

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

Michael Hoyland
ECM28
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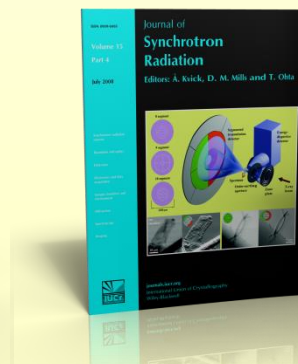
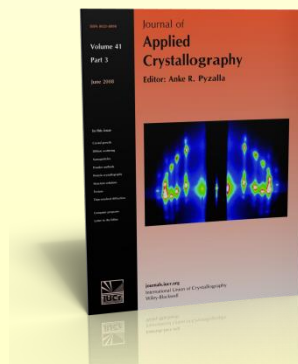
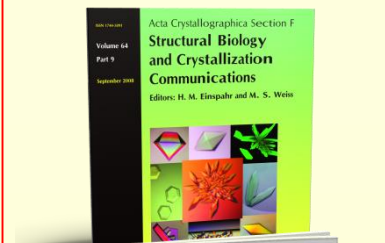
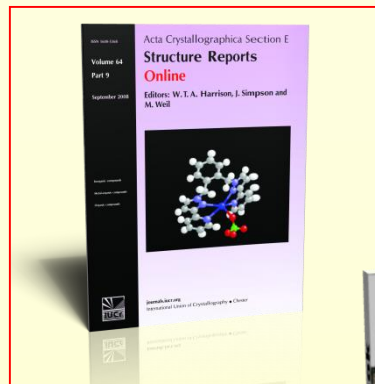
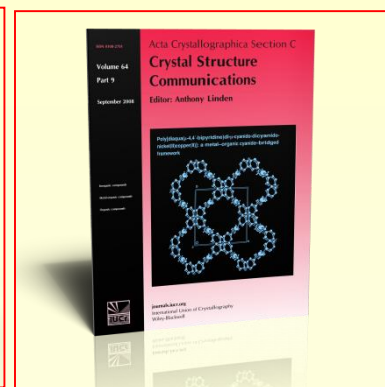
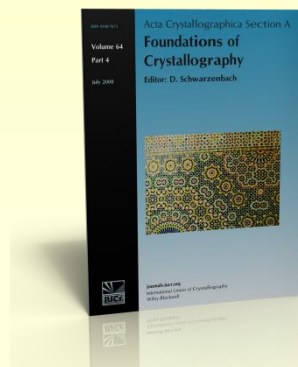
International Union of Crystallography
5 Abbey Square
Chester CH1 2HU
UK



