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# Detecting Li with EDS

October 2014

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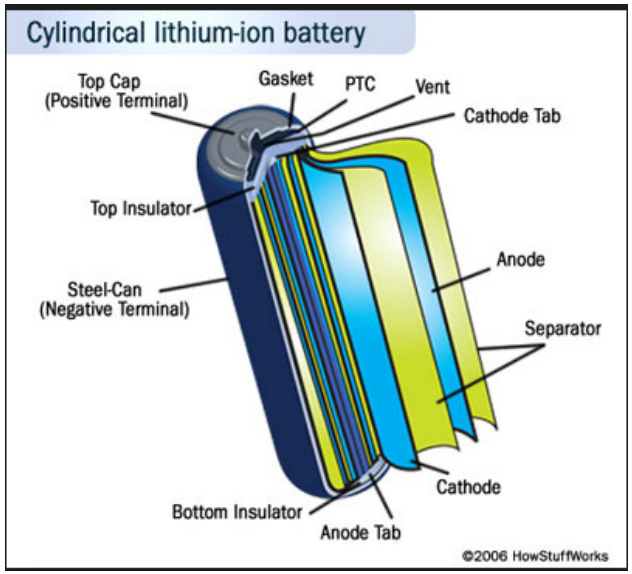
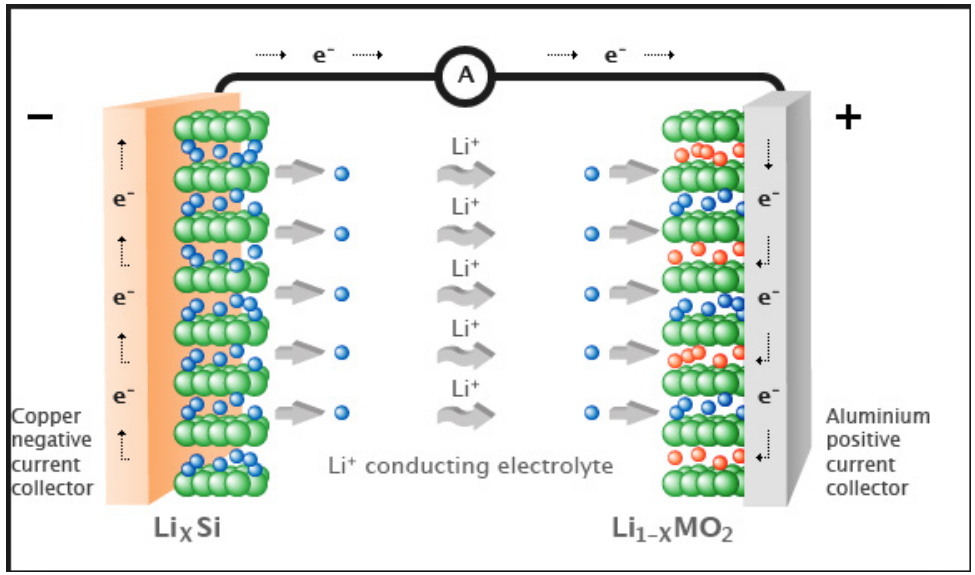
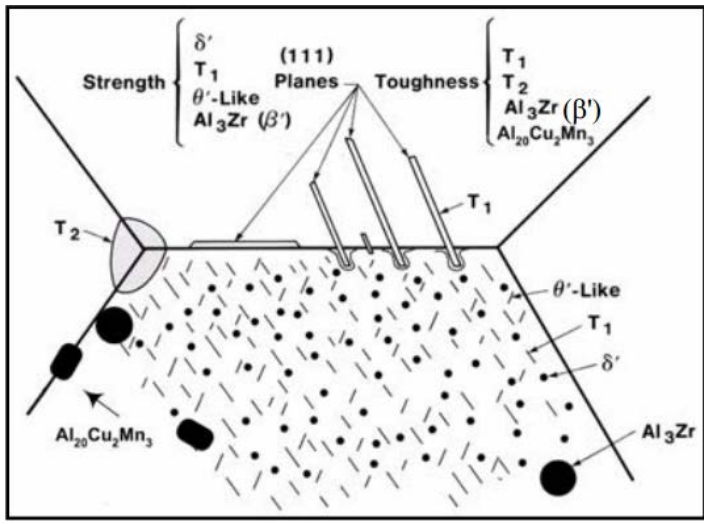
# Why detect Li x-rays? Applications





Lithium floating in oil



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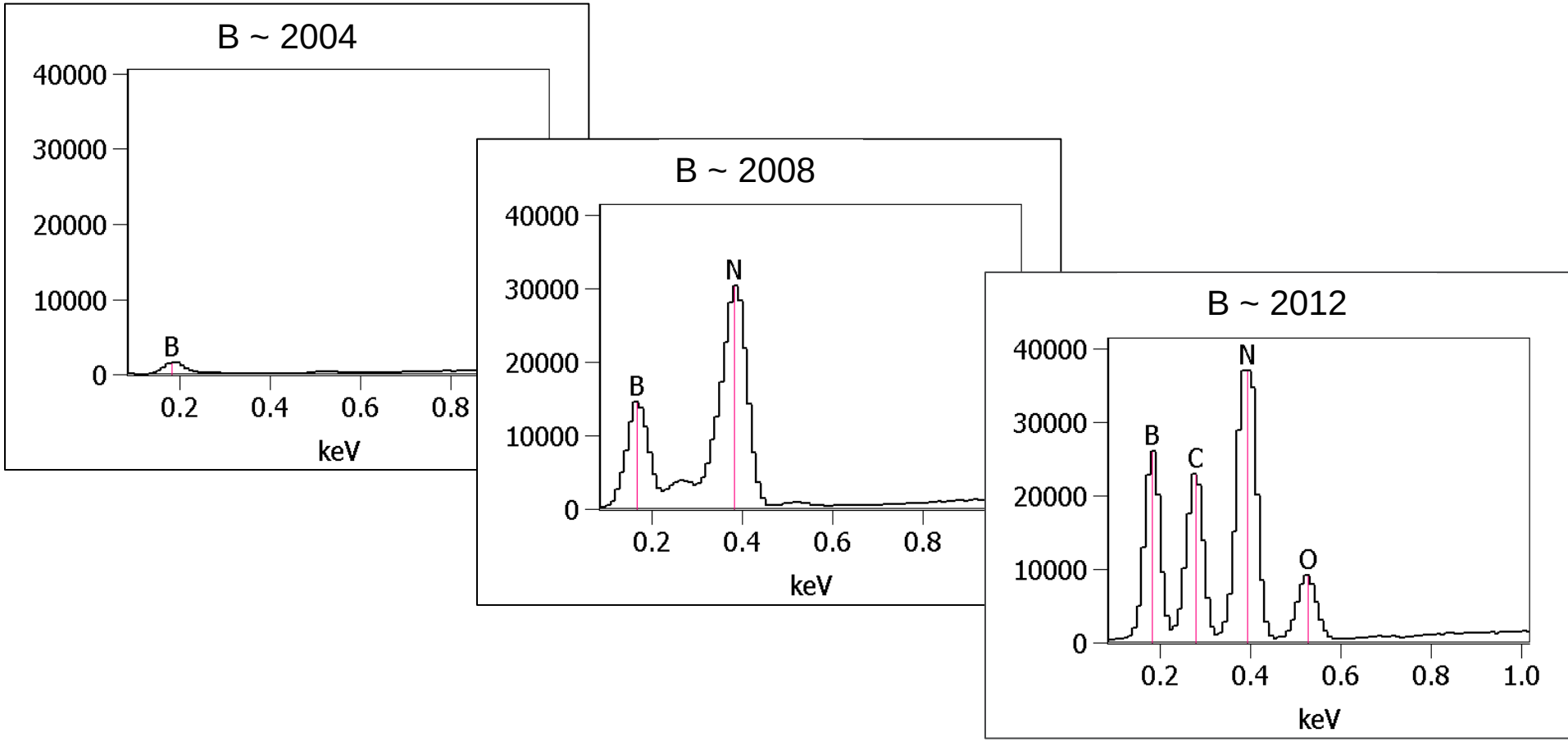
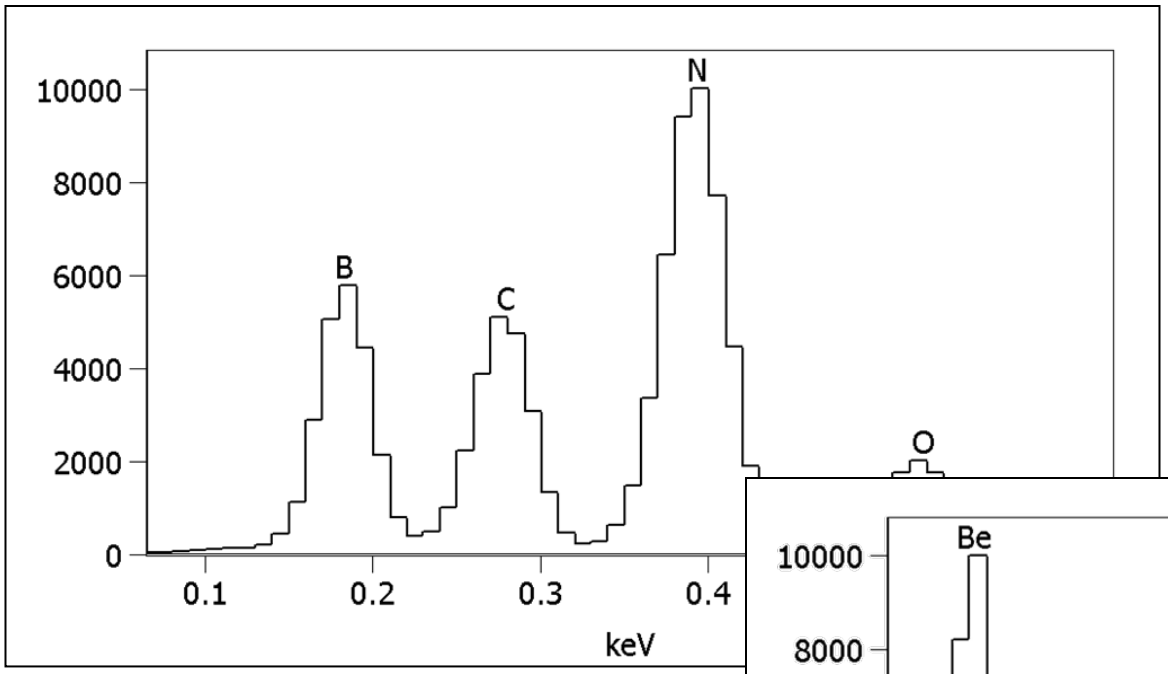
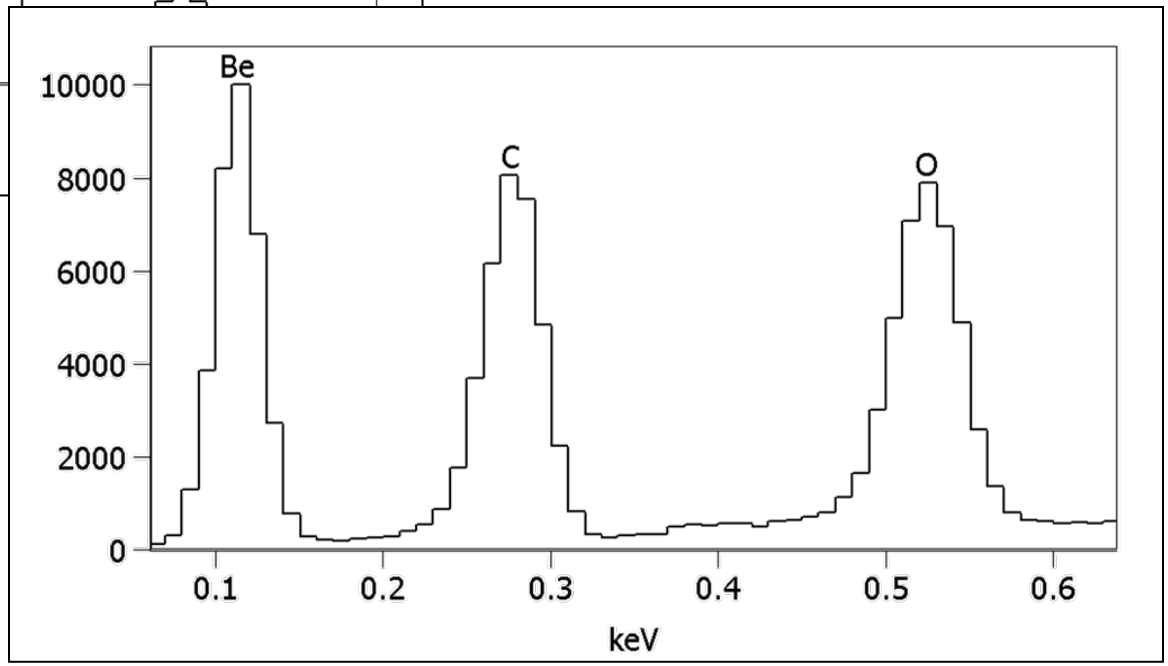
# Why detect Li X-rays? Detector Technology




