Structure Refinement

Starting point: measurement.hkl (Intensities and its deviations for each h k l)

measurement.ins *or* measurement.p4p *or* measurement.crs (cell parameter)

XPrep **Find a potential space group** (via systematic absences)

Output: SpaceGroup.hkl (may be transformed, thus different from measurement.hkl)

SpaceGroup.ins (includes tentative unit cell content, cell parameters, symmetry info)

Olex2 **Structure Solution** (Either with ShelXT or olex.solve: initial guess for the electron dens.)

Output SpaceGroup.res (result-file, will automatically serve as new instruction (.ins) file for each new refinement cycle)

Structure Refinement (least square optimization – minimize difference F_o and F_c)

→assign proper atom types

→allow thermal displacement ("anisotropy")

→calculate H-atom positions

→include weighing scheme (different factors of weak/strong reflexes in residuals)

→include ACTA command (to write a crystallographic information file "CIF")

Structure Validation (PLATON)

→check for missed symmetry ("higher spacegroup"):

CALC ADDSYM, CALC ADDSYM EXACT, CALC NEWSYM

→check for twinning LEPAGE TWIN, TwinRotMat

By now (hopefully):

$R_1 < 5\%$, $wR_2 < 15\%$ and $< 3 \cdot R_1$, GooF ≈ 1, Shift ≈ 0, Max Peak < 5% of Z_{max}, complete ≈ 100%

CheckCIF (online)

Structure Report

No A- or B- Alerts, check other Alerts carefully!

Diamond (and POV-Ray for high resolution graphics)

Structure Representation (show molecule, packing, ellipsoids, selected distances and angles, …)

ToposPro **Network Analysis** (topological analysis, calculate interconnection, H-bridging, etc.)

Mercury **Calculate Powder Diffraction** (and compare with measurement to obtain phase identity and purity)