Structure validation in the submission, review and publication process

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APD - IV,
Gaithersburg, 2013

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Crystallography Journals Online

Part 1

IUCr
CIF (Crystallographic Information File)
Validation
checkCIF
Tools

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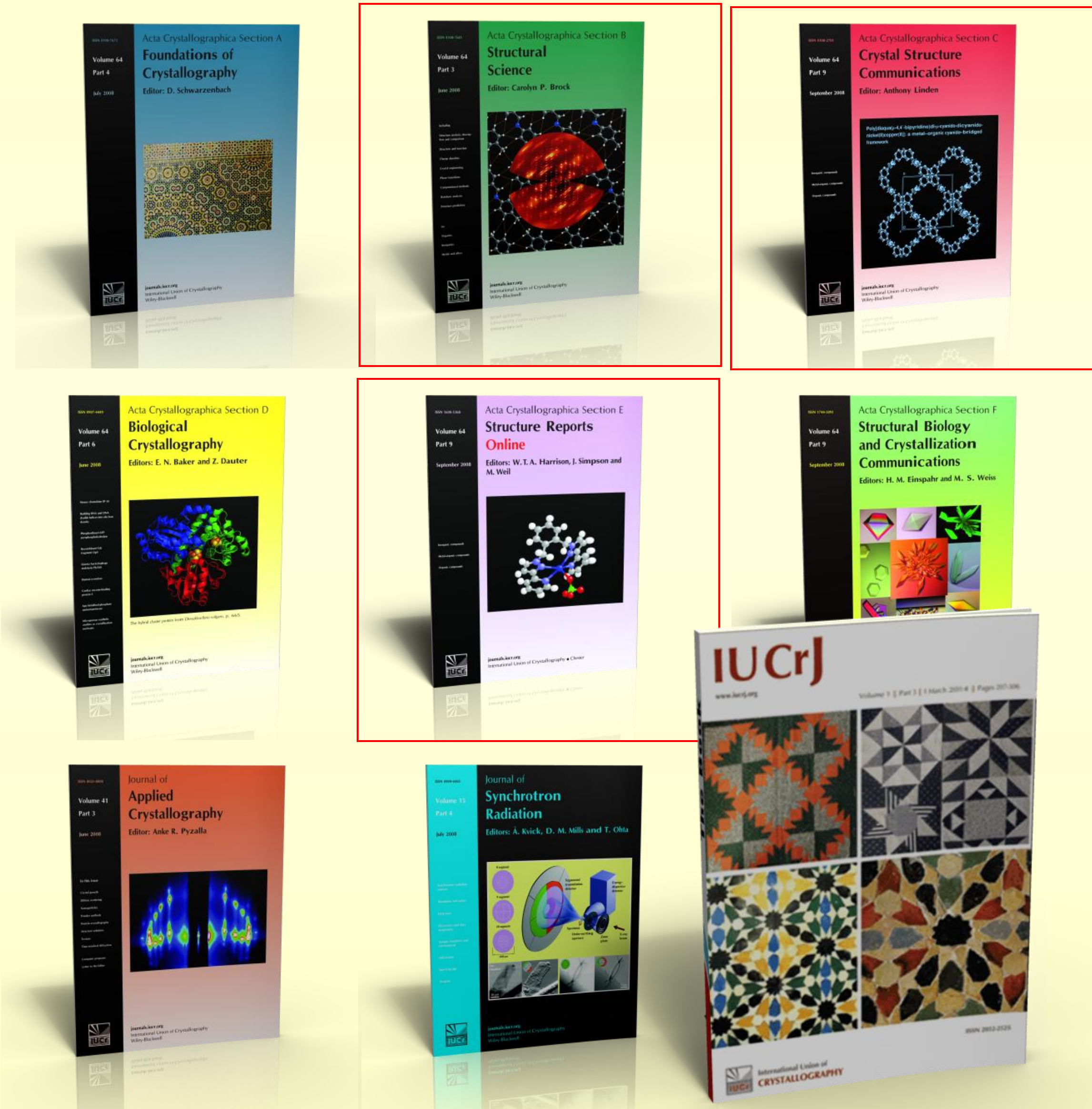
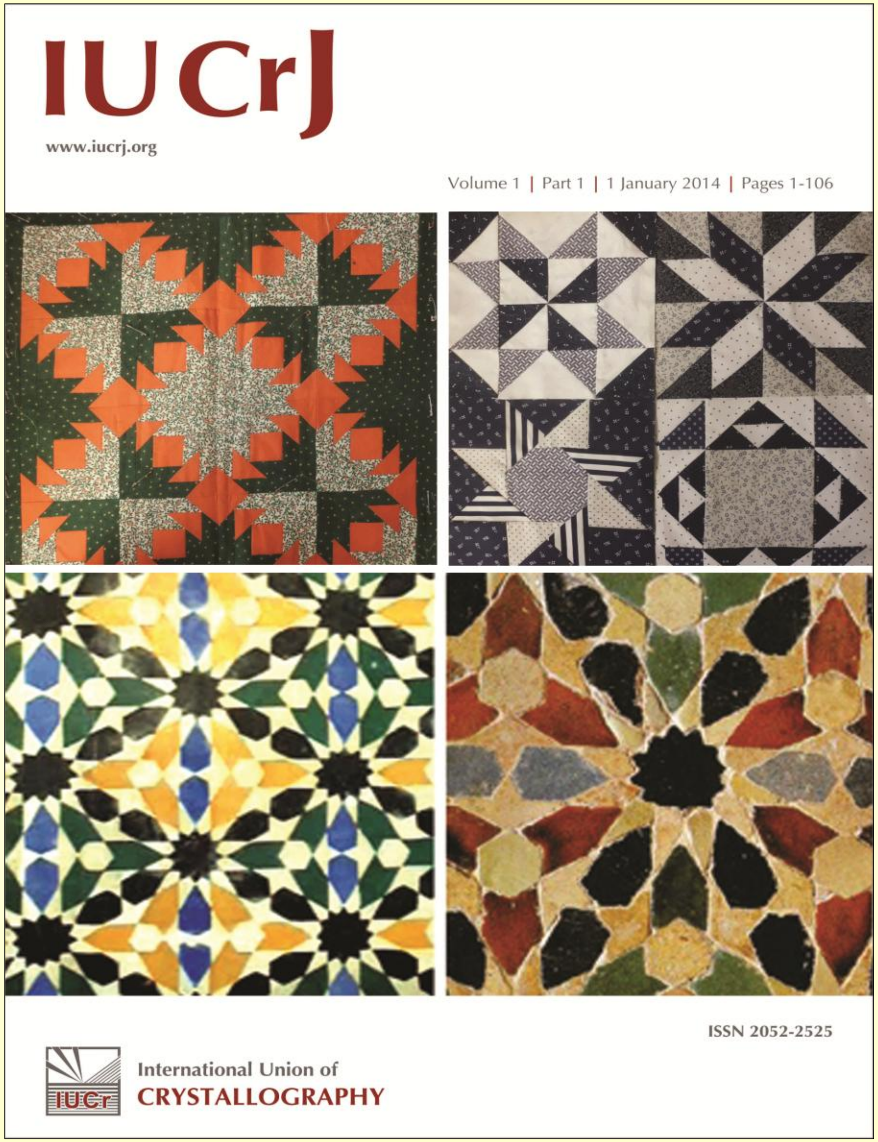
Online

International Union of Crystallography

- International Scientific Union
- Publishes 8 research journals:
    - New title in preparation: IUCrJ
- Major reference work *International Tables for Crystallography*
- *Volume H*: *Powder Diffraction*
- Promotes standard crystallographic data file format (CIF)





IUCrJ

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International Union of
CRYSTALLOGRAPHY

IUCrJ

The new open-access **high-influence** journal from the IUCr **www.iucrj.org** to celebrate the **International Year of Crystallography**

Inaugural issue to be published in 2014

2014
international year of
crystallography

IUCr

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CIF – A timeline (1)

- 1983,1988 – Standard Crystallographic File Structure
  I.D. Brown
- 1990 – Checking of data in *Acta Cryst.* C papers
- 1991 – Publication of first CIF dictionary
- 1991 – Techniques developed for processing CIF data
  First article typeset from a CIF file. Willis *et al.*
- 1992 – First unsolicited CIF submission
- 1994 – Faster processing for CIF submissions
- 1994 – First email versions of checkCIF/printCIF
- 1996 – *Acta Cryst.* C – CIF submission only