Light element sensitivity represents a continued evolution in detector technology.

# Why detect Li X-rays? Detector Technology




From B to Be

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# Challenge # 1

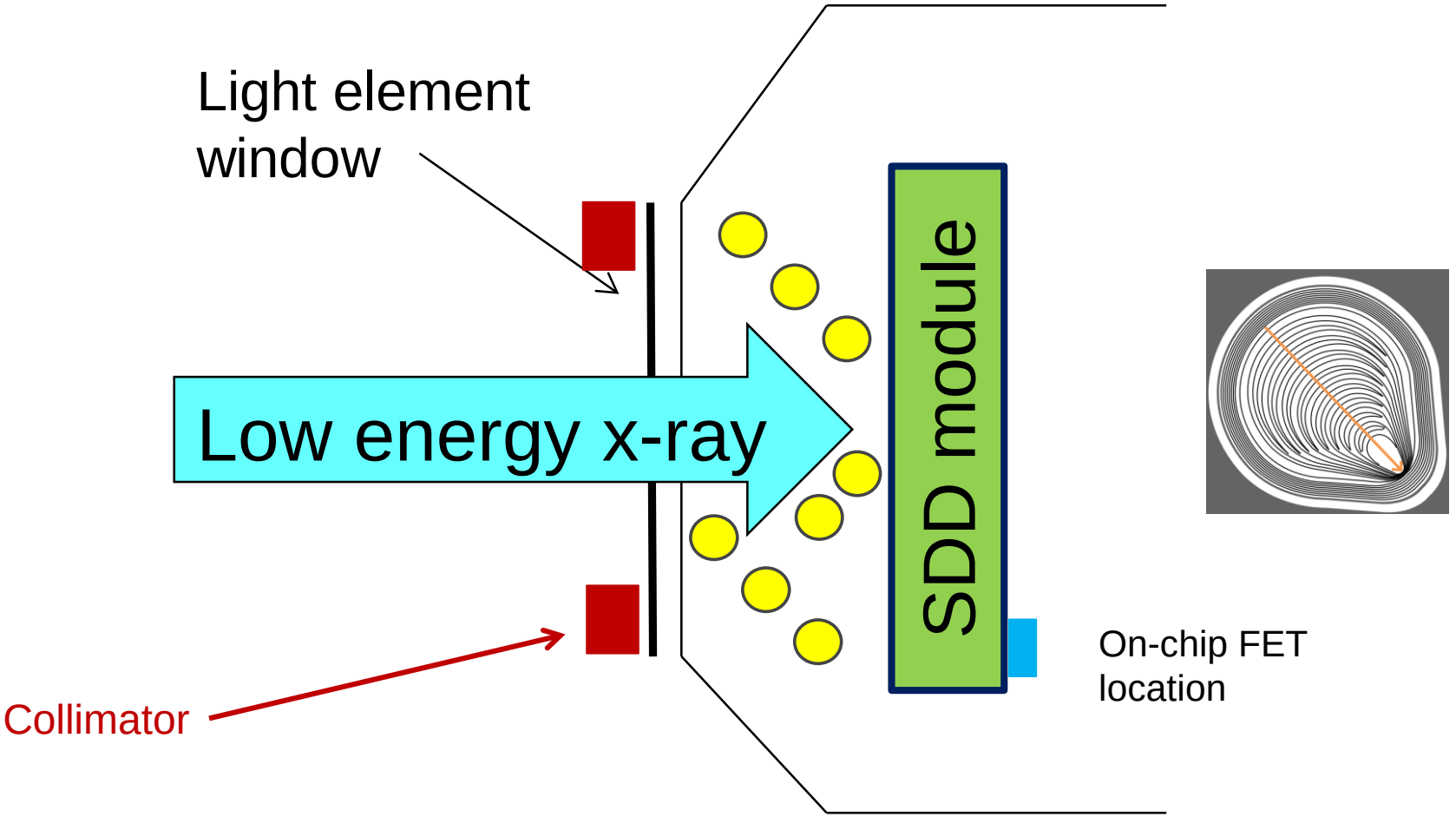
Building an EDS that detects Li x-rays

# Building an EDS that detects Li x-rays

- Spectral performance
  - Zero width < 30 eV
  - Discriminate a 52 eV energy event
  - Peak to background for trace detection

- Windowless
  - Eliminate absorption of Li x-rays in the window

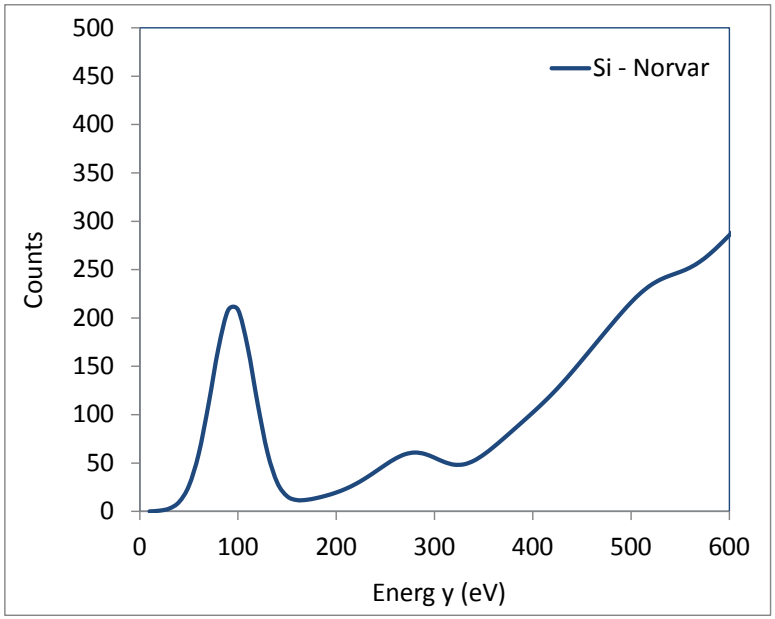
# Building an EDS that detects Li x-rays