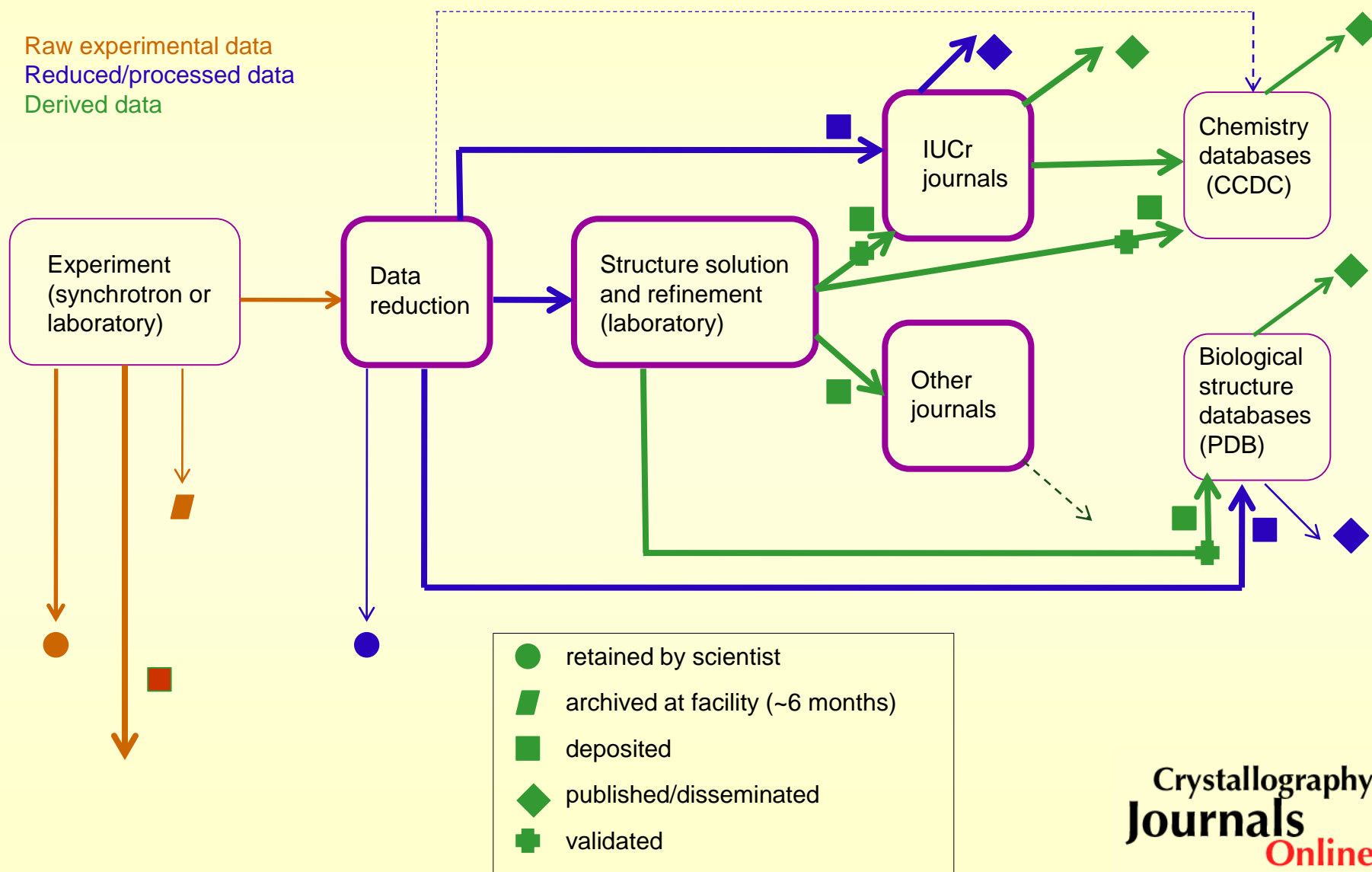
Crystallography
Journals
Online

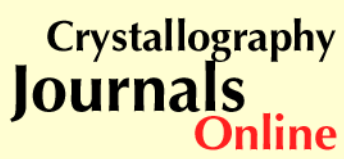
International Union of Crystallography

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



Data flow in crystallography



[illegible]



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

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



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?

