School of Environment X-Ray Centre Induction

THE UNIVERSITY OF AUCKLAND
NEW ZEALAND

Our Goal

Provide a safe and productive research environment, while complying with regulations, policies and law.

Acts, regulations and policies

- Worksafe New Zealand regulations
- Working with sealed/unsealed radiation sources in New Zealand is regulated under the Code of Practice (C17) by the Office of Radiation Safety, Ministry of Health, and the Radiation Safety Regulations (2016) by the National Radiation Laboratory, Ministry of Health.
- The University of Auckland has developed its own radiation policy "University of Auckland Radiation Protection Plan v10 2016"
- The X-Ray Centre provides
  - User guides
  - Training
  - Facility Management
  - Analytical services
- Use of the lab is based on the condition that users fully comply with all of the listed legislation, codes and policies. All users and operators must follow these at all times. Failure to do so can result in access being revoked.

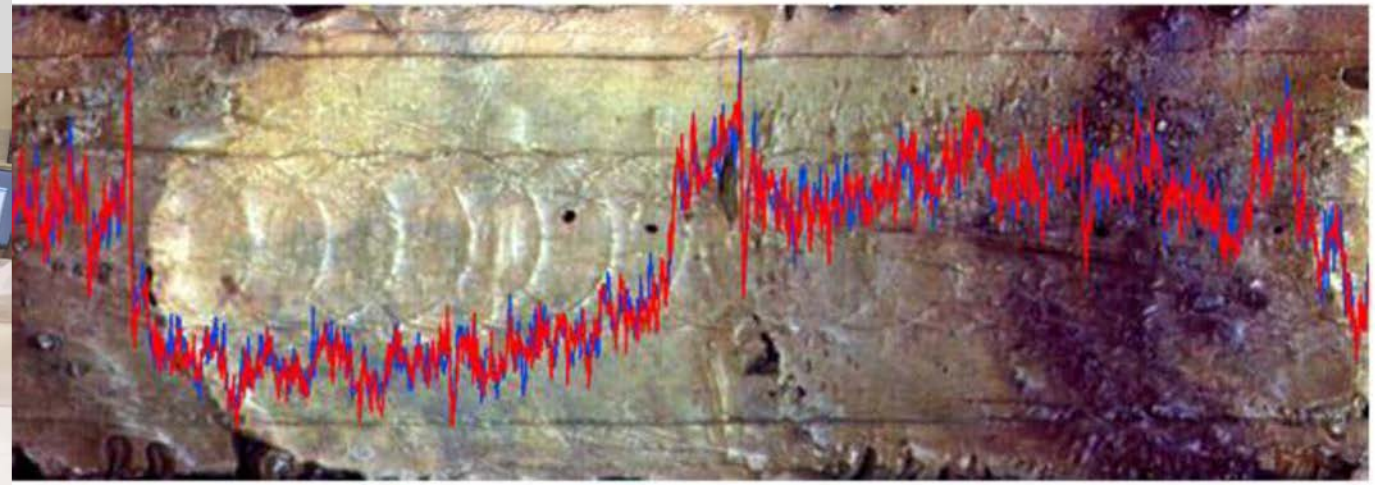
The X-Ray Centre

Empyrean

Axios

