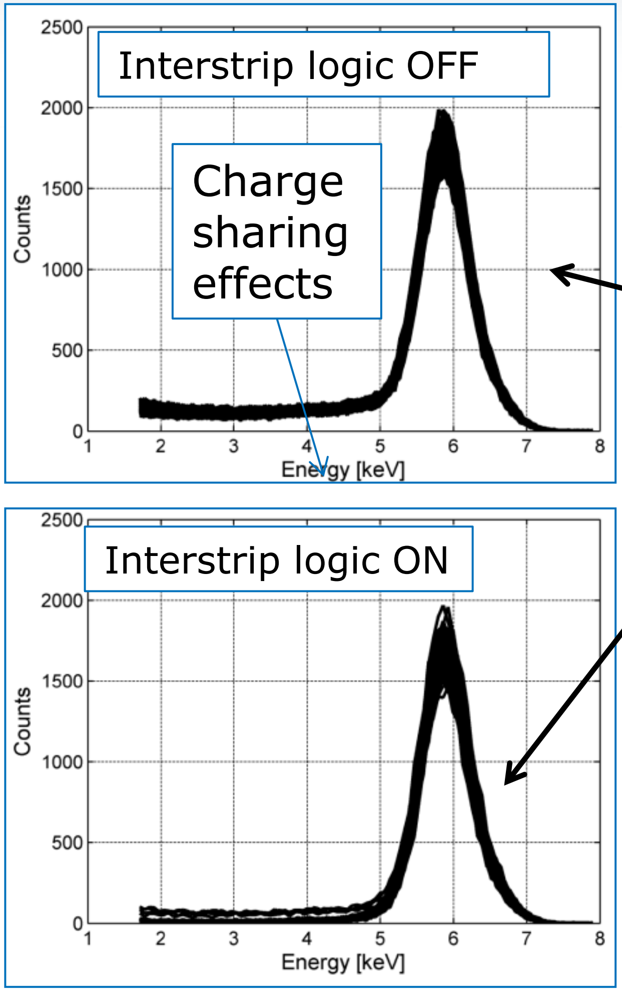
LYNXEYE XE
Technical Data

- New front-end ASIC design (96 channels) with novel interstrip logic circuitry
  - Rejects events resulting in significant charge sharing between neighboring strips
  - Single strip energy spectra almost free of charge sharing effects

- Three independent discriminators in each channel. The discriminators LOW and HIGH are used to set the energy window. The additional discriminator VETO is used to detect and reject events resulting in charge sharing between adjacent strips and minimal intensity losses.

- Highly efficient discriminator threshold trimming to minimize channel-to-channel spread of ASIC parameters
  - The energy resolution of the whole detector is essentially as good as that of a single channel

LYNXEYE XE and Standard LYNXEYE
The Energy Resolution Difference



Device	Resolution (KeV)
Scintillation counter	~3.5
Traditional silicon strip / pixel	~1.6 - 2.0
Proportional counter	~1.1 - 1.6
LYNXEYE XE	**<0.68**
Graphite monochromator	~0.5 - 1.0
Si(Li) detector (Peltier cooled)	<0.2

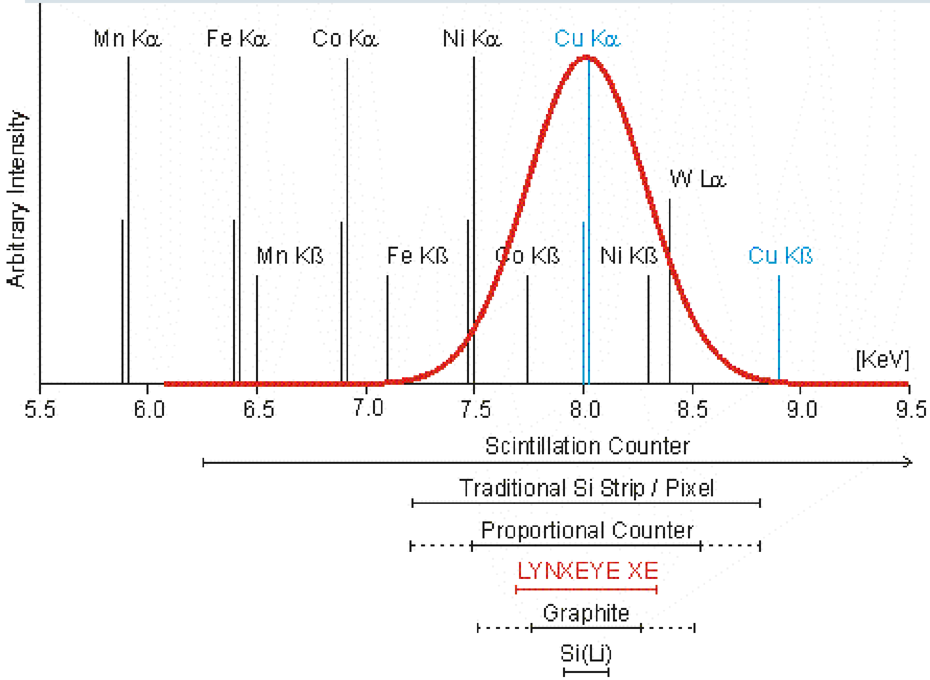
Incorporation in inter-strip logic improves energy resolution 2-3 times compared to conventional Si detector
Comparable to Graphite monochromator

Comparison of Energy Resolution

LYNXEYE XE and Standard LYNXEYE

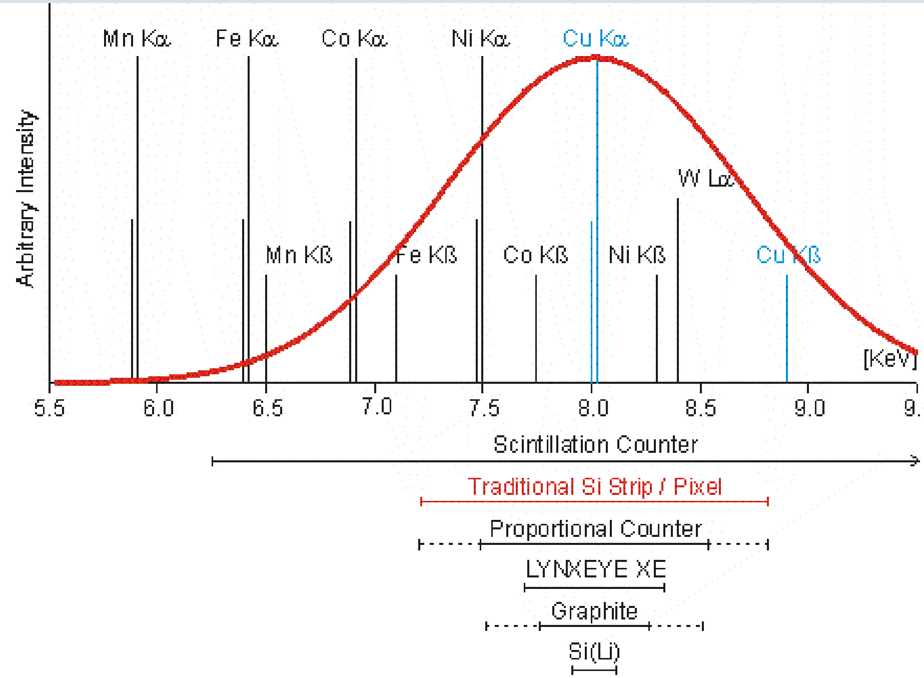
LYNXEYE XE (FWHM <680ev FWHM)

(only available for D8 Advance and Discover)



Standard LYNXEYE detector

(available for D2 Phaser, D4 Endeavor, D8 Advance, D8 Discover)



Cu Kβ gets mostly eliminated (>98%)
No Iron and Manganese fluorescence

Ni filter required
Iron and Manganese fluorescence gets suppressed only partially

Comparison of Technical Data
LYNXEYE XE and Standard LYNXEYE

Detector type	LYNXEYE XE	LYNXEYE
# strips	192 strips, 75 μm pitch	192 strips, 75 μm pitch
Active area	14.4 x 16mm	14.4 x 16mm
Modes	1D and 0D	1D and 0D
Wavelengths	Cr, Co, Cu, Mo, and Ag	Cr, Co, Cu, Mo, and Ag
Maximum Global count rate	> 100,000,000cps	> 100,000,000cps
Energy resolution (Cu)	< 680 eV @ 8 KeV	>1600eV @ 8 KeV
Component recognition	Fully integrated, including Soller slits and/or filter	Detector recognition on D8 DaVinci system
Controller	No external controller	External 19” rack controller
Maintenance	No maintenance, gases or cooling	No maintenance, gases or cooling

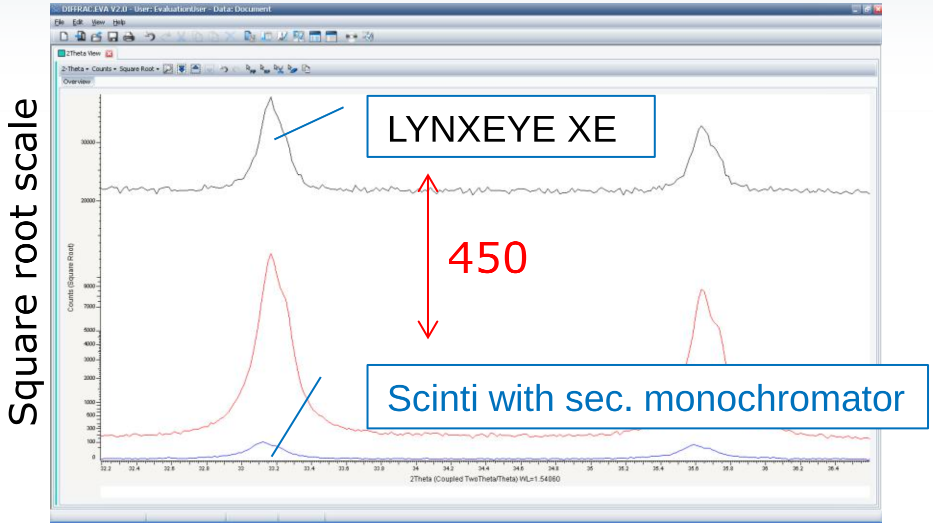
LYNXEYE

LYNXEYE XE

LYNXEYE XE
Speed Advantage

- *"High-speed data acquisition up to 450 times faster than a conventional point detector system (point detector with secondary monochromator)"*

⇨ See LYNXEYE XE Flyer (DOC-H88-EXS055)



Maths:

- Intensity loss secondary monochromator ~70%
- Intensity loss LYNXEYE XE Kß / fluorescence filtering ~20-25%

Factor ~3

- "LYNXEYE is 150 times faster than a point detector system"

3 x 150 = 450

⇨ Confirmed empirically by the data shown in the LYNXEYE XE flyer

Software Integration

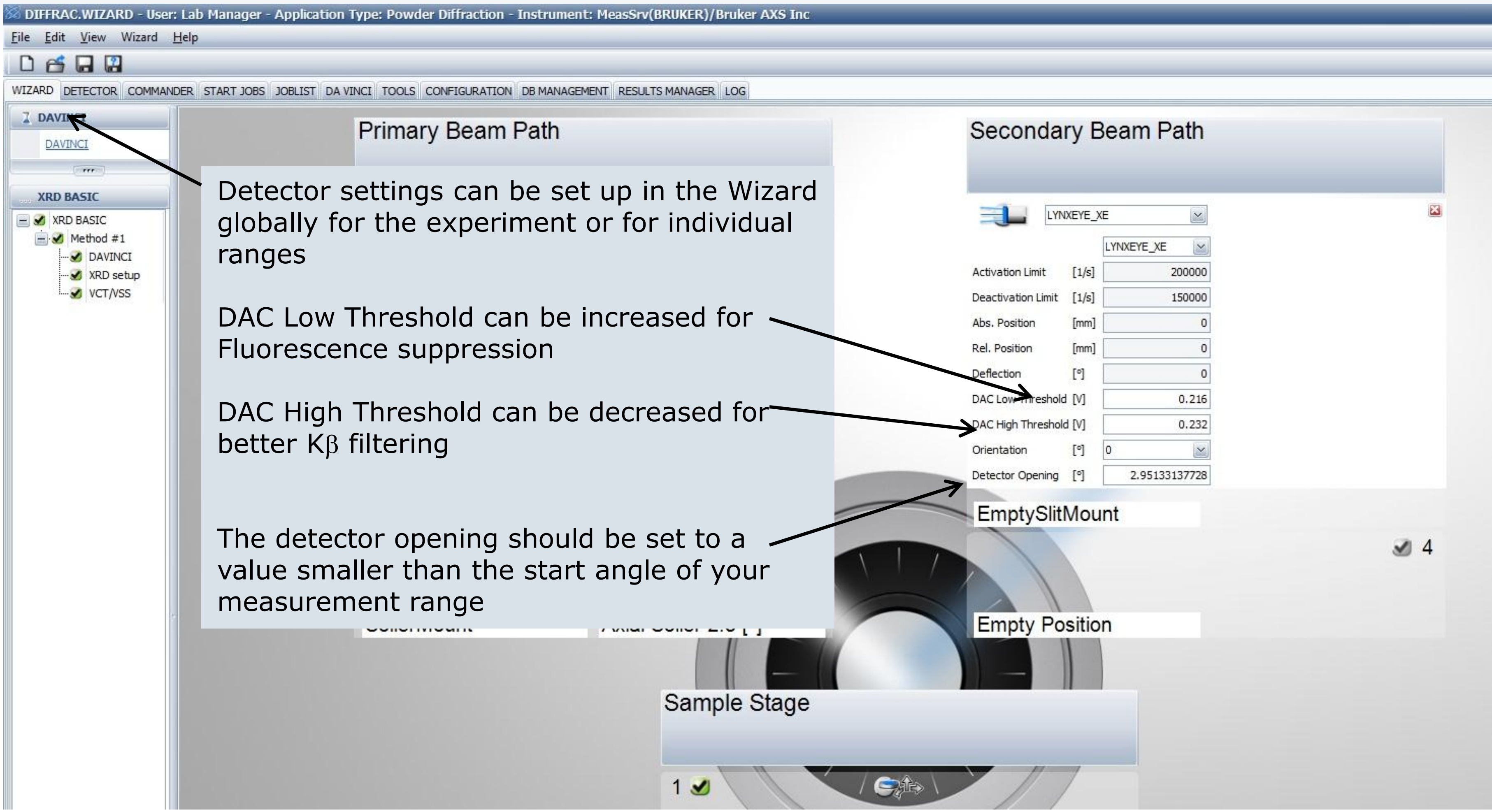
Setting LYNXEYE Detector Opening and Discriminator Settings

Detector settings can be set up in the Wizard globally for the experiment or for individual ranges

