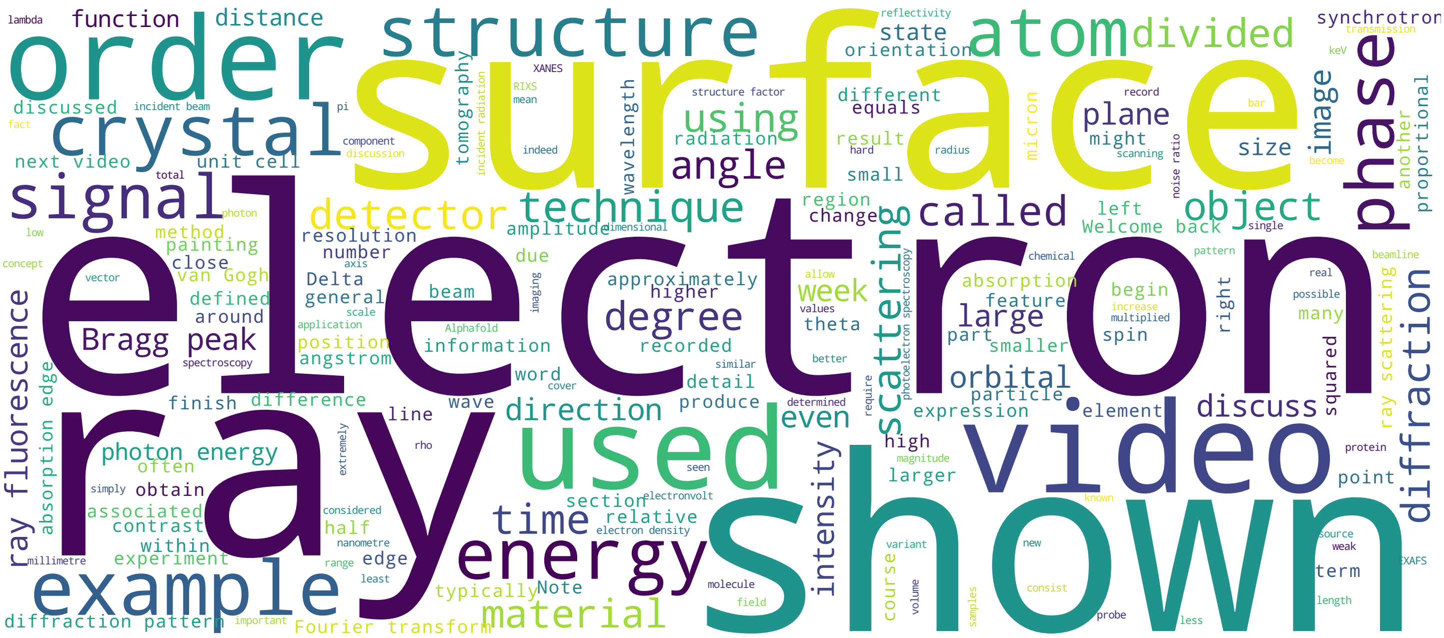
EPFL

École polytechnique fédérale de Lausanne



Search MOOC

Video

EPFL

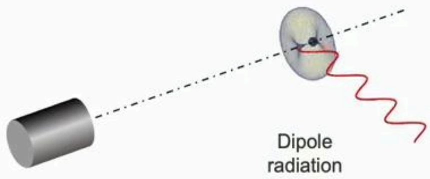
Hello and welcome back to the second video on X-ray fluorescence. In this video, we discuss some experimental factors that need to be considered and finish with an arresting example of the use of scanning X-ray fluorescence in the field of cultural heritage.