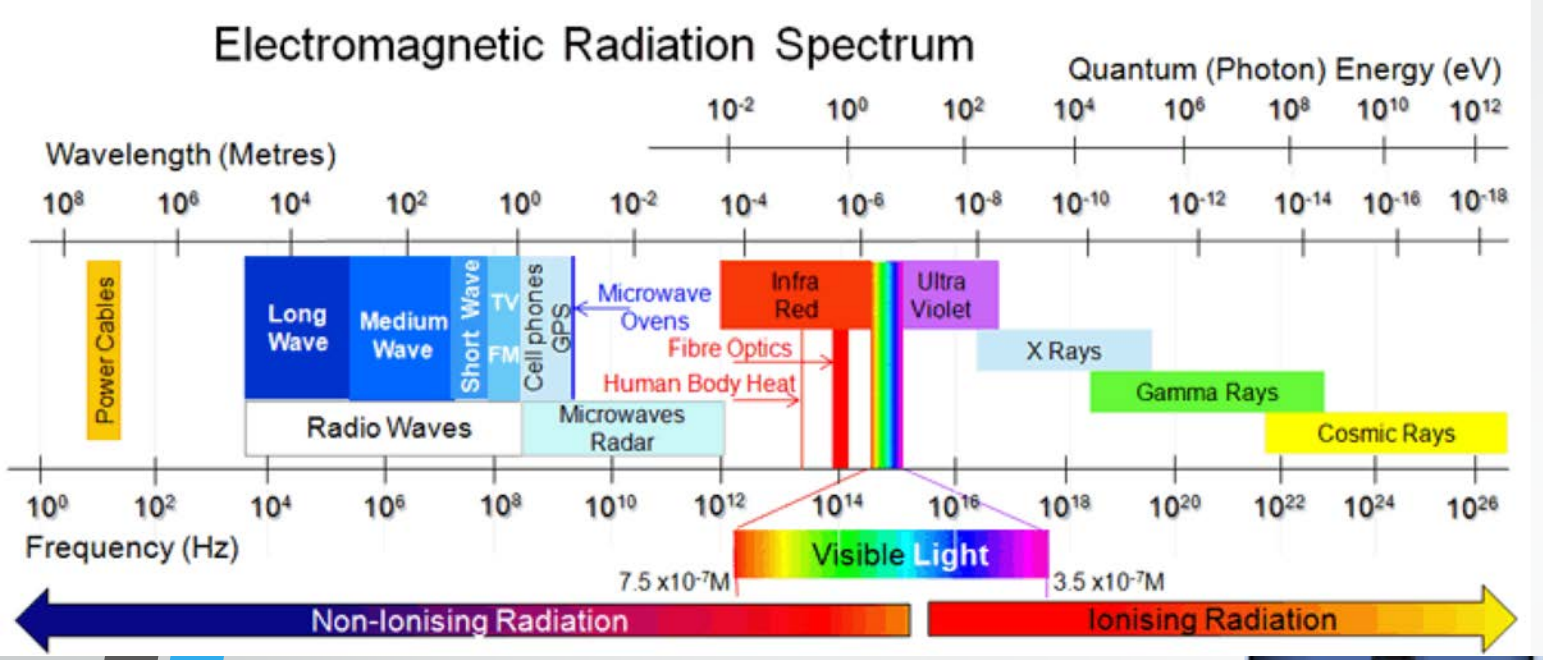
Eagon 2

COX Itrax



What does the X-ray centre offer

Facility	Detail	Main use	Sample type/amount required
XRD	• PANalytical Empyrean powder XRD • Cu X-Ray • Wavelength 1.541Å	• Phase identification of materials/ores etc • Structured determination • Thin films • Nano-materials	• Powder • 0.5g • Non-destructive
XRF	• PANalytical Axios (WDXRF) • Rh X-Ray	• Elemental analysis – qualitative and quantitative	• Powder • 5g Minimum • Destructive
iTrax Core Scanner	• COX Analytical • Mo x-ray • Cr x-ray	• Geochemical analysis	• Almost any solids • Non-destructive
Eagon 2	~1100c	• Automated fusion machine for XRF measurements	
Muffle Furnaces	~1000c	• Loss of ignition (LOI) and heating clay samples	

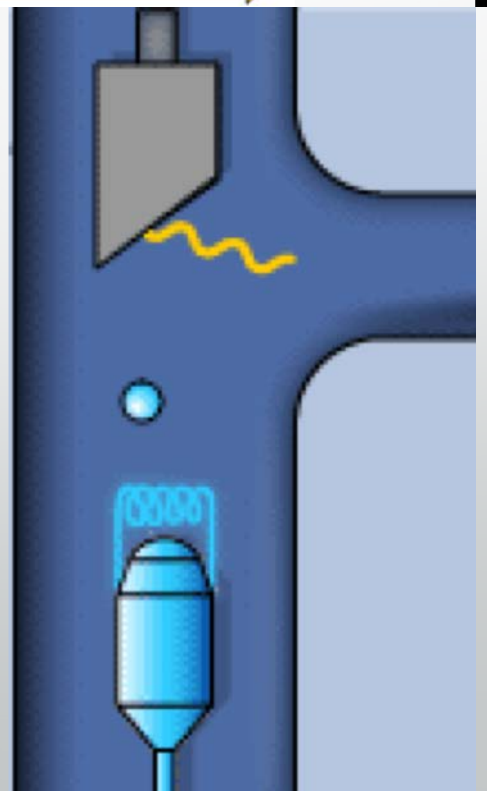


Wilhelm Röntgen
March 1845–
February 1923

X-Rays may be produced when a beam of
Electrons or X-ray photons
Of sufficient energy interact with matter

In generating x-rays:

- 99% of the energy is dissipated as heat. Anode materials are selected to withstand the high temperatures.
- 1% is given off as x-rays

