Publication of small-unit-cell structures in *Acta Crystallographica*

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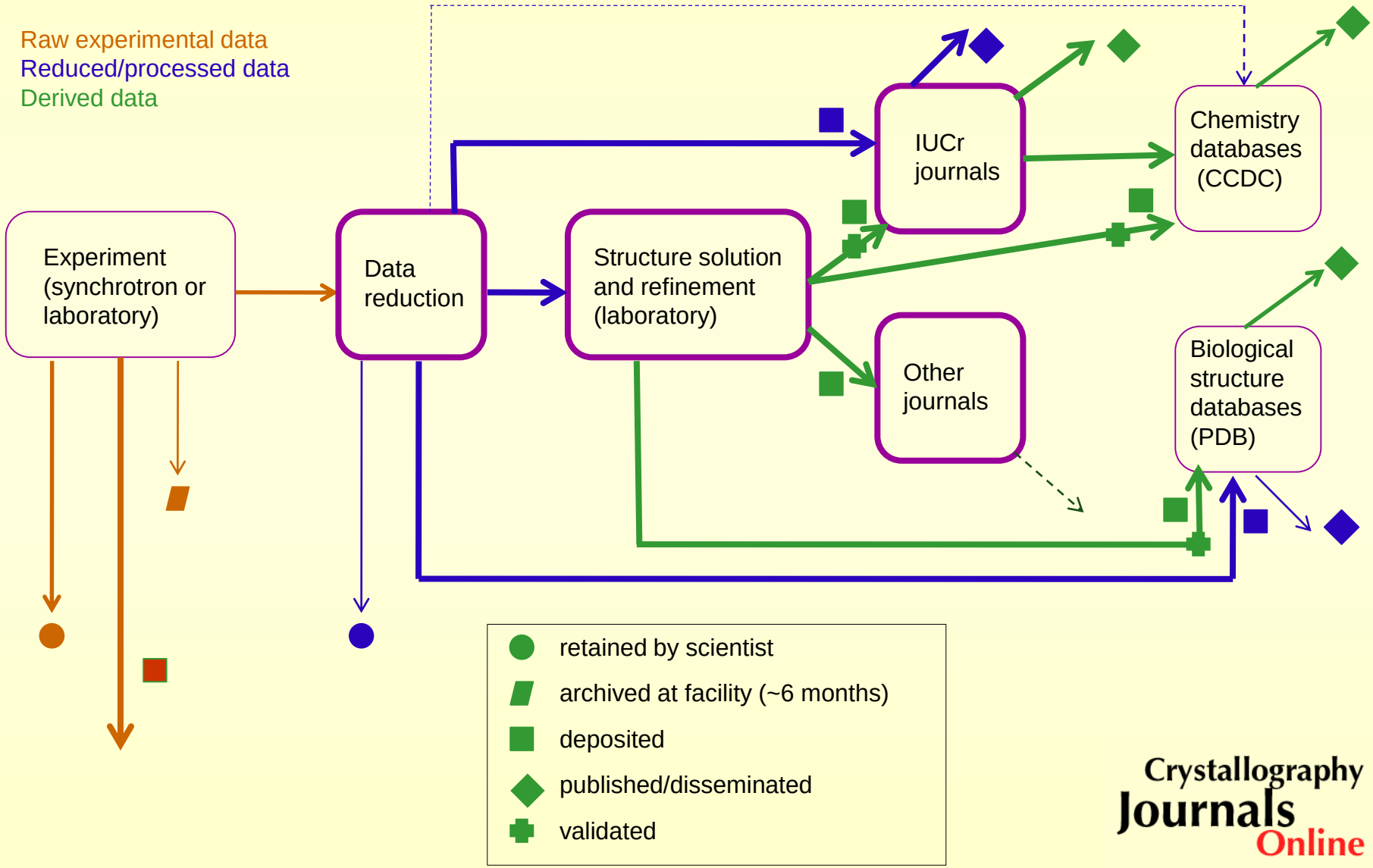
International Union of Crystallography

- International Scientific Union
- Publishes 9 research journals
- Promotes standard crystallographic data file format (CIF)

Data flow in crystallography



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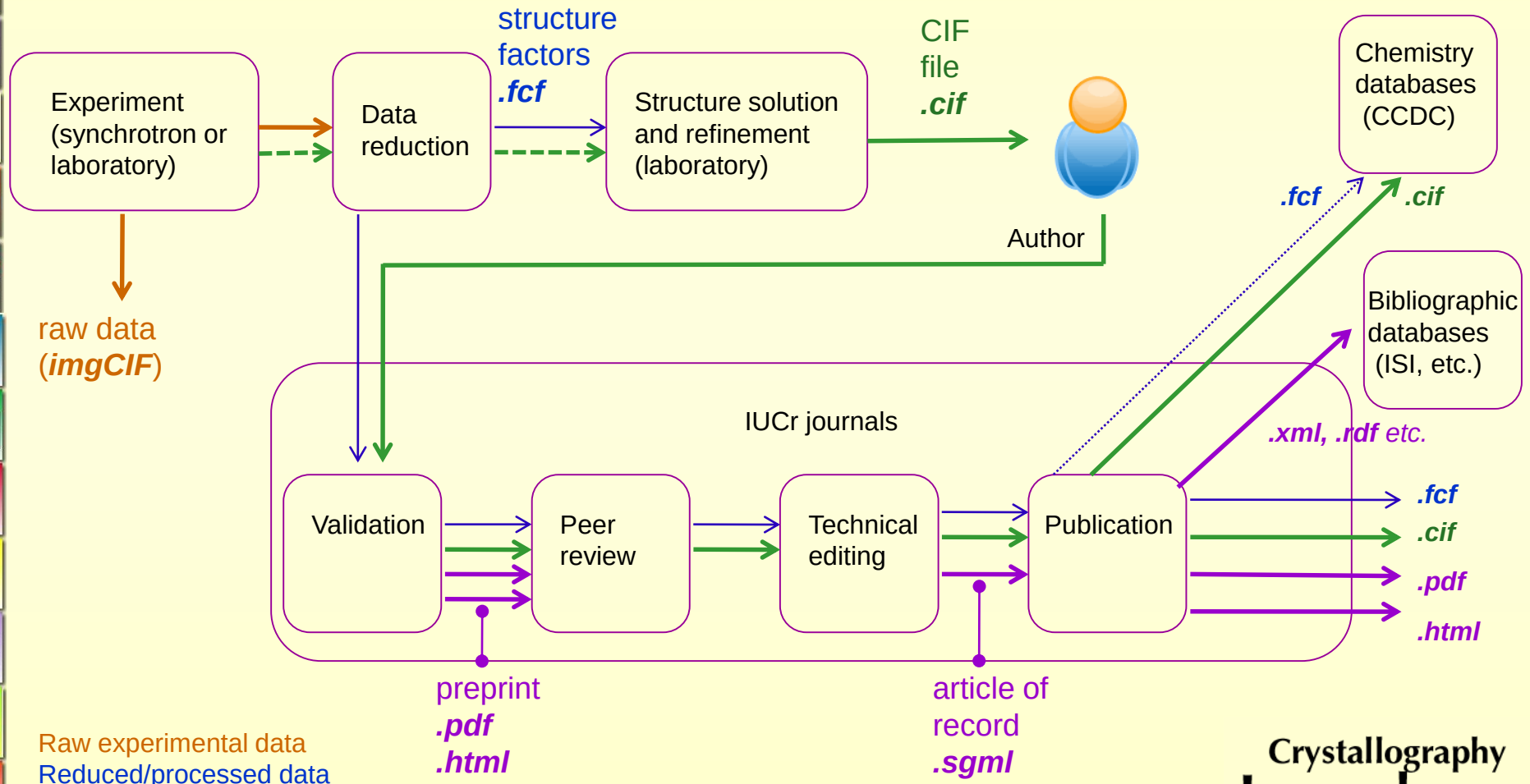
Publication flow in IUCr journals



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CIF – Early Development

- 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
- 1996 – *Acta Cryst.* C – CIF submission only

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The benefits of CIF for publishers