Notes

Summary

0m 05s

Experimental considerations



- Avoid elastic scattering
  - Background artefact signal
- Detector at 90° to incident radiation in the plane of the E-field (assuming linear polarization)
  - Dipole radiation
  - Elastic scattering along E-field axis = 0

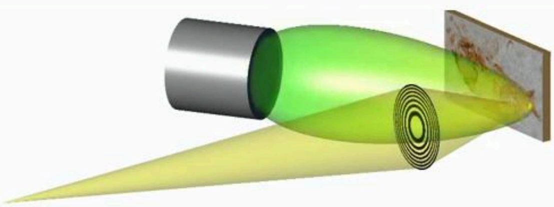
In many, in fact, in most X-ray fluorescence experiments, the incident radiation is not tuned; only the emitted photon energy is scanned. Note that this is not the case in resonant inelastic X-ray scattering, which we discuss in the next video. We want to avoid recording elastically scattered incident radiation from the sample, as this can generate a strong background signal for real X-ray fluorescent signal that might be close in energy to that of the incident beam. This elastic scattering is best suppressed by placing the detector at 90 degrees to the incident beam and in the plane of the electric field of that beam. In this direction, the dipole radiation has a node and the scattered intensity is strongly suppressed.

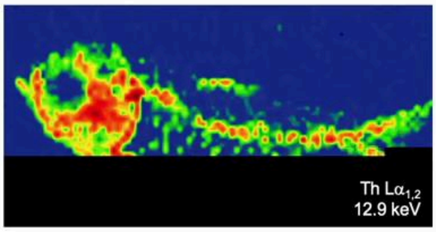
Notes

Summary

0m 25s

Experimental considerations





- Solid-state devices
  - Lithium-doped silicon drift detectors (SDDs)
    - Voltage spikes registered for each photon absorbed
    - Voltage amplitude ∝ hν
  - Fast, parallel acquisition
    - Scanning techniques
  - Large capture cross-section close to 2π sr
  - Low energy resolution
    - ~ 100 eV
    - Elemental presence only (no chemistry)
    - < mg/kg sensitivity (1 ppm)
- Maia detector
  - Array of 384 SDDs in annular configuration
  - Increased S/N ratio, distributed counting rates

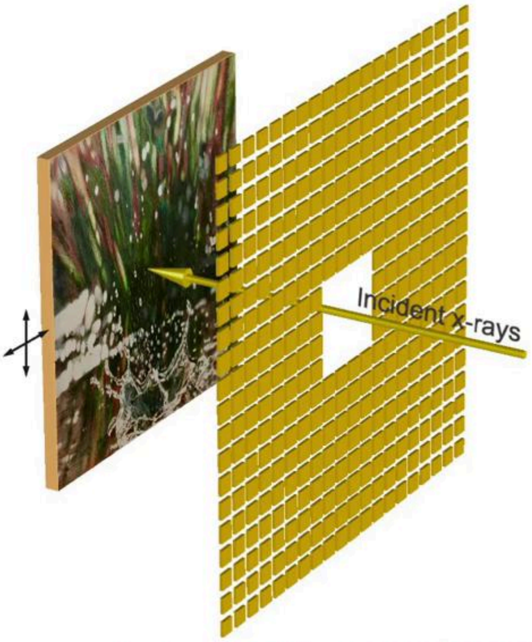
For many scanning X-ray fluorescence experiments in which the elemental composition is sought and subtle shifts in the fluorescent energies is of secondary importance, one can use solid-state devices such as lithium-doped silicon drift detectors or SDDs. For every photon captured by an SDD, a voltage spike is generated with an amplitude which is proportional to the photon energy. This allows fast parallel acquisition of X-ray fluorescent spectra and thus facilitates scanning techniques. If the detector is placed close to the irradiated spot, a large capture cross-section of the fluorescence close to two pi steradians can be achieved. SDDs have a poor energy resolution, however, of the order of around 100 eV, but they do allow sensitivities of trace amounts of material to better than one part in a million.