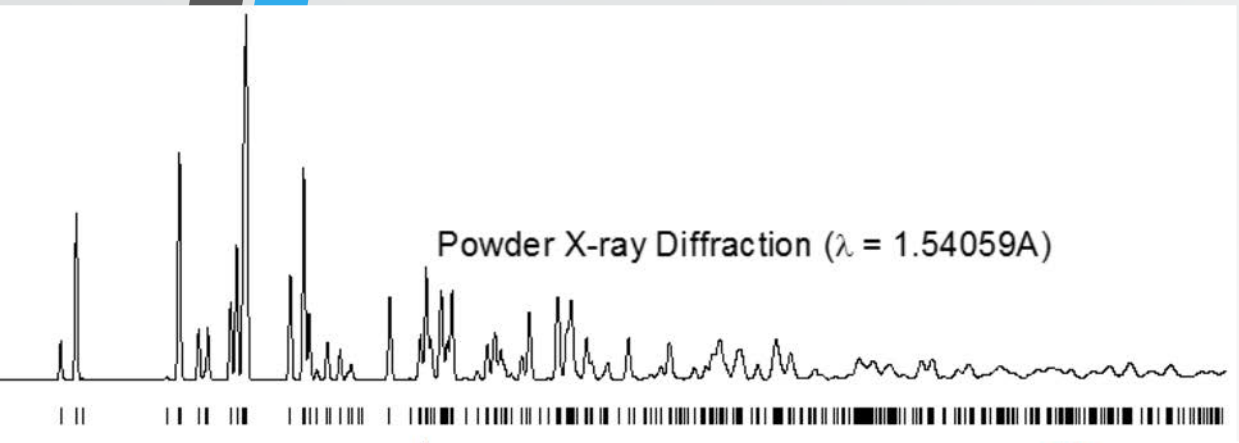


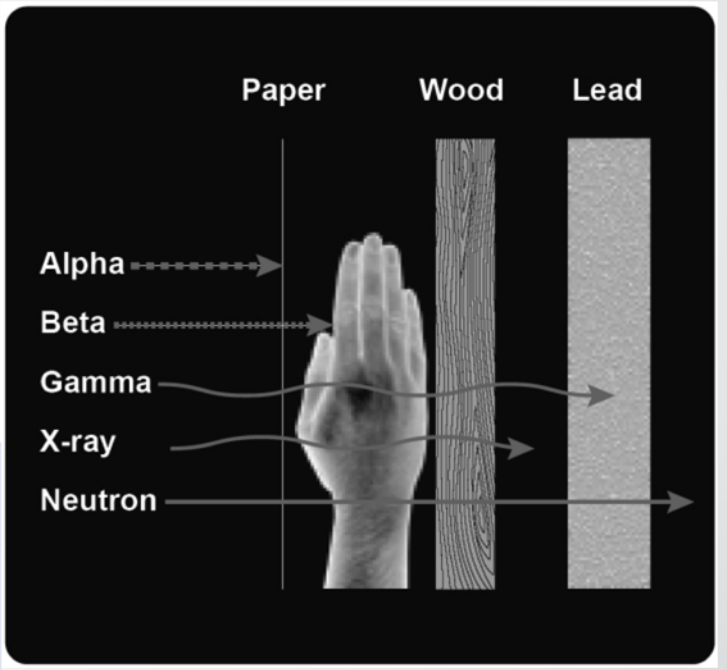
Different anode materials will produce different characteristic x-ray spectra and different amounts of bremsstrahlung radiation.

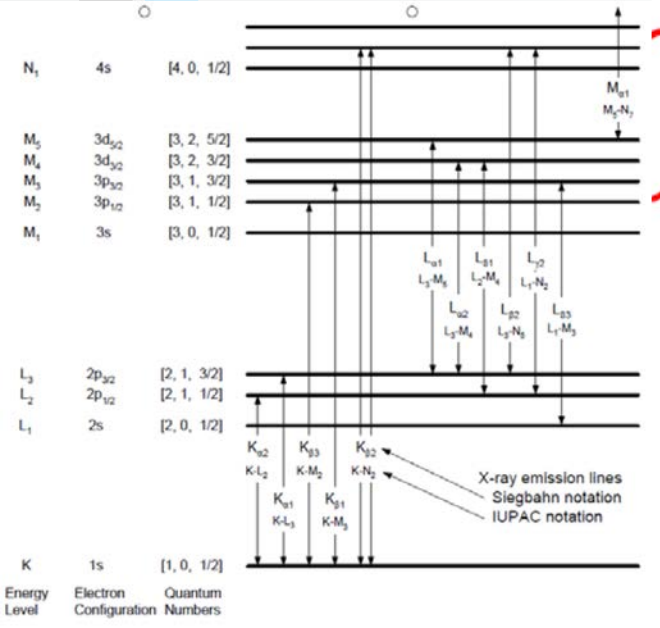
The common elements are

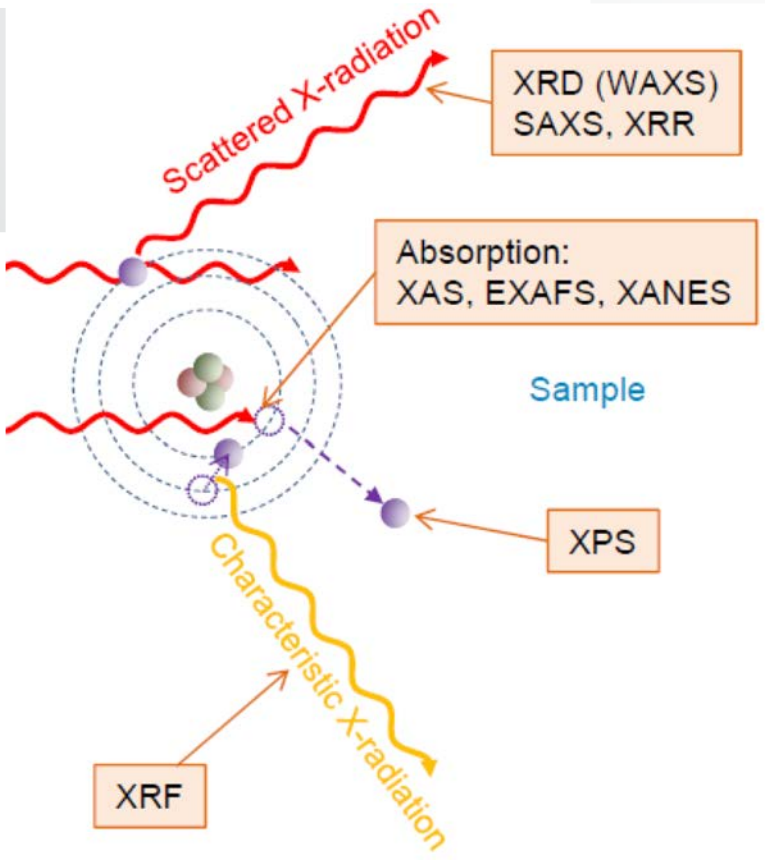
- Cr
- Cu
- Mo
- Rh
- Ag

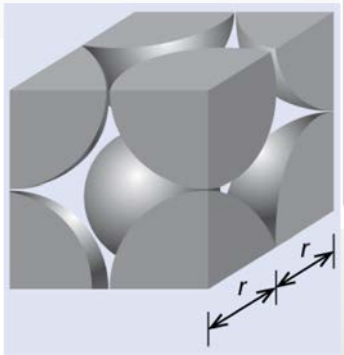
Interaction of ionising radiation and matter

