Chapter 3.3.2

TWINROTMAT

Determine Twin Matrices from Fo/Fc Data

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This function is implemented as an option in PLATON, and carries out an *after-the-fact* analysis for missed or unaccounted for twinning. It is based on an analysis of reflections with large differences between observed and calculated structure factors (i.e. I(obs) >> I(calc)).

TwinRotMat addresses the same issue as the Windows program **ROTAX** by Simon Parsons and Bob Gould but is not identical, both in terms of underlying algorithms and approach. It might be useful to compare the results of both approaches. Ideally, twinning should be already discovered in the early stages of data collection and taken care of. This applies in particular for data collected on CCD detector systems, when not all reflection spots are covered by the assumed lattice. Exceptions are cases of (pseudo)merohedral twinning where the signs of twinning turn up in the data processing stages. Examples of these signs are problems with the structure determination, poor refinement and unexplained density peaks.

This routine addresses cases of (reticular)(pseudo) merohedral twinning. Cases where data are based on an artificially higher volume twin lattice are not covered. (i.e. monoclinic structure in orthorhombic supercell twin lattice). A tentative rotation twin axis and associated matrix is produced.

The analysis is based on the well known fact that unaccounted for twinning shows up in a significant number of reflections with I(obs) much greater than I(calc). In that case, it can be assumed that those (relatively weak) reflection are overlapped by strong reflections with approximately the same theta value. Each such an occurrence leads to a tentative rotation axis. Rotation axis that are observed most frequently are tested for there capability to explain the observed intensity differences. Both an approximate twinning factor (BASF) and approximate effect on the R-value are listed. Suitable matrices are listed in green.

The program needs an '.fcf' file (e.g. shelxl.fcf) [or an .ins + .hkl] for the analysis.
Cases of merohedral twinning are normally handled in a subsequent BASF/TWIN refinement.

Alternatively, an HKLF 5 type of file for the best solution is written to a file with extension '.hkp' to be used for subsequent SHELXL refinement.

Options are provided to display the twinning effect in reciprocal space.

Parameters:

```
Iobs-Icalc/sigma  Crit:       PAR(413)=10.0
Theta             Criterium:  PAR(414)=0.05
Indexfit criterium: PAR(415) = 0.1
```

The analysis can be invoked in various ways. (The -T option bypasses the alternative invocation from the PLATON menu).

- Given an '.fcf' run **platon -T compound.fcf**
- Given an .ins & .hkl run **platon -T compound.ins**. In this case, the missing calculated structure factors will be calculated prior to the analysis for twinning.

Example:s103b.fcf. Monoclinic, twinned about (1 0 0). Overlap in 0, 6 & 12 th l-layer.
PLATON Analysis Result:
2-Rotation about (1 0 0)
Rotation Matrix:

```
1   0   0.834
0  -1   0
0   0  -1
```

An HKLF 5 type SHELXL file is written. In order to proceed with the SHELXL refinement:

1. save the original **.hkl** file
2. copy file **.hkp** to **.hkl**
3. copy latest **.res** to **.ins**
4. add 'BASF 0.0' to the '.ins' instruction set.
5. change 'HKLF' line into 'HKLF 5' (no transformation matrix !