Notes

Summary

1m 21s

Experimental considerations



- Solid-state devices
  - Lithium-doped silicon drift detectors (SDDs)
    - Voltage spikes registered for each photon absorbed
    - Voltage amplitude ∝ hν
  - Fast, parallel acquisition
    - Scanning techniques
  - Large capture cross-section close to 2π sr
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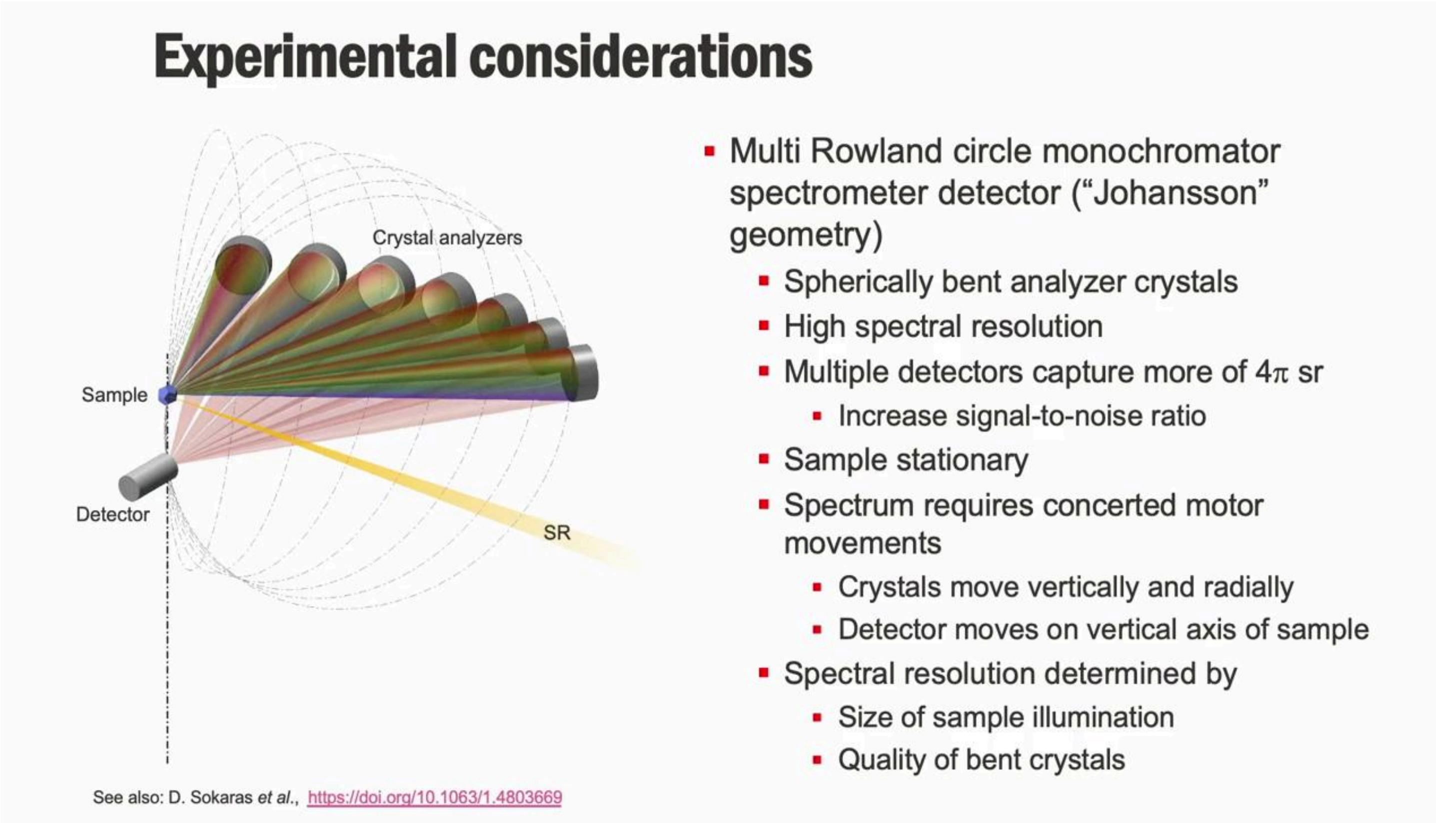
See also C.G. Ryan *et al.*, https://iopscience.iop.org/article/10.1088/1742-6596/499/1/012002

A very sensitive detector that was developed in 2014 is the Maia detector that consists of 384 small SDDs in an annular configuration. The signal-to-noise ratio and dynamic range are improved by distributing the count rates among the individual elements rather than saturating a single device.