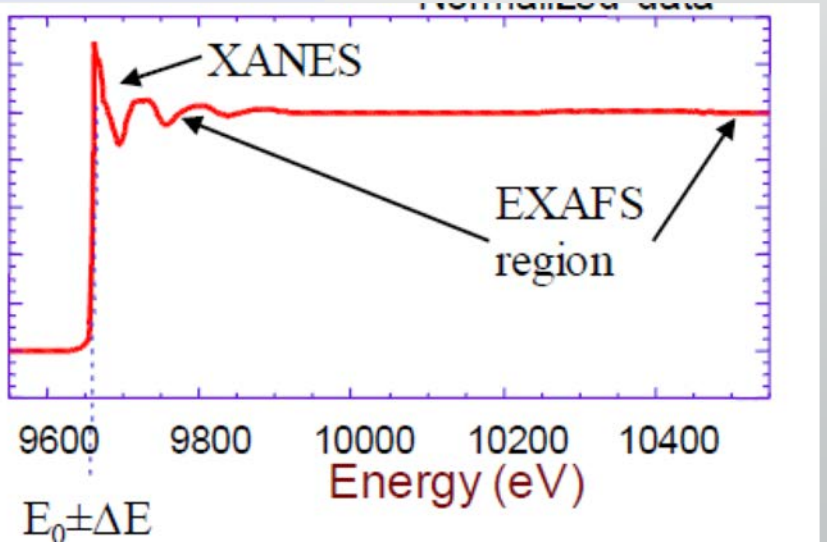












How to gain access to the x-ray centre

1. Safety Inductions
    1. School of Environment General Induction
    2. X-Ray Safety Induction (you are here)
        1. XRD, CRF, iTRAX
2. Theory test (CANVAS)
3. Practical Operation Training
4. Lab book signed and details provided to access control

Why do I need to go through all of this?

- You need to know how to use the equipment correctly, safely, and within guidelines. X-Ray equipment is dangerous if used incorrectly.
- You cannot see or feel the x-rays.
- Personal protective equipment is essential.

Units of radiation exposure and dose

- Exposure - Roentgens
- Absorbed dose - Gray
- Dose Equivalence – Sievert
  - Relative biological effectiveness of different types of ionising radiation
- The effective dose rate - Sievert

Background dose in New Zealand

- Approx 1.8 mSv per annum
- Background depends on activity e.g. number of medical x-rays received

Note that airline crew on international flights are the most occupationally exposed group in NZ; 6-8 mSv per annum. This is received as a result of increaed cosmic radiation received at higher altitudes

What are the effects of radiation?

Everyone receives some ionising radiation exposure!

- Medical x-rays
- Natural radio-activity
- Gamma rays from space (they wont turn you into the hulk)
- Atomic bomb tests
- Aircraft Travel

Biological effect of radiation

X-rays are one form of radiation.
Exposure to ionising radiation can cause cancer, hence we must ensure that exposure to x-rays is kept within safe limits.
Allowable limits of exposure are controlled by national legislation.

Molecular effects of ionising radiation

- Direct effects
  - Radiolysis of DNA
  - Primary feature of high LET radiation
- Indirect effects
  - Free radicals by radiolysis of water
  - Hydroxyl radicals react with other molecules (such as DNA) causing damage.

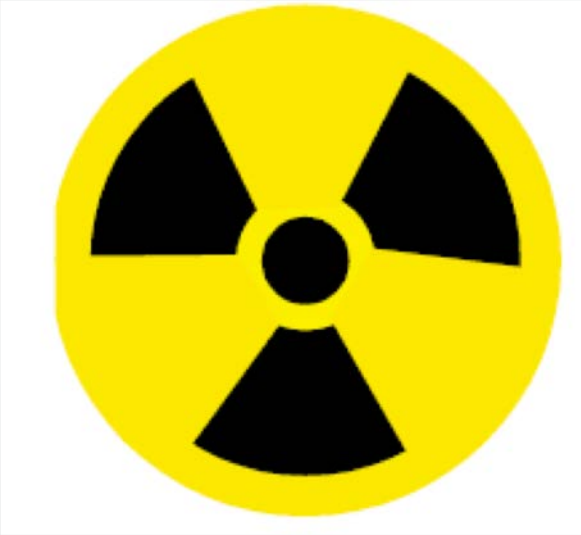
$$2H_2O \longrightarrow H_2O^{+\cdot} + H_2O^-$$

$$H_2O^+ \longrightarrow OH^{\cdot} + H^+$$

Effects of radiation on humans

- Stochastic effects
  - Threshold after which there is an all or nothing effect
  - E.g. cancer or genetic
- Deterministic effects
  - Vary with Dose
  - E.g. lens opacification, blood changes
- Total body irradiation
  - Highly unlikely that an individual would survive a total exposure of more than 3 Gray without intensive medical treatment
- Partial body irradiation
  - Cataracts are formed if eyes are exposed to more than 2 Gray
  - Hair loss occurs at exposures over 3 Gray

- XRF Uses x-rays that are of relatively low energy (4—60KV) but are high intensity.
- Exposure to the primary x-ray beam will cause deep radiation burns within seconds
- Exposure to the secondary (fluorescent) x-ray beam is less hazardous, but exposure limits could be exceeded within a short exposure time.