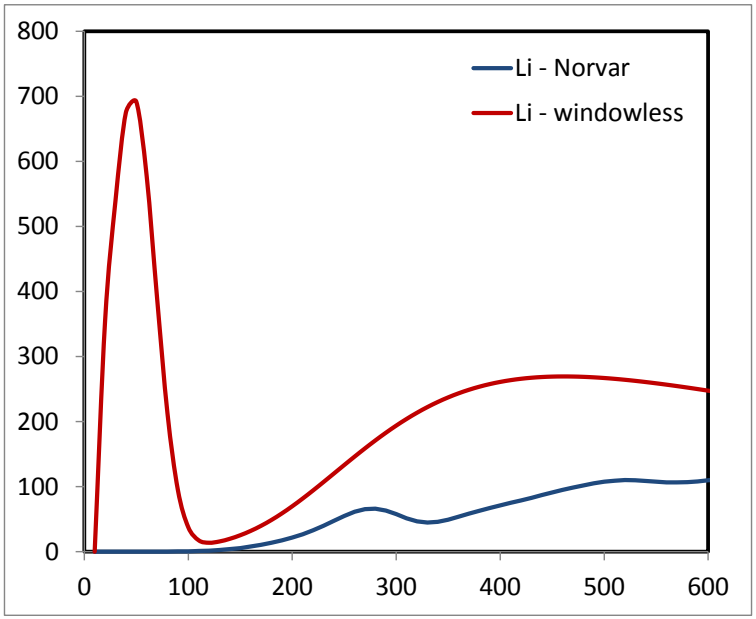

Low energy x-rays have a difficult time getting to the SDD module

# The challenge of the window

## Simulations



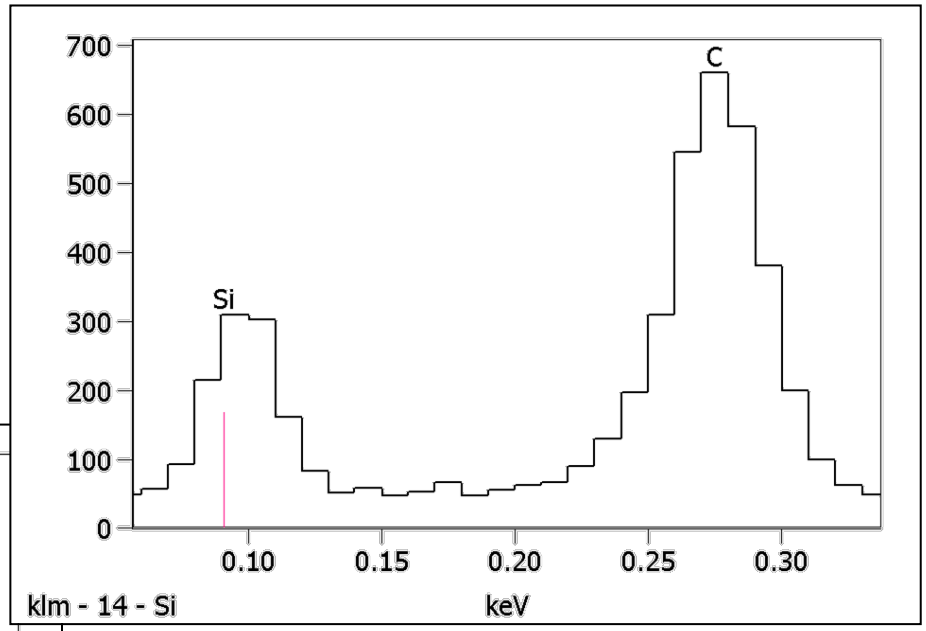

Si-l line with window

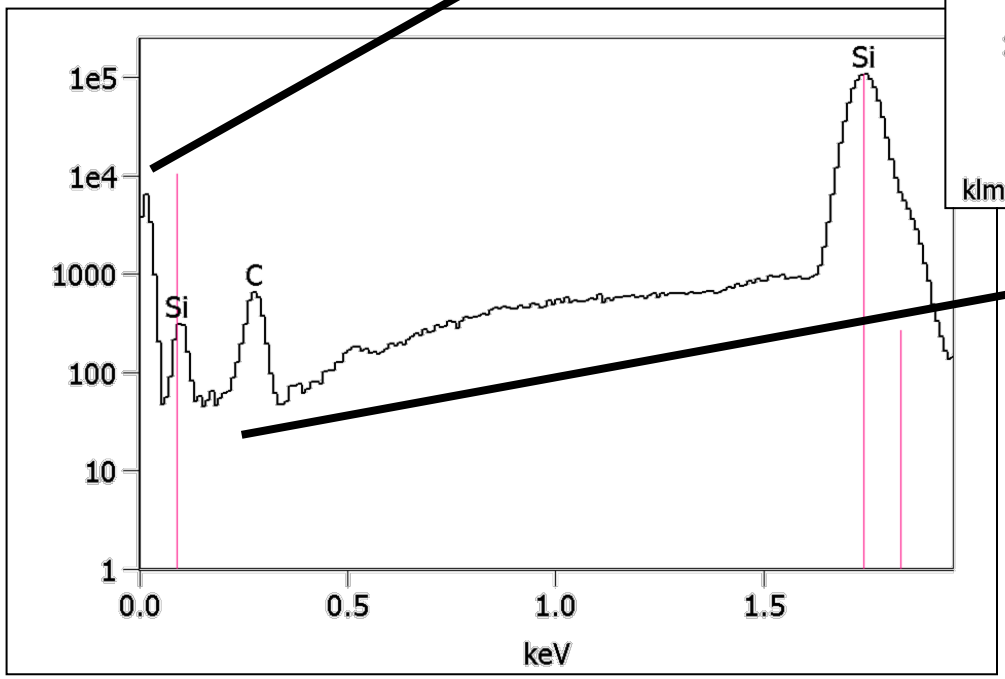



Li-k line with and w/o window

# Actual data: Si-L lines (detector w/window)

Si-L line even with the accelerating voltage at 20 kV

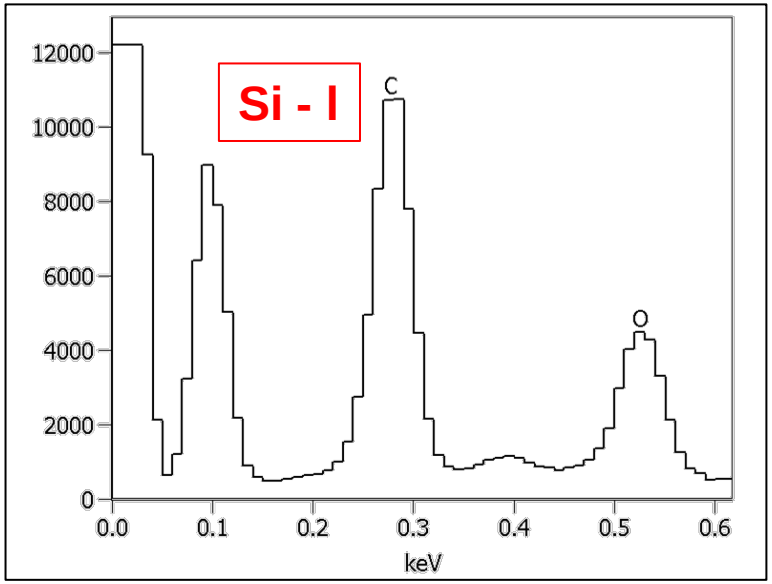


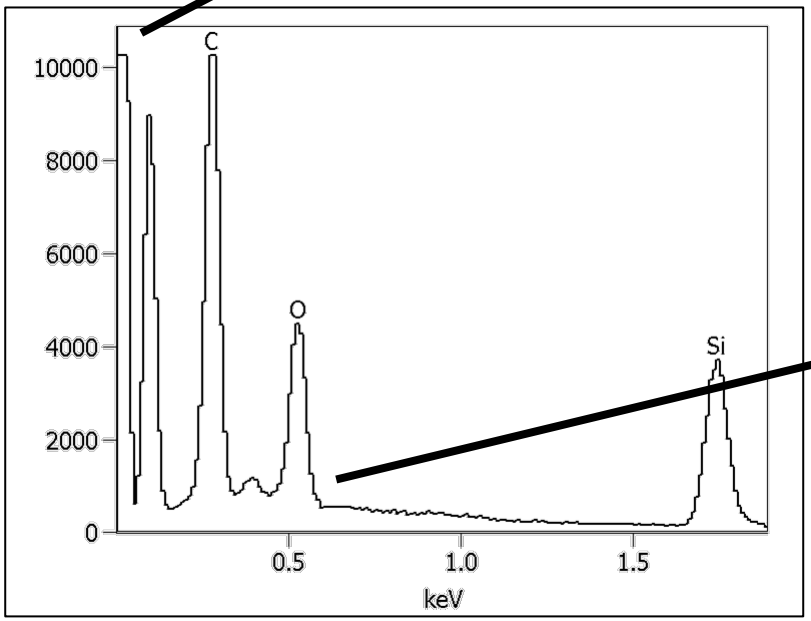




Seeing the Si-L line has become normal even with a window present

# Actual data: Si-L lines (windowless)

Si-L line sharper and cleaner with a windowless EDS detector