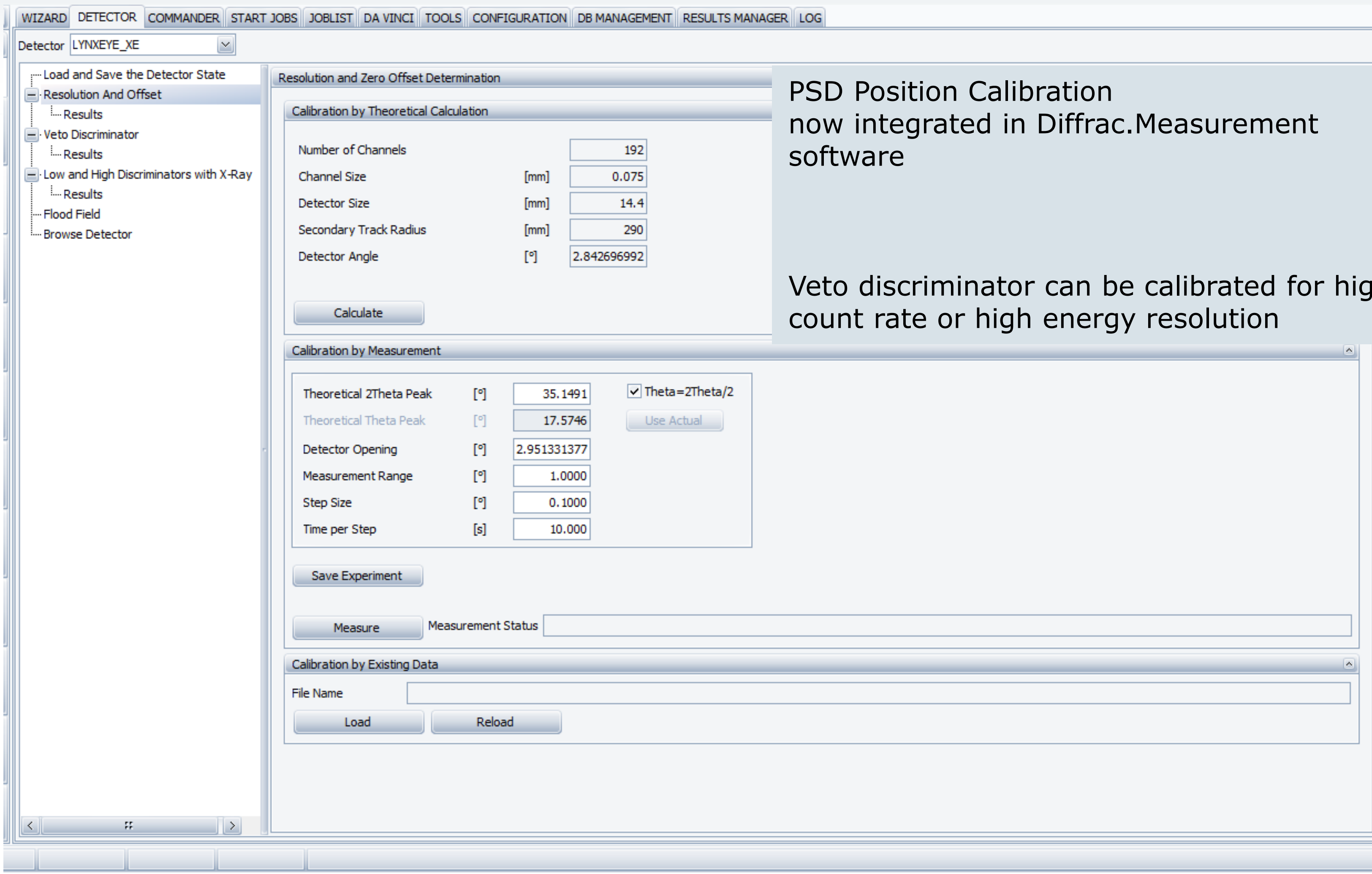
DAC Low Threshold can be increased for Fluorescence suppression

DAC High Threshold can be decreased for better Kβ filtering

The detector opening should be set to a value smaller than the start angle of your measurement range



Software Integration
Detector Plugin in Diffrac.Measurement



PSD Position Calibration
now integrated in Diffrac.Measurement
software

Veto discriminator can be calibrated for high count rate or high energy resolution

LYNXEYE Features
Operation Modi

Operation Mode	Wavelength	Count rate	Application
High Gain/High resolution (Energy)	Cr to Cu radiation	Up to 10kps	Fluorescence and Kβ suppression
High Gain/High Count rate	Cr to Cu radiation	Up to 100kps	Limited Fluorescence suppression
Low Gain/High Resolution	Mo	Up to 10kps	Fluorescence and Kβ suppression
Low Gain/High count rate	Mo	Up to 100kps	Limited Fluorescence suppression

Minimum Energy Resolution	
High Energy Resolution mode:	< 680eV strip (typically 640eV, nearly energy independent)
High Count rate mode:	>1000eV strip (nearly energy independent)

Maximum Count Rate	
High Energy Resolution mode:	ca. 20kcps / strip. Deviation from a straight line ca. 10% at 10kcps.
High Count rate mode:	ca. 500kcps / strip. Deviation from a straight line ca. 10% at 200kcps.

Topics

Introduction

- Monochromatization
  - Common artifacts in PSD data
- Silicon strip detectors
  - How do they work
  - Energy resolution
  - Feature comparison LYNXEYE and LYNXEYE XE

Application examples

- Standard materials SRM1976
- Mineral analysis
  - Fluorescence suppression in Iron, Cobalt or Manganese containing samples
  - Low angle performance in clays
- Steel samples
  - Retained Austenite
  - Corrosion
- Battery materials
  - Fluorescence suppression for Co and Mn

Audience Poll

Please use your mouse to answer the question in the poll window on your screen.

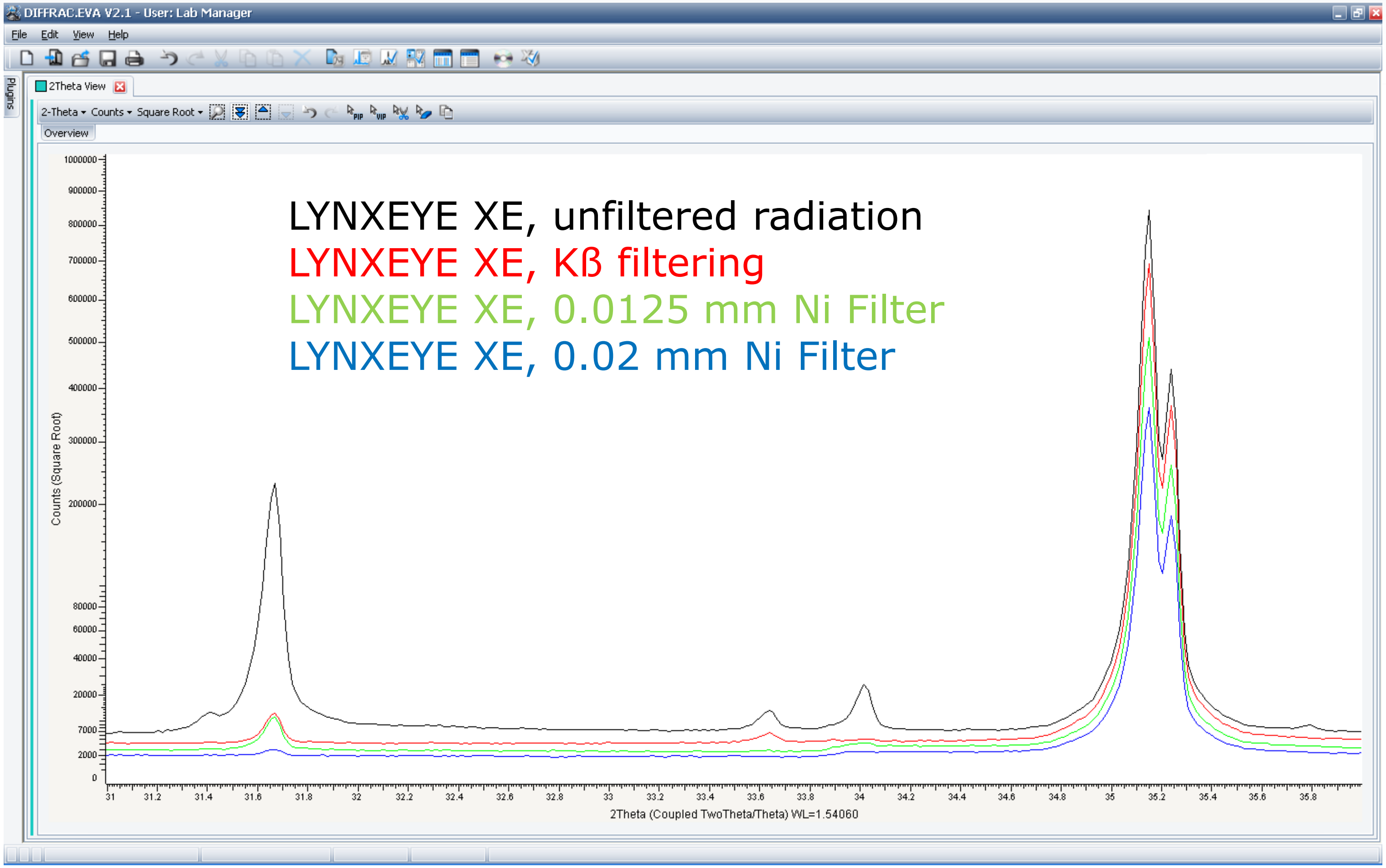
What types of materials do you study where sample fluorescence is an issue? (Select all that apply.)

a. Geological materials
b. Metal materials
c. Battery materials
d. Rare Earth materials
e. Gems and other minerals
f. Other materials

We’ll share the poll results in a few minutes.

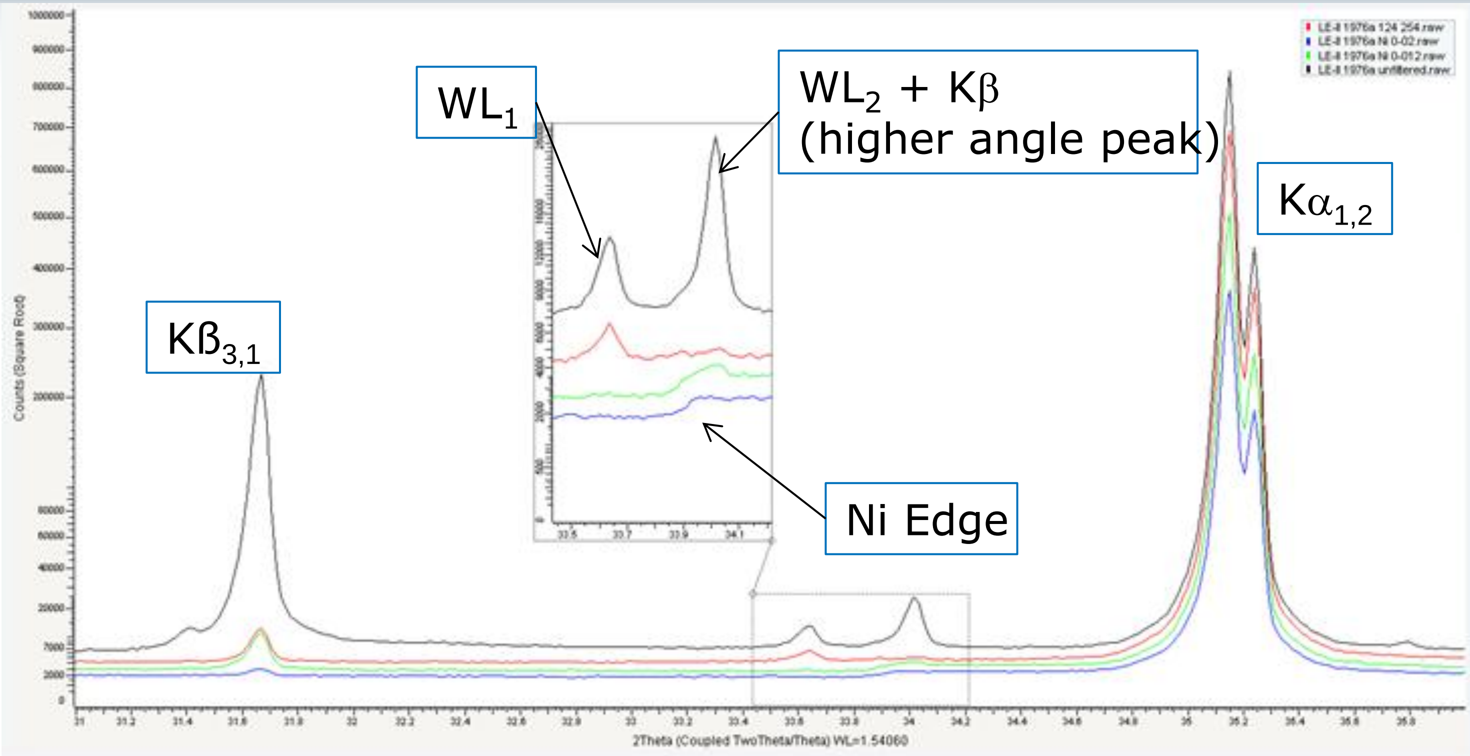
LYNXEYE XE
NIST SRM 1976a (Corundum)

Square root scale to highlight details



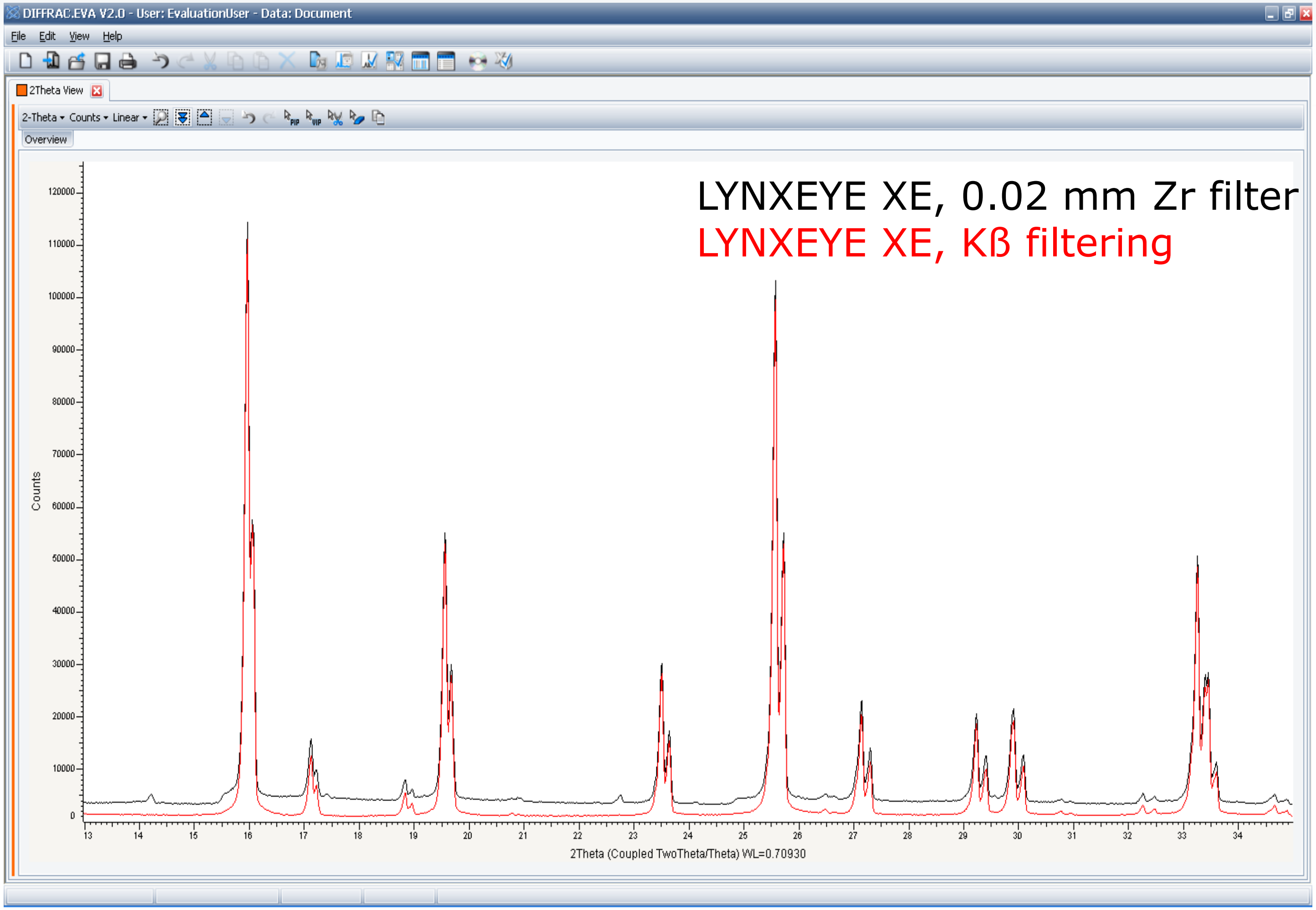
LYNXEYE XE
NIST SRM 1976a (Corundum)