- Submission and deposition of structural and experimental data in a standard format
- Contains the framework for publishing an article directly
- Allows the possibility of automated validation checking against a set of known “standards”
- Allows duplication checking against relevant structural databases (e.g. cellCheckCSD)
- Helps in fraud detection ……

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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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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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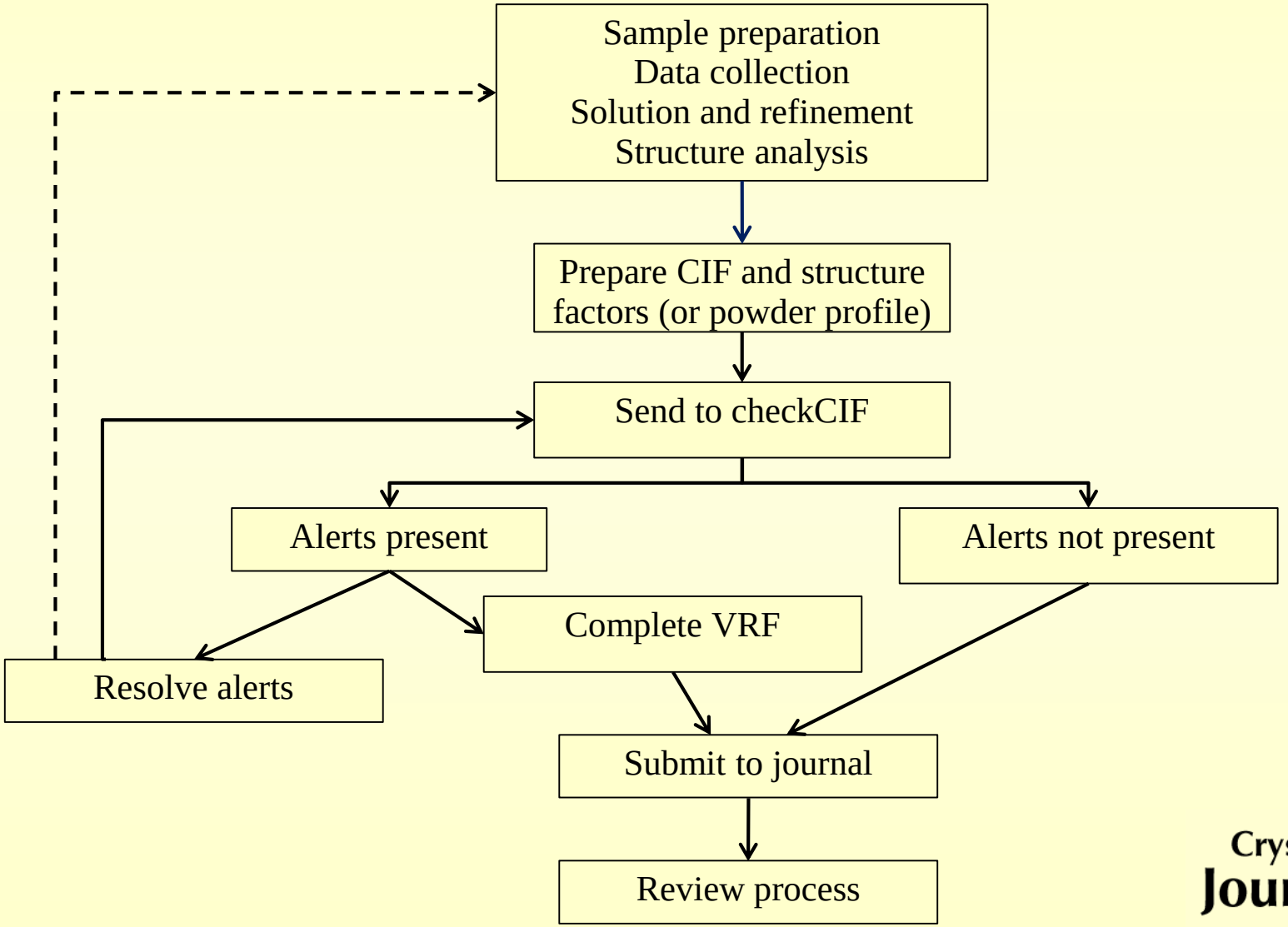
Validation workflow



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CIF as a vehicle for article submission

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  _publ_author_name
