Structure Factor Validation

(fcf validation)

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What can fcf validation detect?

- Mismatch between the data block names in the CIF and .fcf file
- Mismatch between cell parameters in the CIF and .fcf file
- The .fcf file is not from the refinement that produced the CIF
- Incomplete updating of a CIF (e.g. weighting scheme)
- Overlooked twinning
- Atomic coordinates transformed, but not the U^{ij}
- Incorrect element assignment (supplements other tests)
- Element reassignment without re-refining
- Modifying atomic and displacement parameters in the CIF (cheating!)

When might fcf validation not work?

- Weighting scheme cannot be interpreted by PLATON
  – JANA (sometimes), CRYSTALS, RAELS
- Non-merohedral twins
  – SHELXL HKLF5 type input cannot be reconstructed from the .fcf file
- .fcf file is in a format not understood by PLATON

P L A T O N

A Multipurpose Crystallographic Tool

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GRAPHICS	GEOM-CALC	VOIDS FLIP	SYMMETRY	ABSORPTION	REPORT	MISC-TOOLS
PLUTONauto	Calc All	Calc Solv	ADDSYM	MULscanABS	Validation	SYSTEM-S
ORTEP/ADP	Calc Intra	Calc K.P.I	ADDSYM-EQL	ABSPslScan	ASYM-VIEW	FCF2HKL
NewmanPlot	Calc Inter	SQUEEZE	ADDSYM-EXT	ABSTompa	FCF-Valid	Expand2P1
Rlng-Plots	Calc Coord	CALC-FCF	ADDSYM-PLT	ABSGauss	DlfFourier	FCF-Gener
Plane-Plot	Calc Metal	Contour-SQ	ADDSYM-SHX	ABSXtal	ANALofVAR	HKL-Gener
Polyhedra	Calc Geom	SOLV F3D	NEWSYM	ABSSphere	ByvoetPair	HKL-Transf
ContourDlf	Calc Hbond	SOLV PLOT	NONSYM	SHXABS	ASYM-EXPCT	EXOR-RES
Contour-Fo	Calc TMA	CavltyPlot	LePage		ASYM-Valid	ANIS-RES
AutoMolFit	L.S.-PLANE		DelRed		SupplMater	Rename-RES
HKL2Powder	DlhedAngle		MOLSYM		EXPECT-HKL	Auto-Renum
SlmPowderP	AngleLlnes	FLIP MENU	SPGRfromEX		CSD-CELL	SPF -eld
RadDistFun	AngLsplLln	FLIP SHOW	ASYM		CSD-QUEST	SHELXL-res
Patterson	CremerPopl	FLIP PATT	ASYMaverFR		StructTidy	CIF -acc
	BondValenc	FLIPPER 25	LePageTwin		StrainAnal	PDB -pdb
PLUTONatlv	HFIX - RES	STRUCTURE?	TwlnRotMat	Xtal Habit	LocCIF-acc	CIF2SHELXL

Xtal Data (CIF) gg3227.clf- Set 1(1): I
Refl Data (SHELXL) gg3227.hkl [NO-DIRC] (1): I

PLATON MENU
OptionMenus
NoMove
Join-Expand
Organic
Round
Parentheses
Label-Alias
R/S-Determ
Norm-H-bond
NoSymm
NoDisorder
LstARU RCel
LstCellSymm
ListAtoms
ListBonds
LstFlagRadi
Exclude H
MinQPeakHgt
MinQPeakDis
Q-Peak-Incl
KeyInstruct
Prev Next
SAVE-InstrS
ENTRY-LIST
Reset End

WORKING

http://journals.iucr.org/services/cif/checking/checkfull.html

checkCIF/PLATON full publication check

Welcome to the checkCIF/PLATON service, which is operated by the IUCr. You may use this form to upload files for a **full publication check**. Please upload your CIF using the form below.

File name:

Choose File no file selected Send CIF for checking

This full publication check (required for the journals *Acta Crystallographica Section C* and *Acta Crystallographica Section E*) includes checks on:

- CIF syntax and construction
- Cell and geometry details
- Space-group symmetry
- Anisotropic displacement parameters
- Publication items

Structure-factor checking is currently being tested on articles submitted to *Acta Crystallographica Section C* and *Acta Crystallographica Section E*. These tests may be carried out with a local version of PLATON or the trial service here.

If you intend to submit to another section of *Acta Crystallographica*, *Journal of Applied Crystallography* or *Journal of Synchrotron Radiation*, you should make sure that at least basic structural checks are run on the final version of your CIF prior to submission.

If you wish to carry out a full structural check (but without publication checks) please click here.

http://journals.iucr.org/services/cif/checking/checkcifhkl.html

checkCIF check with FCF file

Please upload your CIF and combined FCF files using the form below.
The datablock names in the structure factor files and CIF must match.
If there is more than one structure, then the structure factor files should be concatenated into one file.