Square root scale to highlight details

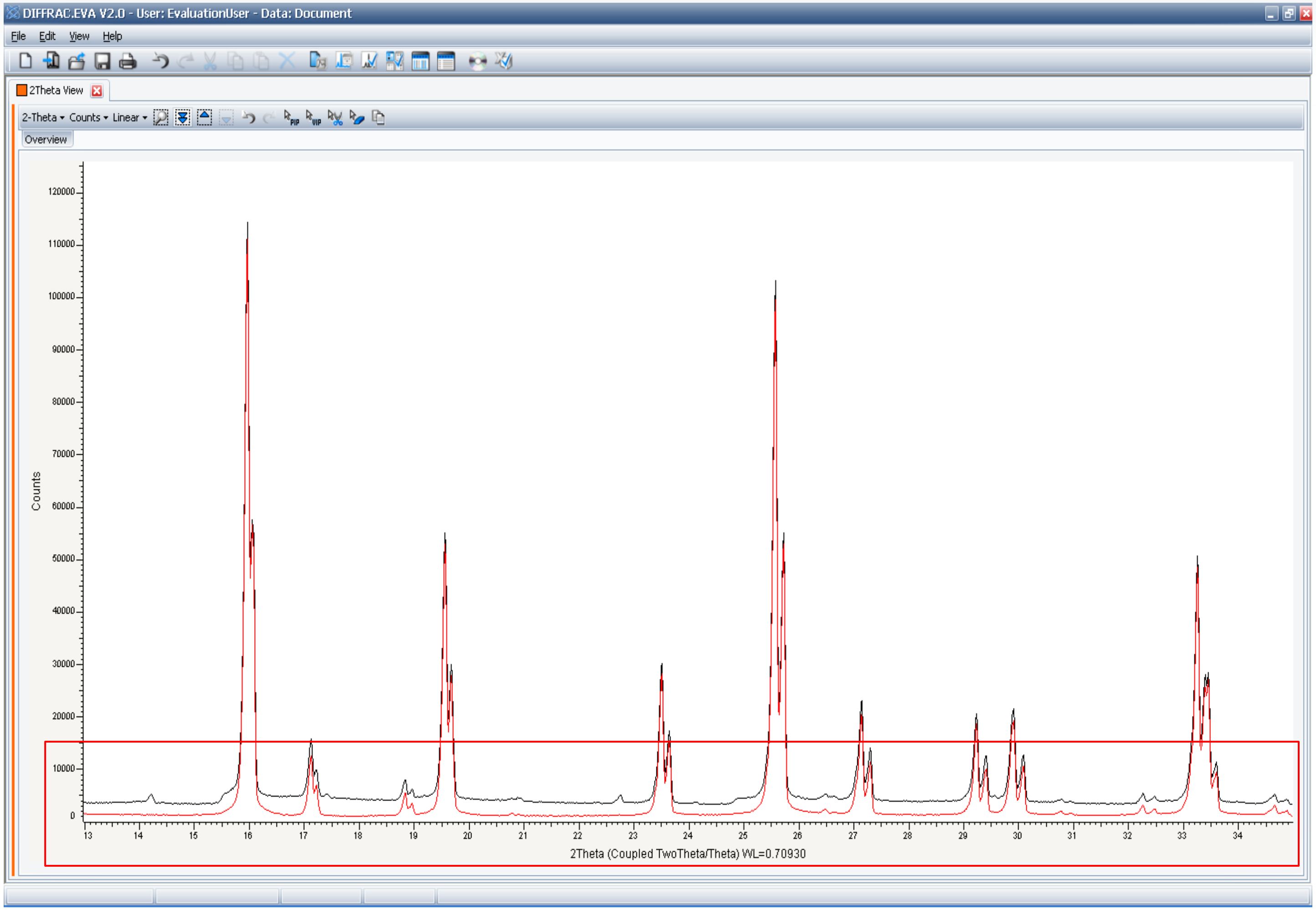


Device	Remnant Kß	Intensity loss
LYNXEYE XE, Kß filtering	0.8%	~20%
LYNXEYE XE, 0.0125 mm Ni Filter	1.2%	~40%
LYNXEYE XE, 0.02 mm Ni Filter	0.3%	~60%

LYNXEYE XE
NIST SRM 1976a, Mo radiation

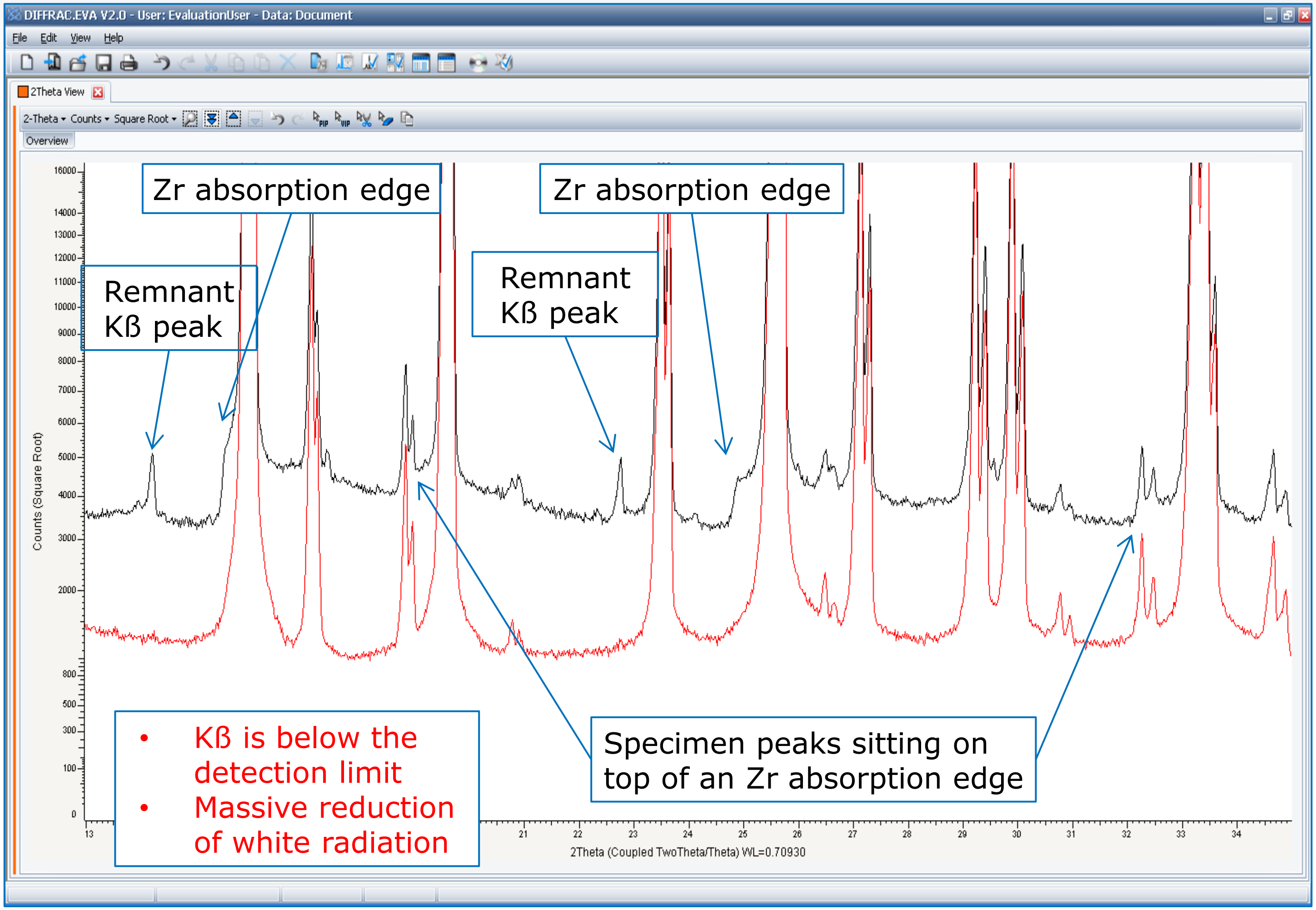


LYNXEYE XE
NIST SRM 1976a, Mo radiation

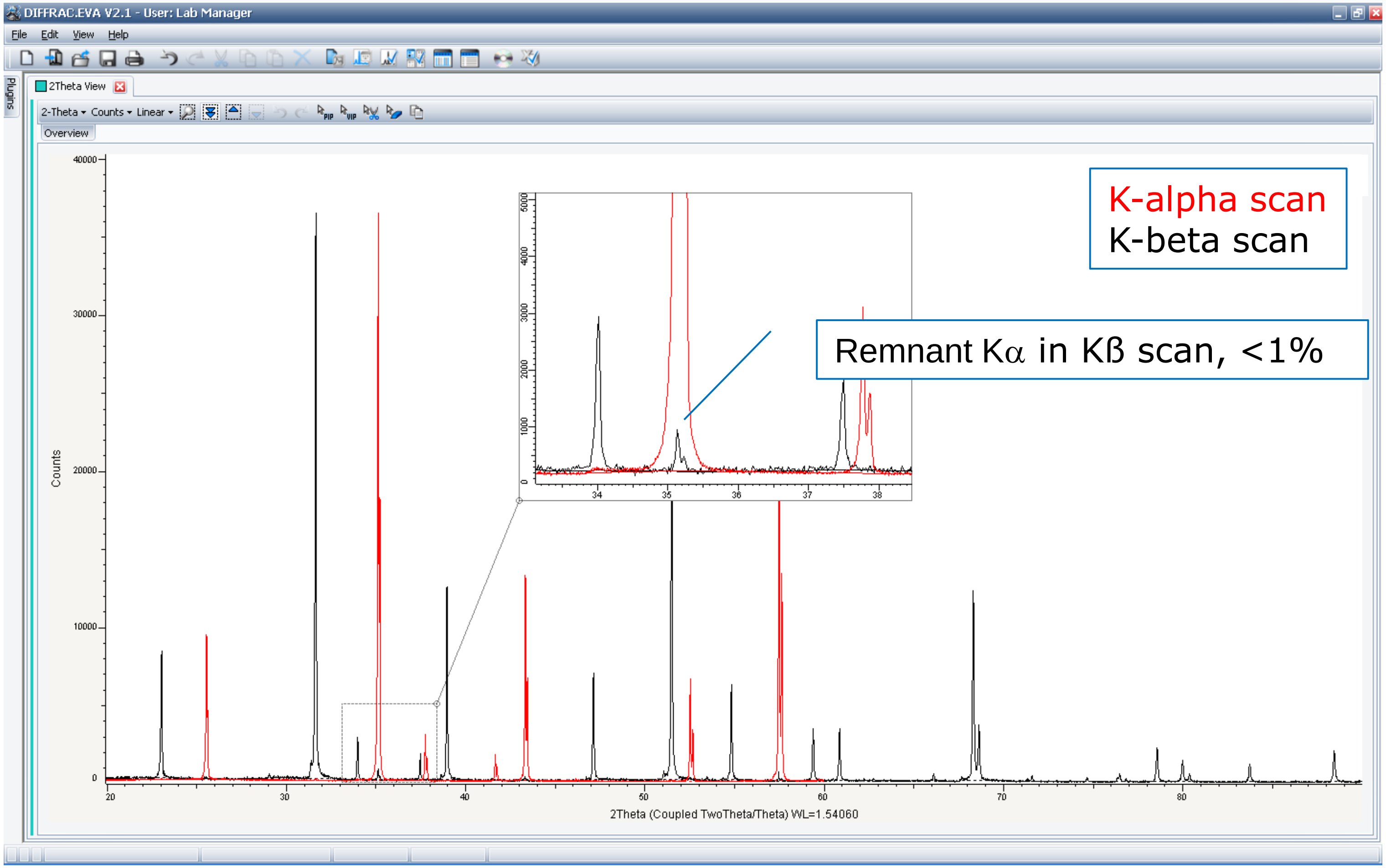


LYNXEYE XE
NIST SRM 1976a, Mo radiation

Square root scale to highlight details



LYNXEYE XE
NIST SRM 1976a, Cu radiation

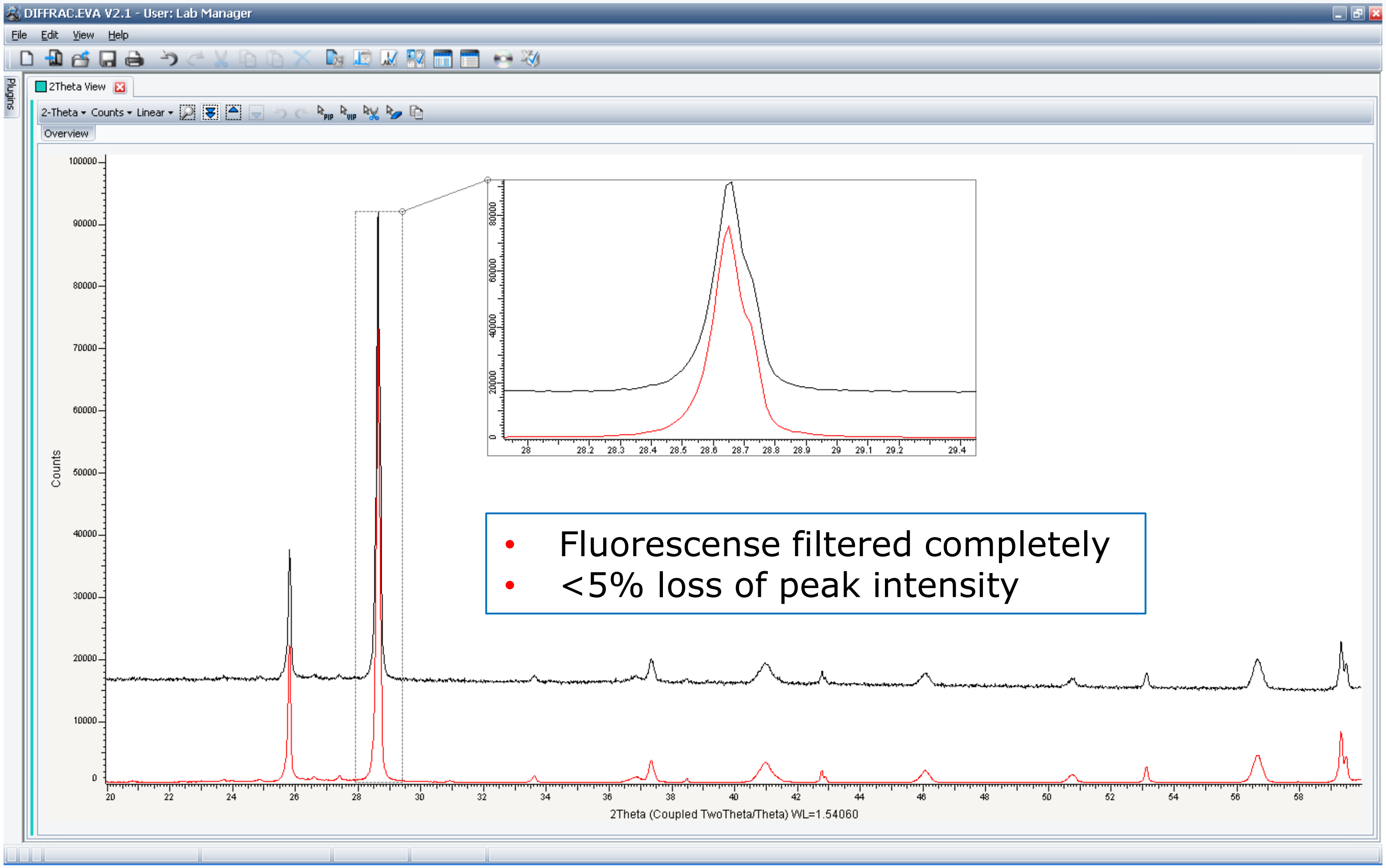


Poll Results

What types of materials do you study where sample fluorescence is an issue? (Select all that apply.)