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Online

CIF – A timeline (2)

- 1997 – Release of powder CIF dictionary (ver. 1.0)
- 1998 – Automated submission by email – use of the VRF (validation response form)
- 1998/9 – web version of checkCIF/printCIF
- 1997-2000 – Electronic only section in *Acta Cryst.* C
- 2001 – Expanded checkCIF to include full PLATON validation test
- 2001 – First edition of *Acta Cryst.* E
- 2007 – Full online submission of CIF, structure factors and figures
- 2008 – *Acta Cryst.* E became an open-access journal

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CIF dictionaries

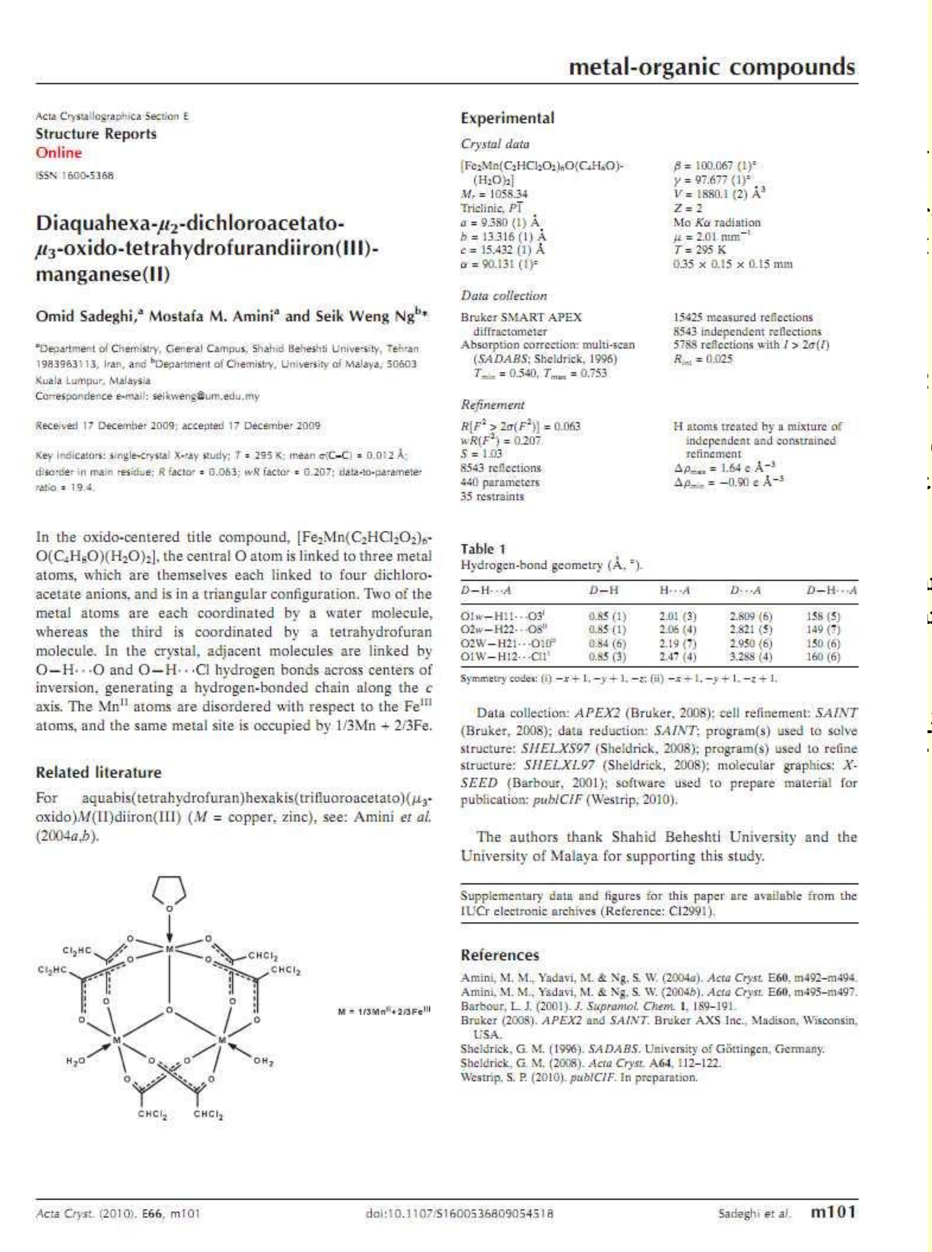
- core CIF 1991
- powder diffraction CIF (pdCIF) 1997
- modulated and composite structures (msCIF) 2002
- electron density (rhoCIF) 2003
- restraints 2011

Crystallography Journals Online

CIF as a vehicle for article submission

```
_publ_section_title
;\
Diaquahexa-\m~2~-dichlo
tetrahydrofurandiiron(I
;

loop_
  _publ_author_name
  _publ_author_address
   'Sadeghi, Omid'
; Department of Chemist
  General Campus
  Shahid Beheshti Unive
  Tehran 1983963113
  Iran
;
   'Ng, Seik Weng'
; Department of Chemist
  University of Malaya
  50603 Kuala Lumpur
  Malaysia
;

_publ_section_abstract
; In the oxido-centere
[Fe~2~Mn(C~2~HCl~2~O~2~
the central O atom is l
which are themselves ea
dichloroacetate anions
configuration. Two of t
coordinated by a water
is coordinated by a tet
the crystal, adjacent r
O---H...O and O---H...C
centers of inversion, g
chain along the <i>c</i
are disordered with res
```

```
                     SHELXL-97
.c

oacetato-\m~3~-oxido-\
I)manganese(II)

)6 O (C4 H8 O) (H2 O)2]'

 C16 H18 Cl12 Fe2 Mn O16'
:                    1058.34

                     triclinic
me_H-M               'P -1'
me_Hall              '-P 1'

xyz
z'

                     9.380(1)
                     13.316(1)
                     15.432(1)
                     90.131(1)
                     100.067(1)
                     97.677(1)
                     1880.1(2)
                     2
```



metal-organic compounds

Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Diaquahexa-μ_2-dichloroacetato-μ_3-oxido-tetrahydrofurandiiron(III)-manganese(II)

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Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.012 Å; disorder in main residue; R factor = 0.063; wR factor = 0.207; data-to-parameter ratio = 19.4.

In the oxido-centered title compound, $[Fe_2Mn(C_2HCl_2O_2)_6O(C_4H_8O)(H_2O)_2]$, the central O atom is linked to three metal atoms, which are themselves each linked to four dichloroacetate anions, and is in a triangular configuration. Two of the metal atoms are each coordinated by a water molecule, whereas the third is coordinated by a tetrahydrofuran molecule. In the crystal, adjacent molecules are linked by O—H···O and O—H···Cl hydrogen bonds across centers of inversion, generating a hydrogen-bonded chain along the *c* axis. The Mn^{II} atoms are disordered with respect to the Fe^{III} atoms, and the same metal site is occupied by 1/3Mn + 2/3Fe.

Related literature

For aquabis(tetrahydrofuran)hexakis(trifluoroacetato)(μ_3-oxido)*M*(II)diiron(III) (*M* = copper, zinc), see: Amini *et al.* (2004*a*,*b*).

M = 1/3Mn^{II}+2/3Fe^{III}

Experimental

Crystal data

$[Fe_2Mn(C_2HCl_2O_2)_6O(C_4H_8O)(H_2O)_2]$
M_r = 1058.34
Triclinic, $P\overline{1}$
a = 9.380 (1) Å
b = 13.316 (1) Å
c = 15.432 (1) Å
α = 90.131 (1)°
β = 100.067 (1)°
γ = 97.677 (1)°
V = 1880.1 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.01 mm^{-1}
T = 295 K
0.35 × 0.15 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.540, T_{max} = 0.753
15425 measured reflections
8543 independent reflections
5788 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.207
S = 1.03
8543 reflections
440 parameters
35 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.64 e Å^{-3}
$\Delta\rho_{min}$ = −0.90 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1w—H11···O3^{i}	0.85 (1)	2.01 (3)	2.809 (6)	158 (5)
O2w—H22···O8^{ii}	0.85 (1)	2.06 (4)	2.821 (5)	149 (7)
O2W—H21···O10^{ii}	0.84 (6)	2.19 (7)	2.950 (6)	150 (6)
O1W—H12···Cl1^{i}	0.85 (3)	2.47 (4)	3.288 (4)	160 (6)

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+1, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The authors thank Shahid Beheshti University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2991).