Notes

Summary

2m 25s



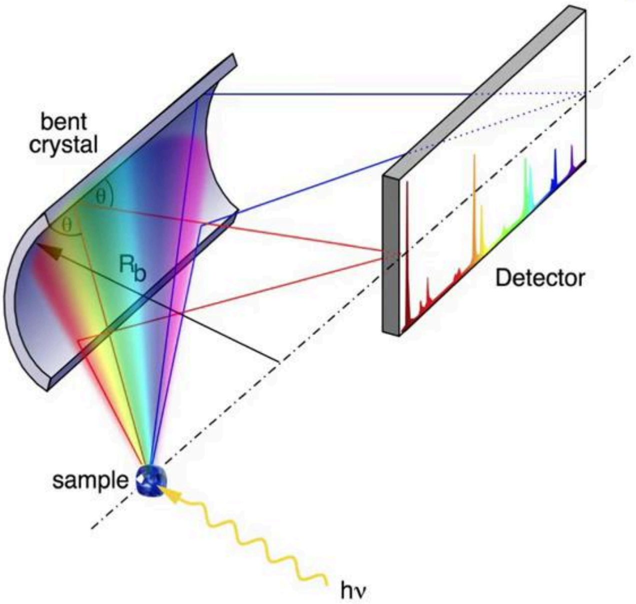
For rapid, high-resolution experiments with good signal-to-noise ratio that permit at least some opportunity for scanning, one can use a Multi Rowland circle spectrometer detector using the Johansson geometry. This consists of an array of spherically diced, bent, or diced analyser crystals that move in concert with the detector around the Rowland circle relative to the stationary sample position. The spectral resolution is determined on the one hand by the size of the sample illumination, the smaller being the better, and on the quality of the bent crystals. The dispersive geometry is discussed in detail in video 5.2.4 of part 1 of this two-part course.

Notes

Summary

2m 48s

Experimental considerations



- Von Hamos polychromator spectrometer
  - Cylindrically bent crystal, radius R_b
    - Maximize arc subtended angle for S/N ratio
    - Sample and 1D/2D detector on cylinder axis
    - Follows Bragg's law
      - Spatially dispersed fluorescence spectrum
      - High resolution
  - No moving parts
    - Fast!
      - Time-resolved studies
      - "Single-shot" experiments
      - XFELs

Still faster is the Von Hamos geometry spectrometer, also discussed in video 6.2.5 in the sister course. This setup provides rapid high resolution spectra and has a geometry with a cylindrical symmetry. By maximising the fraction of a cylinder covered by the bent crystal, the signal-to-noise ratio is enhanced. The detector need only be a 1D strip detector, but is often in actuality a 2D area detector. Importantly, there are no moving parts making Von Hamos detectors especially attractive for single-shot and XFEL experiments.

Notes

Summary

3m 41s

Vincent van Gogh's paintings are perhaps the foremost example of post-impressionist art. The man himself led a very troubled life, which ended in his suicide at the age of 37. Van Gogh only sold one painting during his lifetime. This painting now resides at the Pushkin Museum in Moscow. The rest of van Gogh's more than 900 paintings were not sold or made famous until after his death. A corollary of this is that he lived most of his life in poverty, forcing him on many occasions to reuse old canvases, thus obliterating almost all of his earlier oeuvre.