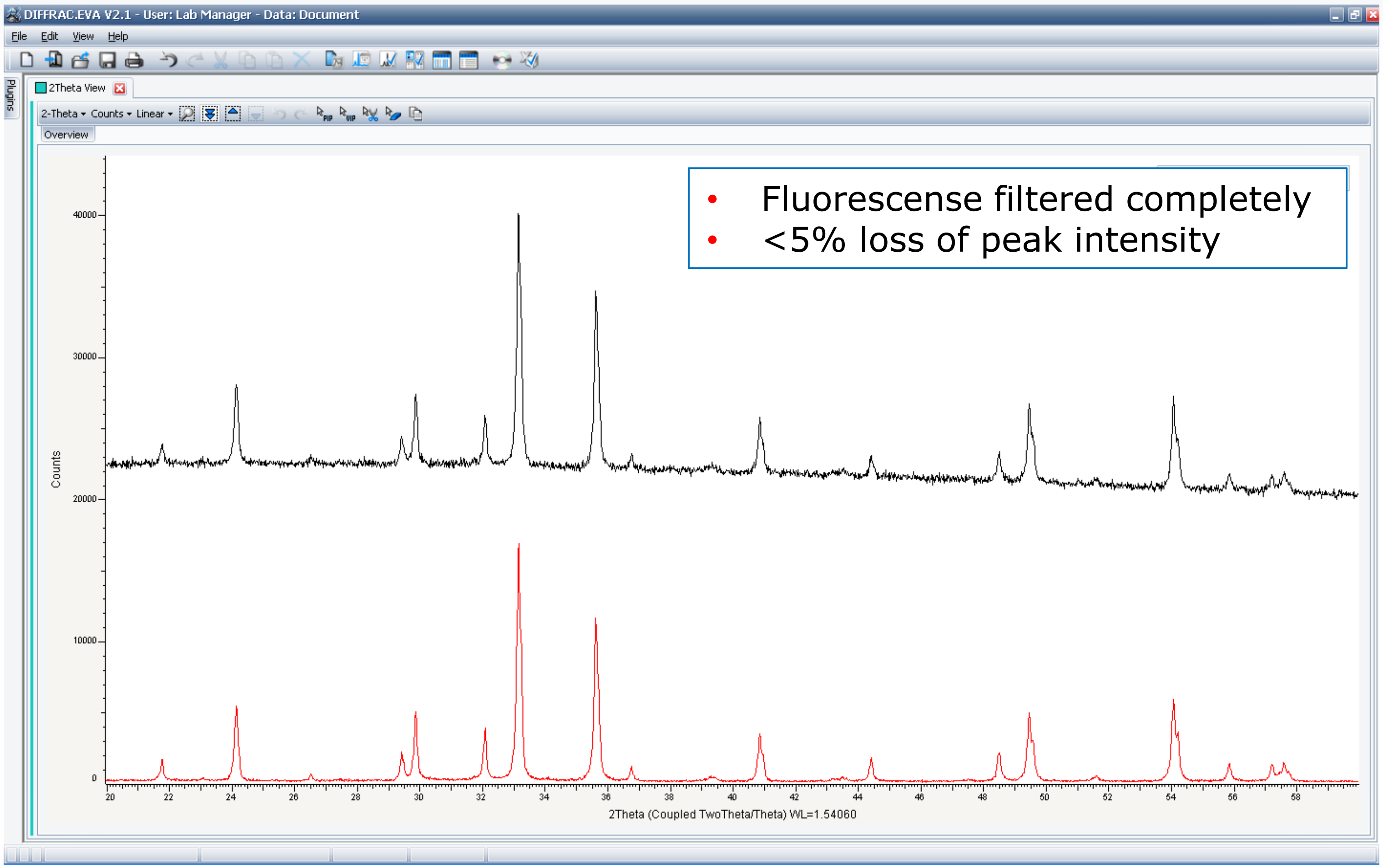
a. Geological materials
b. Metal materials
c. Battery materials
d. Rare Earth materials
e. Gems and other minerals
f. Other materials

LYNXEYE XE
Manganese Ore

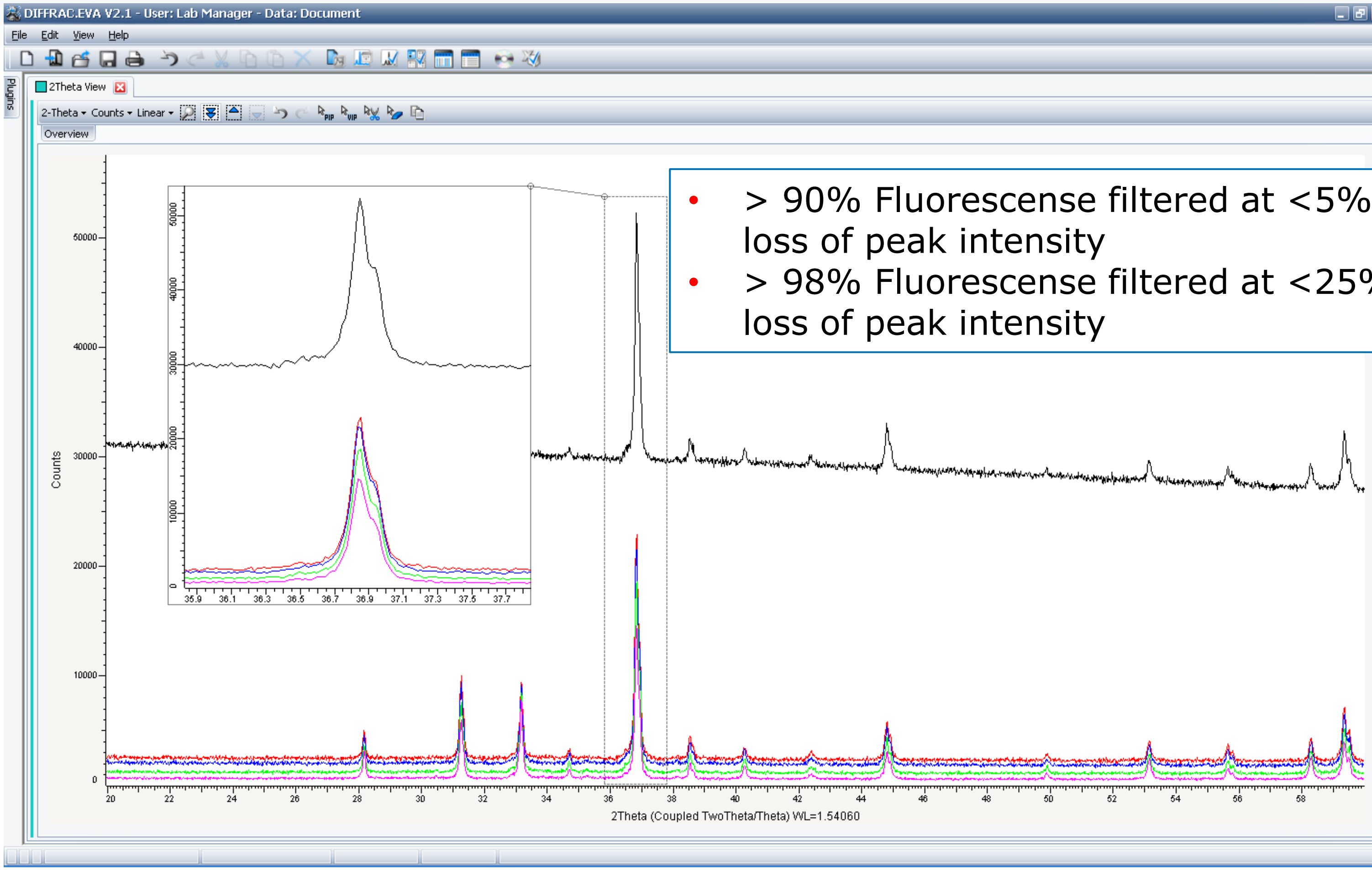


- Fluorescense filtered completely
- <5% loss of peak intensity

LYNXEYE XE
Iron Ore

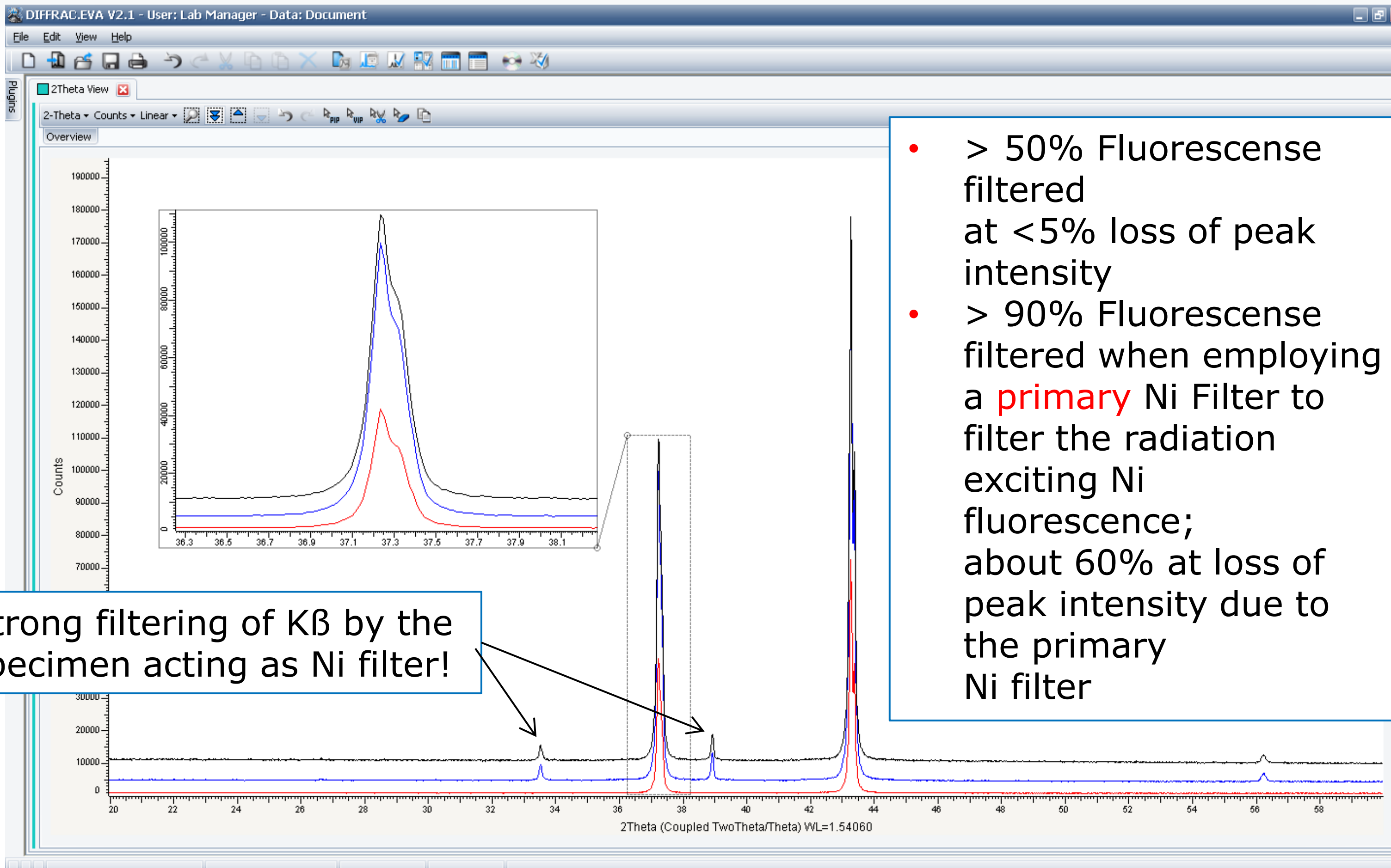


LYNXEYE XE
Cobalt Ore



- > 90% Fluorescense filtered at <5% loss of peak intensity
- > 98% Fluorescense filtered at <25% loss of peak intensity

LYNXEYE XE
Nickel Ore



Strong filtering of Kß by the specimen acting as Ni filter!