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  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-centered
[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 m
O---H...O and O---H...C
centers of inversion, g
chain along the <i>c</i>
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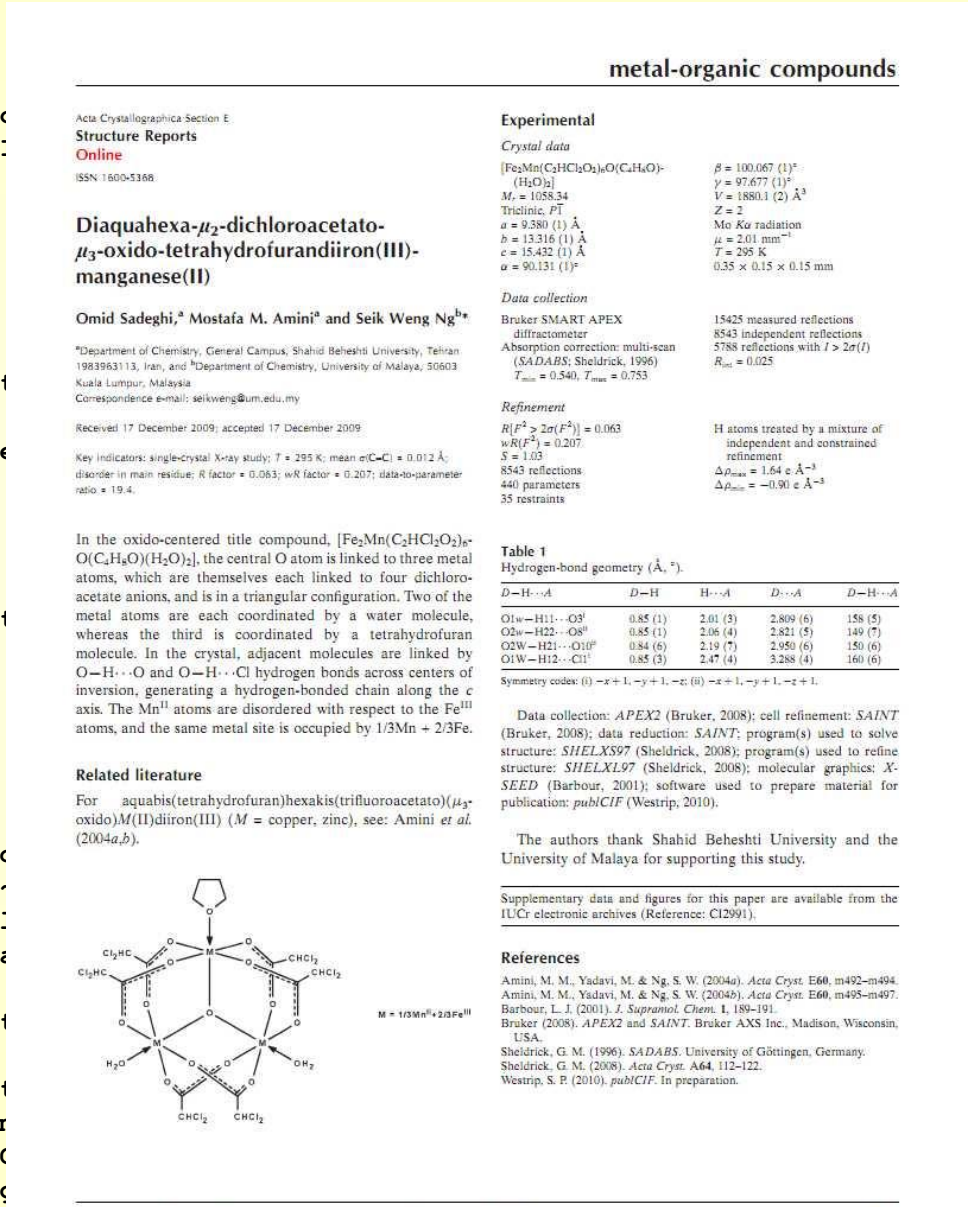

metal-organic compounds

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–Cl) = 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*).

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
O1*w*—H11···O3^{i}	0.85 (1)	2.01 (3)	2.809 (6)	158 (5)
O2*w*—H22···O8^{ii}	0.85 (1)	2.06 (4)	2.821 (5)	149 (7)
O2*W*—H21···O10^{ii}	0.84 (6)	2.19 (7)	2.950 (6)	150 (6)
O1*W*—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.