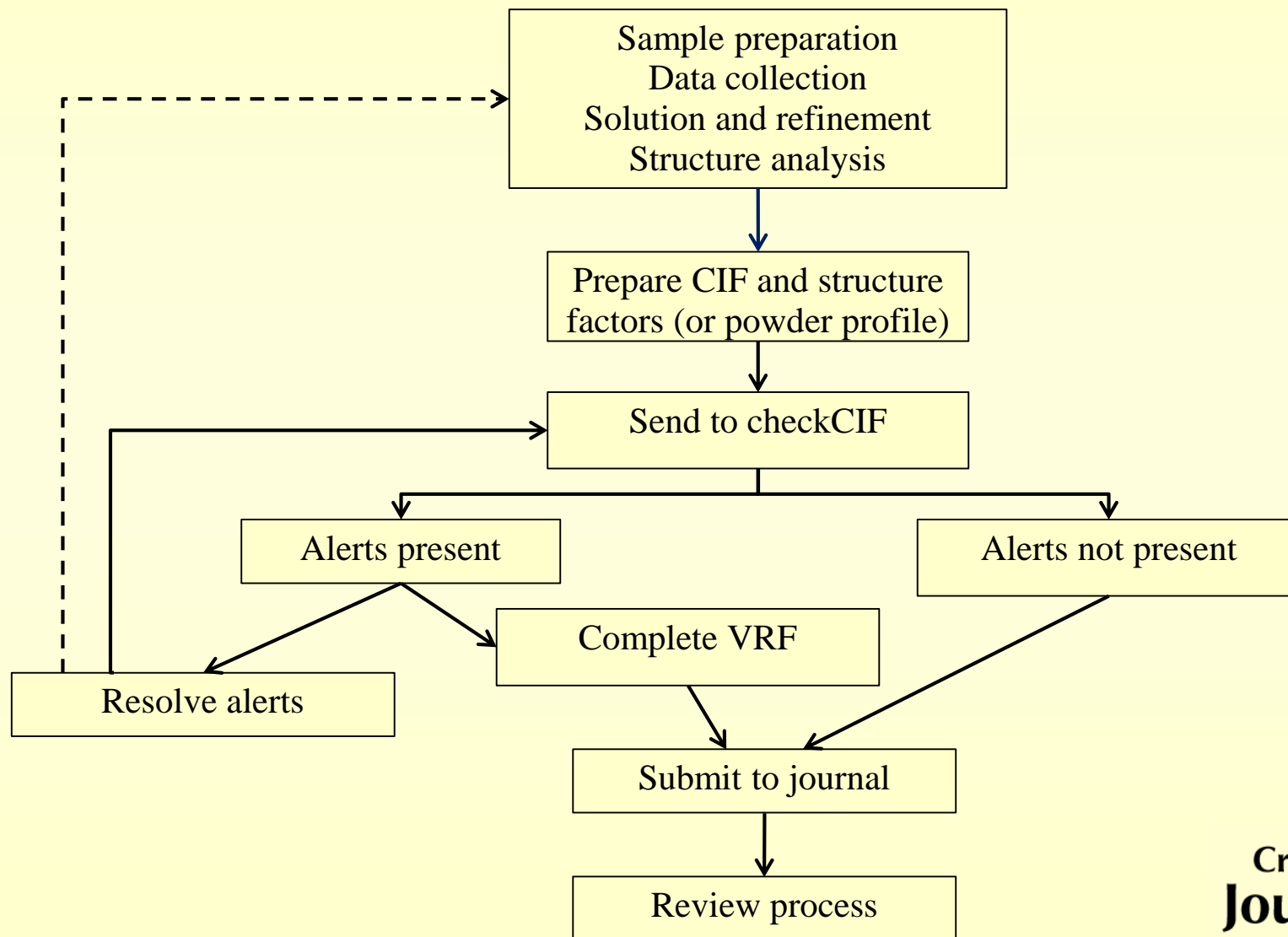
Automation of validation

- 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!**

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

Validation workflow



CIF as a vehicle for article submission

_publ_section_title

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Diaqua-hexa- μ_2 -dichloro-tetrahydrofuran- μ_2 -iron(III)-manganese(II)

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loop_

_publ_author_name

_publ_author_address

'Sadeghi, Omid'

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General Campus

Shahid Beheshti University

Tehran 1983963113

Iran

;

'Ng, Seik Weng'

; Department of Chemistry

University of Malaya

50603 Kuala Lumpur

Malaysia

;

_publ_section_abstract

; In the oxido-centered

[Fe μ_2 -Mn(C μ_2 -HCl μ_2 -O μ_2 -

the central O atom is

which are themselves each

dichloroacetate anions

configuration. Two of

coordinated by a water

is coordinated by a tet

the crystal, adjacent

O---H...O and O---H...O

centers of inversion,

chain along the *c*-axis

are disordered with

Acta Crystallographica Section E

Structure Reports

Online

ISSN: 1600-5368

Diaqua-hexa- μ_2 -dichloroacetato- μ_2 -oxido-tetrahydrofuran- μ_2 -iron(III)-manganese(II)

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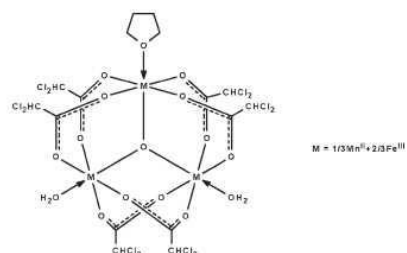
Received 17 December 2009; accepted 17 December 2009

Key indicators: single-crystal X-ray study; *T* = 295 K; mean $\sigma(\text{C}-\text{C})$ = 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, $[\text{Fe}_2\text{Mn}(\text{C}_2\text{H}_3\text{Cl}_2\text{O}_2)_6(\text{H}_2\text{O})_2] \cdot (\text{C}_2\text{H}_3\text{Cl}_2\text{O}_2)_2$, 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 $\text{O}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{Cl}$ 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_2 -oxido)- $M(\text{II})$ diiron(III) (*M* = copper, zinc, see: Amini *et al.* (2004a,b).



metal-organic compounds

Experimental

Crystal data

$[\text{Fe}_2\text{Mn}(\text{C}_2\text{H}_3\text{Cl}_2\text{O}_2)_6(\text{H}_2\text{O})_2] \cdot (\text{C}_2\text{H}_3\text{Cl}_2\text{O}_2)_2$
 $M_r = 1058.34$
 Triclinic, *P*1
a = 9.380 (1) Å
b = 13.316 (1) Å
c = 15.432 (1) Å
 $\alpha = 90.131 (1)^\circ$

$\beta = 100.067 (1)^\circ$
 $\gamma = 97.677 (1)^\circ$
V = 1880.1 (2) Å³
Z = 2
 Mo *K* α radiation
 $\mu = 2.01 \text{ mm}^{-1}$
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 σ (*I*)
*R*_{int} = 0.025

Refinement

$R[\text{F}^2] > 2\sigma(\text{F}^2) = 0.063$
 $wR(\text{F}^2) = 0.207$
S = 1.03
 8543 reflections
 440 parameters
 35 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 1.64 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.90 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> — <i>H</i> ... <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> — <i>H</i> ... <i>A</i>
O1W—H11...O3 ⁱ	0.85 (1)	2.01 (3)	2.809 (6)	158 (5)
O2W—H22...O8 ⁱ	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. (2004a). *Acta Cryst.* E60, m492–m494.
- Amini, M. M., Yadavi, M. & Ng, S. W. (2004b). *Acta Cryst.* E60, 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.* A64, 112–122.
- Westrip, S. P. (2010). *publCIF*. In preparation.