- > 50% Fluorescense filtered at <5% loss of peak intensity
- > 90% Fluorescense filtered when employing a primary Ni Filter to filter the radiation exciting Ni fluorescence; about 60% at loss of peak intensity due to the primary Ni filter

Filtering of Fluorescence with Copper radiation Summary

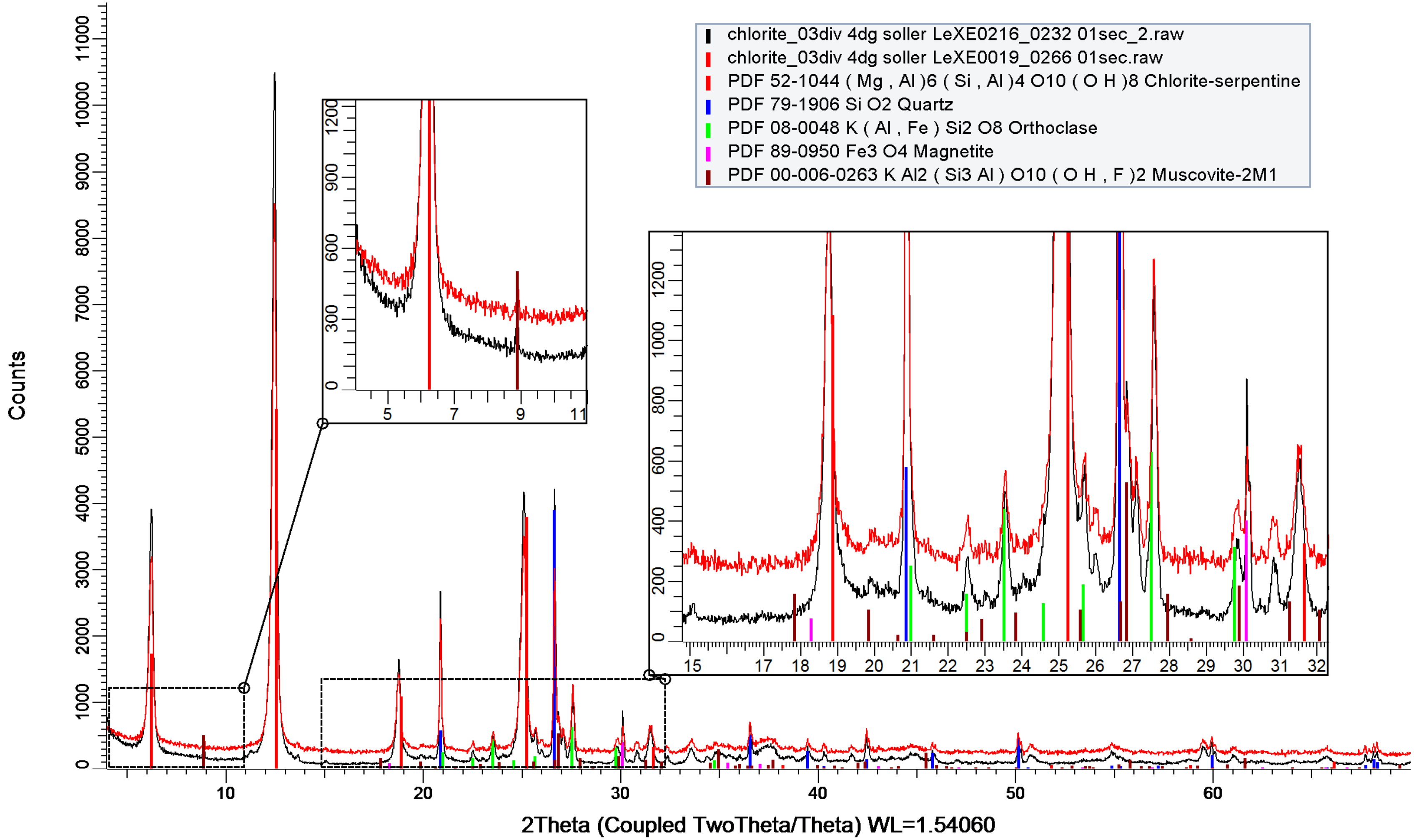
Manganese:
Fluorescence filtered completely at <5% loss of peak intensity

Iron:
Fluorescence filtered completely at <5% loss of peak intensity

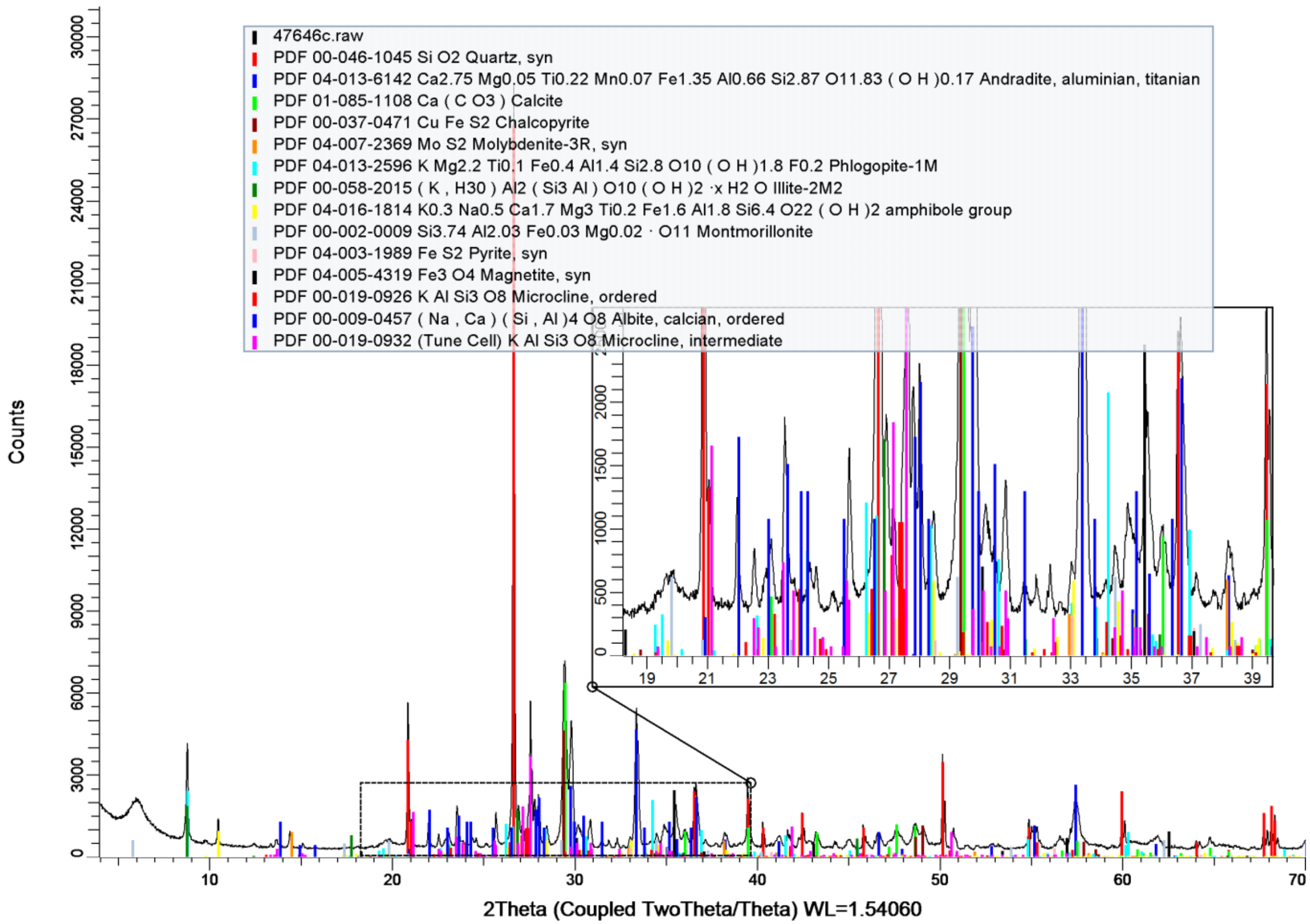
Cobalt:
> 90% Fluorescence filtered at loss of <5% peak intensity
> 98% Fluorescence filtered at loss of <25% peak intensity

Nickel:
> 50% Fluorescence filtered at loss of <5% peak intensity
> 90% Fluorescence filtered at a loss of ~ 60% peak intensity using a primary Ni filter

Comparison Standard LYNXEYE and LYNXEYE XE Chlorite Sample



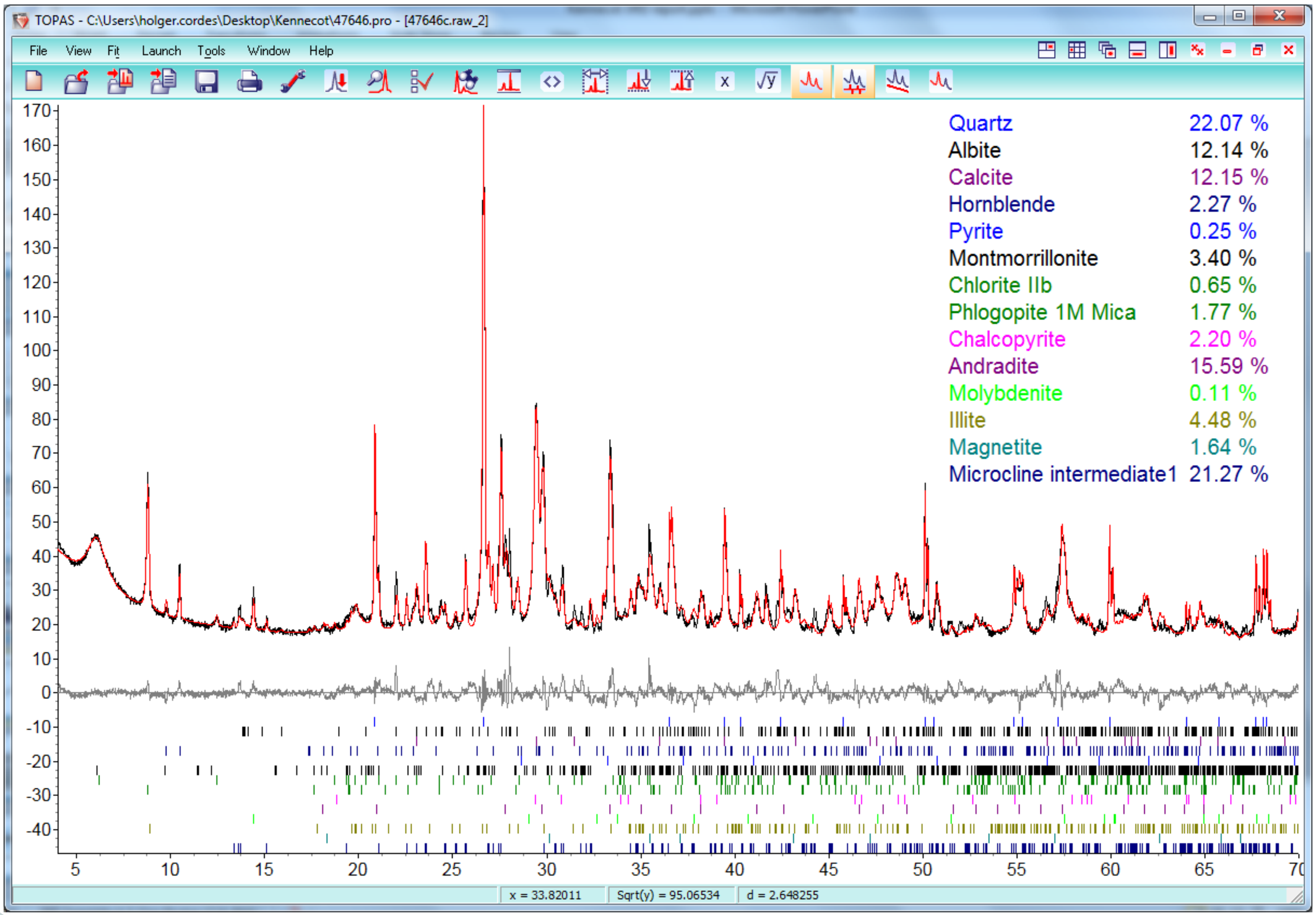
Mineral Sample
Phase identification with Diffrac.Eva



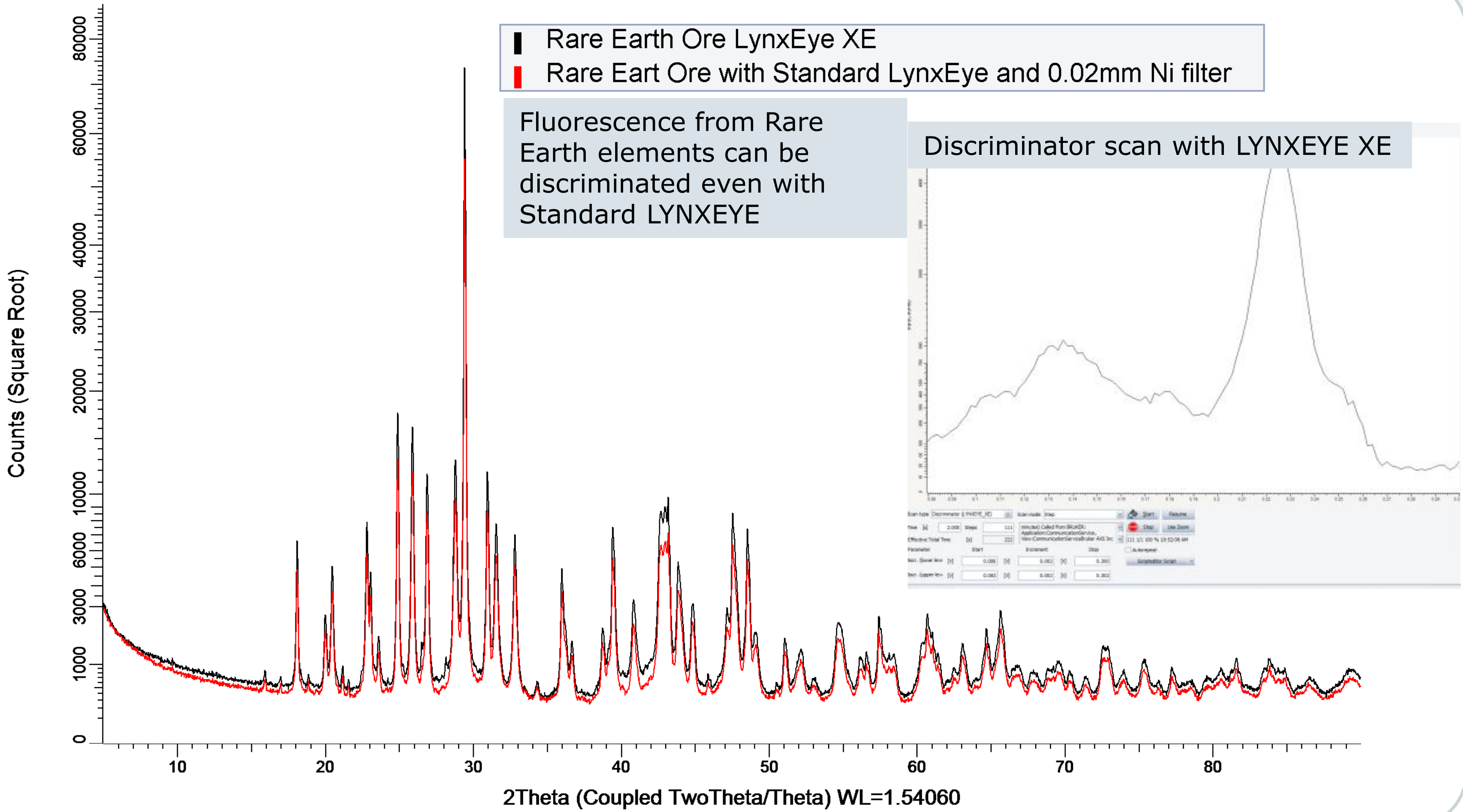
Configuration:

- D8 ADVANCE
- Cu radiation with 40kV, 40mA
- 0.3° divergence slit
- 0.015° steps,
- 1 sec/step
- Front loaded sample holder
- Autochanger
- 2.5° Soller slits
- LYNXEYE XE detector