```
                    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
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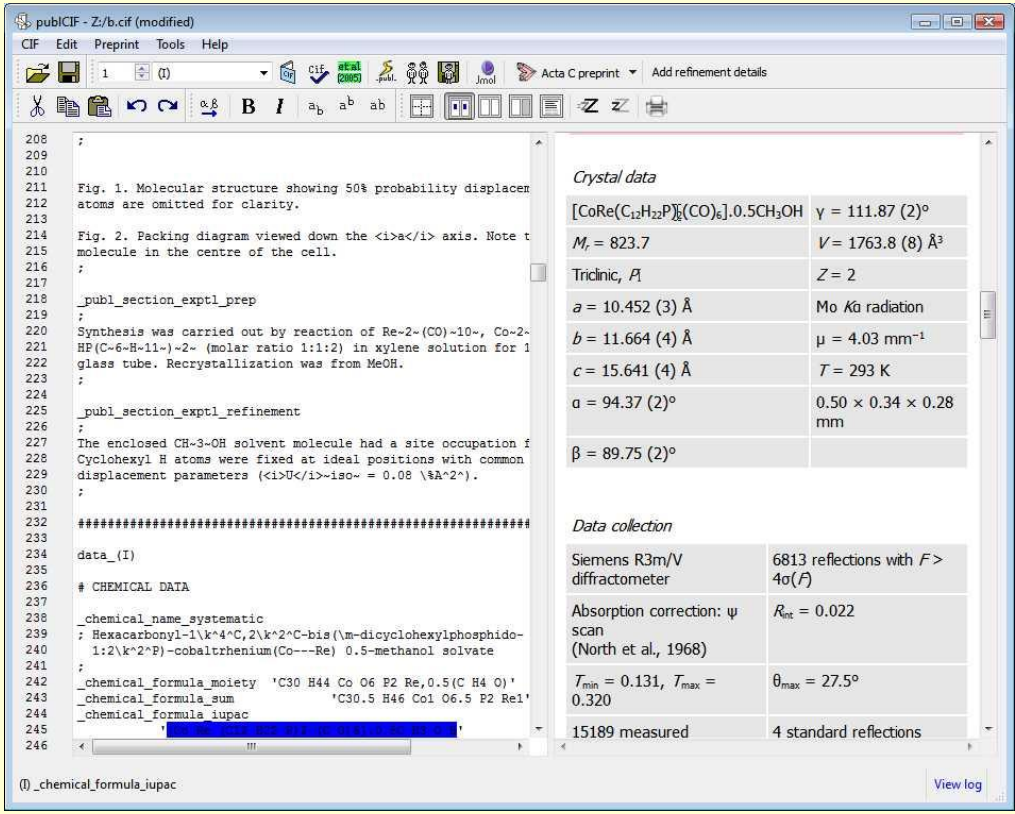
Authoring tools
(1) *publCIF*

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



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

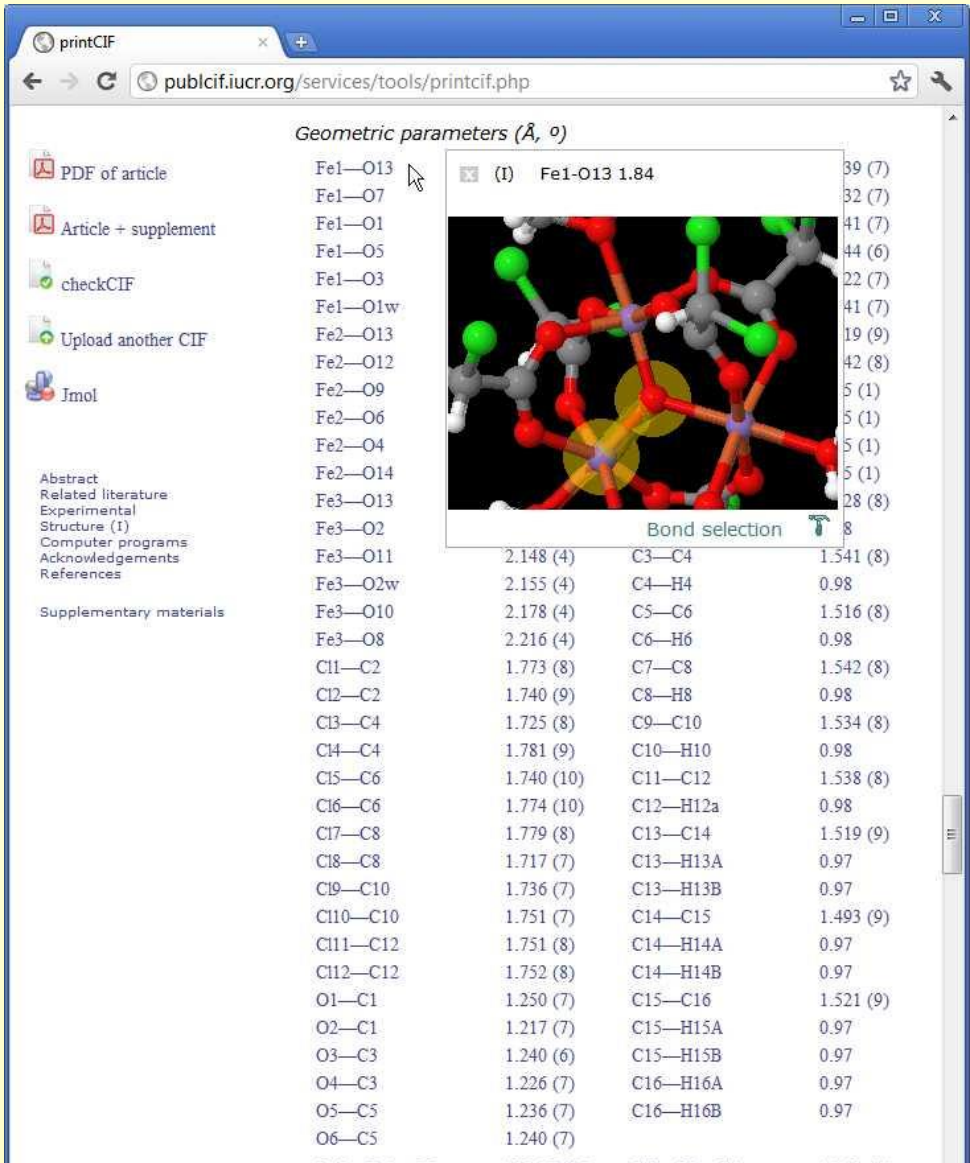
Crystallography Journals Online