CIF file name:
Choose File no file selected
HKL file name:
Choose File no file selected

Send Files for checking

Useful links
How to use checkCIF/PLATON reports
Details of checkCIF/PLATON tests
Submit to IUCr Journals
CIF dictionary

“Normal” feedback in the absence of a .fcf file (or if .fcf file not recognised)

```
# PLATON/CHECK-( 30310) versus check.def version of 260210  for entry: I
# Data From: x.cif - Data Type: CIF              Bond Precision   C-C = 0.0040 A
#                                                                  Temp =  93 K
# UCL  3.7797(17)   10.362(3)   11.297(3)     62.92(2)     82.30(3)     87.60(4)
# WaveLength 0.71075    Volume Reported      390.3(2)  Calculated      390.3(2)
# SpaceGroup from Symmetry P -1          Hall: -P 1
#                 Reported P -1                -P 1
# MoietyFormula C12 H12 N2, 2(F3 H2 O2 V)
#      Reported C12 H12 N2 2+ , 2H2 F3 O2 V -
#    SumFormula C12 H16 F6 N2 O4 V2
#      Reported C12 H16 F6 N2 O4 V2
# Mr         =    468.15[Calc],    468.15[Rep]
# Dx,gcm-3   =     1.992[Calc],     1.992[Rep]
# Z          =        1[Calc],        1[Rep]
# Mu (mm-1)  =     1.288[Calc],     1.290[Rep]
# F000       =     234.0[Calc],     234.0[Rep]  or F000' =    234.76[Calc]
# Reported   T Limits: Tmin=0.864               Tmax=1.000  AbsCorr=MULTI-SCAN
# Calculated T Limits: Tmin=0.857 Tmin'=0.857   Tmax=0.975
# Reported   Hmax=  4, Kmax= 12, Lmax= 13, Nref=  1385        , Th(max)= 25.340
# Calculated Hmax=  4, Kmax= 12, Lmax= 13, Nref=  1435        , Ratio  =  0.965
# Reported   Rho(min) = -0.34, Rho(max) =  0.36 e/Ang**3 (From CIF)
# w=1/[sigma**2(Fo**2)+(0.0393P)**2+  0.0941P], P=(Fo**2+2*Fc**2)/3
# R= 0.0329(  1215), wR2= 0.0800(  1385), S = 1.081, Npar= 126
```

Enhanced feedback in the presence of a .fcf file

```
...
# Reported   Hmax=  4, Kmax= 12, Lmax= 13, Nref=  1385        , Th(max)= 25.340
# Obs in FCF Hmax=  4, Kmax= 12, Lmax= 13, Nref=  1385        , Th(max)= 25.341
# Calculated Hmax=  4, Kmax= 12, Lmax= 13, Nref=  1435        , Ratio  =  0.965
# Reported   Rho(min) = -0.34, Rho(max) =  0.36 e/Ang**3 (From CIF)
# Calculated Rho(min) = -0.35, Rho(max) =  0.30 e/Ang**3 (From CIF+FCF data)
# w=1/[sigma**2(Fo**2)+(0.0393P)**2+  0.0941P], P=(Fo**2+2*Fc**2)/3
# R= 0.0329(  1215), wR2= 0.0800(  1385), S = 1.081      (From CIF+FCF data)
# R= 0.0329(  1215), wR2= 0.0800(  1385), S = 1.081      (From FCF data only)
# R= 0.0329(  1215), wR2= 0.0800(  1385), S = 1.081, Npar= 126
```

Structure Factor Validation Output

```
# Reported   Hmax=  4, Kmax= 12, Lmax= 13, Nref=  1385        , Th(max)= 25.340
# Obs in FCF Hmax=  4, Kmax= 12, Lmax= 13, Nref=  1385        , Th(max)= 25.341
# Calculated Hmax=  4, Kmax= 12, Lmax= 13, Nref=  1435        , Ratio  =  0.965

# Reported   Rho(min) = -0.34, Rho(max) =  0.36 e/Ang**3 (From CIF)
# Calculated Rho(min) = -0.35, Rho(max) =  0.30 e/Ang**3 (From CIF+FCF data)

# w=1/[sigma**2(Fo**2)+(0.0393P)**2+  0.0941P], P=(Fo**2+2*Fc**2)/3

# R= 0.0329(  1215), wR2= 0.0800(  1385), S = 1.081      (From CIF+FCF data)
# R= 0.0329(  1215), wR2= 0.0800(  1385), S = 1.081      (From FCF data only)
# R= 0.0329(  1215), wR2= 0.0800(  1385), S = 1.081, Npar= 126
```

From CIF + FCF data = recalculated from Fobs and atomic coordinates + U's in CIF

From FCF data only = recalculated solely from Fobs, Fcalc and weights

From CIF = reported values in the CIF

Structure Factor Validation Output

Mismatch between the data block names in the CIF and .fcf file

```
900_ALERT_1_A No Matching Reflection File Found ..............      !
902_ALERT_1_A No (Interpretable) Reflections found in FCF ....      !
```

Mismatch between cell parameters in the CIF and .fcf file

```
901_ALERT_1_A Cell Parameters in CIF and FCF do not Match ....      !
902_ALERT_1_A No (Interpretable) Reflections found in FCF ....      !
```

In both cases, no extended summary of R-factors, delta-Rho, etc.

