Application Note

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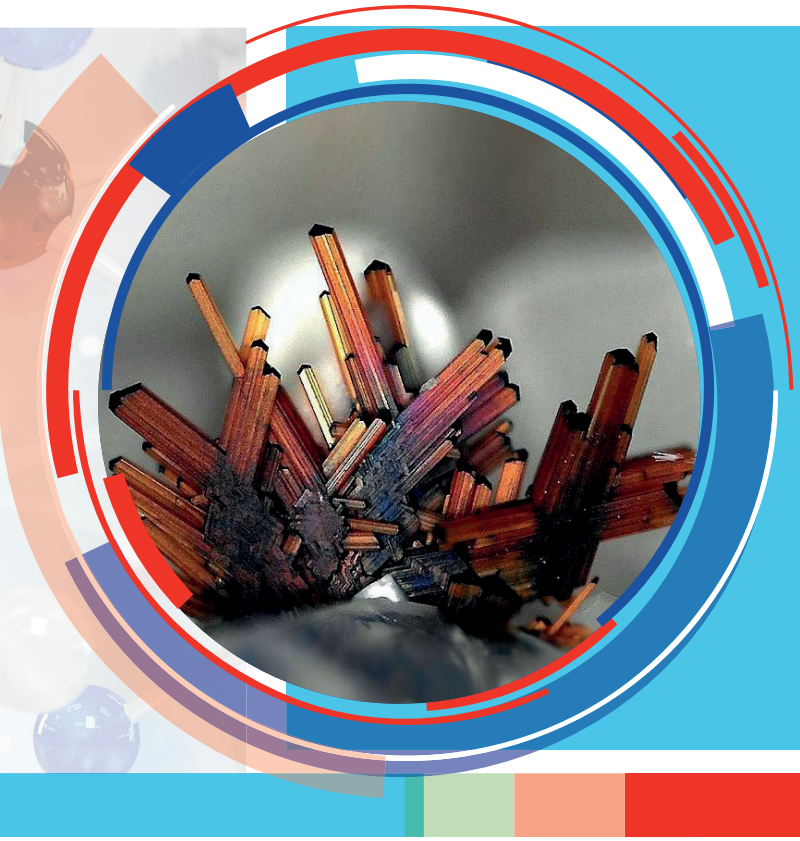
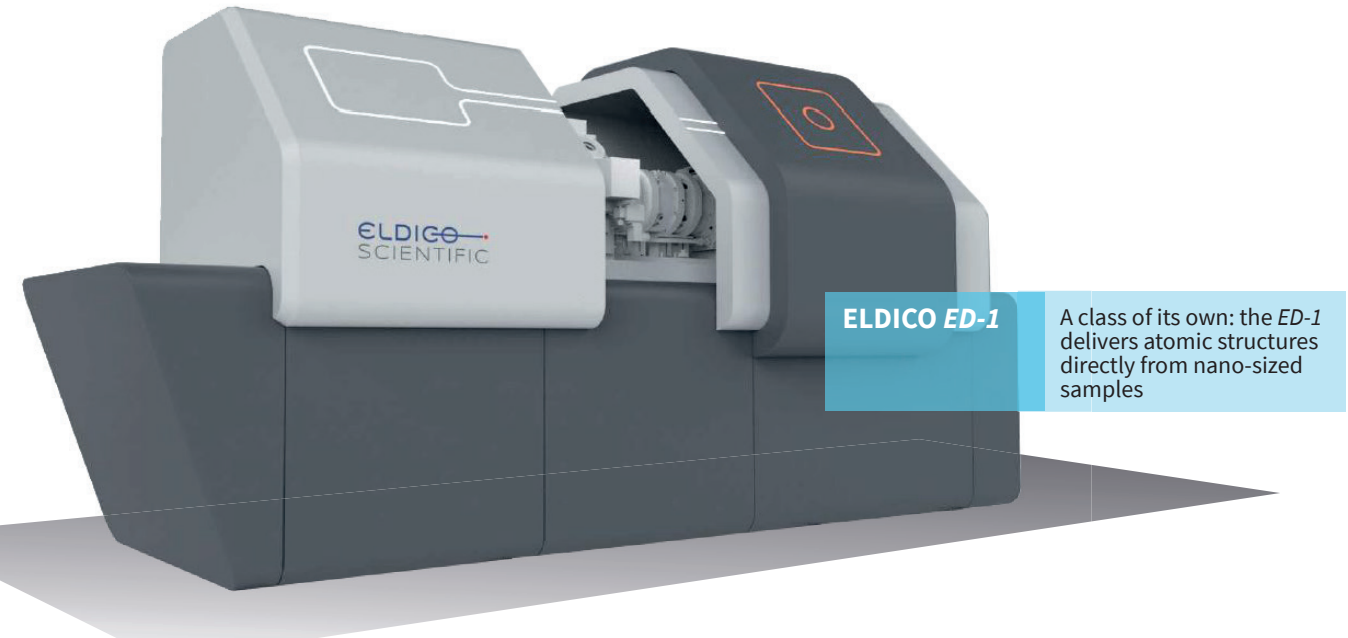