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



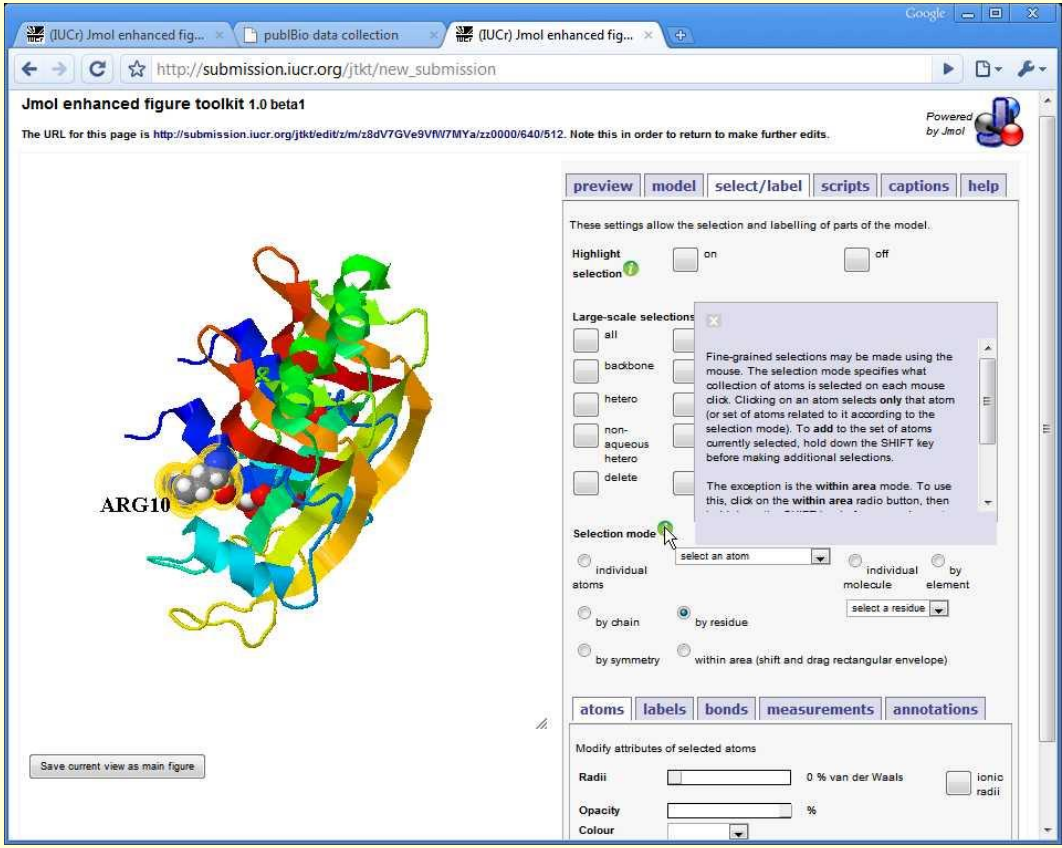
Authoring tools

(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



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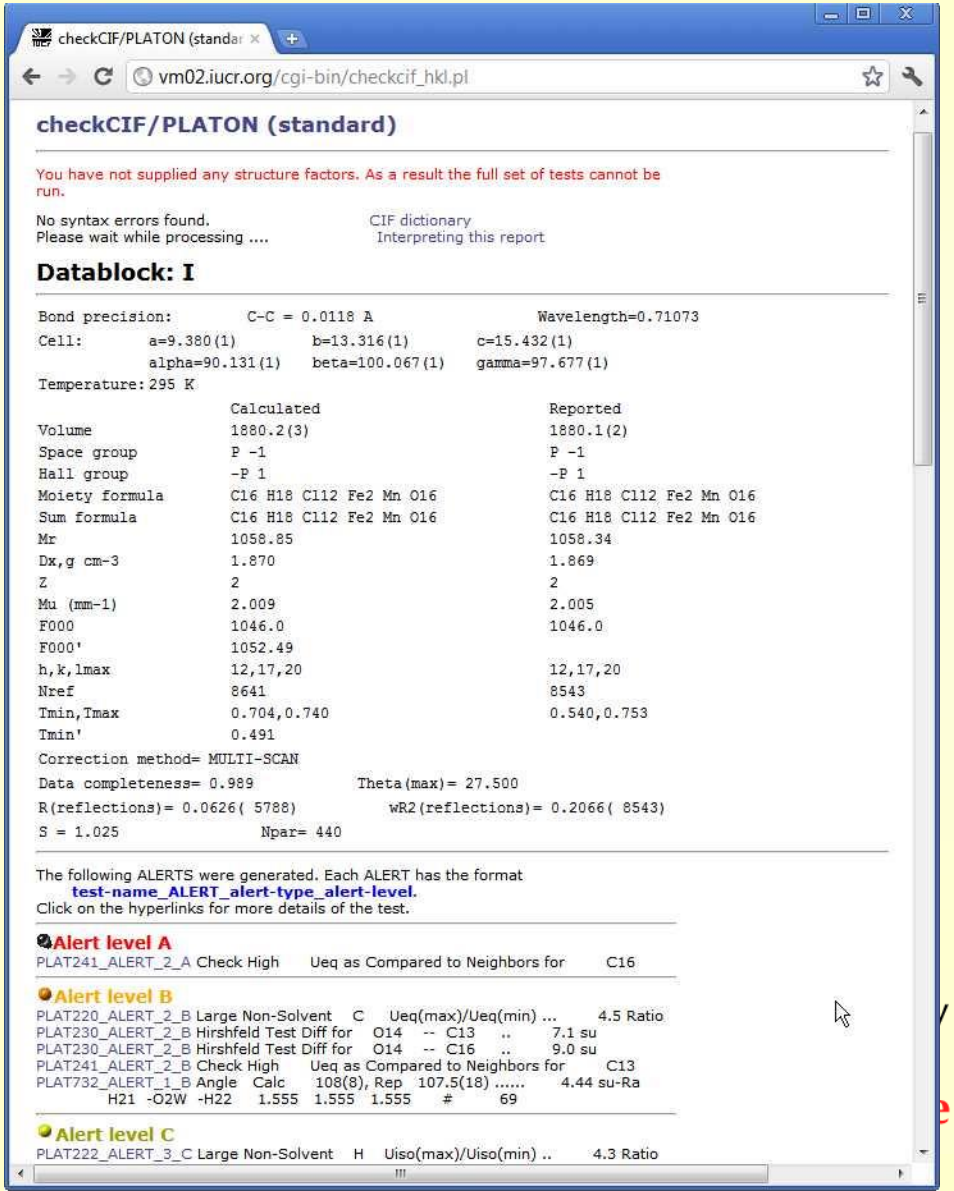
Authoring tools

(4) *checkCIF: validation of structural model*

http://checkcif.iucr.org



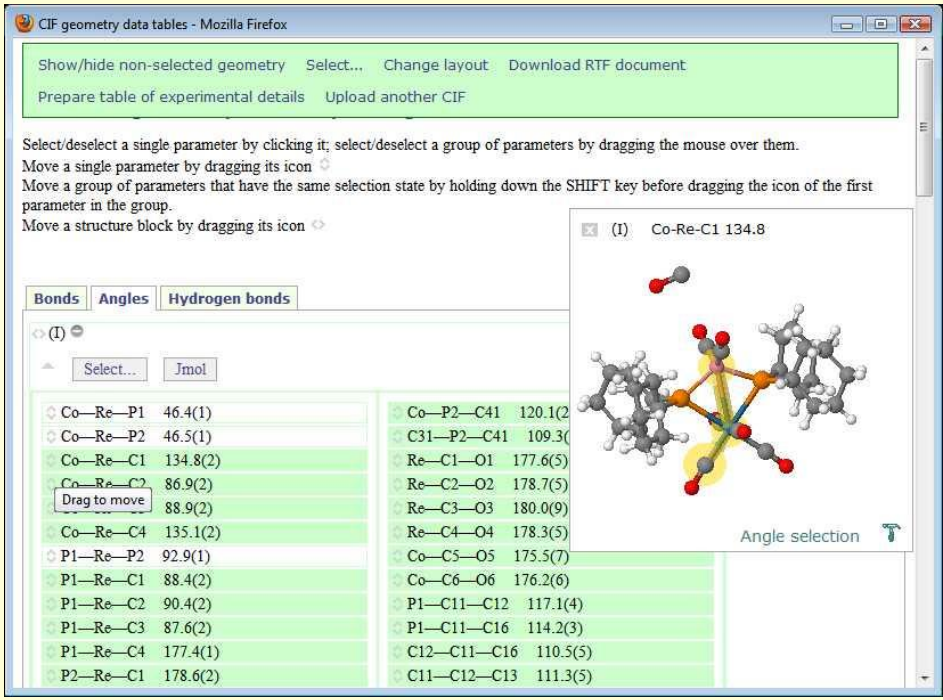