X-rays are only produced with the x-ray tube is switched on.

In case of emergency

- Switch off the x-ray unit
- Do not take any remedial action
- Tag unit – do not use or alter
- Notify users, technician, RSO and the relevant authorities
- Refer exposed person for medical examination

Safe operation

- Only trained operators who are aware of the dangers and rules are allowed to operate these machines.
- The safe operation and functionality of the interlocks are check regularly
- X-ray leakage and scatter are monitored on a regular basis
- Equipment is secured against unauthorised and untrained use

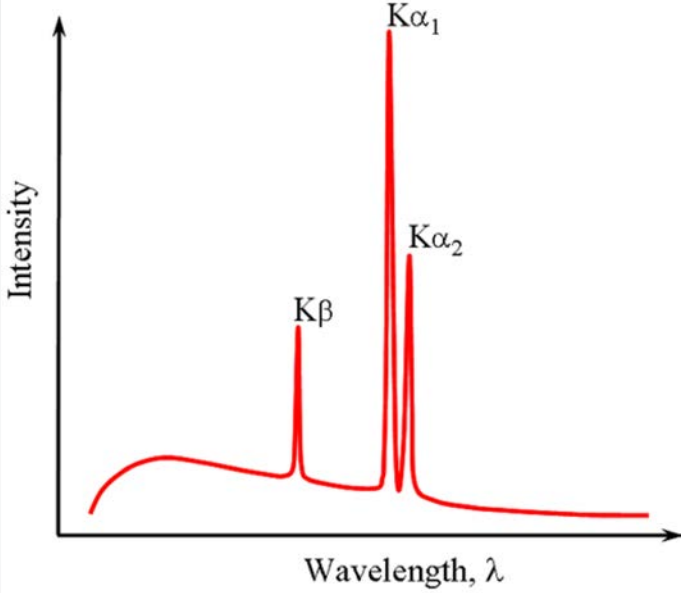
X-ray analytical equipment

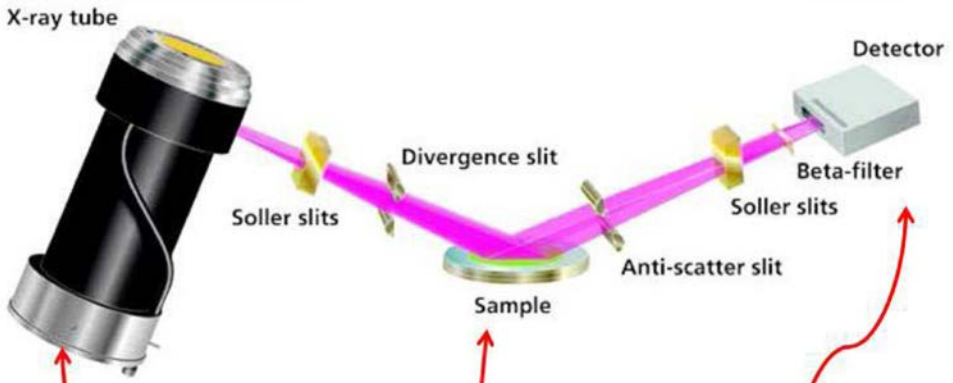
- While most x-rays generated by analytical x-ray units are of low energy and the beam is narrow, their intensity is very high. If an operator was able to put their hand in the path of the beam, then they would sustain x-ray burns.
- In order to ensure safe operation of such machines, the manufacturers have installed interlocks and designed the operation of the machine so that is is extremely difficult for the operator to come in contact with the beam.
- X-ray tubes employ special anodes (Mo, Cu, Fe, CO and Cr) to produce the correct type of characteristic radiation.
- Special windows are employed; Beryllium, Mica or low absorption glass, to minimise the loss of low energy radiation.
- Voltages are characteristically low (30-50kV) but amperages are high (15-20mA)
- Incidents with x-ray diffraction equipment are rare, but those that have occurred are serious.

Powder XRD

Sample types:

- Powder ~1 gram
- Thin film
- Liquid





Setup of the PANalytical x-ray diffractor used for monitoring phase purity

Standard XRD

Powder

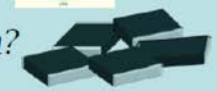
0.05 μm to 50 μm

Powder X-ray diffractometery (PXRD)

Phase identification: *What is this stuff?*

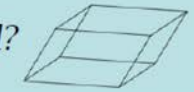
Phase fraction quantification via RIR: *How much is there?*