Notes

Summary

4m 24s

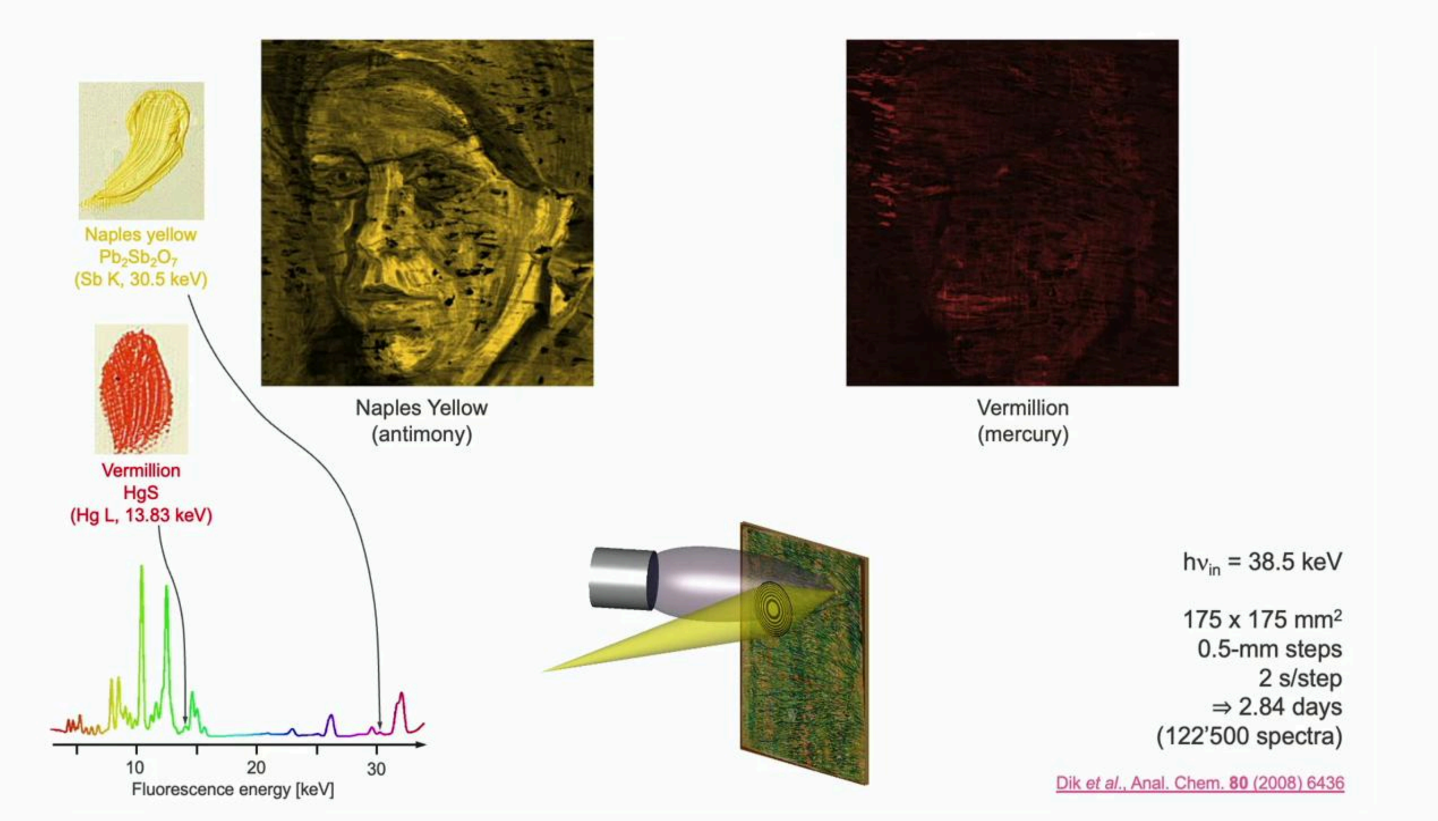
XRF example – The head under the grass

The painting, Patch of Grass, which was painted by van Gogh in the spring of 1887, now resides in the Kröller-Müller Museum in the Netherlands. As can be seen here, a vague outline of the features of a three-quarter profile head could be discerned when the painting was imaged using infrared reflectography. The goal was thus to use both X-ray fluorescence and XANES to reveal the underlying painting in more detail. Here, we only discuss the XRF results, though the XANES data proved to be also crucial in a deeper understanding of the materials used in the original painting.