Authoring tools

(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/



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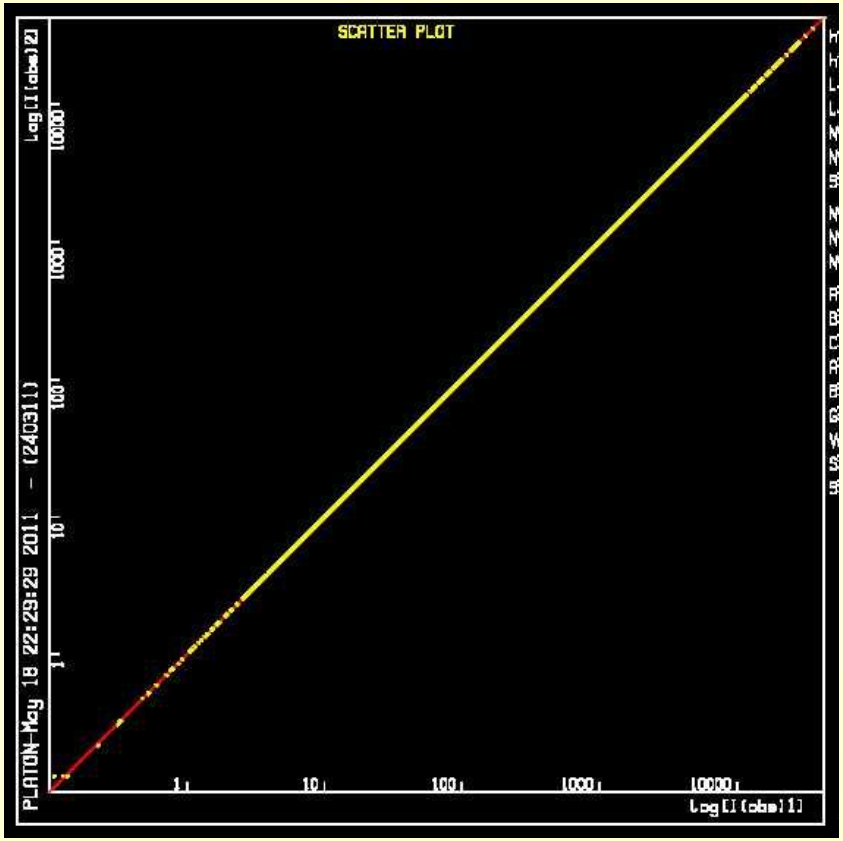
Internal consistency *versus* external context

- *Acta Cryst. E* 2007-2009 published over 100 fraudulent structure determinations



- Nature of fraud only became apparent when able to correlate different structure factor files.