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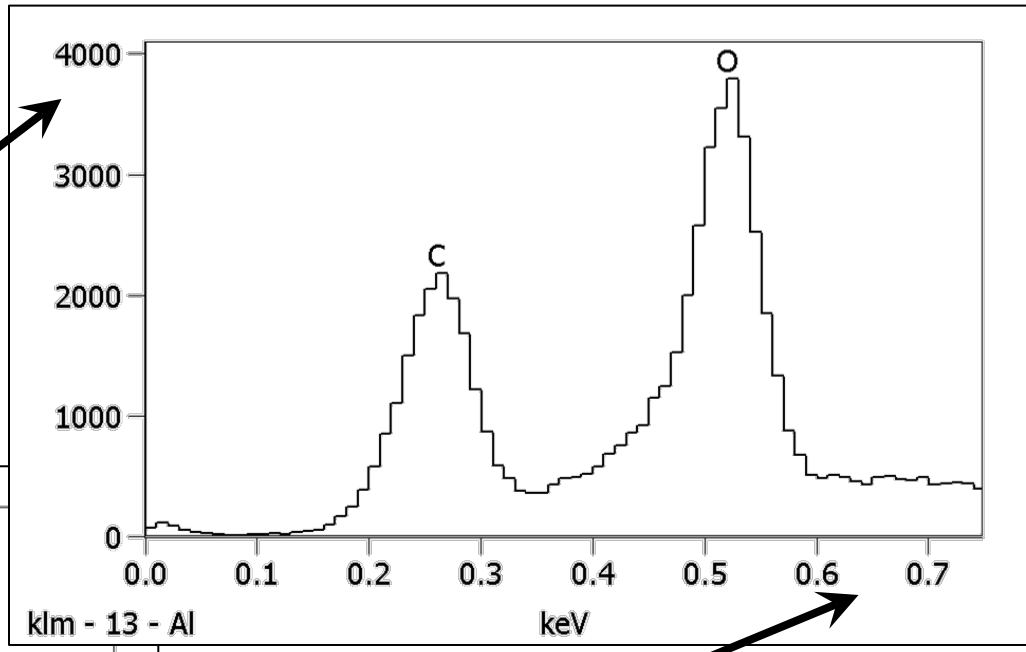
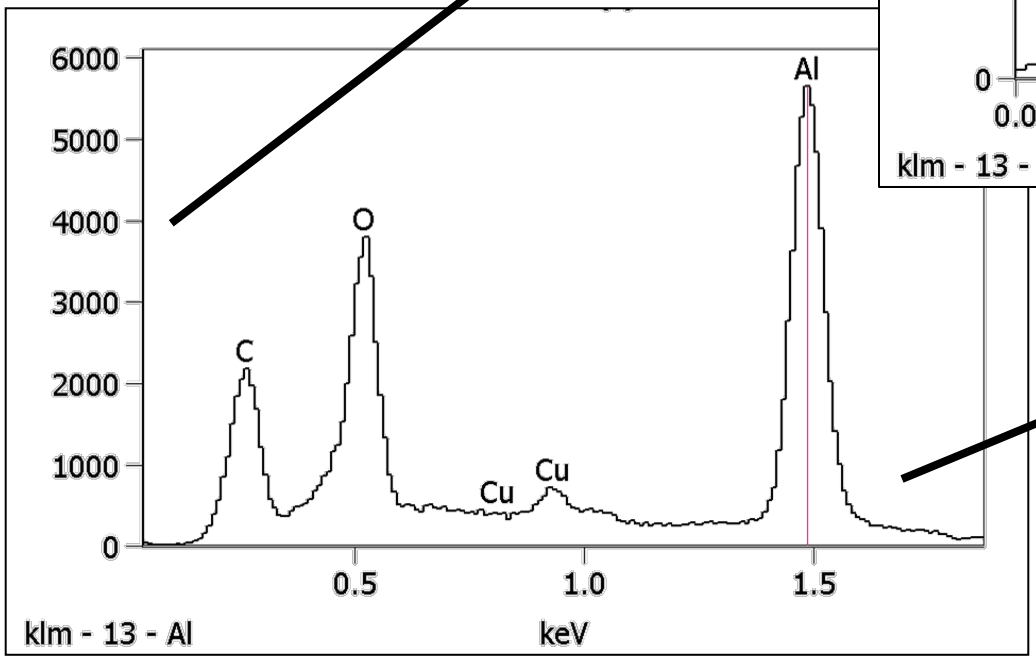
# Actual data: Al lines (detector w/window)

Aluminum

Strong Al-k

No Al-L




The Al-l line is still out of reach with a window present

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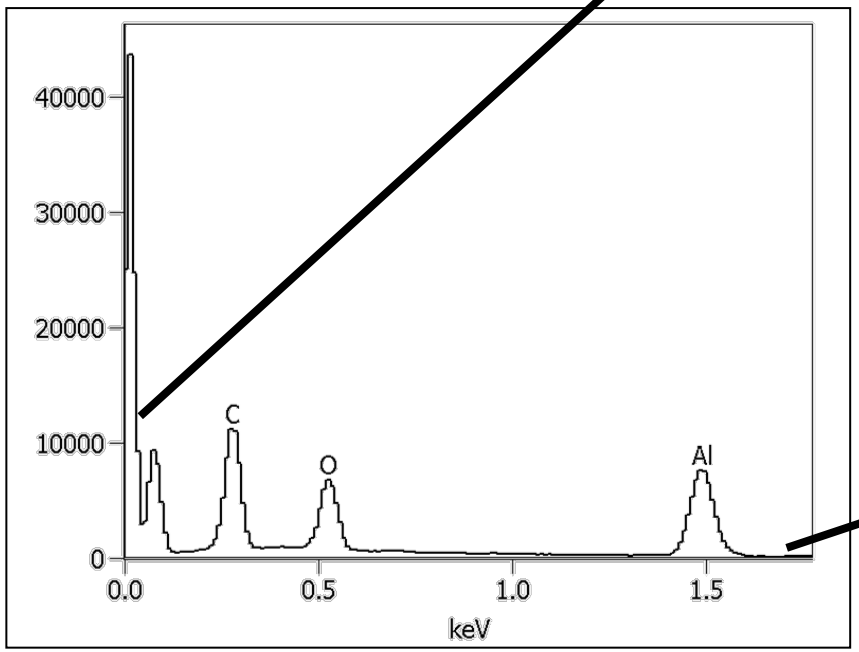
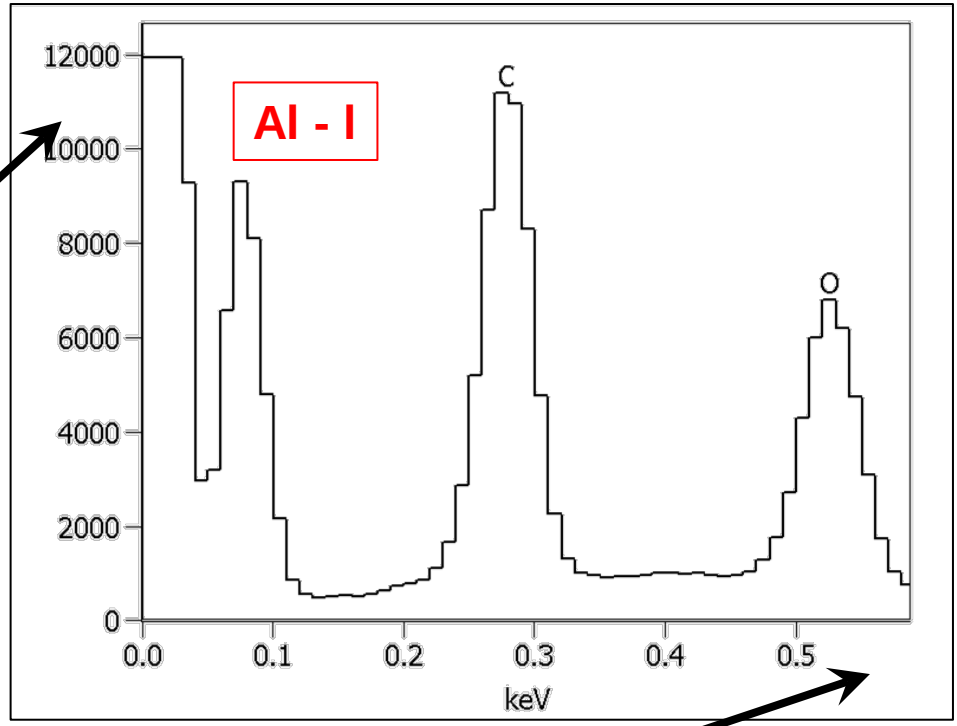
# Actual data: Al lines (windowless)

Aluminum

Strong Al-k

Strong Al-l




The Al-l line is cleanly detected

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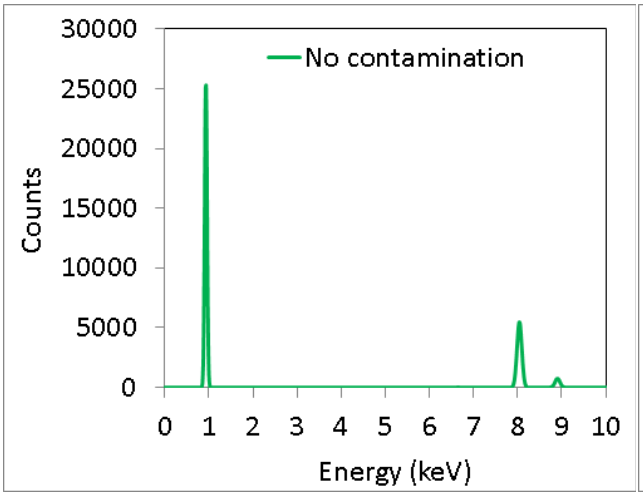
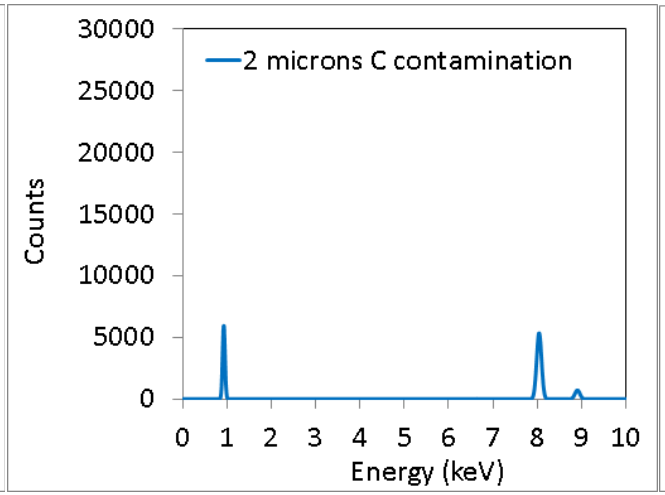
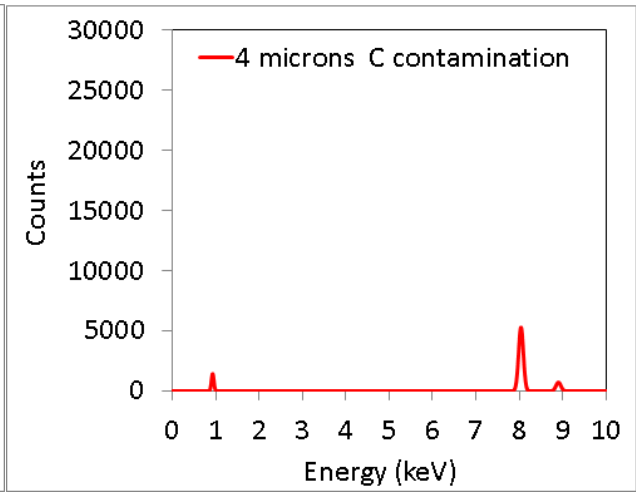
# The challenge of no window