References

Amini, M. M., Yadavi, M. & Ng, S. W. (2004*a*). *Acta Cryst.* E**60**, m492–m494.
Amini, M. M., Yadavi, M. & Ng, S. W. (2004*b*). *Acta Cryst.* E**60**, m495–m497.
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *publCIF*. In preparation.

Acta Cryst. (2010). E**66**, m101 doi:10.1107/S1600536809054518 Sadeghi *et al.* **m101**

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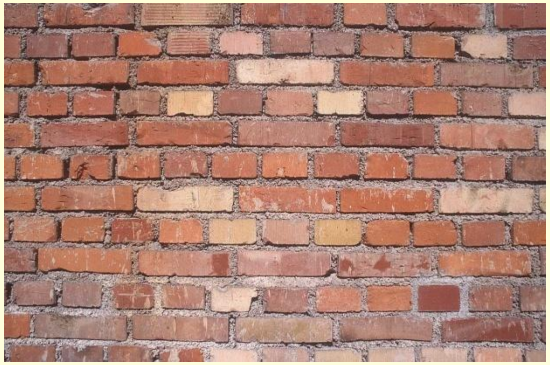
Validation with checkCIF



Brick Wall

OR

Useful set of tools

What is validation?

Comparison against a set of test criteria

- Are all the usually expected data and information present?
- Are related parameters consistent?
- Is the space group correct?
- Has the refinement converged?
- Are the assigned atom types correct?
- Is the structure reasonable?
- Has the structure been determined before?

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Is validation needed?

CIF made possible the automated checking of structure determinations.

- Incomplete CIFs
- Syntax errors in CIFs
- Too many avoidable oversights
- Doesn’t meet accepted standards
- More non-experts determining structures

- Still some avoidable mistakes

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Automation of validation

- Allows authors to get anonymous and instant feedback
- Detect and fix problems prior to submission
- Fewer and shorter revision cycles
- Consistent application of criteria
- Known application of criteria (**no hidden hurdles to jump**)
- Allows editors and referees to focus on the science

- **Benefit – faster publication times!**

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What does validation software do?

- Identifies possible problems via ALERTs
- Provides explanations of ALERTs
- Suggests interpretations and possible solutions
- A tool to help the author
  - efficiently check their work
  - avoid simple mistakes

- Not intended as a hurdle to make life difficult
- Not intended to hinder publication of correct results
- Also a useful tool for reviewers

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Alert indicators

```
380 ALERT 4 C Likely Unrefined X(sp2)-Methyl Moiety ...... C18
412 ALERT 2 C Short Intra XH3 .. XHn : H19B .. H30A = 1.81 Ang.
720 ALERT 4 C Number of Unusual/Non-Standard Label(s) .... 1
```

Alert numbers 1-5
indicate the type of issue

Alerts levels A, B, C and G indicate
the severity of the issue

Alert levels and types

ALERT level A = Most likely a serious problem – resolve or explain
ALERT level B = A potentially serious problem, consider carefully
ALERT level C = Check. Ensure it is not caused by an omission or oversight
ALERT level G = General information/check it is not something unexpected

ALERT type 1 CIF construction/syntax error, inconsistent or missing data
ALERT type 2 Indicator that the structure model may be wrong or deficient
ALERT type 3 Indicator that the structure quality may be low
ALERT type 4 Improvement, methodology, query or suggestion
ALERT type 5 Informative message, check

Alert level A

Alert A Most likely a serious problem – resolve or explain

Required item omitted, large deviation from usually expected value, or inconsistent values

Alert A	No crystal dimensions have been given
Alert A	Atom C58A ADP max/min Ratio 18.00
Alert A	H…A calc 5.82(3); rep 1.915; dev 3.91 Å
Alert A	Space group symbol does not match sym. ops.

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Alert level B

Alert B   A potentially serious problem, consider carefully

Item is a significant or unexpected outlier

Alert B	The formula has elements in wrong order
Alert B	ADDSYM detects Cc to Fdd2 transformation
Alert B	Refined extinction parameter < 1.9σ
Alert B	Structure contains VOIDS of 130.00 Å^3

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Validation Response Form

- Indicates that there are issues which have triggered a level A alert
- Provides a field for the author to respond to the alert

PLAT355_ALERT_3_A Long O-H Bond (0.82A) O22 - H220 ... 1.14 Ang.

_vrf_PLAT355_I
; PROBLEM: Long O-H Bond (0.82A) O22 - H22O ... 1.14 Ang.
RESPONSE: The H22O atom participates in a very strong nearly symmetric hydrogen bond which is discussed in details in the paper.
;

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Example of an unsuitable VRF

Alert A Given & expected crystal density differ
Alert A Given & expected absorption co-efficient differ
Calculated density = 3.377 density in CIF = 1.689
Calculated mu = 2.063 mu in CIF = 1.031

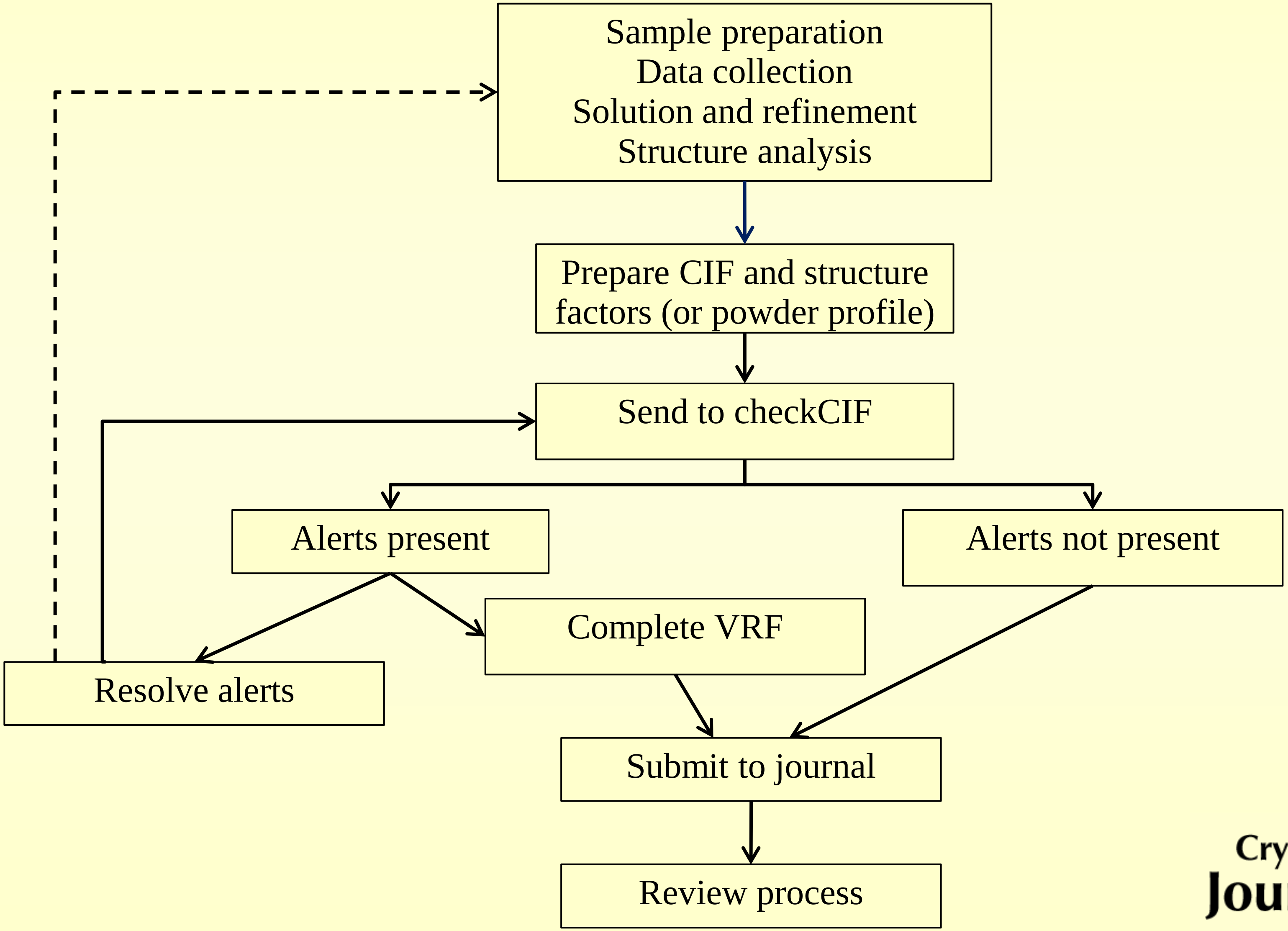
Author Response:

It appears that the absmu- and the density-problem are related. No explanation other than it is related to the disordered triflate groups and the refinement over several partially occupied sites

Actual cause of alert:

Molecule sits over inversion centre in $P2_1/n$
Z given as 4, instead of 2

checkCIF workflow



Sample preparation
Data collection
Solution and refinement
Structure analysis

Prepare CIF and structure factors (or powder profile)

Send to checkCIF

Alerts present

Alerts not present

Complete VRF

Resolve alerts

Submit to journal

Review process

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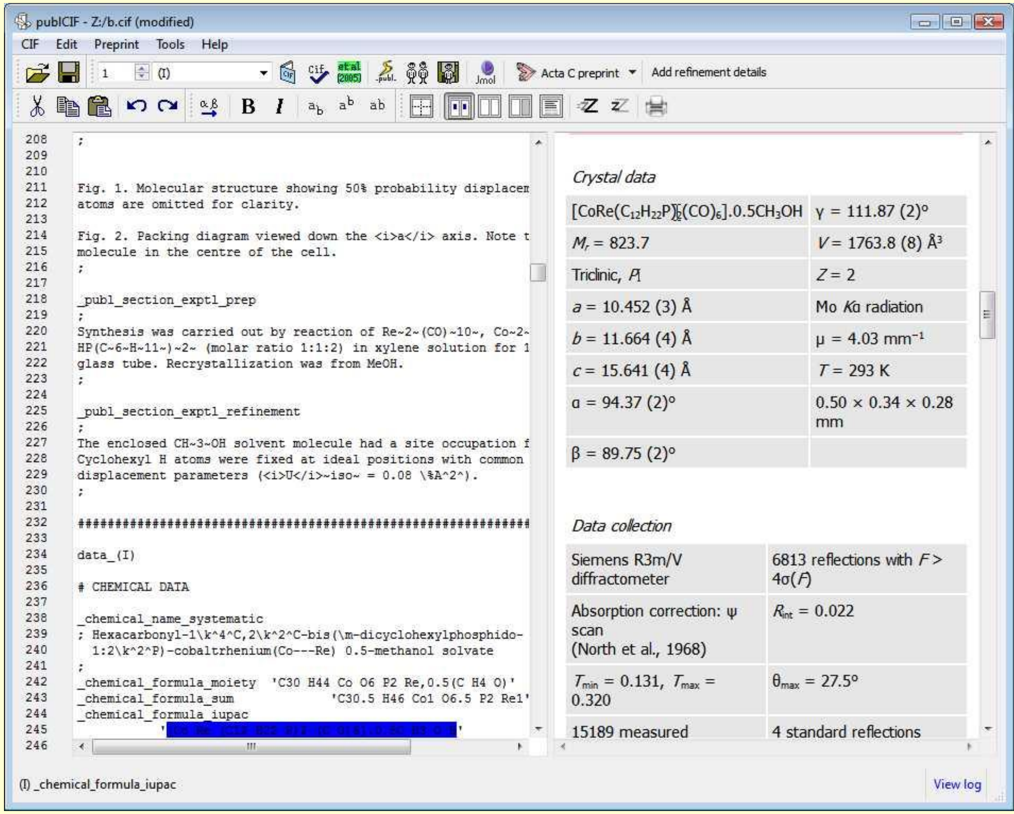
Limitations of validation

- Test is not yet implemented
- Test is not practical in these circumstances
- Error is not a validation issue
- Mistake cannot be detected from data in the CIF
- Nonsense entries in the CIF

Authoring tools

(1) *publCIF*

Desktop CIF publishing editor, validator and formatter for small-molecule, powder, modulated and incommensurate structure CIFs



```
;


Fig. 1. Molecular structure showing 50% probability displacem
atoms are omitted for clarity.

Fig. 2. Packing diagram viewed down the <i>a</i> axis. Note t
molecule in the centre of the cell.
;

_publ_section_exptl_prep
;
Synthesis was carried out by reaction of Re~2~(CO)~10~, Co~2~
HP(C~6~H~11~)~2~ (molar ratio 1:1:2) in xylene solution for 1
glass tube. Recrystallization was from MeOH.
;

_publ_section_exptl_refinement
;
The enclosed CH~3~OH solvent molecule had a site occupation f
Cyclohexyl H atoms were fixed at ideal positions with common
displacement parameters (<i>U</i>~iso~ = 0.08 \%A^2^).
;

#################################################################

data_(I)

# CHEMICAL DATA

_chemical_name_systematic
; Hexacarbonyl-1\k^4^C,2\k^2^C-bis(\m-dicyclohexylphosphido-
  1:2\k^2^P)-cobaltrhenium(Co---Re) 0.5-methanol solvate
;
_chemical_formula_moiety  'C30 H44 Co O6 P2 Re,0.5(C H4 O)'
_chemical_formula_sum              'C30.5 H46 Co1 O6.5 P2 Re1'
_chemical_formula_iupac
            '[illegible]'
```

Crystal data

$[CoRe(C_{12}H_{22}P)_2(CO)_6].0.5CH_3OH$	γ = 111.87 (2)°
M_r = 823.7	V = 1763.8 (8) Å^3
Triclinic, $P\bar{1}$	Z = 2
a = 10.452 (3) Å	Mo $K\alpha$ radiation
b = 11.664 (4) Å	μ = 4.03 mm^{-1}
c = 15.641 (4) Å	T = 293 K
α = 94.37 (2)°	0.50 × 0.34 × 0.28 mm
β = 89.75 (2)°	

Data collection

Siemens R3m/V diffractometer	6813 reflections with $F > 4\sigma(F)$
Absorption correction: ψ scan (North et al., 1968)	R_{int} = 0.022
T_{min} = 0.131, T_{max} = 0.320	θ_{max} = 27.5°
15189 measured	4 standard reflections