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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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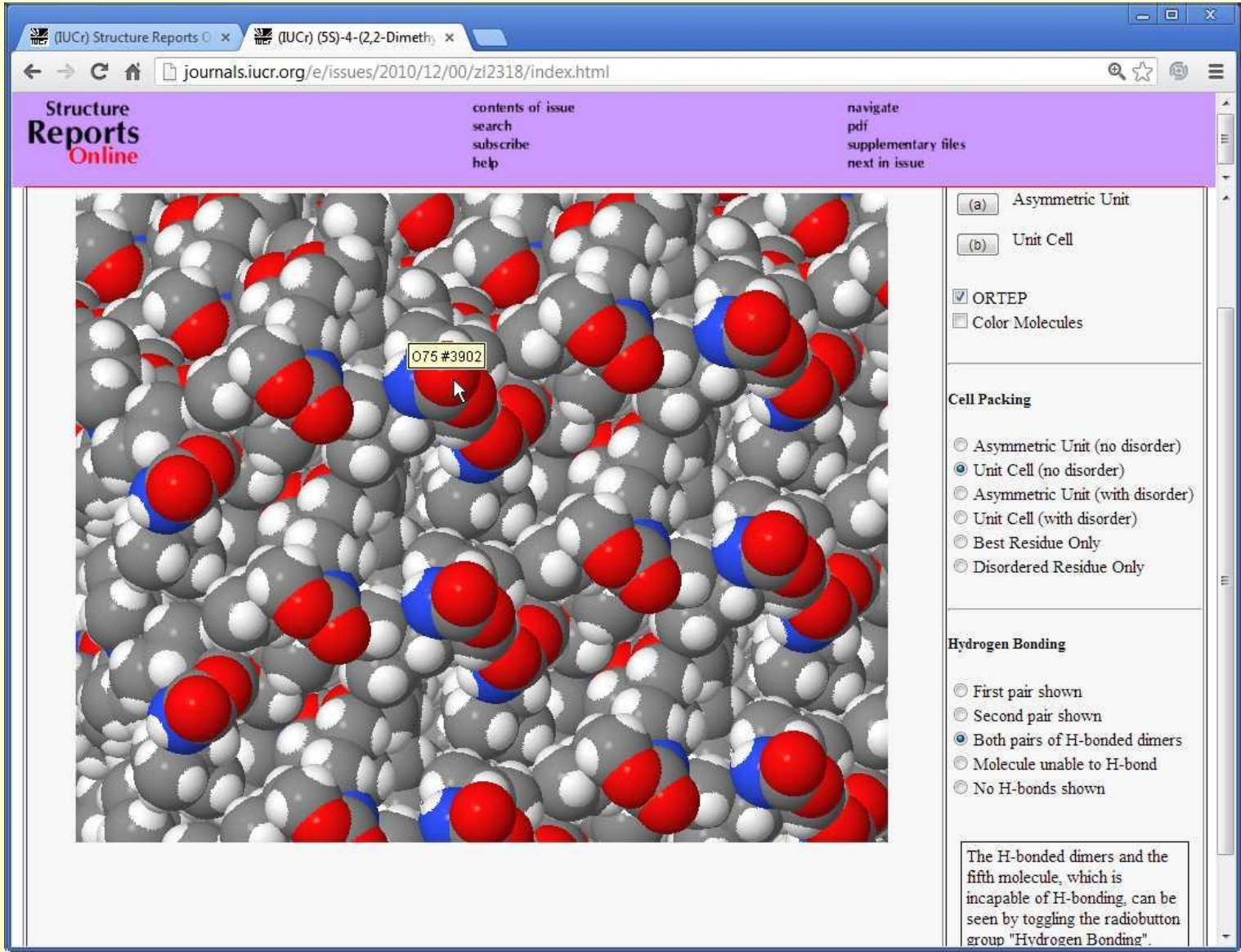
Reading the data

Interactive figures as an *integral* part of the article

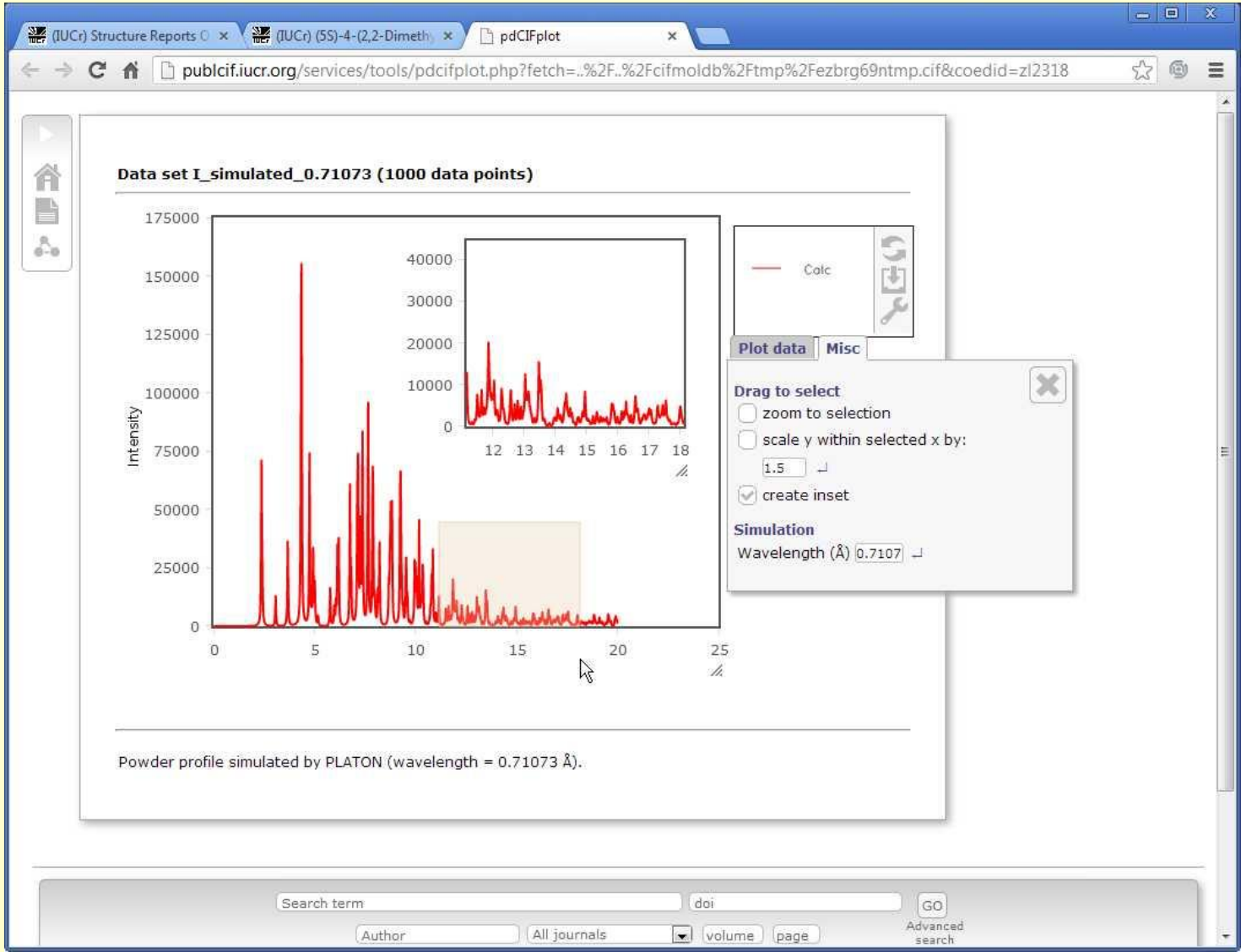
Author views

Reader freedom to explore



Working with the data

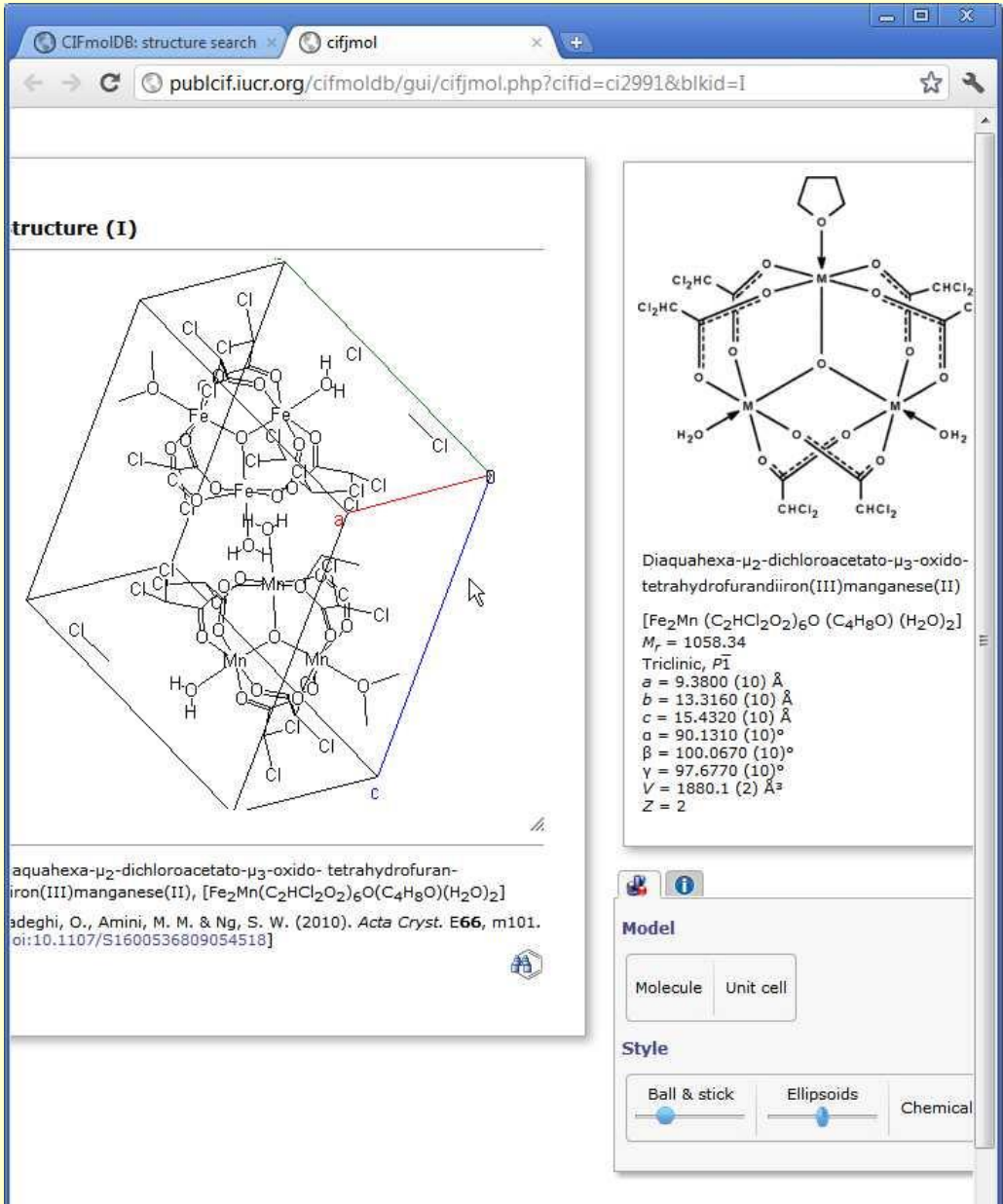
For *any* published structure, the reader can *generate* a predicted powder diffraction pattern



Visualising crystallography and chemistry

Direct access to the data means that visualisation is not restricted to different views/perspectives of a static scene

Can interactively explore atomic structure and motions, chemical connectivity, lattice symmetry, disorder, molecular dimensions, bond lengths and angles . . .



Thank you

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