## Impact of module contamination
### Simulations



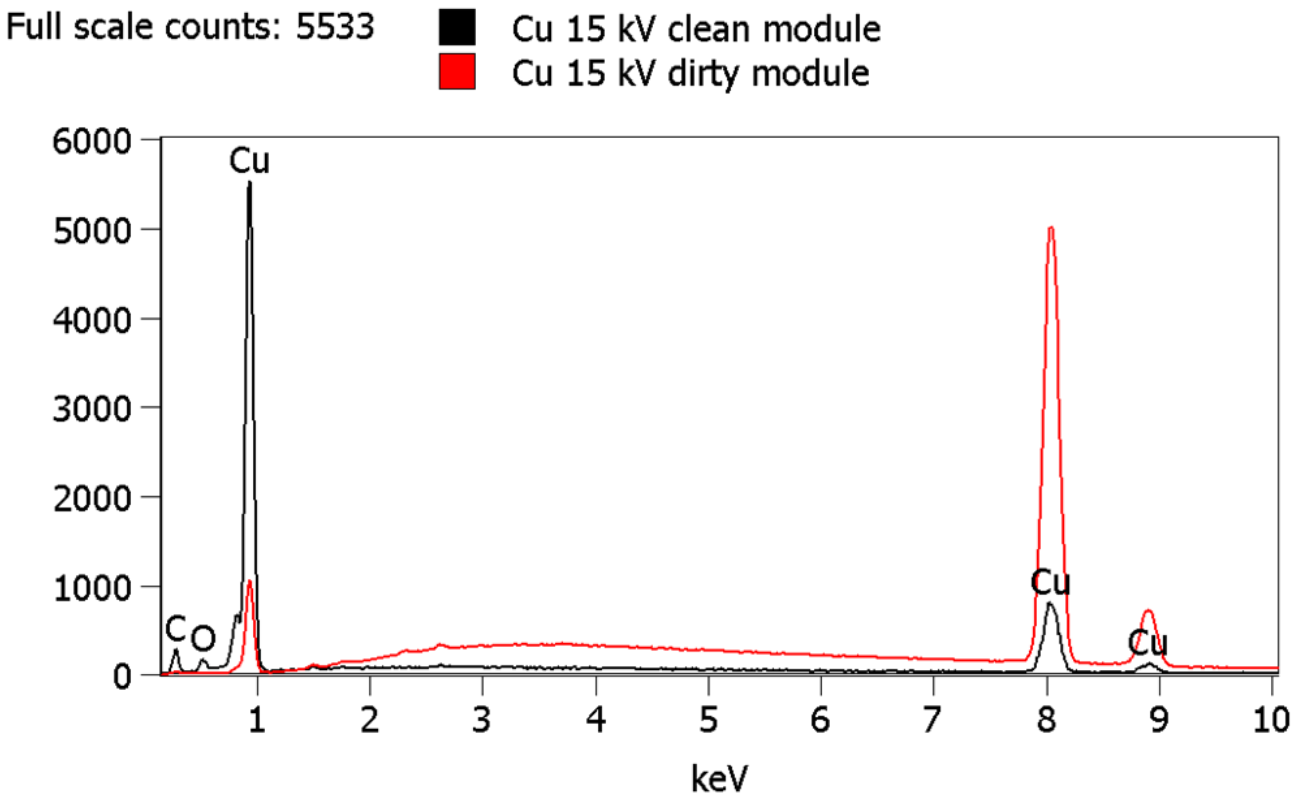

Copper k (~ 8 keV) and l (~ 1 keV) lines

Module is -25$^{o}$ C cold
Window is insulated and therefore relatively warm

# The challenge of no window

## Impact of module contamination

12 months exposure to rotary pump




Module is -25° C cold

Window is insulated and therefore relatively warm

# Detector requirements for Li detection

- Spectral performance
  - Zero width < 30 eV
  - Discriminate a 52 eV energy event
  - Peak to background for trace detection

- Safe Windowless operation
  - Eliminate absorption of Li x-rays in the window
  - Exposed (-25$^{o}$ C cold) SDD module must be protected when not in use with interlocks against chamber vent at all levels.
    - Pressure sensor, software time-outs, etc.
    - Shutter when not in use
  - SEM must be a dry-pumped system. Oil in the chamber will contaminate the module and ruin the Li detection capability.

# Challenge #2

Getting Li x-rays out of the sample

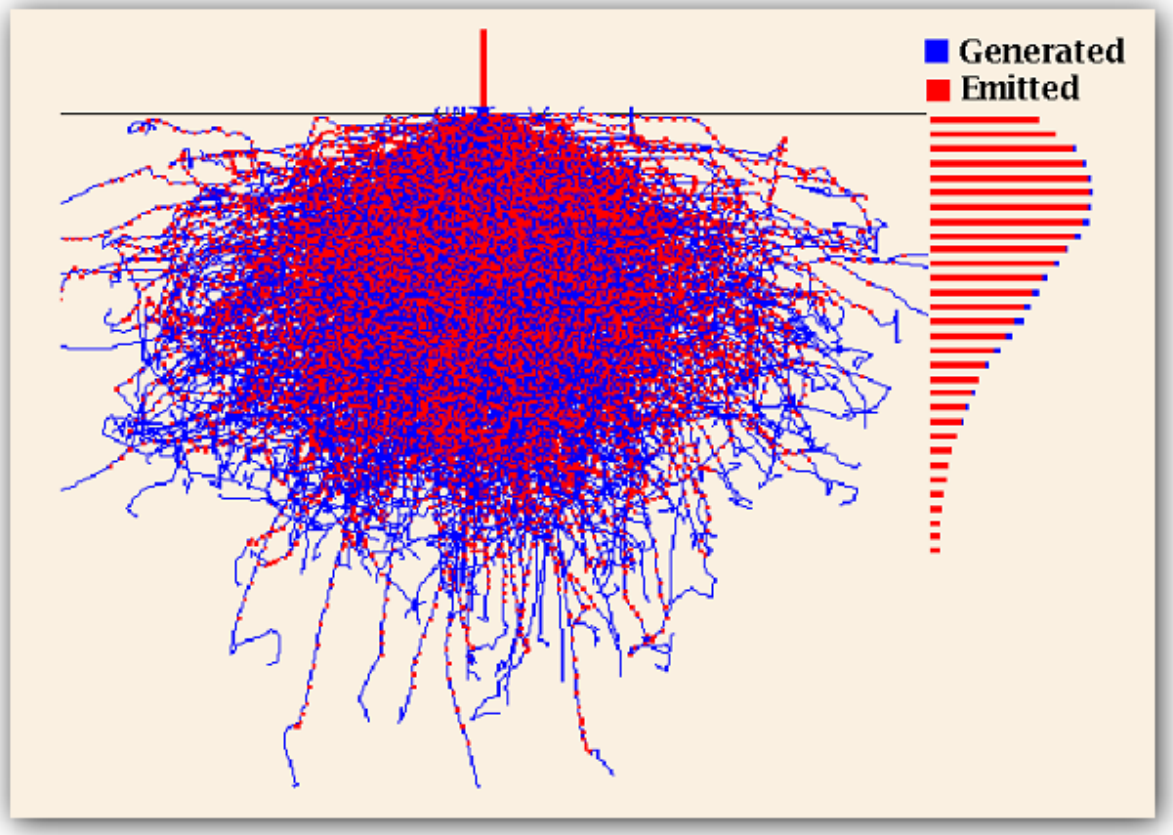
# Sample requirements

- Must get x-rays out of the samples to measure them
  - X-ray absorption within the sample
  - Heavy elements are really effective at absorbing low energy x-rays
- Li is highly reactive. When exposed to air, it oxidizes almost immediately.
- The escape depth of Li is only 40 nm – 50 nm. As the surface oxidizes, the Li x-rays are increasingly absorbed in the oxidized layer.

# Photon - Sample Interactions

## Absorption example

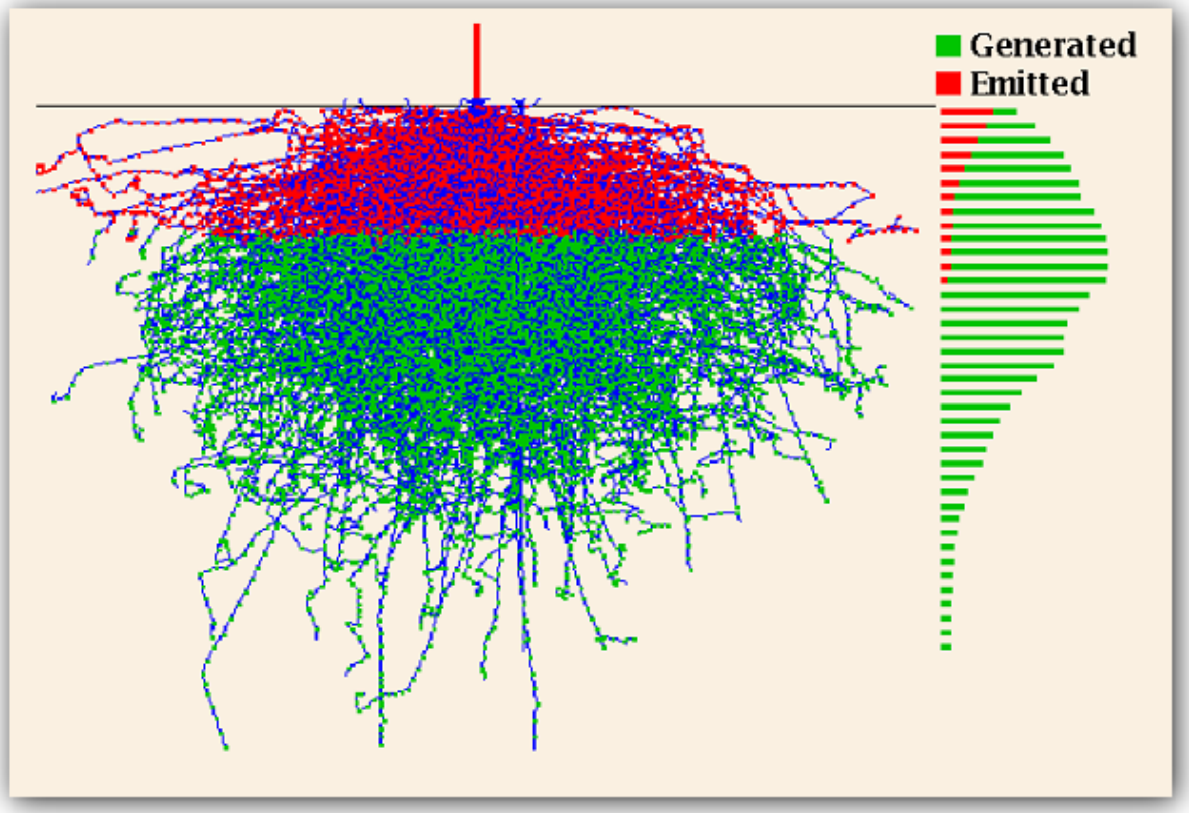
- Fe Kα X-Ray emission from an FeB sample



