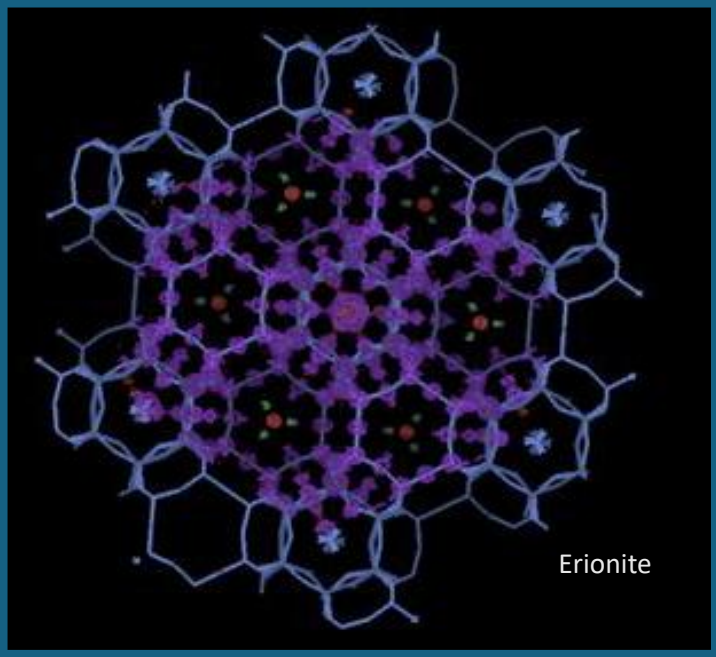

TEM-SAED for Identifying Erionite Fibres in Environmental Sample Protocols

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Erionite in New Zealand
Assessing and Managing Risk

Disclaimer

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Purpose

To confirm the presence of erionite fibres in environmental surface dust samples by analysing their crystallographic unit-cell parameters using Transmission Electron Microscopy with Selected Area Electron Diffraction (TEM-SAED). The key distinguishing feature is erionite's c-spacing of **15.1 Å**, which helps differentiate it from similar zeolite fibres that are difficult to distinguish through SEM-EDX or standard chemical composition methods.

1. TEM Setup

- **TEM model**: Tecnai F20 field-emission TEM (FEI Company, OR, USA)
- **Operating voltage**: 200 kV
- **Sample holder**: High-tilt holder
- **Camera**: TVIPS 16k CMOS, rolling-shutter mode
- **Software**: TVIPS MicroED.exe (for rotation control), EM2EM, ADXV, XDS (for indexing)

2. Grid Preparation and Fibre Transfer

2.1 Grid Type

- Use **300-mesh copper (Cu) TEM grids** with a **carbon-film coating** (hole diameter: 1.2 µm; Protochips, NC, USA).

2.2 Sample extraction process Method

1. Put a leaf sample into a 200 mL beaker with DI water.
2. Sonicate for 2 minutes.
3. Filter the suspension onto a PC filter (0.2 µm pore size).
4. Place the PC filter into a beaker with 30 mL hydrogen peroxide (30%, Fisher Scientific) for 48 hours, followed by heating at 90 °C for 8 hours.
5. Add 100 mL DI water into the sample beaker, sonicate for 2 minutes, and filter the suspension onto a new PC filter.

2.3 Indirect Transfer Method

1. Cut a quarter of the PC filter that containing mineral dust particles and place it in a 2 ml test tube.
2. Add ~2 mL of ethanol into the test tube.
3. Sonicate the sample tube for 2 minutes.

4. Plasma-clean the TEM grid for 20 seconds and place the grid on clean filter paper in a petri dish, ensuring the carbon-film-coated side is facing up.
5. Use a 0.1–2.5 µL pipette to drop the suspension onto the TEM grid inside a fume hood.
6. Cover the petri dish and let the ethanol evaporate for 10 minutes before placing it in the TEM sample holder.

3. TEM-SAED Data Acquisition

3.1 Low-Dose Setup

- Conduct fibre screening using **low-dose search mode** at **1700× magnification**.
- Use **spot size 7** and insert a **40 µm selected area aperture**.
- Set **virtual camera distance** to **975 mm** (resolution potential: 0.75 Å).

3.2 Data Collection

- Put the select area aperture on the fibre
- Set up the rotation from **–40° to +60°** in the MicroED interface.
- Acquire **170 consecutive exposure images**.
- Each frame:
    - **Exposure time**: 1.6 seconds
    - **Rotation step**: 0.6° per frame

4. Data Conversion and Inspection

4.1 File Format Conversion

- Convert acquired TIFF image files to **.img format** using **EM2EM**: http://www.ImageScience.de/em2em

4.2 Manual Inspection

- Use **ADXV**: http://www.scripps.edu/tainer/arvai/adxv.html
- Inspect and record **beam centres** for each dataset.

5. Indexing and Unit-Cell Determination

5.1 Indexing

- Process datasets in **XDS** (X-ray Detector Software; Kabsch 1993, 2010).
- Determine potential **unit-cell constants** and assign a **space group**.
- XDS will default to **P1 symmetry** unless a match is found.

5.2 Reference Unit Cell (Erionite)

If known erionite parameters are detected:

- Re-integrate using:

- a ≈ 13.3 Å
- b ≈ 13.3 Å
- c ≈ 15.1 Å
- α = β = 90°, γ = 120°
  (Ballirano et al., 2017; Gualtieri et al., 1998)

6. Data Quality and Refinement

6.1 Evaluation Criteria

After integration, assess the index statistics for the whole data set (images 1 to 170 in the report)

- **I/σ (signal-to-noise ratio)**
- **R-measure** (data consistency)
- **CC 1/2** (cross-correlation in resolution shells)

6.2 Acceptable indexing results Determination

- Accept data **only up to the resolution shell** where:
  - I/σ > 1
  - R-measure < 100%
  - CC 1/2 > 50%

6.3 Final Output

- Reprocess datasets to include **only reliable resolution shells**.
- Summarise results in **tabular form**, including:
  - Indexed unit cell parameters
  - Completeness
  - Data quality statistics