Mineral Sample
Rietveld analysis with Diffrac.TOPAS

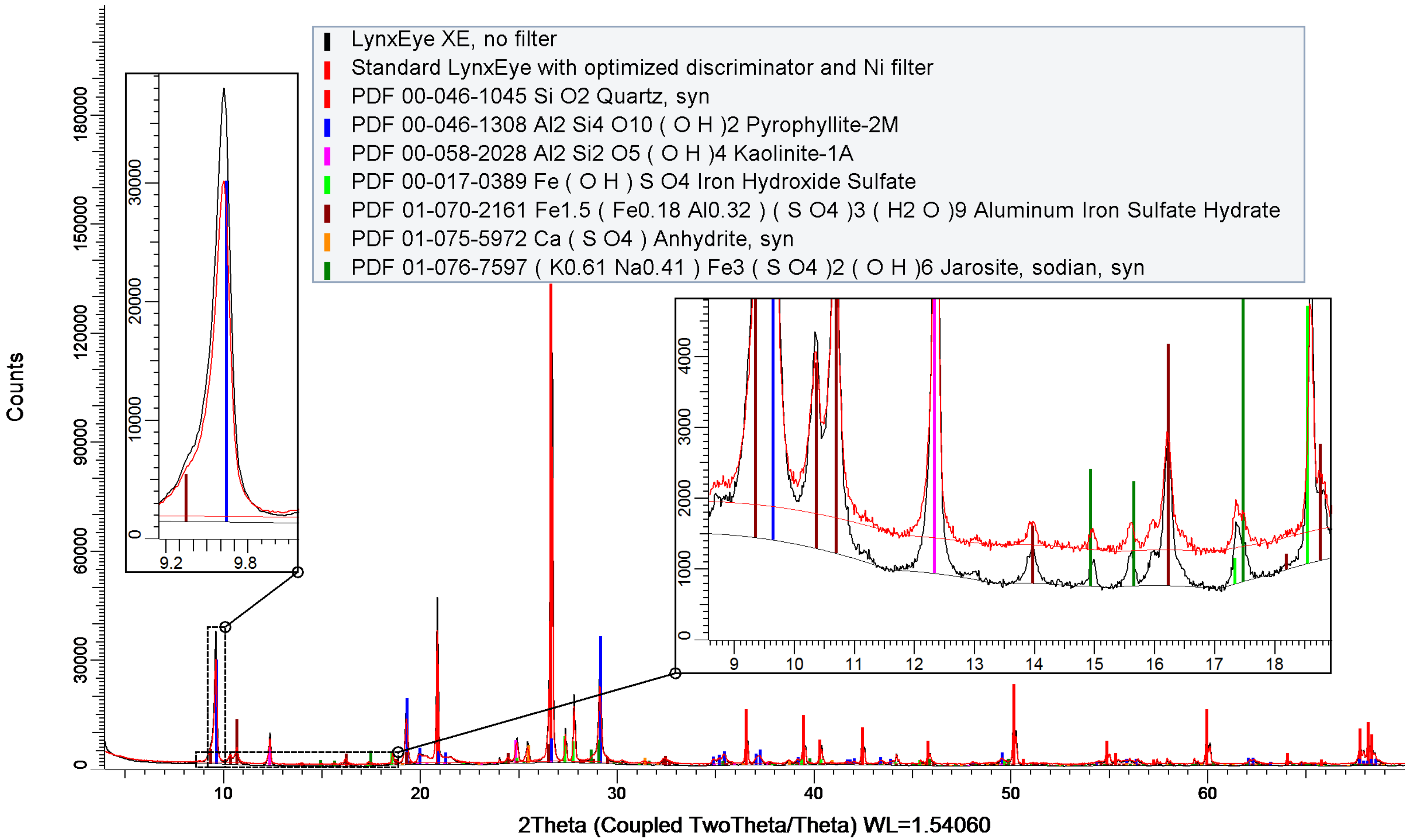


Comparison Standard LYNXEYE and LYNXEYE XE
Rare Earth Ore



Comparison Standard LYNXEYE and LYNXEYE XE

Sample from gold ore processing plant



Fluorescent Minerals

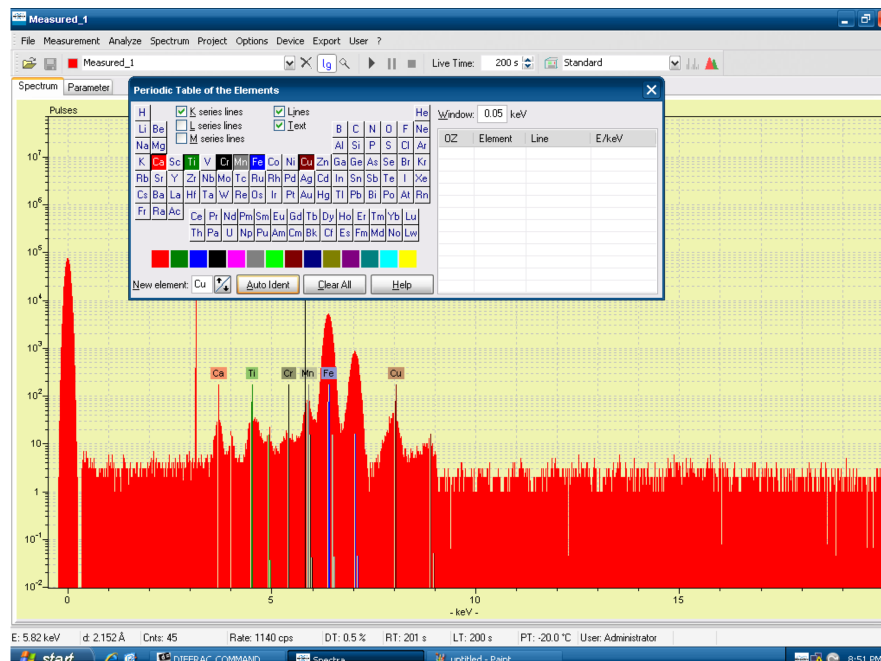
Garnet Solid Solution Series

Pure Pyrope: $Mg_3Al_2(SiO_4)_3$ typically with Ca, Cr, Fe and Mn (red, purple)

Pure Almandine: $Fe_3Al_2(SiO_4)_3$

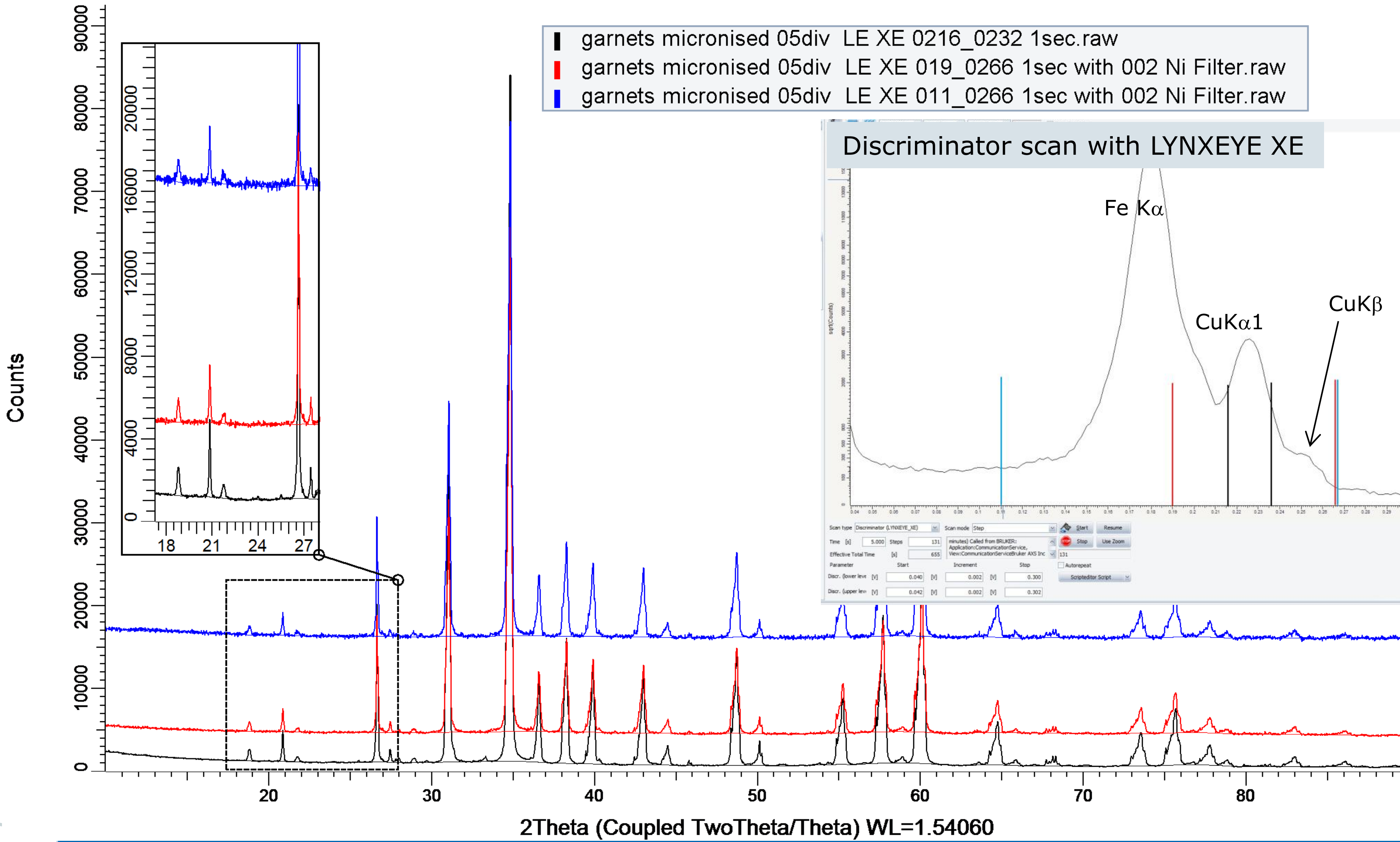
Pure Spessartine: $Mn^{2+}{}_3Al_2(SiO_4)_3$.