(I) _chemical_formula_iupac

http://journals.iucr.org/services/cif/publcif/

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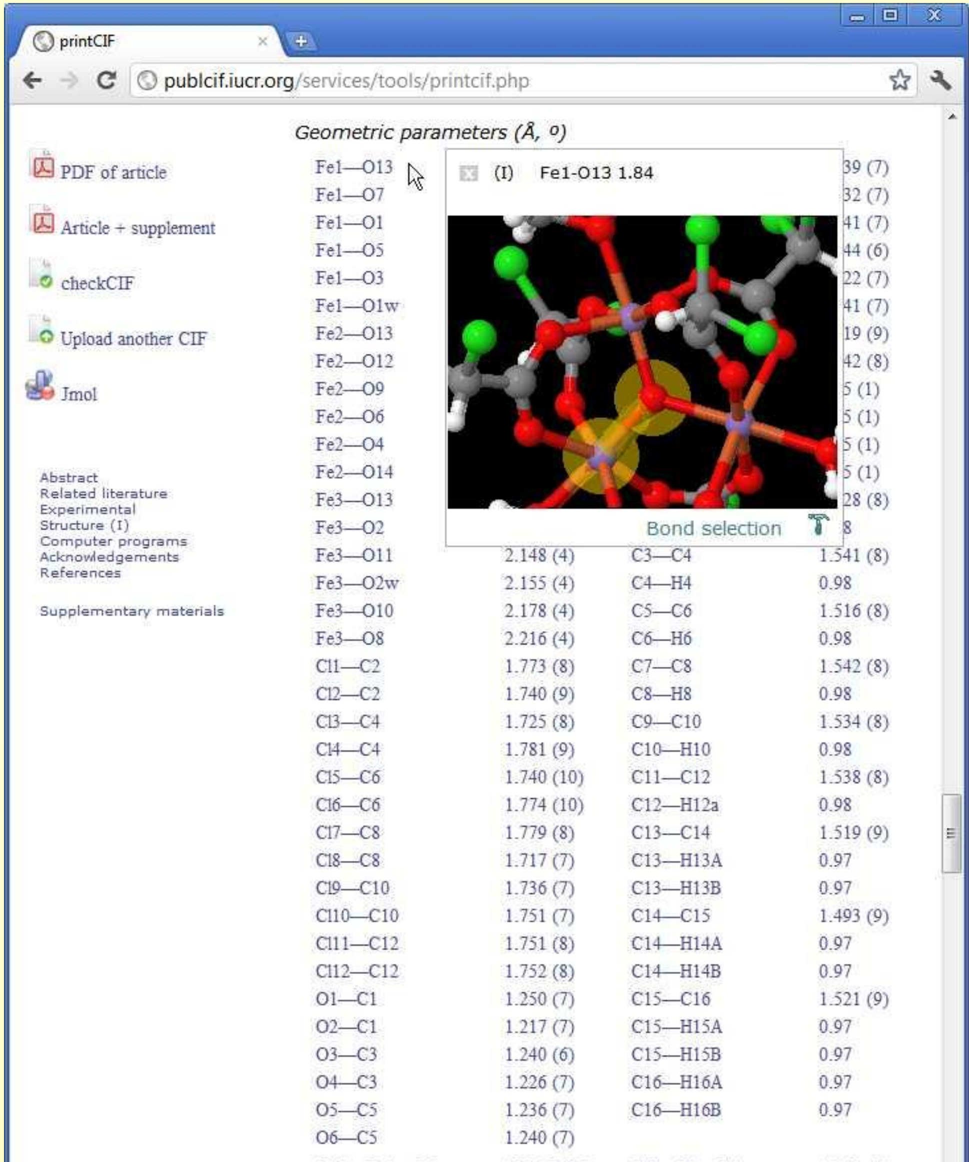
Authoring tools

(2) *printCIF*

Online CIF publishing validator and formatter for small-molecule, powder, modulated and incommensurate structure CIFs

http://publcif.iucr.org/services/tools/printcif.php



Geometric parameters (Å, º)

Fe1—O13			39 (7)
Fe1—O7			32 (7)
Fe1—O1			41 (7)
Fe1—O5			44 (6)
Fe1—O3			22 (7)
Fe1—O1w			41 (7)
Fe2—O13			19 (9)
Fe2—O12			42 (8)
Fe2—O9			5 (1)
Fe2—O6			5 (1)
Fe2—O4			5 (1)
Fe2—O14			5 (1)
Fe3—O13			28 (8)
Fe3—O2			8
Fe3—O11	2.148 (4)	C3—C4	1.541 (8)
Fe3—O2w	2.155 (4)	C4—H4	0.98
Fe3—O10	2.178 (4)	C5—C6	1.516 (8)
Fe3—O8	2.216 (4)	C6—H6	0.98
Cl1—C2	1.773 (8)	C7—C8	1.542 (8)
Cl2—C2	1.740 (9)	C8—H8	0.98
Cl3—C4	1.725 (8)	C9—C10	1.534 (8)
Cl4—C4	1.781 (9)	C10—H10	0.98
Cl5—C6	1.740 (10)	C11—C12	1.538 (8)
Cl6—C6	1.774 (10)	C12—H12a	0.98
Cl7—C8	1.779 (8)	C13—C14	1.519 (9)
Cl8—C8	1.717 (7)	C13—H13A	0.97
Cl9—C10	1.736 (7)	C13—H13B	0.97
Cl10—C10	1.751 (7)	C14—C15	1.493 (9)
Cl11—C12	1.751 (8)	C14—H14A	0.97
Cl12—C12	1.752 (8)	C14—H14B	0.97
O1—C1	1.250 (7)	C15—C16	1.521 (9)
O2—C1	1.217 (7)	C15—H15A	0.97
O3—C3	1.240 (6)	C15—H15B	0.97
O4—C3	1.226 (7)	C16—H16A	0.97
O5—C5	1.236 (7)	C16—H16B	0.97
O6—C5	1.240 (7)		

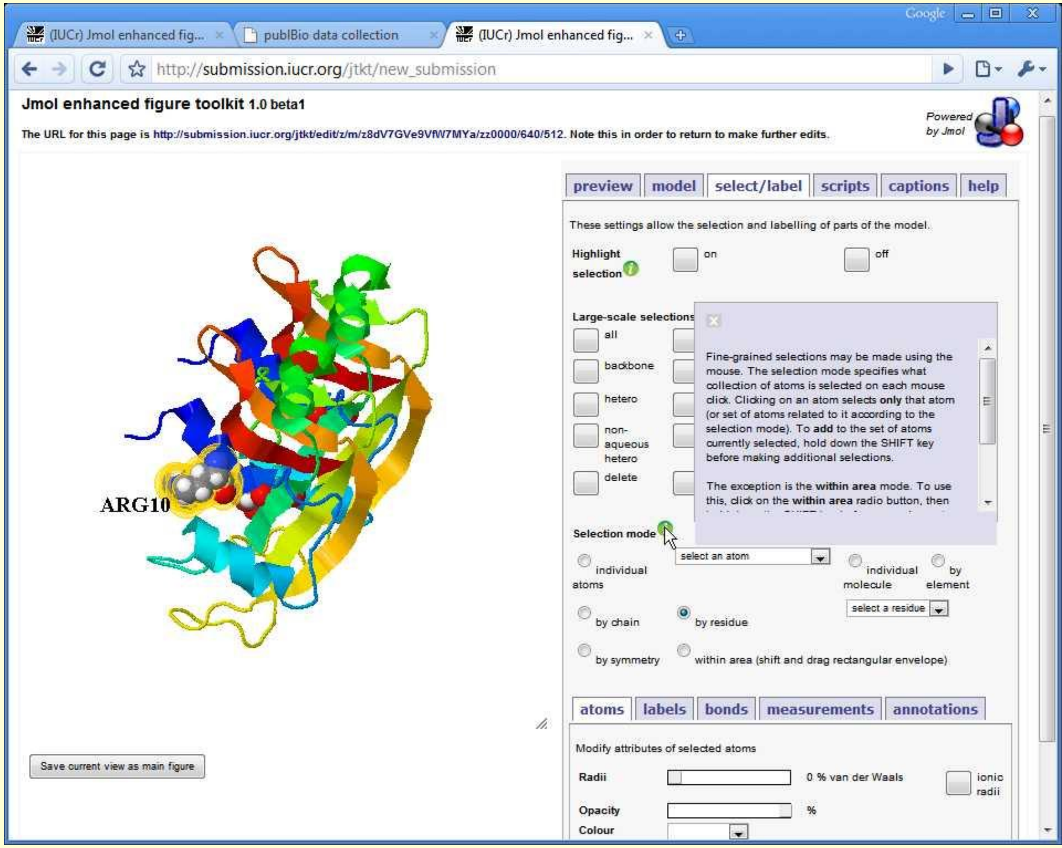
Authoring tools

(3) *Enhanced figure toolkit*

Create interactive three-dimensional visualisations with *Jmol*, a viewer that allows direct interaction with the underlying data

These figures form an *integral* part of the online published article

http://submission.iucr.org/jtkt



Jmol enhanced figure toolkit 1.0 beta1

The URL for this page is http://submission.iucr.org/jtkt/edit/z/m/z8dV7GVe9VfW7MYa/zz0000/640/512. Note this in order to return to make further edits.

These settings allow the selection and labelling of parts of the model.

Highlight selection ☐ on ☐ off

Large-scale selections

☐ all

☐ backbone

☐ hetero

☐ non-aqueous hetero

☐ delete

Fine-grained selections may be made using the mouse. The selection mode specifies what collection of atoms is selected on each mouse click. Clicking on an atom selects **only** that atom (or set of atoms related to it according to the selection mode). To **add** to the set of atoms currently selected, hold down the SHIFT key before making additional selections.