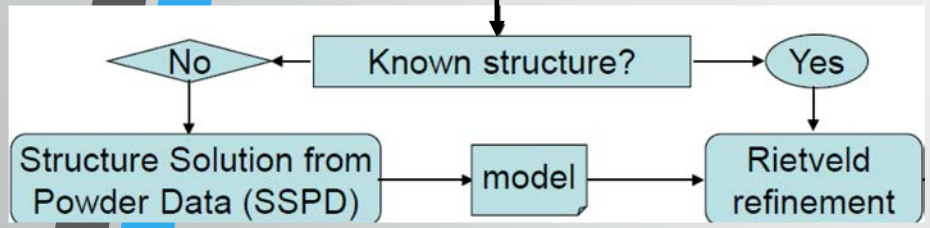
Out-of-plane preferred orientation: *Is sample non-random?*



crystallite size / micro-strain: *What can I learn about the crystallites?*

Lattice parameter indexation / cell refinement: *What is the cell?*

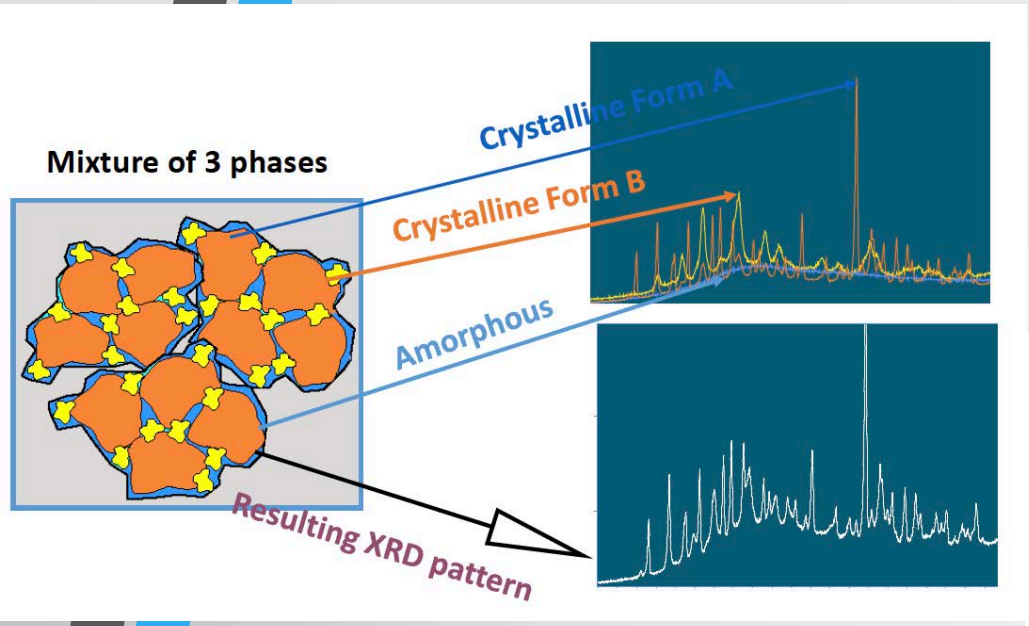




High temperature XRD up to 1200c

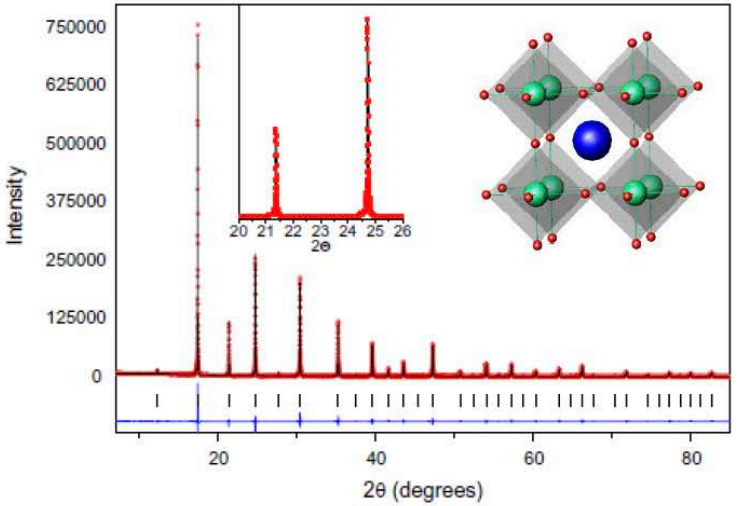
X-ray reflectivity for thin films

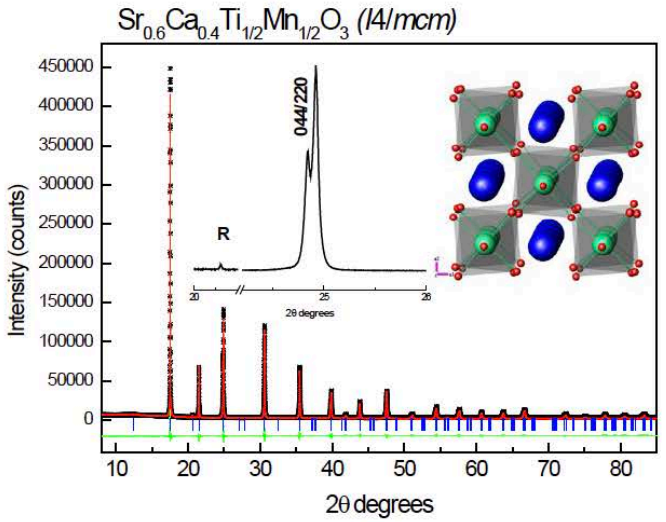
XRD for Phase Analysis



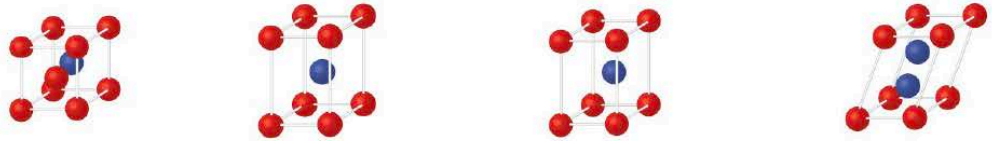