Twinned Crystals: FROM PROBLEM TO OPPORTUNITY

Twinning is a common challenge for single crystal X-ray diffraction and electron diffraction alike. Besides real twins (i.e. intergrown crystals) electron crystallographers also frequently find themselves confronted with pseudo-twins (i.e. separated crystals which are in the beam at the same time) due to the sample preparation based on dispersing a powder on a TEM grid. On the example of a pseudo-twin of the amino acid tyrosine measured on our *ED-1* system we demonstrate that electron diffraction cannot only handle twinning, but can also draw a benefit from it as 2 datasets are collected simultaneously, thus producing a higher completeness from a single φ scan.

Measurement Conditions

temperature	ambient
electron energy	160 keV
wavelength	0.02851 Å
data collection method	single scan continuous rotation
φ range	-70° to 30°
φ increment	1°
exposure time	0.5 s per frame
total measurement time	50 s

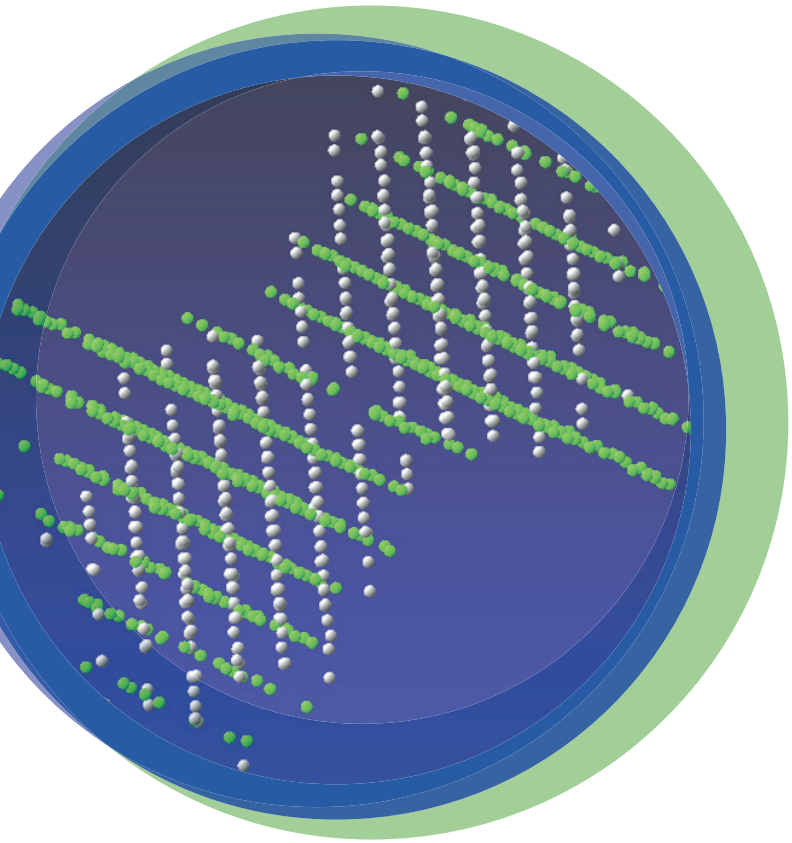


ELDICO *ED-1* A class of its own: the *ED-1* delivers atomic structures directly from nano-sized samples

Data processing and twin handling was performed using the APEX4 software package and implemented programs.[1] The reciprocal space visualization in RLATT was used to divide the reflections into two groups corresponding to the two twin domains. Indexing of these groups then yielded the same unit cell (within standard deviations), but with different orientations (rotation by ca. 47°). Both domains were then integrated and corrected for Lorentz effects, scan speed, background, and absorption using SAINT and SADABS. Space group determination