The exception is the **within area** mode. To use this, click on the **within area** radio button, then

Selection mode

○ individual atoms [select an atom] ○ individual molecule ○ by element

○ by chain ● by residue [select a residue]

○ by symmetry ○ within area (shift and drag rectangular envelope)

Modify attributes of selected atoms

Radii 0 % van der Waals ☐ ionic radii

Opacity %

Colour

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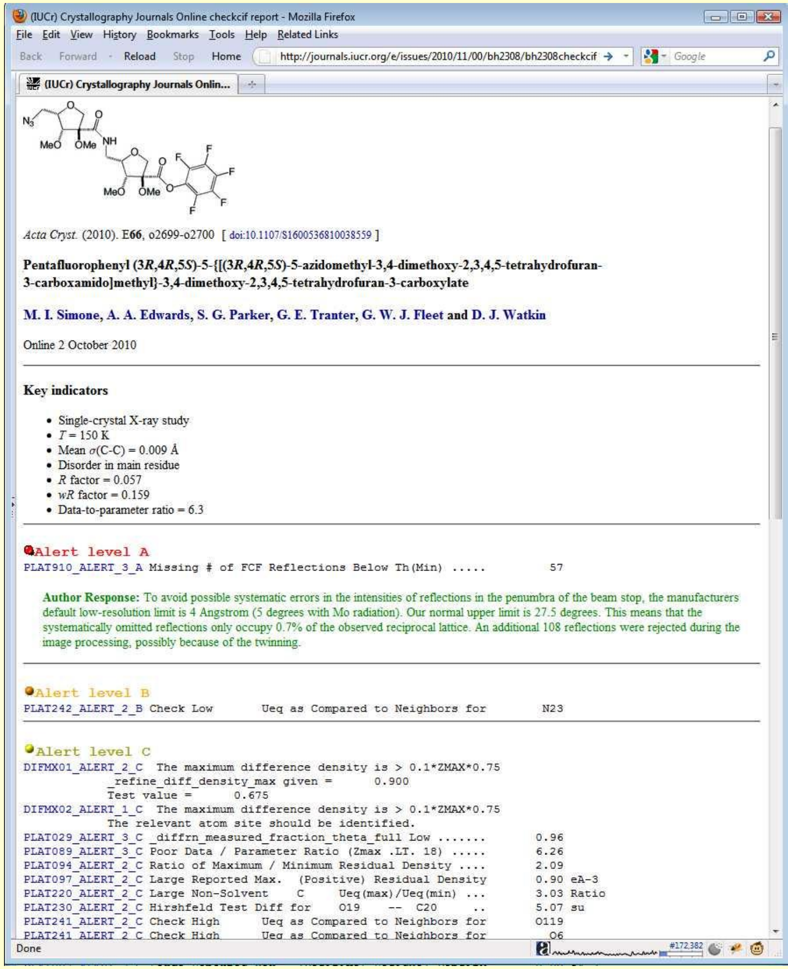
(4) *checkCIF*

- checkCIF reports are generated following the upload of a CIF to the checkCIF service
  - For single-crystal studies the structure factors are also validated

- Within the submission system the checkCIF report is made available to the reviewers

http://checkcif.iucr.org/



Acta Cryst. (2010). E**66**, o2699-o2700 [doi:10.1107/S1600536810038559]

Pentafluorophenyl (3*R*,4*R*,5*S*)-5-{[(3*R*,4*R*,5*S*)-5-azidomethyl-3,4-dimethoxy-2,3,4,5-tetrahydrofuran-3-carboxamido]methyl}-3,4-dimethoxy-2,3,4,5-tetrahydrofuran-3-carboxylate

M. I. Simone, A. A. Edwards, S. G. Parker, G. E. Tranter, G. W. J. Fleet and D. J. Watkin

Online 2 October 2010

Key indicators

- Single-crystal X-ray study
- T = 150 K
- Mean σ(C-C) = 0.009 Å
- Disorder in main residue
- R factor = 0.057
- wR factor = 0.159
- Data-to-parameter ratio = 6.3

```
Alert level A
PLAT910_ALERT_3_A Missing # of FCF Reflections Below Th(Min) .....         57
```

Author Response: To avoid possible systematic errors in the intensities of reflections in the penumbra of the beam stop, the manufacturers default low-resolution limit is 4 Angstrom (5 degrees with Mo radiation). Our normal upper limit is 27.5 degrees. This means that the systematically omitted reflections only occupy 0.7% of the observed reciprocal lattice. An additional 108 reflections were rejected during the image processing, possibly because of the twinning.

```
Alert level B
PLAT242_ALERT_2_B Check Low       Ueq as Compared to Neighbors for        N23
```

```
Alert level C
DIFMX01_ALERT_2_C  The maximum difference density is > 0.1*ZMAX*0.75
           _refine_diff_density_max given =       0.900
           Test value =       0.675
DIFMX02_ALERT_1_C  The maximum difference density is > 0.1*ZMAX*0.75
           The relevant atom site should be identified.
PLAT029_ALERT_3_C _diffrn_measured_fraction_theta_full Low .......       0.96
PLAT089_ALERT_3_C Poor Data / Parameter Ratio (Zmax .LT. 18) .....       6.26
PLAT094_ALERT_2_C Ratio of Maximum / Minimum Residual Density ....       2.09
PLAT097_ALERT_2_C Large Reported Max.  (Positive) Residual Density       0.90 eA-3
PLAT220_ALERT_2_C Large Non-Solvent    C     Ueq(max)/Ueq(min) ...       3.03 Ratio
PLAT230_ALERT_2_C Hirshfeld Test Diff for    O19    --  C20     ..       5.07 su
PLAT241_ALERT_2_C Check High       Ueq as Compared to Neighbors for      O119
PLAT241_ALERT_2_C Check High       Ueq as Compared to Neighbors for        O6
```

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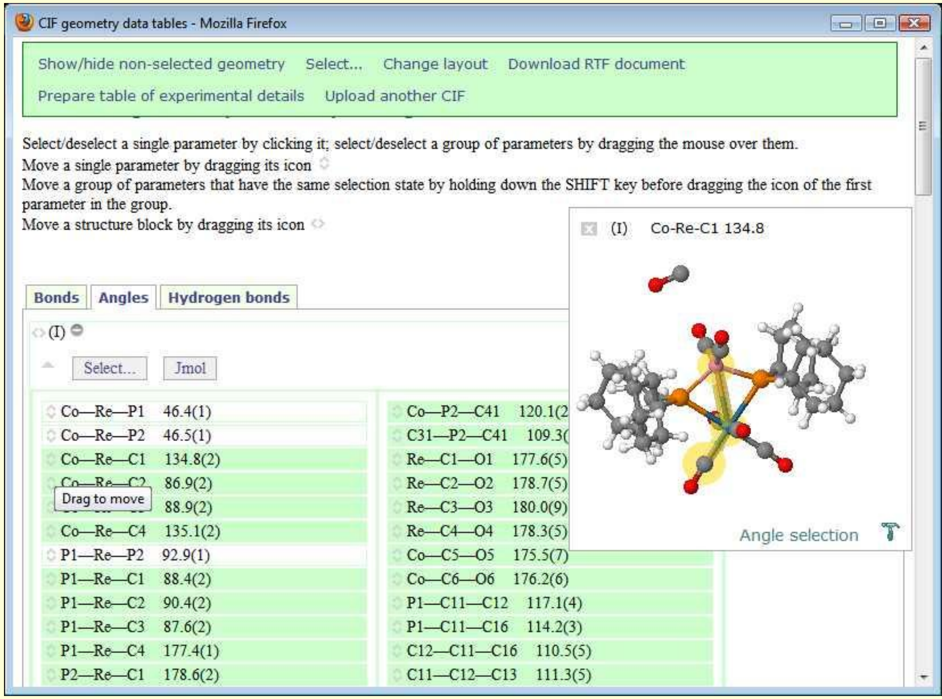
Authoring tools

(5) *Experimental tables*

Online service for formatting complex geometry and experimental tables for small-molecule, powder, modulated and incommensurate structure CIFs

Output available in rtf format

http://publcif.iucr.org/services/tools/



Select/deselect a single parameter by clicking it; select/deselect a group of parameters by dragging the mouse over them.
Move a single parameter by dragging its icon
Move a group of parameters that have the same selection state by holding down the SHIFT key before dragging the icon of the first parameter in the group.
Move a structure block by dragging its icon