XRD for Rietveld refinement

Example: Structures in Sr1-*x* *Ax* Ti1/2 Mn1/2 O3 (*A = Ca, La, Ba)*





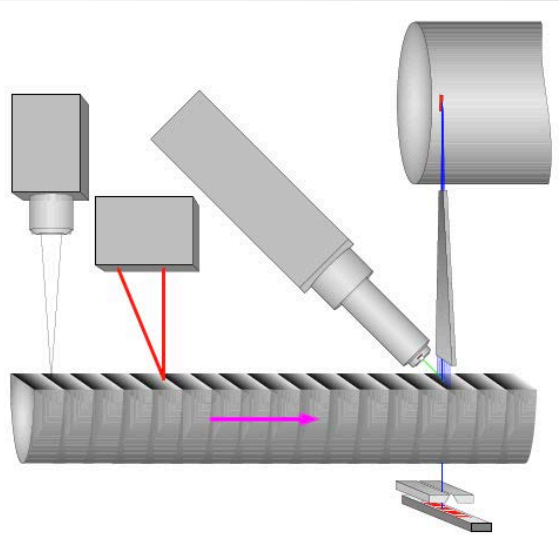
cubic tetragonal orthorhombic Monoclinic …. etc.

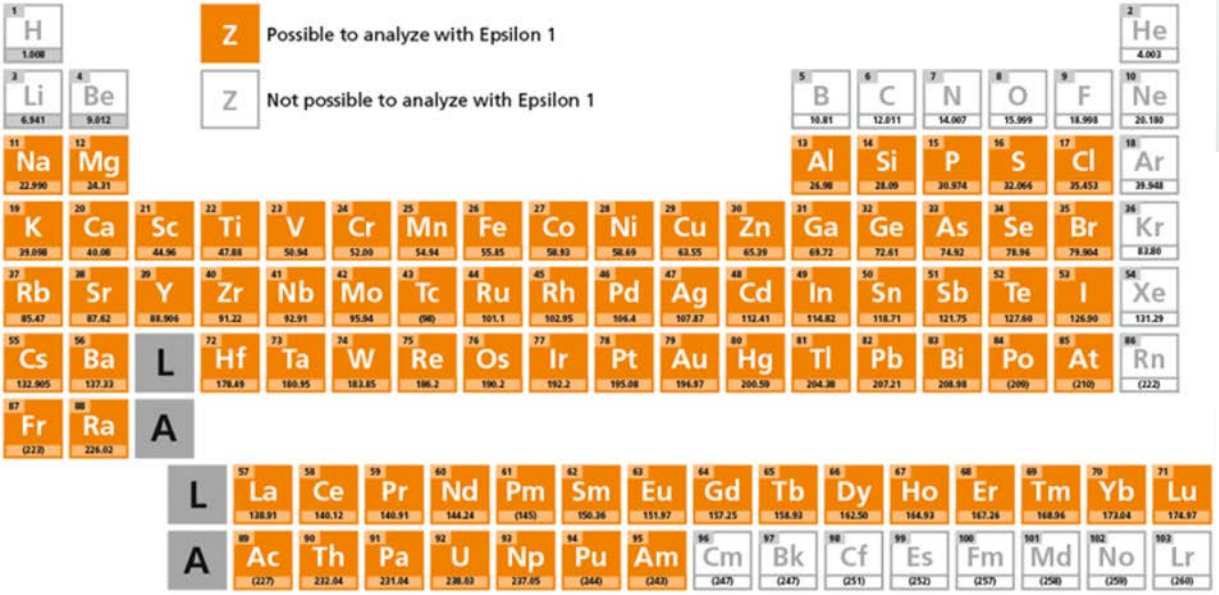


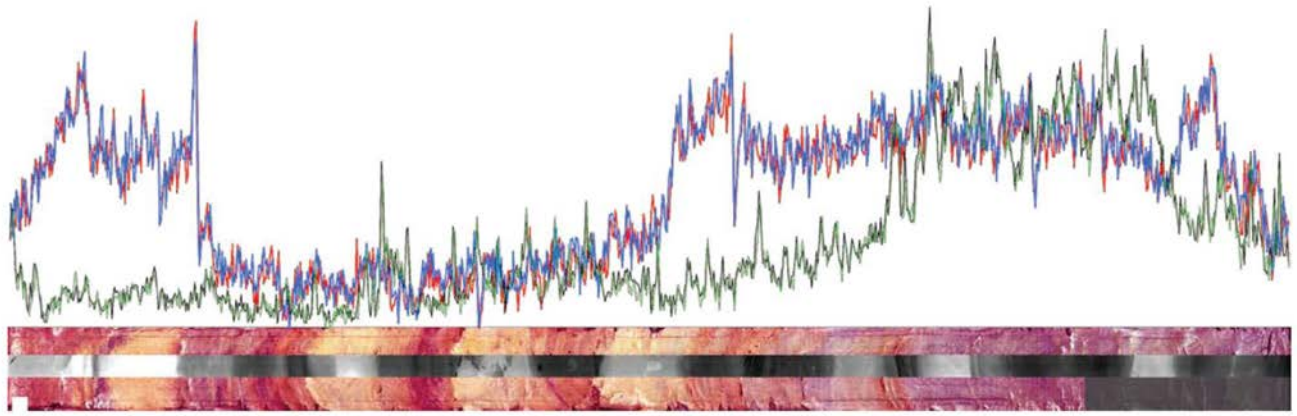
iTRAX Core Scanner

Instrument key features

- XRF Sensitivity – PP levels (transition metals)
- Radiographical image resolution 20 – 200 µm
- Any sample size (shorter than 1.8m)
- Completely non-destructive
- Magnetic susceptibility (optional)



