Application Note

ELDICO SCIENTIFIC
THE ELECTRON DIFFRACTION COMPANY

Closing the R Value Gap:

R_1 VALUES <10% FROM ELECTRON DIFFRACTION DATA

R values are used as quality indicators for crystal structures and the most common one is the conventional R_1 value based on reflections with significant intensity (usually >2σ). So far there is a substantial gap between the R values that can be obtained with X-ray diffraction (XRD) and electron diffraction (ED) data.
A case study of 1,3,5-triphenylbenzene (TPB) measured on the ELDICO *ED-1* electron diffractometer at room temperature now shows that ED can achieve R_1 values lower than 10% with standard kinematic refinement.

There are several reasons why structures from ED tend to be of lower quality than XRD data, the main one of which is dynamic scattering. Further ones are beam damage, stronger absorption, lower resolution, and lower completeness which then requires merging of datasets from different crystals. Of course, instrumentation also plays a role as electron diffraction experiments are often performed on modified transmission electron microscopes (TEMs) which are not optimized for diffraction. To verify the benefits of a dedicated electron diffractometer, TPB was measured, which is a medium-sized organic compound with low beam-sensitivity and high symmetry, so some of the other factors can be ruled out and mainly the instrumentation determines the data quality.

Measurement Conditions

temperature	ambient
electron energy	160 keV
wavelength	0.02851 Å (160 keV)
data collection method	single scan continuous rotation
φ range	-70° to 65° (-47.5° to 37.5° used)
φ increment	0.5°
exposure time	1 s per frame
total measurement time	270 s

ELECTRON SOURCE AND OPTICS
Imaging (STEM) mode (focussed beam) with low dose, diffraction mode (parallel beam), fast switching of beam modes

PRECISE GONIOMETER
For accurate nano-rotation, 4 translation stages and 1 rotation axis

OCTAGON
For cryo option and other custom attachments

HYBRID PIXEL DETECTOR
Low noise, high dynamic range, stable towards primary beam

ELDICO *ED-1*

Designed by crystallographers for crystallographers

A 135° φ scan was performed from which the high and low angle regions were cut due to shading of the beam by other crystals and the TEM grid on which the sample was prepared. Data were processed and evaluated using the APEX4 software package and implemented programs.[1]

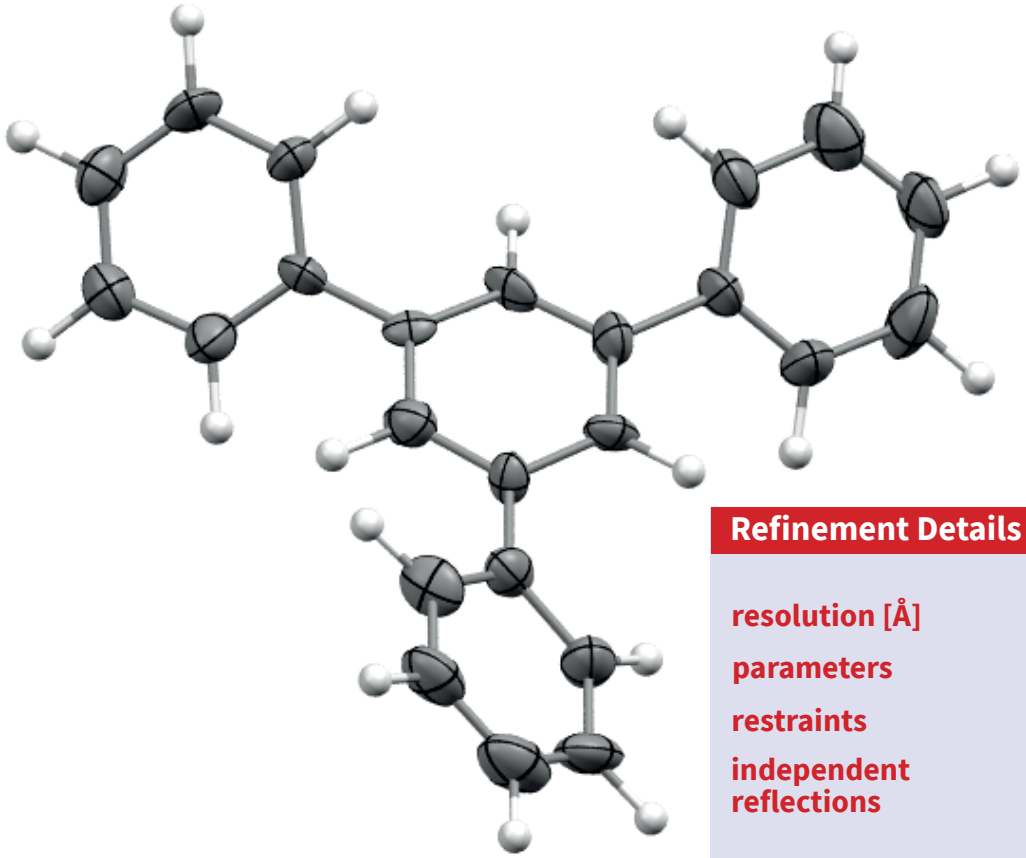
The frames were integrated and corrected for Lorentz effects, scan speed, background, and absorption using SAINT and SADABS. Space group determination based on systematic absences and E statistics was performed with XPREP. The structure was solved by SHELXD and refined with SHELXL in conjunction with SHELXLE. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were placed in calculated positions and refined with a riding model based on neutron diffraction distances and $U_{iso}(H) = 1.2*U_{iso}(C)$.

The refinement converged with a final R_1 value of 9.70% (wR_2 = 28.05%), thus staying below the important barrier of 10% which is often considered as a threshold for accepting XRD data. Notably this was achieved by standard kinematic refinement without taking dynamic scattering into account, illustrating that even without addressing this intrinsic challenge of ED physics, improved instrumentation can provide high data quality.

Crystal Information

chemical formula	$C_{24}H_{18}$
molecular weight	306.38
crystal system	orthorhombic
space group	$Pna2_1$
a, b, c [Å]	7.60(4), 19.68(11), 11.25(6)
α, β, γ [°]	90, 90, 90
V [Å3]	1681(16)
Z	4
F (0 0 0)	648

Molecular structure



Refinement Details

resolution [Å]	0.99
parameters	218
restraints	307
independent reflections	1069
R_{int} [%]	5.60
R_1 [I > 2σ(I)] [%]	9.70
wR_2 [all data]	28.05
goodness of fit	1.145

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