Co—Re—P1	46.4(1)	Co—P2—C41	120.1(2
Co—Re—P2	46.5(1)	C31—P2—C41	109.3(
Co—Re—C1	134.8(2)	Re—C1—O1	177.6(5)
Co—Re—C2	86.9(2)	Re—C2—O2	178.7(5)
[illegible]	88.9(2)	Re—C3—O3	180.0(9)
Co—Re—C4	135.1(2)	Re—C4—O4	178.3(5)
P1—Re—P2	92.9(1)	Co—C5—O5	175.5(7)
P1—Re—C1	88.4(2)	Co—C6—O6	176.2(6)
P1—Re—C2	90.4(2)	P1—C11—C12	117.1(4)
P1—Re—C3	87.6(2)	P1—C11—C16	114.2(3)
P1—Re—C4	177.4(1)	C12—C11—C16	110.5(5)
P2—Re—C1	178.6(2)	C11—C12—C13	111.3(5)

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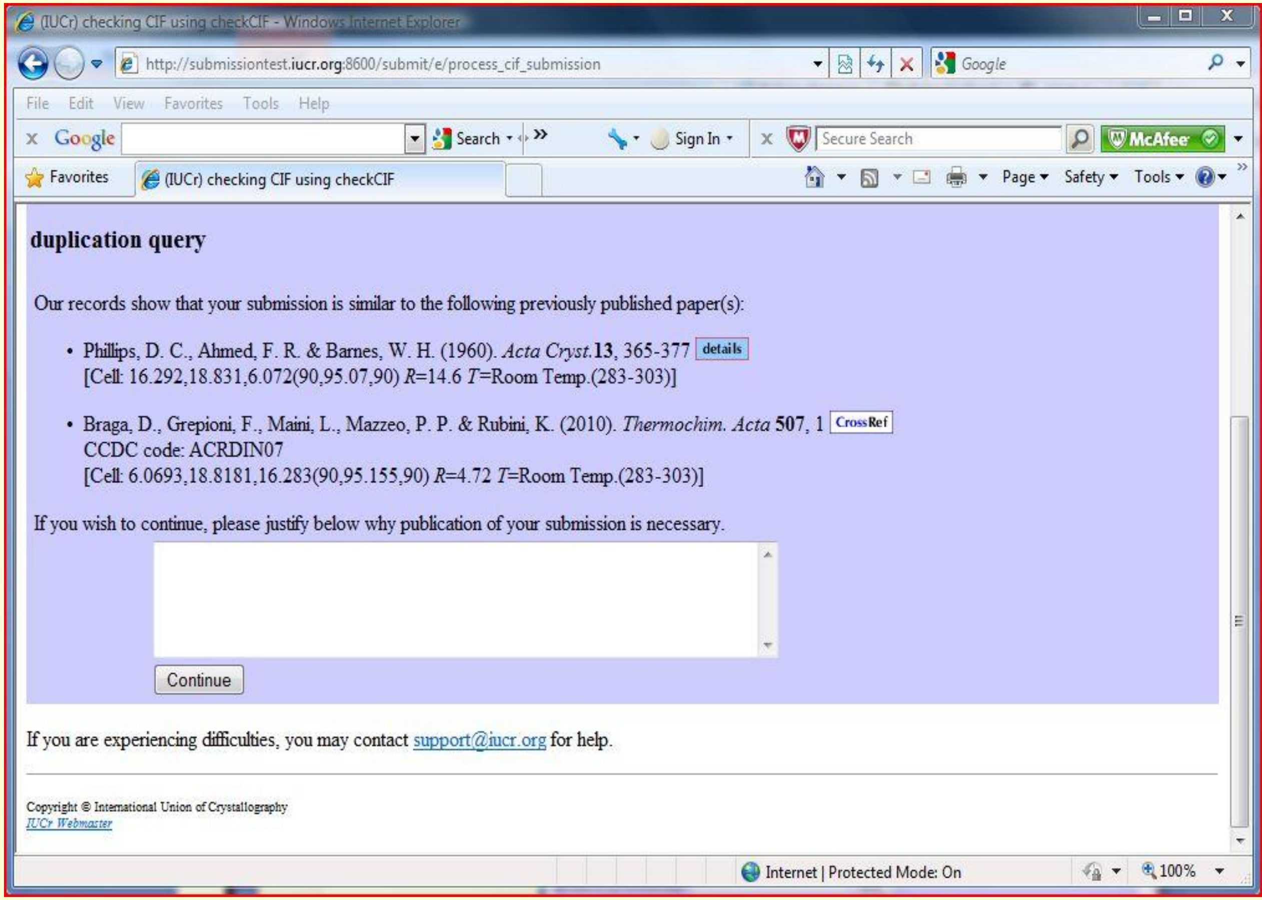
The system checks the article against an internal database to determine if the structure has been submitted to or published by the IUCr previously.

In addition an automated check is performed against the CCDC database.

If duplicates occur, then the author is able to note why their article should be considered for publication.



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Our records show that your submission is similar to the following previously published paper(s):

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- Braga, D., Grepioni, F., Maini, L., Mazzeo, P. P. & Rubini, K. (2010). *Thermochim. Acta* **507**, 1 CrossRef
  CCDC code: ACRDIN07
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Continue

If you are experiencing difficulties, you may contact support@iucr.org for help.

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Why publish data?

Some reasons:

- To enhance the reproducibility of a scientific experiment
- To verify or support the validity of deductions from an experiment
- To safeguard against error
- To safeguard against fraud
- To allow other scholars to conduct further research based on experiments already conducted
- To allow reanalysis at a later date, especially to extract 'new' science as new techniques are developed
- To provide example materials for teaching and learning
- To provide long-term preservation of experimental results and future access to them
- To permit systematic collection for comparative studies

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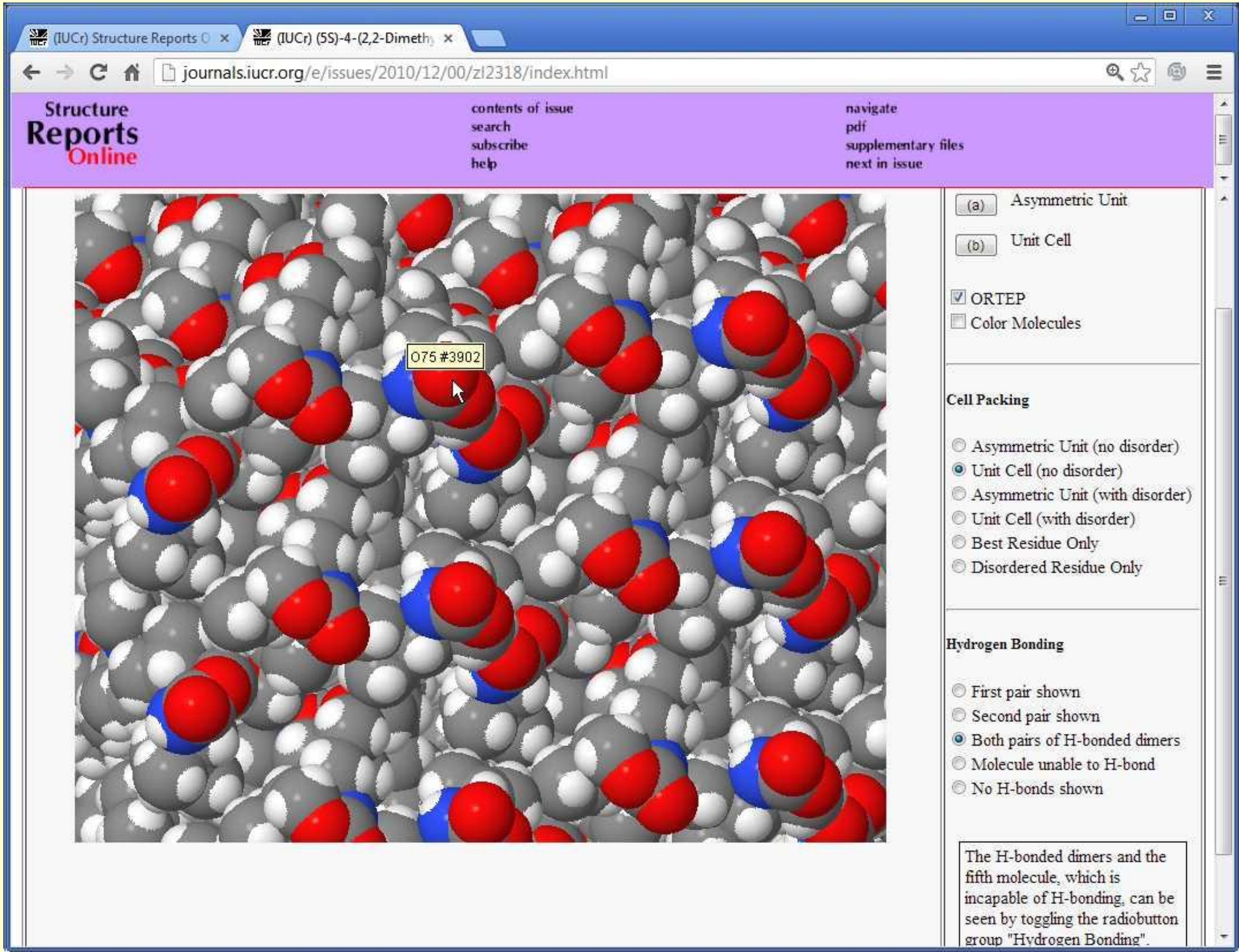
Reading the data