Notes

Summary

5m 08s

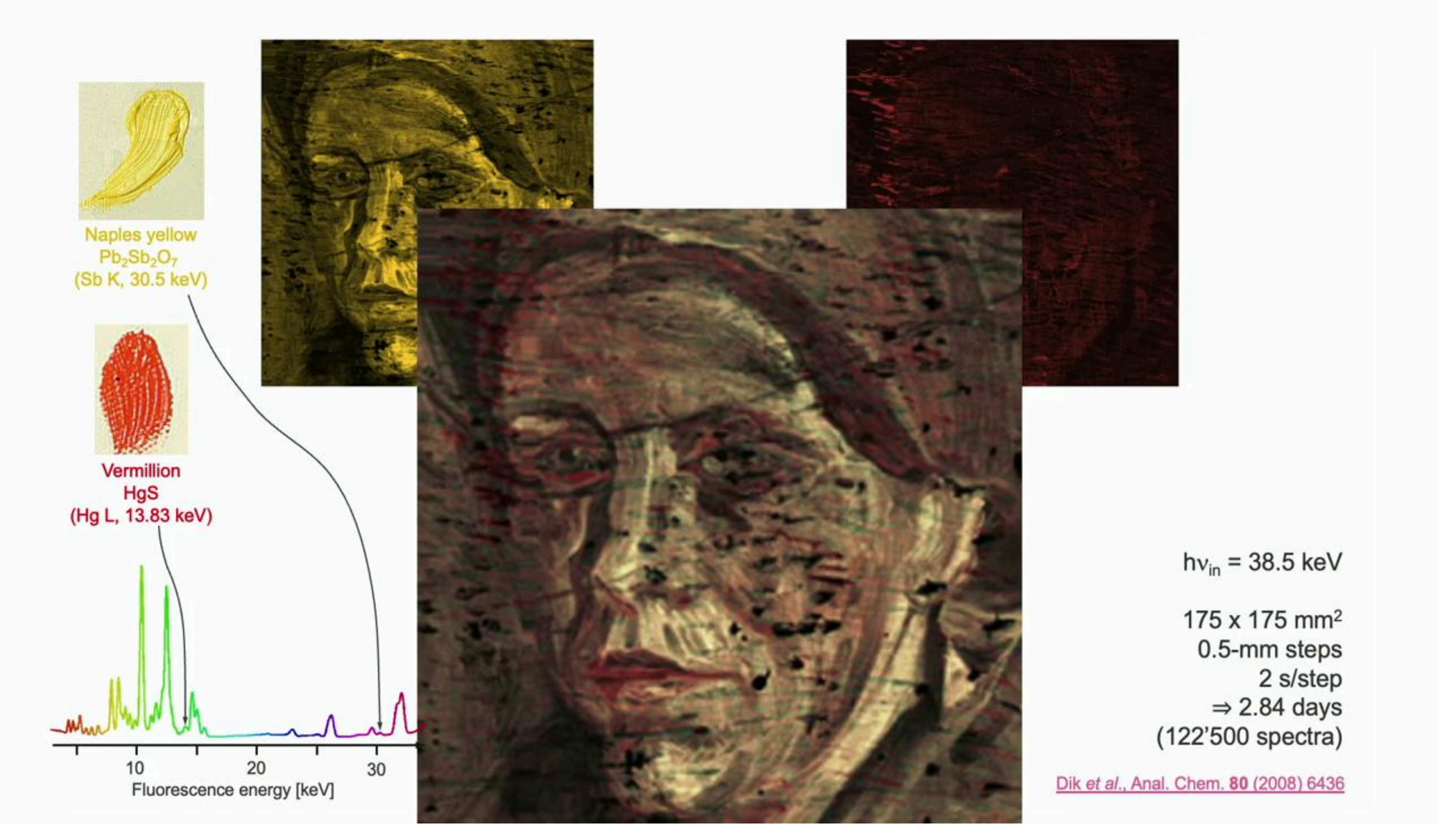


A typical fluorescent spectrum after absorption of 38.5 keV radiation is shown on the left. Two weak but important signals were selected as fingerprint markers of Naples Yellow and Vermilion. The former is uniquely identified by the antimony K line at 30.5 keV, while the latter is identified by the mercury L line at 13.83 keV. The painting was scanned within the red square shown here. This has a side length of 175 millimetres and 0.5-millimetre steps were taken, each step recording a two-second fluorescent spectrum. In total, the scanned region thus required 2 times 350 squared seconds to record, or almost three days. The Naples Yellow map on the left and the Vermilion map on the right showing the intensity of the respective fluorescent lines as they emerge are shown, using much larger step sizes for the sake of brevity.

Notes

Summary

5m 52s



These images could be combined to produce a tritonal colour reconstruction of the antimony and mercury intensity distributions representing the flesh colour of the hidden face.

Notes

Summary

7m 11s

In the next video, we discuss resonant inelastic X-ray scattering, or RIXS, a photon-in-photon-out method that is used to study a wide range of materials, including metals, semiconductors, and insulators, and is particularly useful for studying materials with strong electronic correlations such as high-temperature superconductors.

Notes

Summary

7m 23s