ELDICO SCIENTIFIC
THE ELECTRON DIFFRACTION COMPANY

PXRD and 3D-ED: GET MORE OUT OF YOUR POWDERS

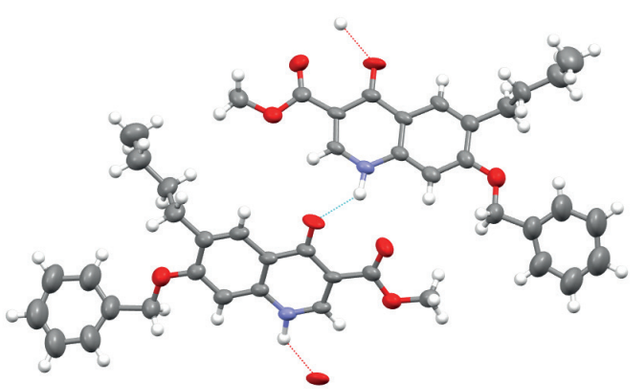
SHAPING THE FUTURE OF CRYSTALLOGRAPHY

If compounds cannot be crystallized in suitable size for single-crystal X-ray diffraction (SC-XRD), structure determination by powder X-ray diffraction (PXRD) has been a commonly applied method. However, structure solution by PXRD can be challenging as structural information could be difficult to retrieve due to peak overlap. Single crystal electron diffraction (SC-ED) has the potential and benefit to complement structure solutions from PXRD by providing additional structural insight on atom position and displacement parameters.

The promising combination of PXRD and SC-ED is here shown on the example of Nequinate. The compound is an antiprotozoan used as a coccidiostat for poultry and rabbits. The structure solution of Nequinate ($C_{22}H_{23}NO_4$) from PXRD experiments was published in 2022[1]. The compound was analysed as purchased from a commercial vendor; no additional crystallisation experiments were needed.

Structure solution took less than 60 min.

By conducting SC-ED experiments on the *ELDICO ED-1*, the published structure from PXRD of Nequinate is supported with additional and more accurate structural information regarding the hydrogen positions and the anisotropic displacement parameters. The process from sample preparation to structure solution took less than 60 mins. The sample was of good crystallinity and after checking a few particles, two suitable particles were found for full measurement runs of 120° rotation each. The collected datasets were integrated to a resolution of up to 0.83 Å and with a completeness of 79 % and an R_{int} of 9.9 %. After finding all non-hydrogen atoms from the initial solution, the hydrogen atoms involved in the hydrogen-bonding network were found from the difference Fourier maps and refined with a low number of restraints (FLAT, DFIX). The remaining hydrogen atoms were placed in calculated positions (AFIX). The structure was refined kinematically with anisotropic displacement parameters for all non-hydrogen atoms to an R_1 of 20.2 %.



[1] J. A. Kaduk, A. M. Gindhart, S. Gates-Rector & T. N. Blanton, Powder *Diffr.* **2022**, 37(4), 211.

Electron diffraction — pushing the boundaries of crystallography

As every crystallographer knows: Crystallization is a tricky thing. Obtaining crystals large enough for X-ray diffraction experiments is not that easy. In many cases, it isnt even possible. And there is more: Only electron diffraction can provide the fastest and most convenient access to a variety of analytical approaches that will shape the future of crystallography.

Electron diffraction supports sophisticated research challenges such as

- Nano-crystals
- Location of light elements
- Detection of mixtures and impurities