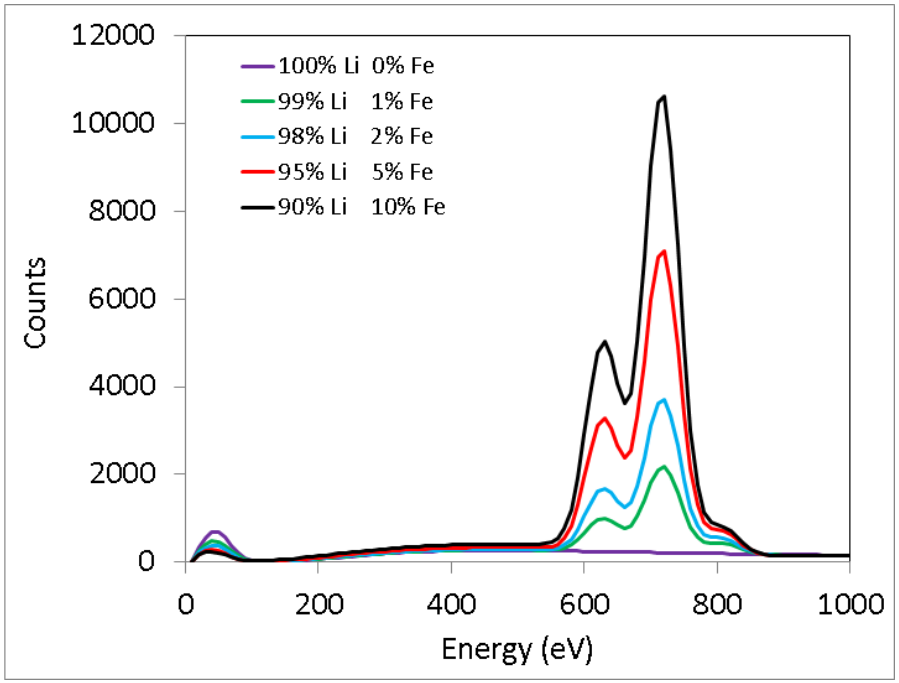
# Photon - Sample Interactions

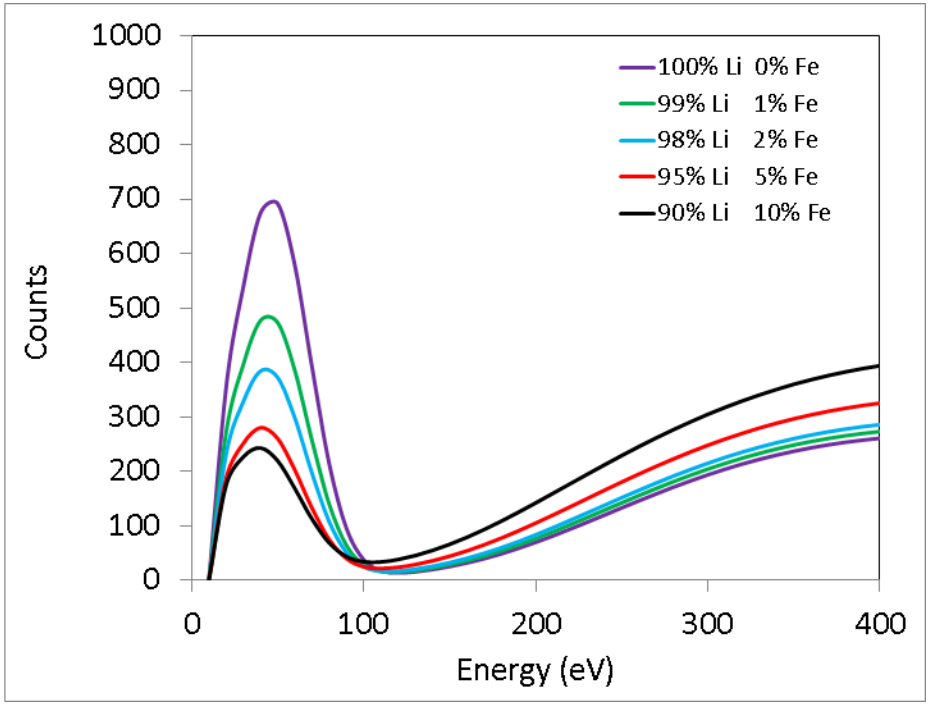
## Absorption example

- B Kα X-Ray emission from an FeB sample



# Absorption of fluoresced x-rays



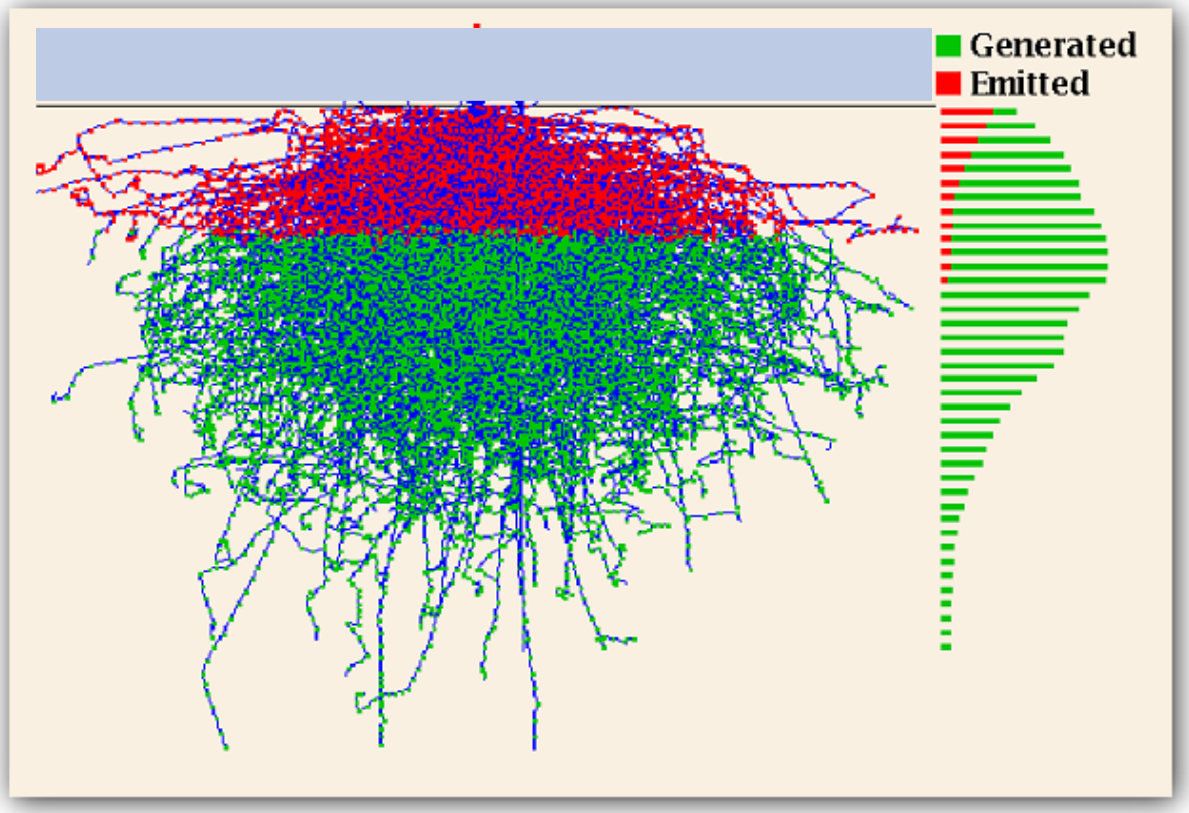




- Relative peak heights of Fe and Li as a small amount of Fe is added
- As we add even a small amount of a heavier element, the Li is strongly absorbed