based on systematic absences and E statistics as well as data merging was performed in XPREP. The structure was solved by SHELXT and refined with SHELXL in conjunction with SHELXLE against all data using the twin law derived from the relation of the two domains. All hydrogen atoms could be located in the difference Fourier map and were refined with a riding model based on neutron diffraction distances.

The refinement converged at a final R_1 value of 14.05% (wR_2 = 37.68%), which is a good result for normal electron diffraction data and even better for a twin. Whereas the single data sets from integration of the two domains yielded a completeness of 80.4% and 59.9%, respectively, the merged dataset reached 86.7% based on a single measurement that took less than one minute.

Reciprocal space view



Determined Twin Law

$$\begin{bmatrix} 0.940 & -0.094 & 0.088 \\ -0.167 & 0.734 & 0.218 \\ -1.132 & -2.042 & 0.675 \end{bmatrix}$$

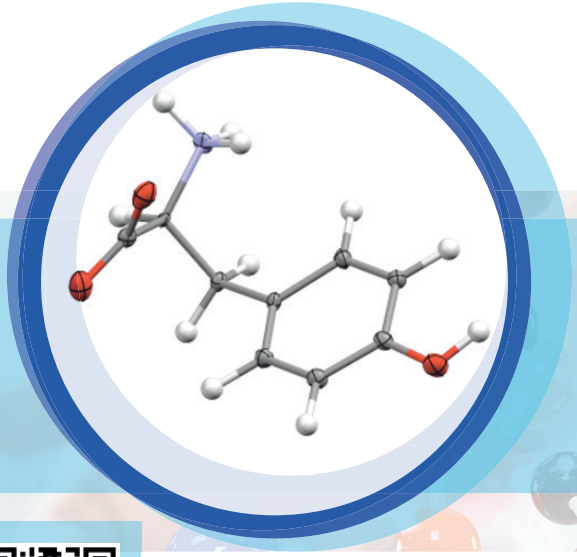
Crystal Information

chemical formula	$C_9H_{11}NO_3$
molecular weight	181.19
crystal system	orthorhombic
space group	$P2_12_12_1$
a, b, c [Å]	5.80(6), 6.92(6), 21.1(2)
α, β, γ [°]	90, 90, 90
V [Å^3]	846(14)
Z	4
F (0 0 0)	384

Refinement Details

independent reflections	1112
parameters	122
restraints	246
resolution [Å]	0.9
R_{int} [%]	26.26
R_1 [I > 2σ(I)] [%]	14.05
wR_2 [all data]	37.68
goodness of fit	1.068

Molecular structure



Crystal 1		80,4%
Crystal 2		59,9%
Merged dataset		86.7%

[1] *APEX suite of crystallographic software, APEX4 Version 2021.4-0, Bruker AXS Inc., Madison, Wisconsin, USA, 2021.*

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