The challenge

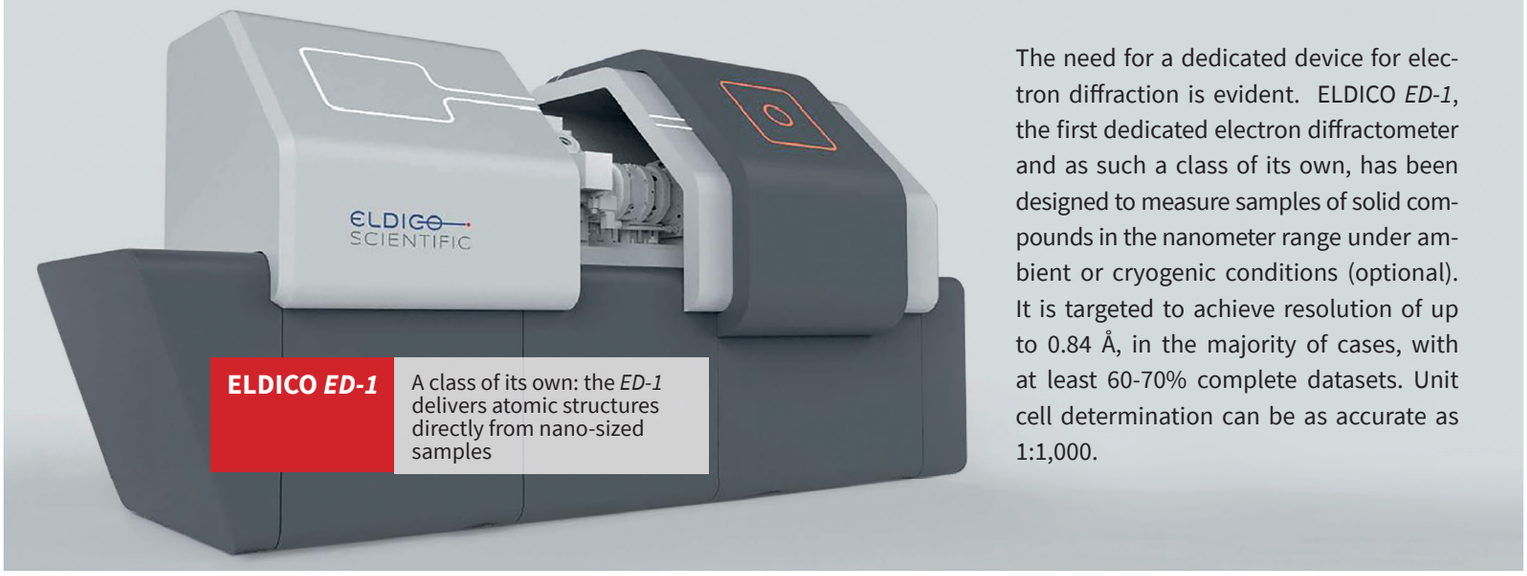
Growing crystals large enough for the X-ray experiments can take weeks, months or even years. This proves to be a true bottleneck for crystallographic experiments at nano-scale.

The solution

Electron diffraction is the method of choice to elucidate structures as small as 50 to 500 nanometers, directly from the flask.

APPLICATIONS FOR ELECTRON DIFFRACTION

1. Structure **ELUCIDATION**
2. Assist **CRYSTAL STRUCTURE PREDICTION**
3. Impurity **IDENTIFICATION**
4. Crystal mapping: **MICRO CRYSTALLINITY OF AMORPHOUS SOLID DISPERSIONS**
5. Absolute **STRUCTURE DETERMINATION**
6. Identification of **UNKNOWN PHASES**
7. Quality **CONTROL**



ELDICO *ED-1* A class of its own: the *ED-1* delivers atomic structures directly from nano-sized samples

The need for a dedicated device for electron diffraction is evident. ELDICO *ED-1*, the first dedicated electron diffractometer and as such a class of its own, has been designed to measure samples of solid compounds in the nanometer range under ambient or cryogenic conditions (optional). It is targeted to achieve resolution of up to 0.84 Å, in the majority of cases, with at least 60-70% complete datasets. Unit cell determination can be as accurate as 1:1,000.

- **ELDICO *ED-1* – rapid data collection under ambient or cryogenic conditions**
- **Superior performance for advanced crystallographic applications**

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