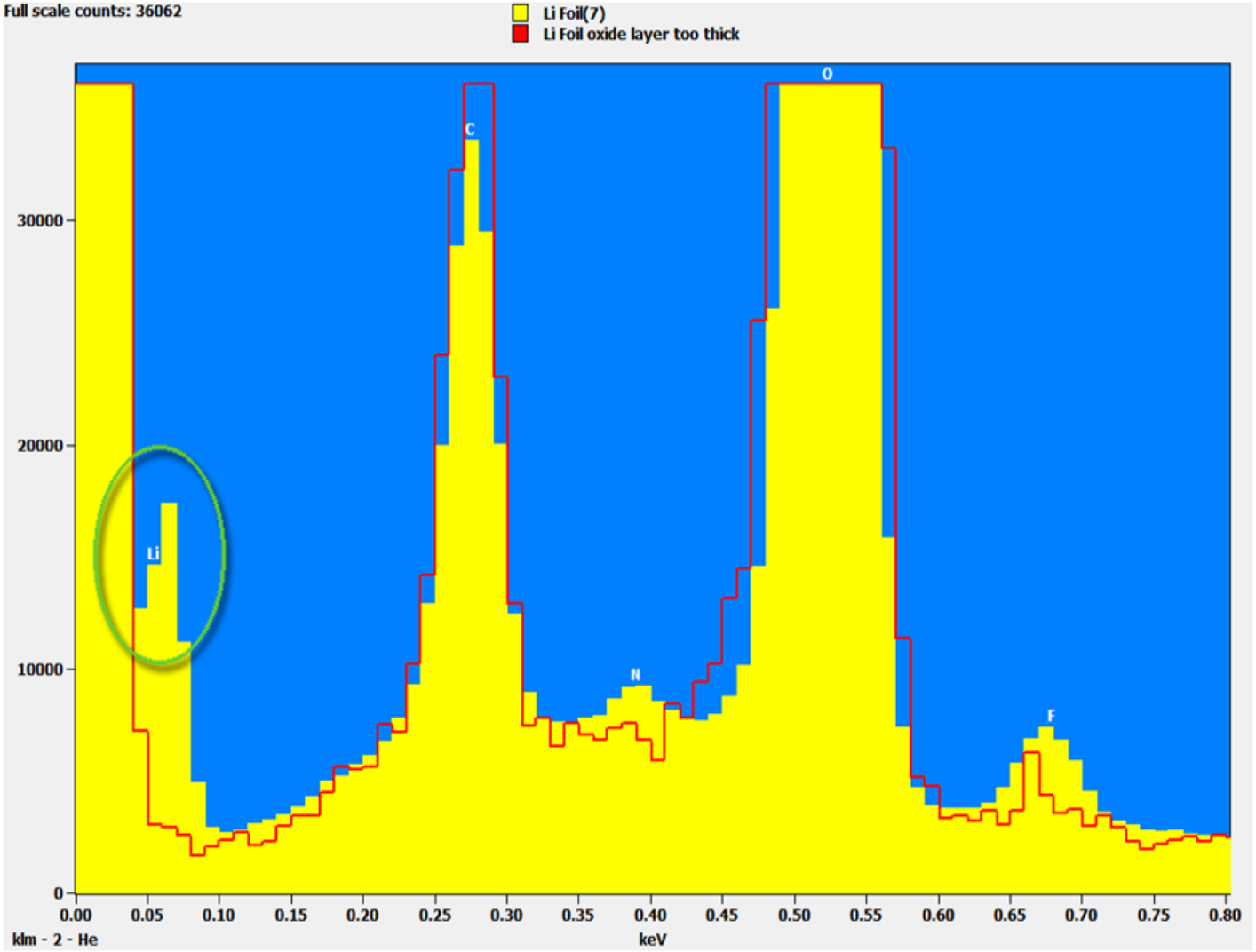
# Effect of a Thin layer on surface

## Absorption example

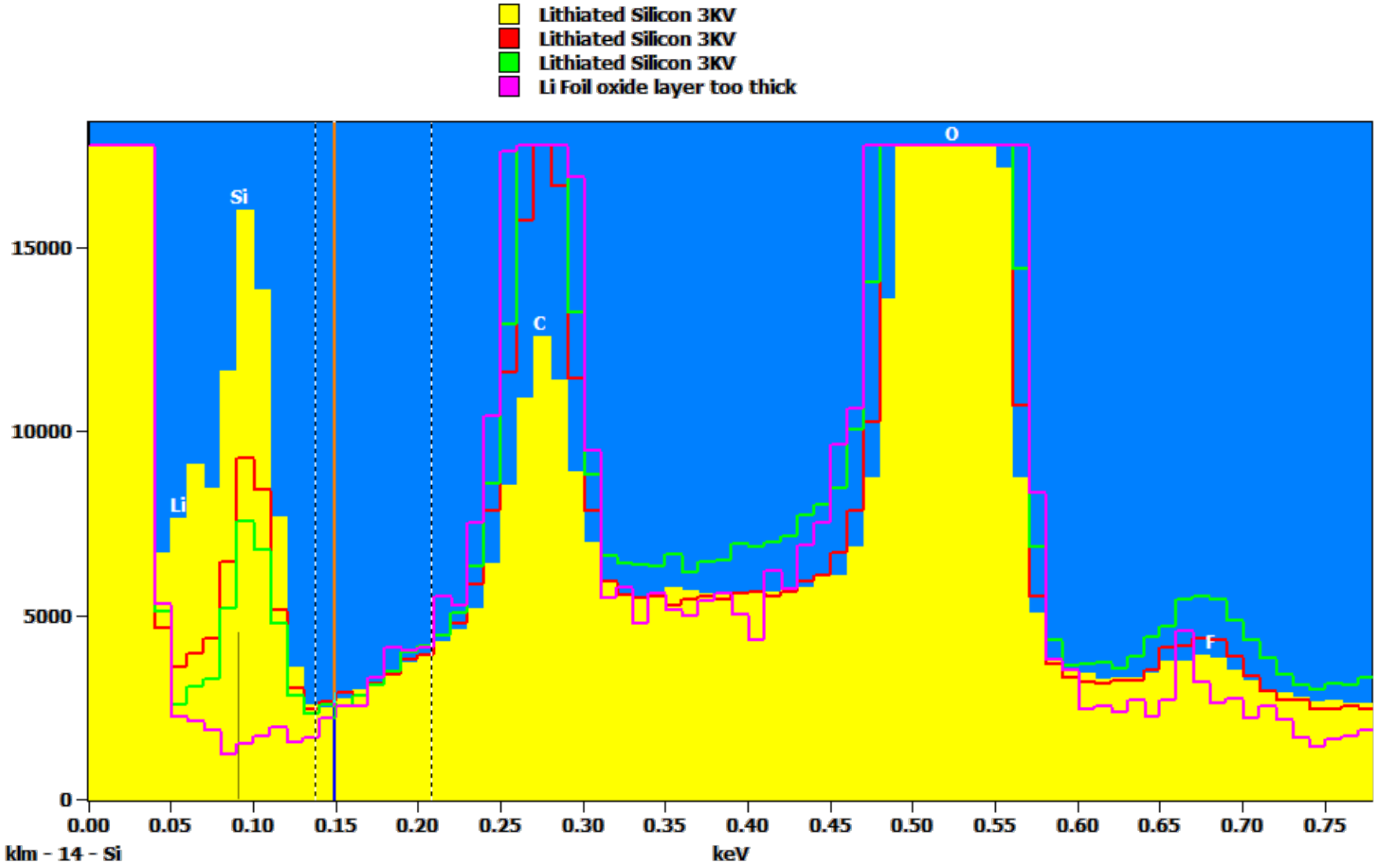
- As a thin layer grows on the surface, low energy x-rays are absorbed



# Actual data: Li foil …. as it oxidizes



# Actual data as the Lithiated-Si surface oxidizes







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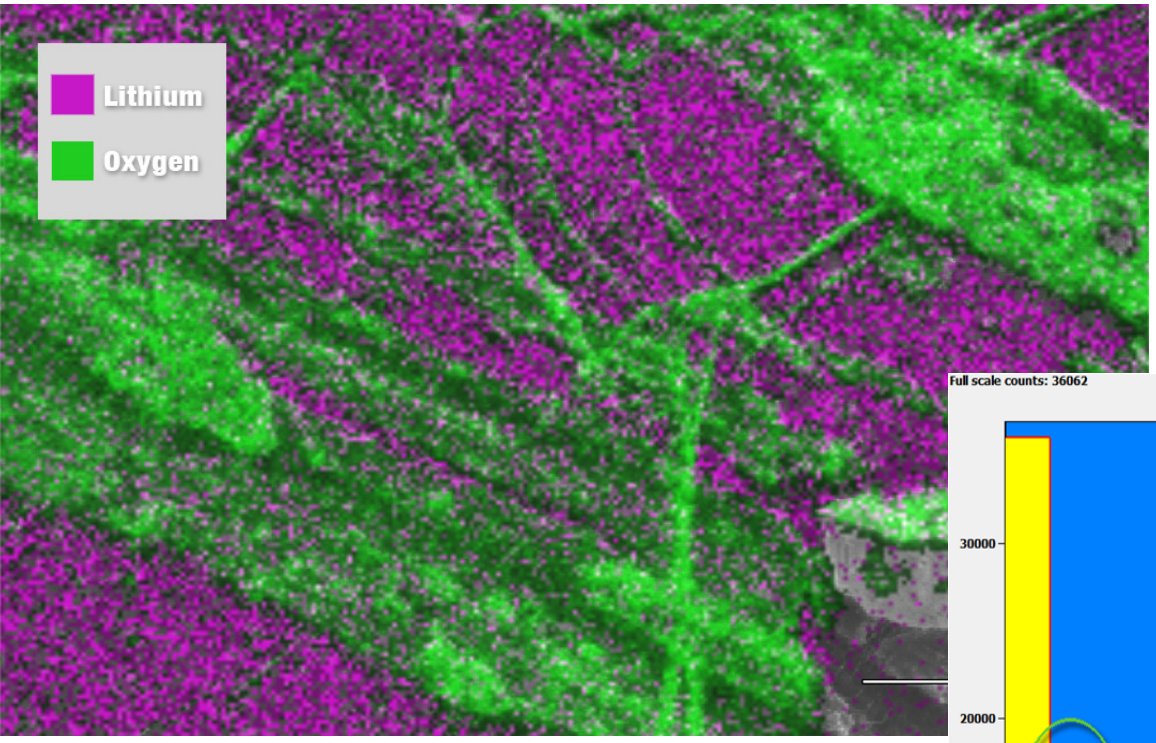
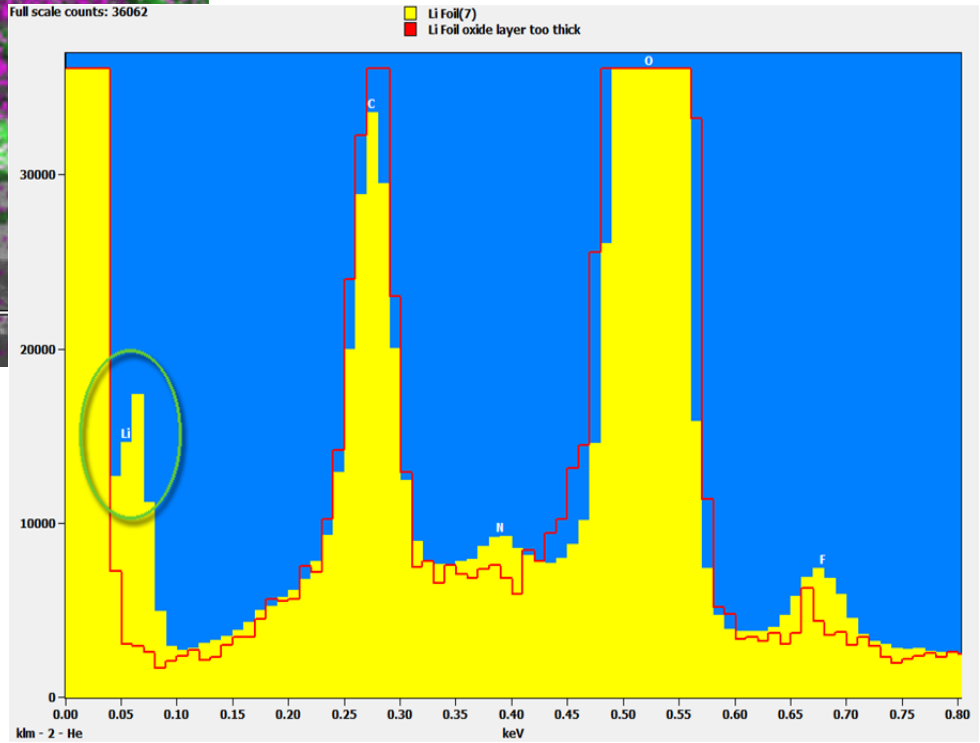
# Challenge #3