Interactive figures as an *integral* part of the article

Author views

Reader freedom to explore



(IUCr) Structure Reports O ×

(IUCr) (5S)-4-(2,2-Dimethy ×

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(a) Asymmetric Unit

(b) Unit Cell

☑ ORTEP
☐ Color Molecules

Cell Packing

○ Asymmetric Unit (no disorder)
● Unit Cell (no disorder)
○ Asymmetric Unit (with disorder)
○ Unit Cell (with disorder)
○ Best Residue Only
○ Disordered Residue Only

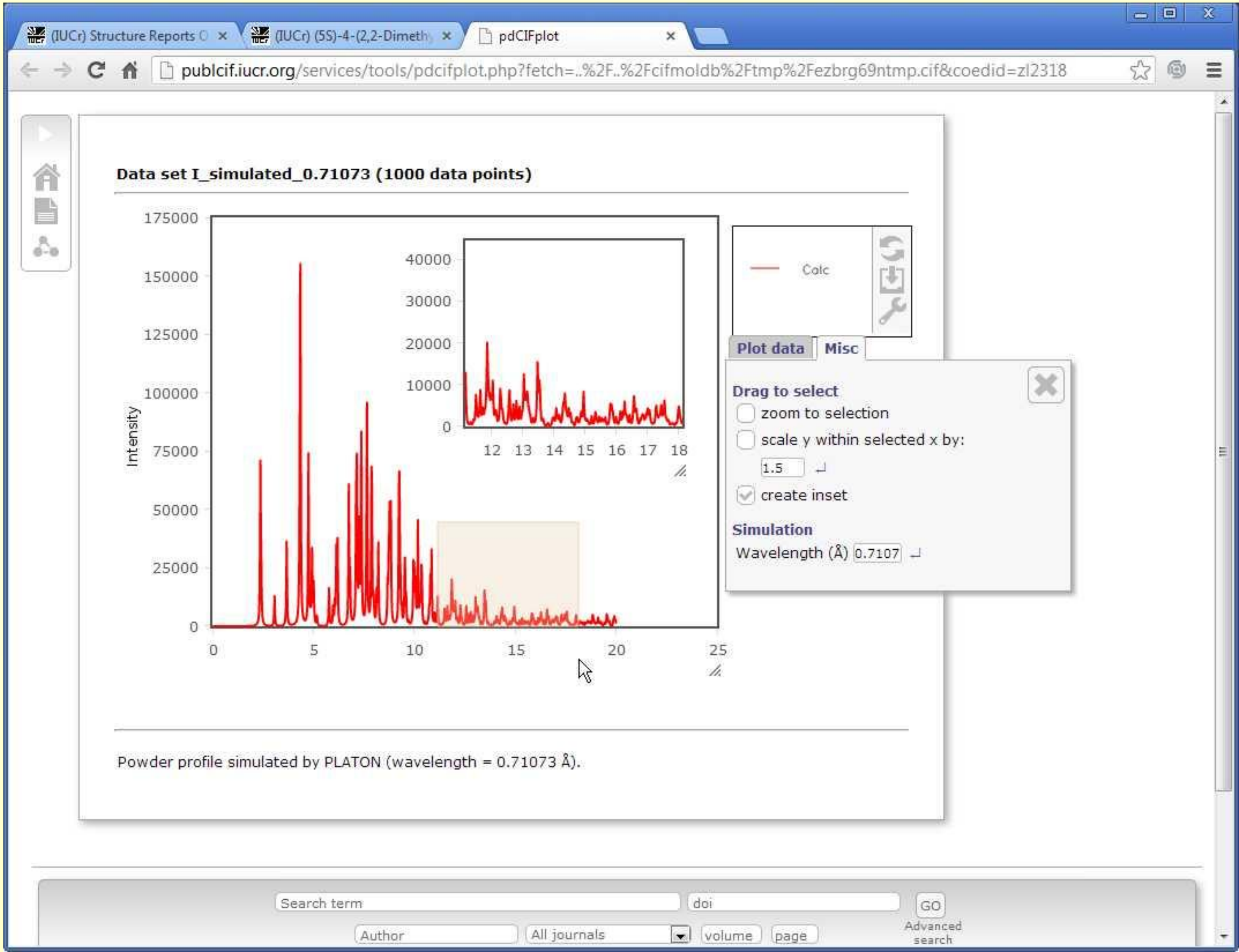
Hydrogen Bonding

○ First pair shown
○ Second pair shown
● Both pairs of H-bonded dimers
○ Molecule unable to H-bond
○ No H-bonds shown

The H-bonded dimers and the fifth molecule, which is incapable of H-bonding, can be seen by toggling the radiobutton group "Hydrogen Bonding".

Working with the data

For *any* published structure, the reader can *generate* a predicted powder diffraction pattern



publcif.iucr.org/services/tools/pdcifplot.php?fetch=..%2F..%2Fcifmoldb%2Ftmp%2Fezbrg69ntmp.cif&coedid=zl2318

Data set I_simulated_0.71073 (1000 data points)

Drag to select

☐ zoom to selection

☐ scale y within selected x by: 1.5

☑ create inset

Simulation

Wavelength (Å) 0.7107

Powder profile simulated by PLATON (wavelength = 0.71073 Å).

Part 2

CIF for powder studies
Current perspective
The future

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Advantages of CIF for powder studies

- Data in CIF ideally written directly by refinement software – fewer errors
- Forms a complete record of the experiment
- Data validation possible
- Can use to typeset experimental details, coordinates, bond lengths, angles etc. for journal article or thesis – fewer errors
- Easier and quicker for authors

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Successful?

- Single-crystal studies – yes (aided by a small number of refinement programs)
- Single phase
- Single data set
- Straightforward experiment
- Can validate with checkCIF – useful results

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Powder studies

- Several refinement programs
- Different output (different residuals etc.)
- One phase/multiple phases
- One data set/multiple data sets (and weighting)
- Multiple wavelengths (Cu Kα, synchrotron, neutron, time of flight…)
- Diverse experiments (but all can be described using the powder CIF dictionary)

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- Refinement program might not write all necessary details to CIF
- CIF may have to be created by hand – errors!
- checkCIF struggles with multiple phase studies, time of flight experiments, ‘non-standard’ wavelengths…
- Powder diffractionists go elsewhere…

Short term – improve checkCIF

- Suppress irrelevant tests
- Add powder-specific tests
- Develop checkCIF so can handle multiple data blocks/`non-standard’ wavelengths
- Expertise requested for validation of powder data based structures and incommensurate structures
- Feedback and suggestions welcome

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Long-term measures

- Could existing refinement programs write more details to the CIF?
- Could we ask for other file formats as a record of the experiment?
- Develop a tool to read the most common file formats (which are these?) and convert them to powder CIF

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Data deposition

- Raw data? (Too unwieldy?)
- Profile (not necessarily in CIF format)
- Structure factors, if available?
- IUCr Data Deposition Working Group – suggestions welcome

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International Tables for Crystallography

- Volume H on powder diffraction
- Editors: Chris Gilmore, Jim Kaduk and Henk Schenk
- Due 2014 (International Year of Crystallography)

INTERNATIONAL TABLES for CRYSTALLOGRAPHY

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Thank you

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