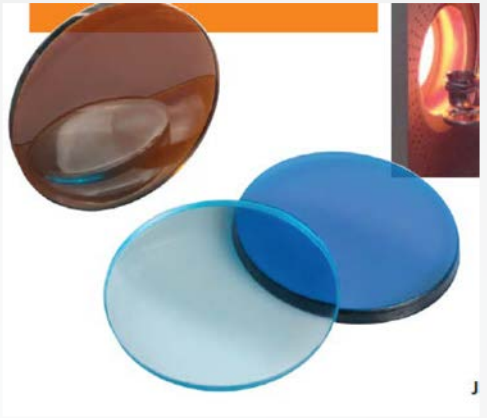




Itrax optical image, radiographic image and XRF data from an Arctic sediment core

XRF

Sample + Flux



Sample : Flux Ratio

1:100

..

1:20

1:10

1:5

1:3

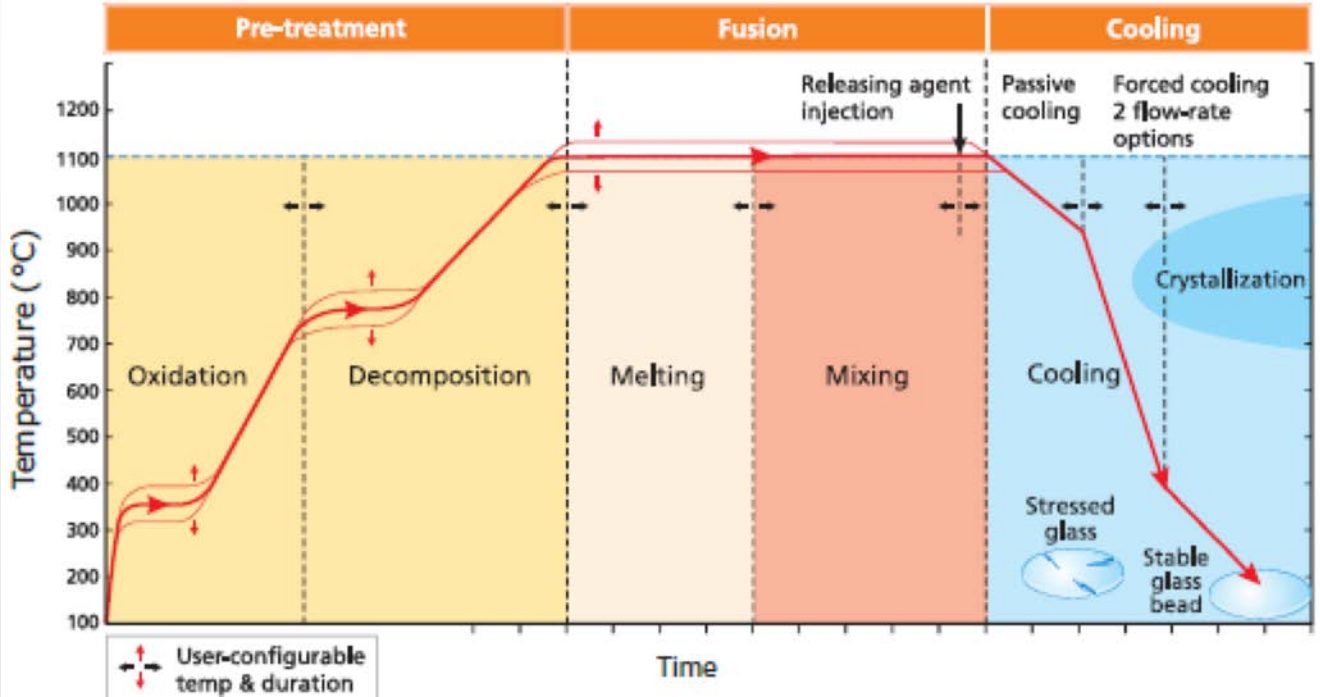
1:2

- 5-10g sample will be destroyed
- Glass disk
- Sample composition – contains sulphur?
- Organic?
- Flammable?
- Explosive?
- Elements of interest?

If you aren't careful this can happen!

Fused bead XRF

Eagon 2 Automated Fusion Machine



Notes

- Booking and scheduling for all experiments is required
- ilab is the University booking system for the x-ray centre. Do not use equipment without a booking confirmation or before talking to the technician in charge.
- All x-ray machines require payment, cash is not accepted
- Users must make sure to finish work within the booked time, and clean up work area after use
- Your first training session is free, after that you will be charged
- X-ray labs are operated during building hours (7.30am-6.30pm weekdays).
- After hours access will need to be approved by the Technical Manager
- Technical support is available 9am - ~4.30pm

Practical training

- Dedicated 2-3 hours for each piece of equipment (XRD, XRF, iTRAX)
- This includes
  - Hazard ID within the lab
  - Sample preparation
  - Programming for measurements
  - Instrument operation
  - Necessary data conversion
  - iLab booking and scheduling
  - Lab user rules and policies
- You can bring your own sample during training (1 sample only for XRD)
- Training does not include detailed data analysis or formatting

QUESTIONS?