Qualitative XRF spectrum collected in D2 PHASER with XFlash detector

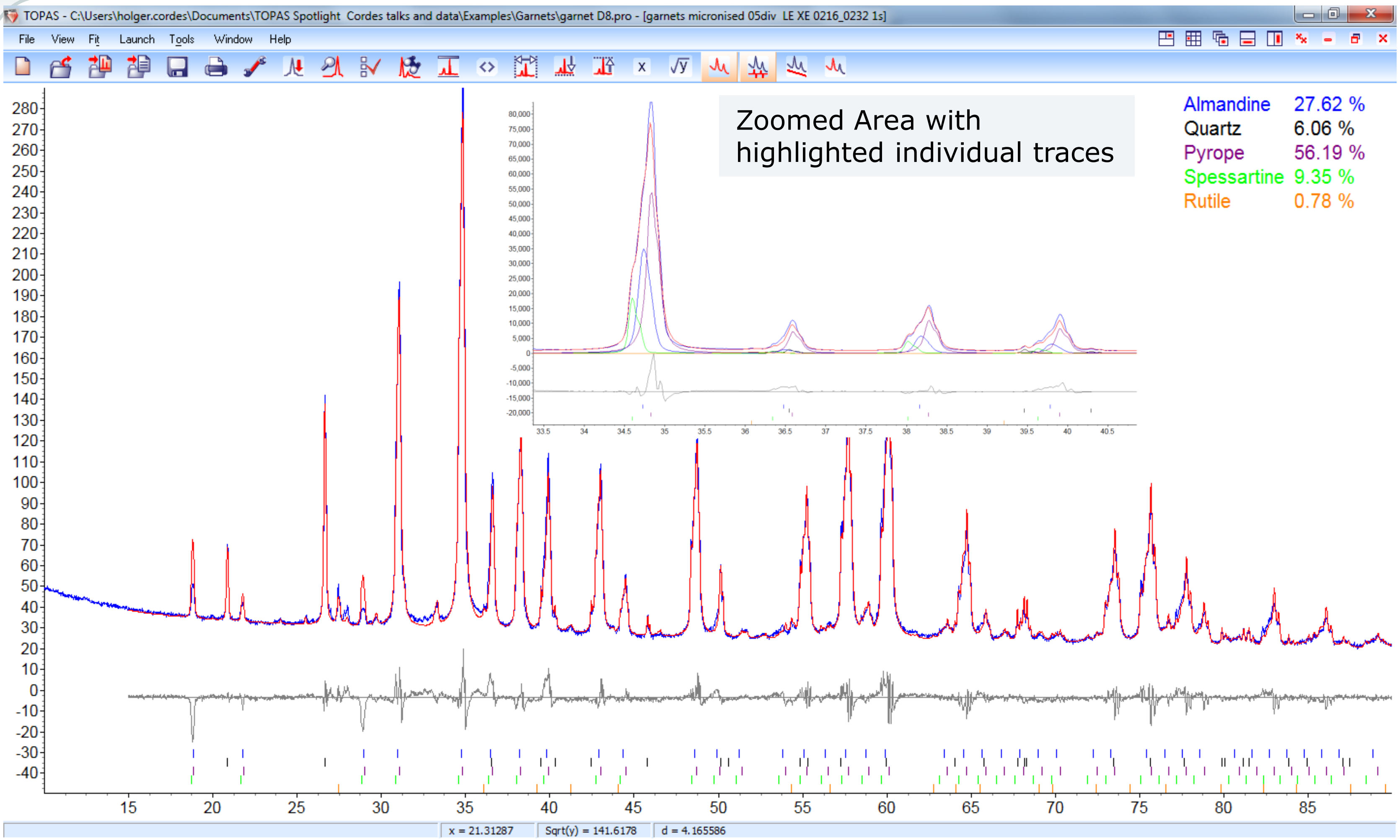


Comparison Standard LYNXEYE and LYNXEYE XE

Garnet solid solution series

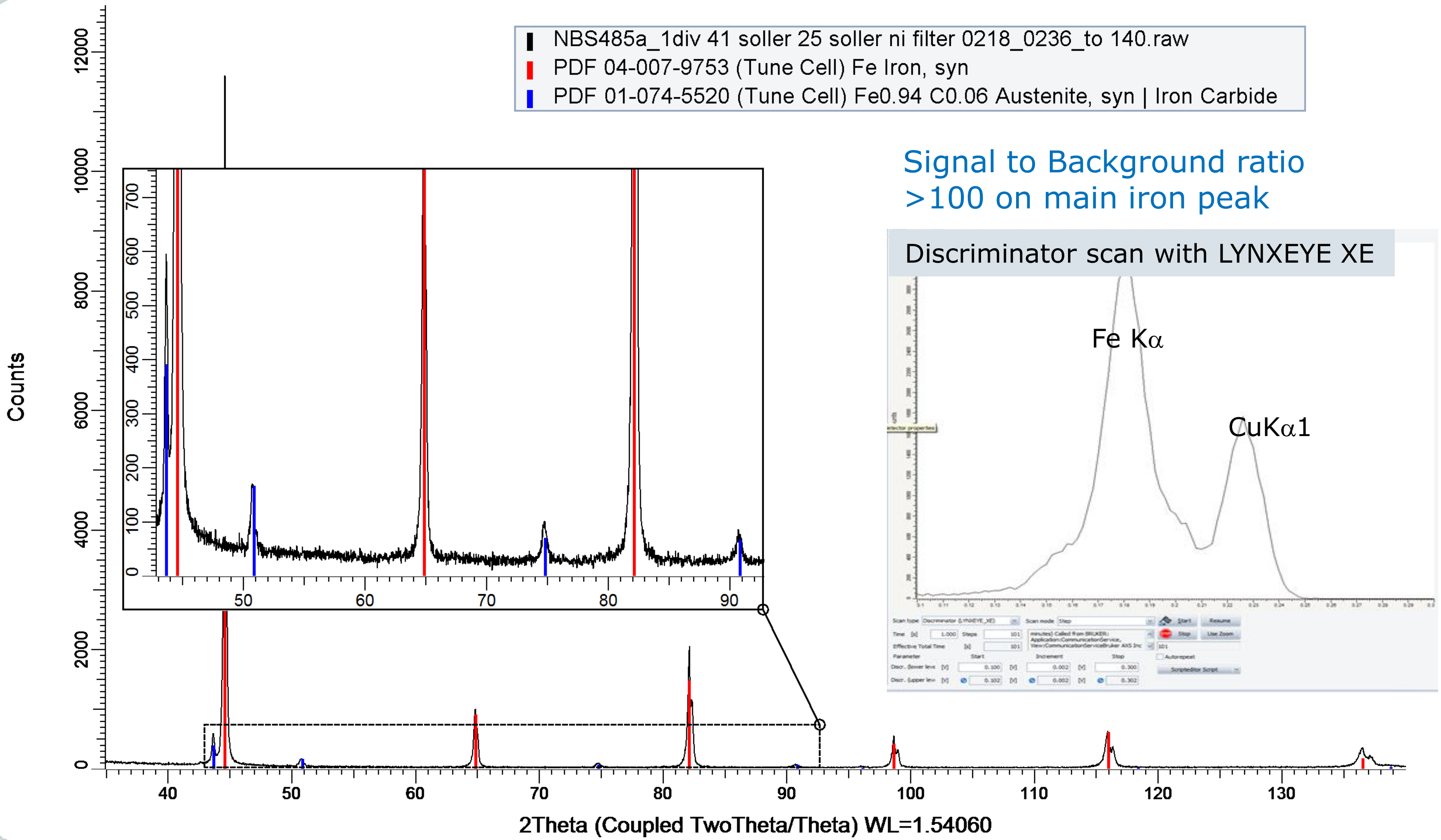


Garnet Sample
Quantitative Rietveld Analysis



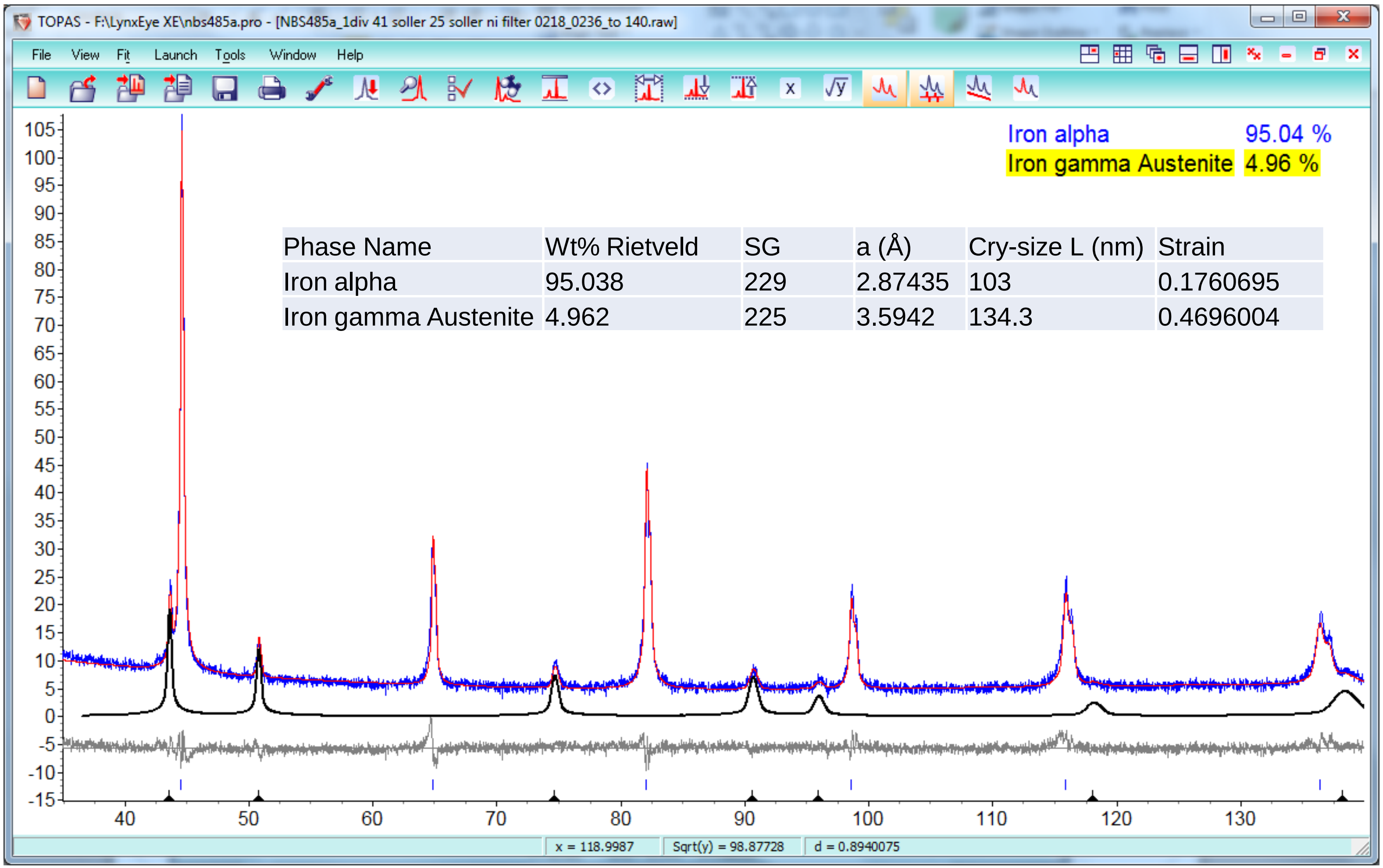
Steel Sample

5% Austenite in Ferrite, NBS485a



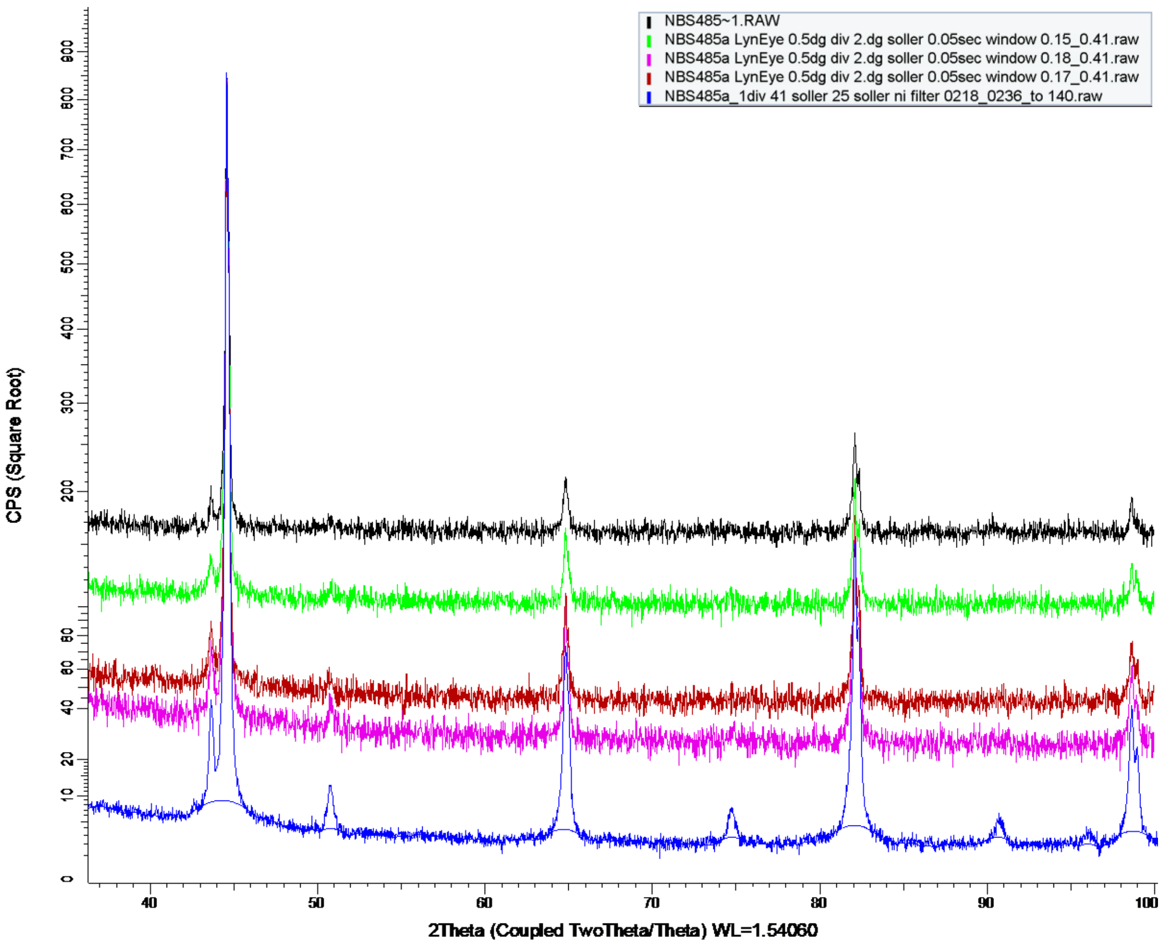
Steel Sample

5% Austenite in Ferrite, NBS485a



Phase Name	Wt% Rietveld	SG	a (Å)	Cry-size L (nm)	Strain
Iron alpha	95.038	229	2.87435	103	0.1760695
Iron gamma Austenite	4.962	225	3.5942	134.3	0.4696004

NBS485a
Comparison with 1. Generation LYNXEYE



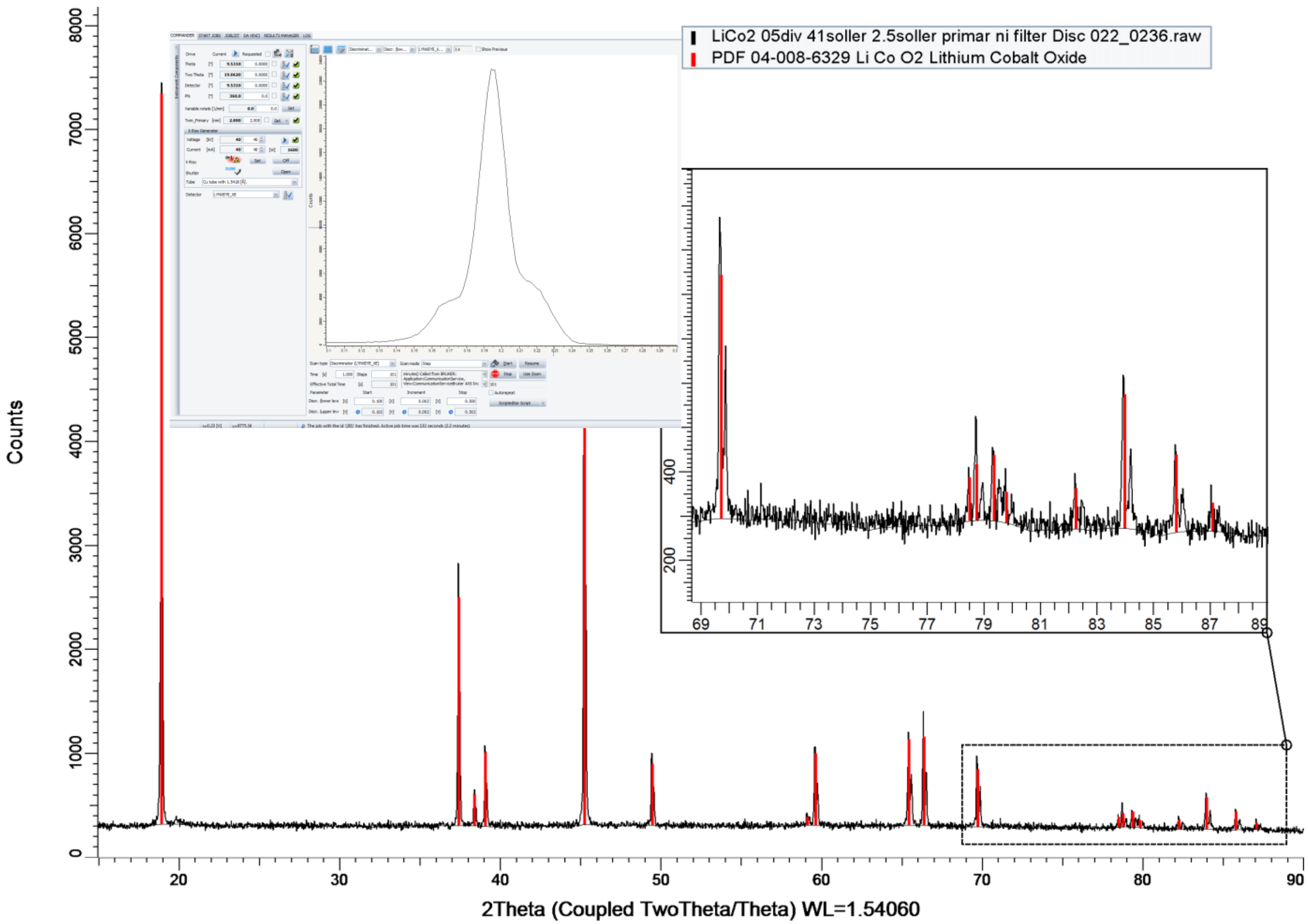
LYNXEYE XE detector

Other scans were collected with 1. generation LYNXEYE with various discriminator settings

All scans were normalized to the same maximum peak height. (counting statistic slightly lower on 1. generation scans due to shorter measurement time)

Lower Detection limit for retained Austenite with LYNXEYE XE

Battery Materials
LiCoO2

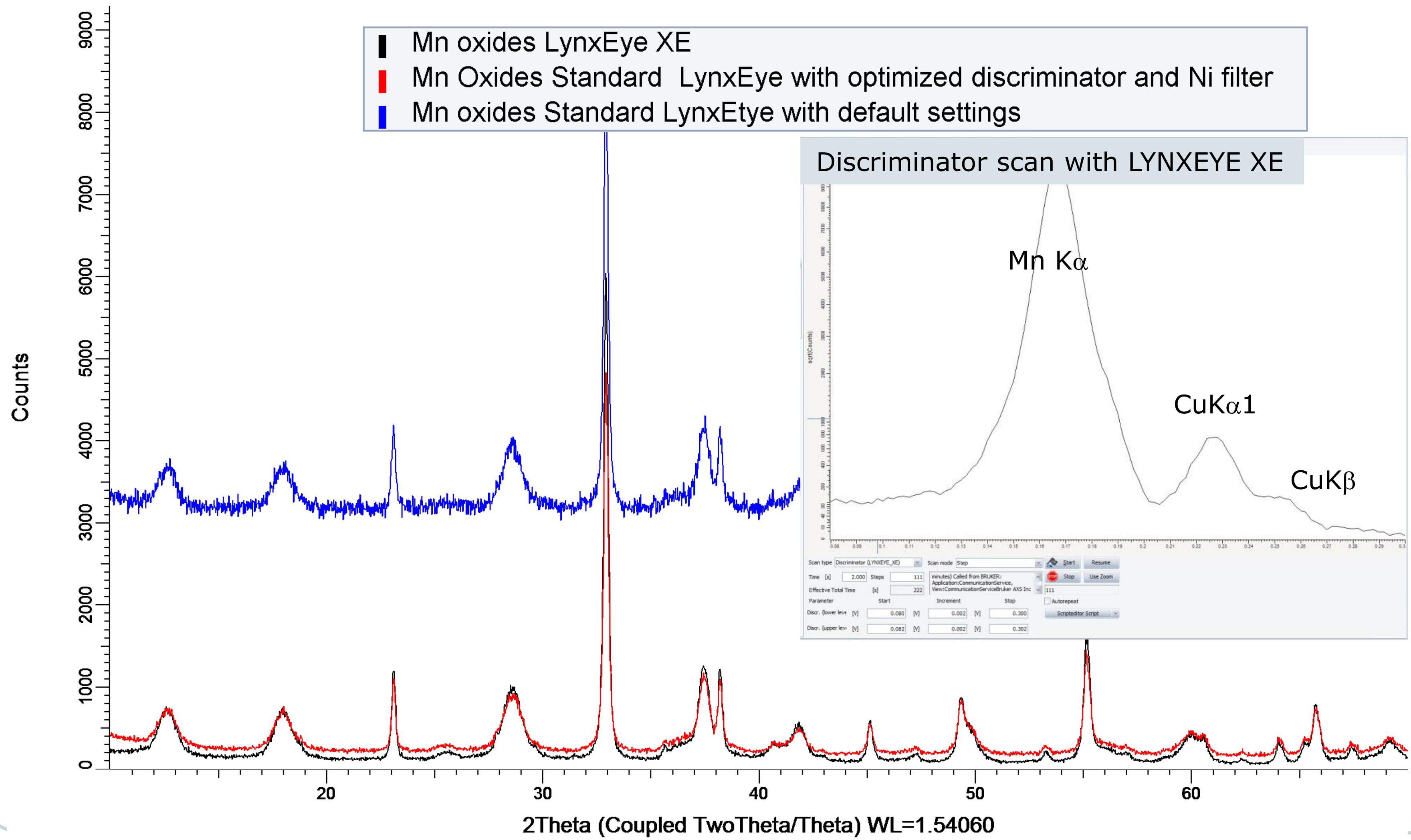


Configuration:

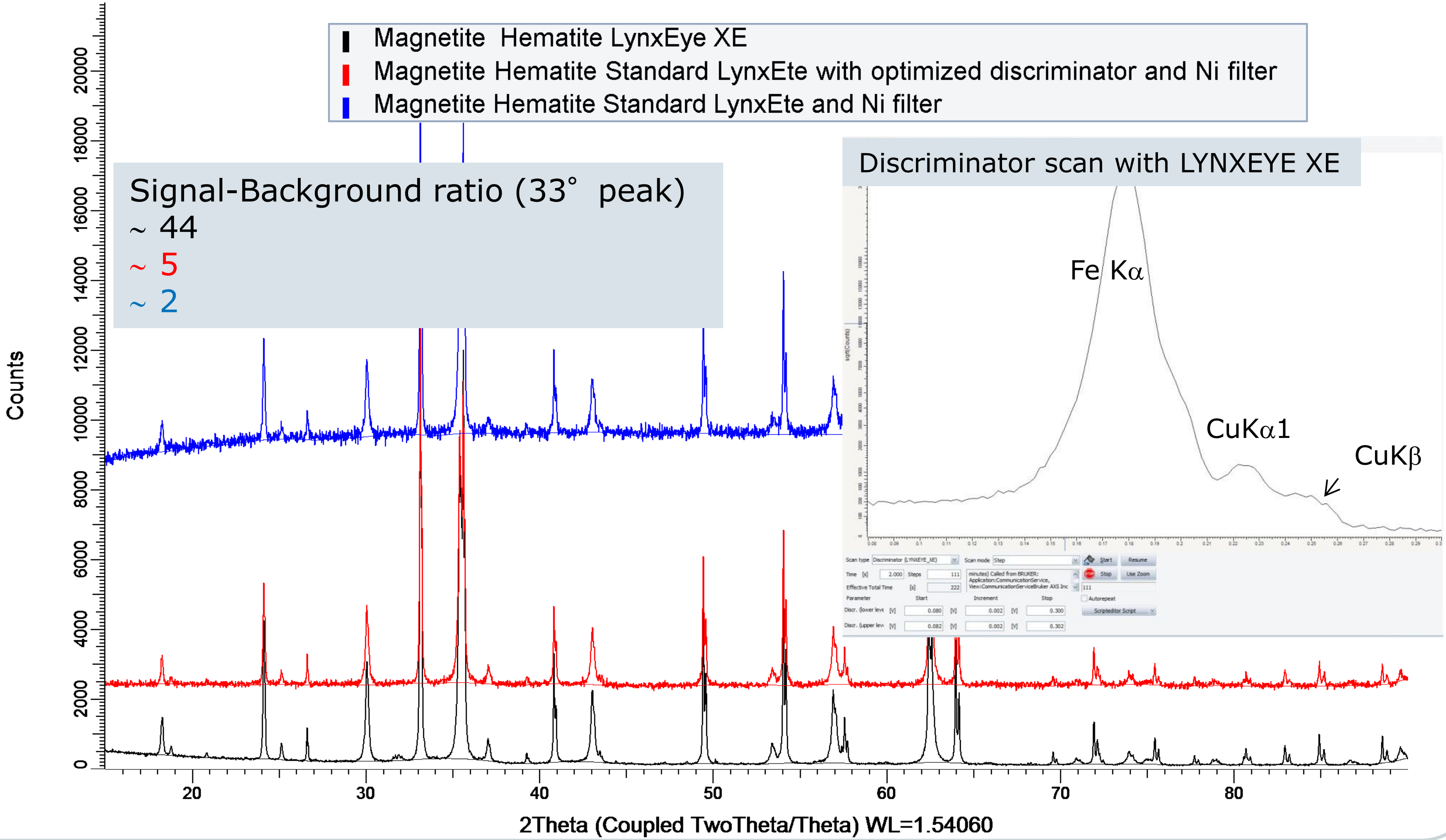
- LYNXEYE XE detector
- Disc 0.22 and 0.28
- 4.1° primary Soller
- 2.5° secondary Soller
- 0.5° divergence slit
- 0.02° steps, 0.1 sec/step
- Rotated 15 rpm

- Signal to background ratio approx. 25 on main $LiCoO_2$ peak

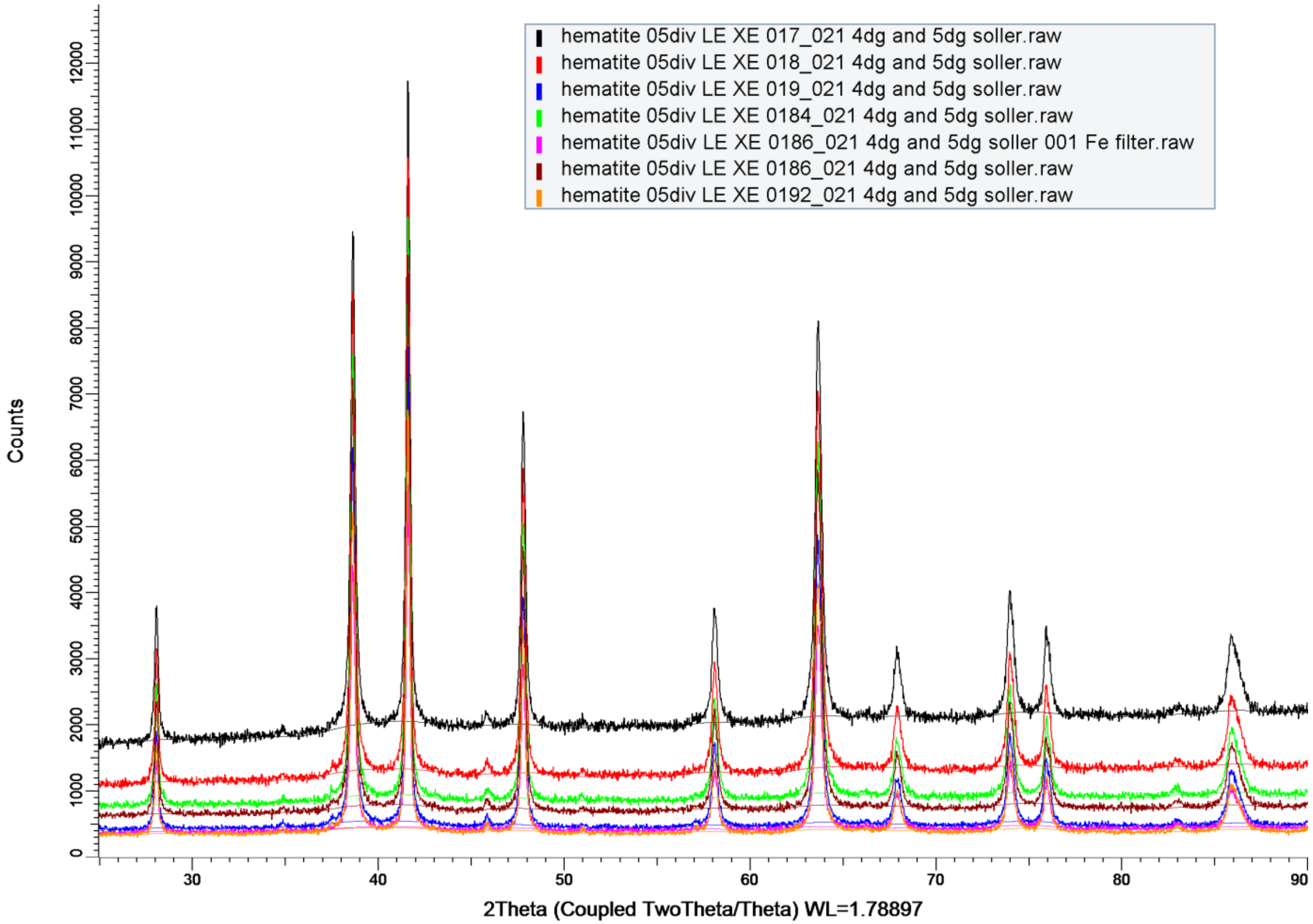
Battery materials
Manganese oxides



Corrosion Samples
Iron oxides



Iron oxide sample measured with Co radiation
Various discriminator settings

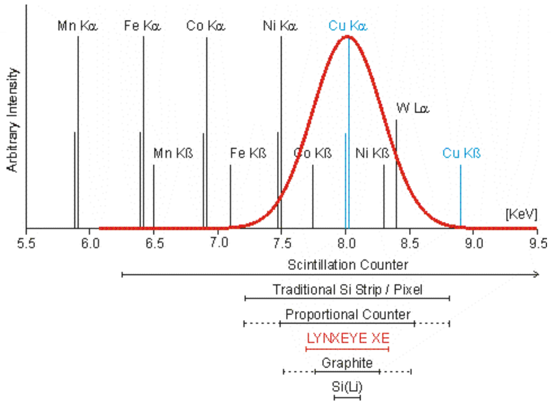


- Co radiation for better sample penetration
- Signal-Background ratio not as high as with Cu radiation but significantly better than with Standard LYNXEYE detector

Questions?

Any questions?

Please type any questions you may have for our speakers in the Q&A panel and click Send.



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Webinar	Content
Jun 27, 2013 **Good Diffraction Practice IX: LYNXEYE XE - Combining 1D Speed with 0D Background**	Join Sr. Applications Scientist Holger Cordes for a 45-minute overview of the new LYNXEYE XE 1D energy-discriminating detector. Holger will present XRD application examples, using different sample types, illustrating how the LYNXEYE XE can help you better characterize your material samples. Register now

Webinar	Content
Sep 30, 2010 **Good Diffraction Practice III: Powder XRD Instrumentation and Data Quality**	Join Dr. Lutz Bruegemann as he explains important aspects of X-ray powder diffraction workflow: from mechanical setup of the goniometer to data collection and evaluation of data quality. View recording Download slides
	X-ray diffraction analysis with a two-dimensional

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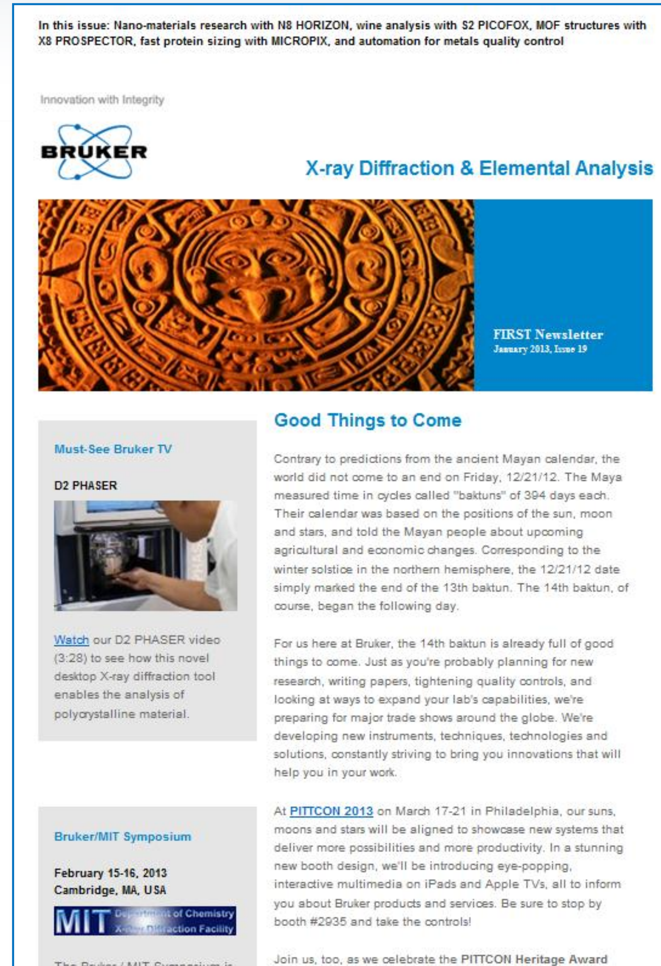

In this issue: Nano-materials research with N8 HORIZON, wine analysis with S2 PICOFOX, MOF structures with X8 PROSPECTOR, fast protein sizing with MICROPIX, and automation for metals quality control

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X-ray Diffraction & Elemental Analysis

FIRST Newsletter
January 2013, Issue 19

Must-See Bruker TV

D2 PHASER

Watch our D2 PHASER video (3:28) to see how this novel desktop X-ray diffraction tool enables the analysis of polycrystalline material.

Bruker/MIT Symposium

February 15-16, 2013
Cambridge, MA, USA

MIT Department of Chemistry X-ray Diffraction Facility

The Bruker / MIT Symposium is

Good Things to Come

Contrary to predictions from the ancient Mayan calendar, the world did not come to an end on Friday, 12/21/12. The Maya measured time in cycles called "baktuns" of 394 days each. Their calendar was based on the positions of the sun, moon and stars, and told the Mayan people about upcoming agricultural and economic changes. Corresponding to the winter solstice in the northern hemisphere, the 12/21/12 date simply marked the end of the 13th baktun. The 14th baktun, of course, began the following day.

For us here at Bruker, the 14th baktun is already full of good things to come. Just as you're probably planning for new research, writing papers, tightening quality controls, and looking at ways to expand your lab's capabilities, we're preparing for major trade shows around the globe. We're developing new instruments, techniques, technologies and solutions, constantly striving to bring you innovations that will help you in your work.

At PITTCON 2013 on March 17-21 in Philadelphia, our suns, moons and stars will be aligned to showcase new systems that deliver more possibilities and more productivity. In a stunning new booth design, we'll be introducing eye-popping, interactive multimedia on iPads and Apple TVs, all to inform you about Bruker products and services. Be sure to stop by booth #2935 and take the controls!

Join us, too, as we celebrate the PITTCON Heritage Award

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