Mapping Li (and other light elements)

# Li mapping



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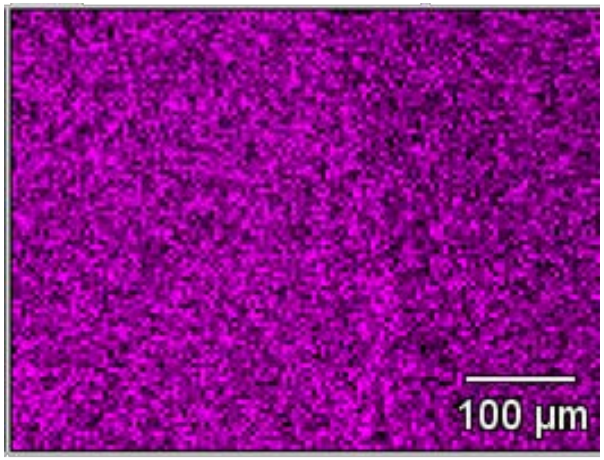
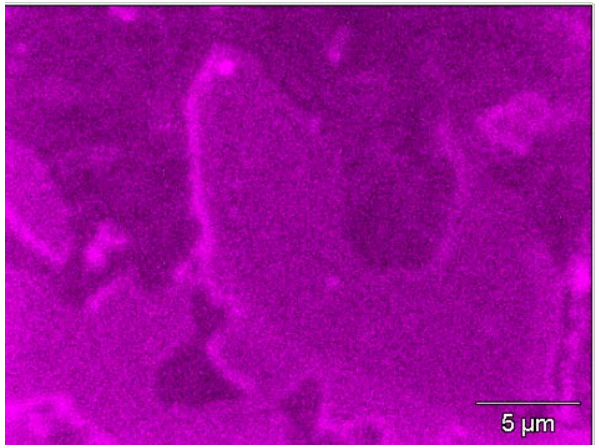
# The “real” application of a Li detector

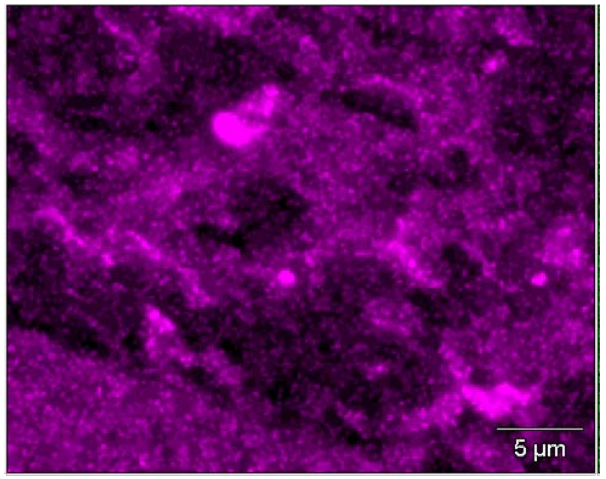
B circa 2008



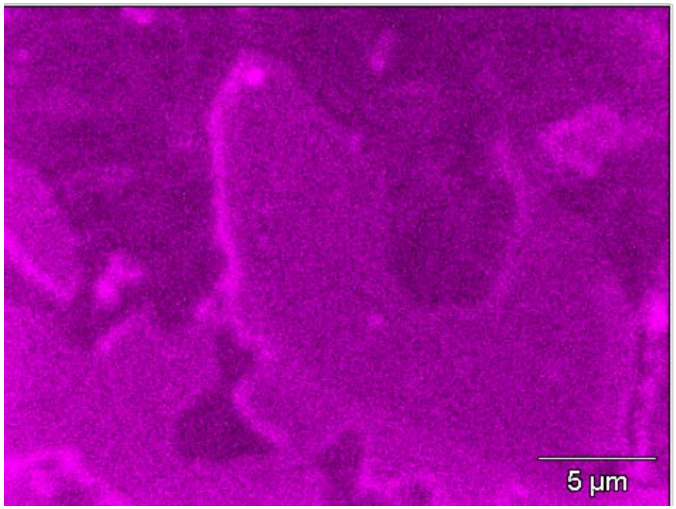

B circa 2012

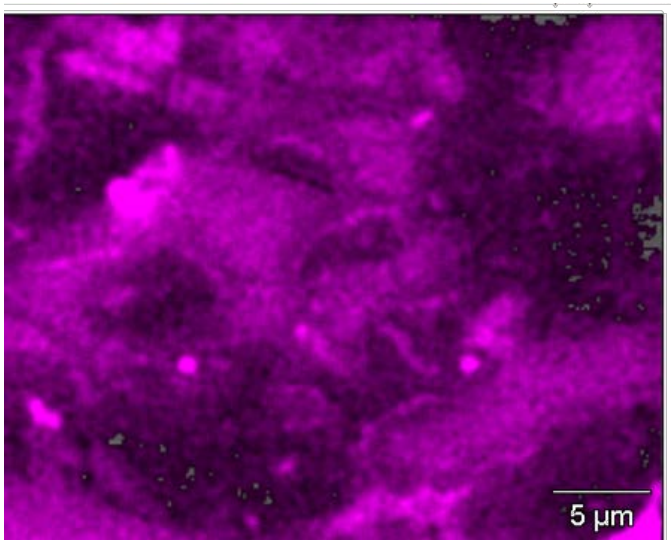


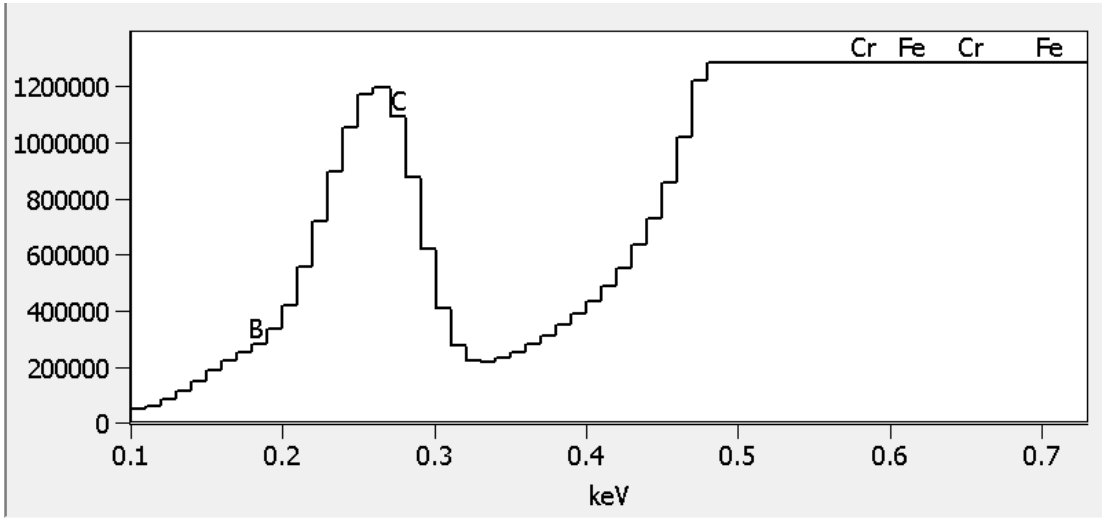

B circa 2014

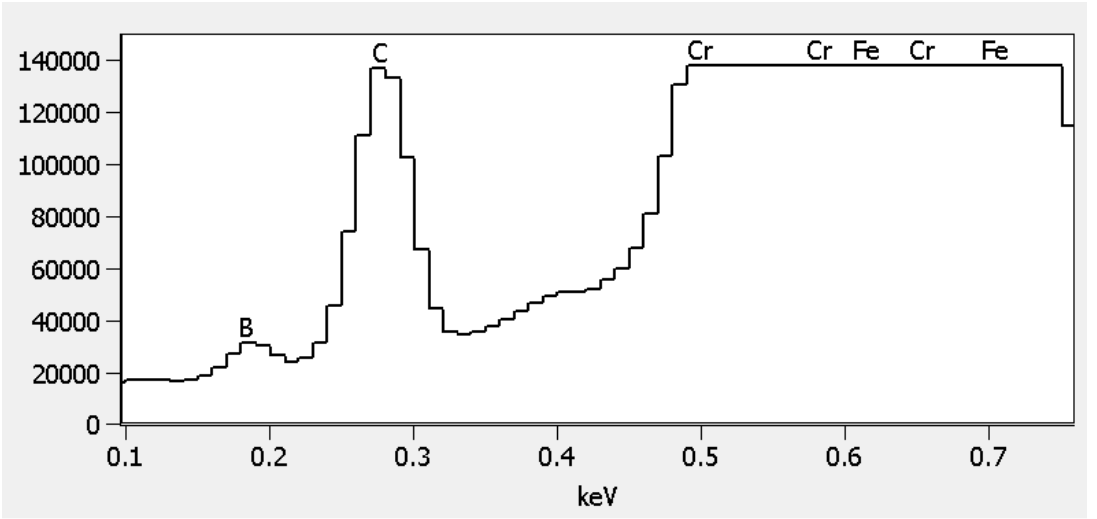


# The “real” application of a Li detector



# Summary

- Li detection is possible

- 3 main challenges
  - The detector has specific requirements.
  - The sample must be prepared and maintained very carefully.
  - The sample must be such that the x-rays can escape from the sample and be measured.

- The biggest gain with a Li detector is an overall improvement in light element detection and mapping.