Weighting scheme in CIF not understood

```
808_ALERT_5_G No Parsable  SHELXL Style Weighting Scheme Found       !
929_ALERT_5_G No Weight Pars,Obs and Calc R1,wR2,S not checked       !
```

May cause mismatches in the extended summary of R-factors, residual electron density peaks, etc., but no other alerts in this regard.

Little to be done if not using SHELXL.

New CIF datanames to robustly document weights are needed;
the current dataname is a free text item.

Twinning detected

```
931_ALERT_5_G Check Twin Law ( 1 0  0)[       ] Estimated BASF    0.18

931_ALERT_5_G Check Twin Law (       )[ 3 0  1] Estimated BASF    0.17
```

Alert may be generated even if twinning has been handled.

Twinning may also cause mismatches in the extended summary of R-factors, e.g. where non-merohedral twinning is treated with the HKLF5 method.

CIF datanames to enable proper reporting and validation of twins urgently needed – currently in development.

The .fcf file is not from the refinement that produced the CIF

A water molecule was omitted from the refinement used to generate the .fcf file, but the finished model is in the CIF

```
# Reported   Rho(min) = -0.34, Rho(max) =  0.36 e/Ang**3 (From CIF)
# Calculated Rho(min) = -1.18, Rho(max) = 10.08 e/Ang**3 (From CIF+FCF data)
# w=1/[sigma**2(Fo**2)+(0.0393P)**2+  0.0941P], P=(Fo**2+2*Fc**2)/3

# R= 0.1442(  1215), wR2= 0.2787(  1385), S = 4.255      (From CIF+FCF data)
# R= 0.2189(  1215), wR2= 0.5046(  1385), S = 7.612      (From FCF data only)
# R= 0.0329(  1215), wR2= 0.0800(  1385), S = 1.081, Npar= 126

973_ALERT_2_A Large Calcd. Positive Residual Density on     V1      10.08 eA-3
971_ALERT_2_B Large Calcd. Non-Metal Positive Residual Density       3.14 eA-3

921_ALERT_1_A R1  * 100.0 in the CIF and FCF Differ by .......     -18.60
922_ALERT_1_A wR2 * 100.0 in the CIF and FCF Differ by .......     -42.46
923_ALERT_1_A S    values in the CIF and FCF Differ by .......      -6.53
925_ALERT_1_A The Reported and Calculated Rho(max) Differ by .       9.72 eA-3
926_ALERT_1_A Reported and Calculated   R1 * 100.0 Differ by .     -11.13
927_ALERT_1_A Reported and Calculated  wR2 * 100.0 Differ by .     -19.87
928_ALERT_1_A Reported and Calculated    S value   Differ by .      -3.17
```

Weights not updated in CIF after a new refinement

A common fault!!

```
# R= 0.0329(  1215), wR2= 0.0640(  1385), S = 1.849      (From CIF+FCF data)
# R= 0.0329(  1215), wR2= 0.0640(  1385), S = 1.848      (From FCF data only)
# R= 0.0329(  1215), wR2= 0.0800(  1385), S = 1.081, Npar= 126

923_ALERT_1_A S    values in the CIF and FCF Differ by .......      -0.77

922_ALERT_1_B wR2 * 100.0 in the CIF and FCF Differ by .......       1.60
927_ALERT_1_B Reported and Calculated  wR2 * 100.0 Differ by .       1.60
928_ALERT_1_B Reported and Calculated    S value   Differ by .      -0.77
```

Weights not updated in CIF after a new refinement

A common fault!!

```
_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'w = 1/[\s^2^(Fo^2^)+(0.0393P)^2^+0.0941P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     mixed
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_number_reflns          1385
_refine_ls_number_parameters      126
_refine_ls_number_restraints      2
_refine_ls_R_factor_all           0.0395
_refine_ls_R_factor_gt            0.0329
_refine_ls_wR_factor_ref          0.0800
_refine_ls_wR_factor_gt           0.0765
_refine_ls_goodness_of_fit_ref    1.081
```

Improper editing of a CIF

- Atomic coordinates transformed through a symmetry operation other than inversion, but not the U^{ij}

  – always re-refine and generate a new CIF, avoid piecemeal cut/paste or hand-editing of the CIF itself.

- Element reassignment without re-refining

- Modifying atomic and displacement parameters in the CIF (cheating!)

Such manipulations lead to mismatches of R-factors, goodness-of-fit and residual electron density.