SHELXL-97

c

oacetato- μ_3 -oxido- μ_2 -iron(III)-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)

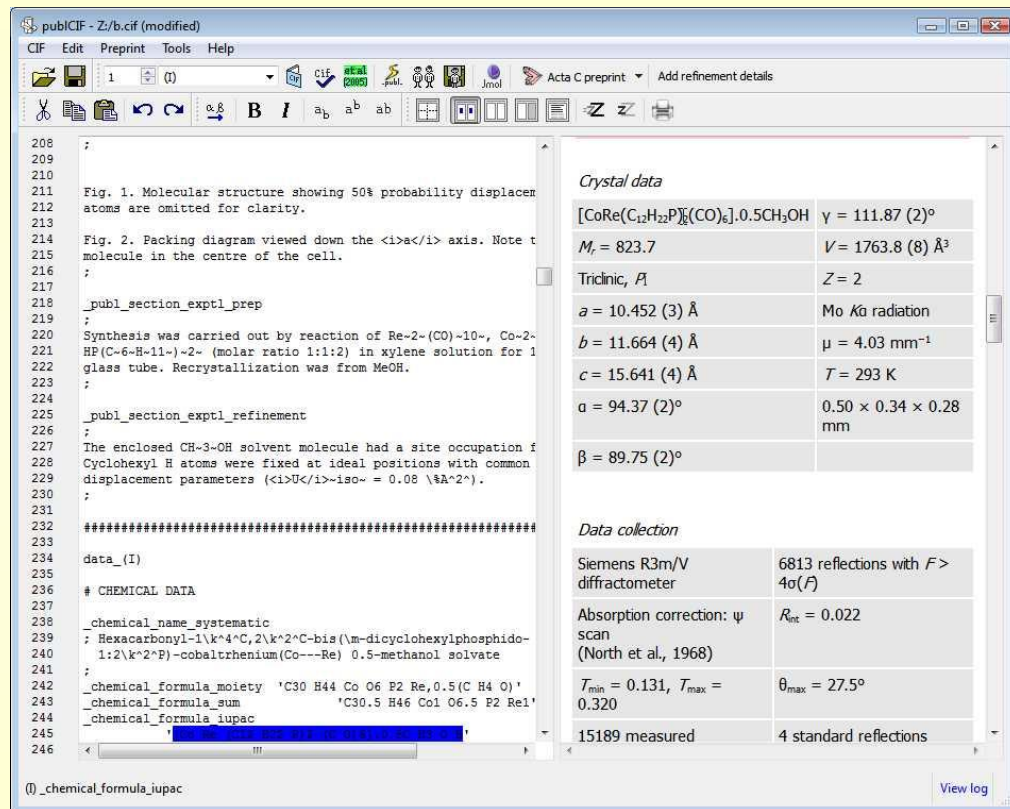
1880.1 (2)

Crystallography
Journals
Online

Authoring tools

(1) *publCIF*

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



The screenshot shows the publCIF application window. The left pane displays the CIF file content, which includes figure captions, synthesis details, and chemical data. The right pane provides a summary of the crystal and data collection parameters.

Crystal data

[CoRe(C ₁₂ H ₂₂ P) ₆](CO) ₆ · 0.5CH ₃ OH	$\gamma = 111.87 (2)^\circ$
$M_r = 823.7$	$V = 1763.8 (8) \text{ \AA}^3$
Triclinic, $P1$	$Z = 2$
$a = 10.452 (3) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 11.664 (4) \text{ \AA}$	$\mu = 4.03 \text{ mm}^{-1}$
$c = 15.641 (4) \text{ \AA}$	$T = 293 \text{ K}$
$\alpha = 94.37 (2)^\circ$	$0.50 \times 0.34 \times 0.28 \text{ mm}$
$\beta = 89.75 (2)^\circ$	

Data collection

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

At the bottom of the left pane, the chemical formula is displayed: [CoRe(C12H22P)6](CO)6.O5CH3OH.

<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>

printCIF

publCIF.iucr.org/services/tools/printcif.php

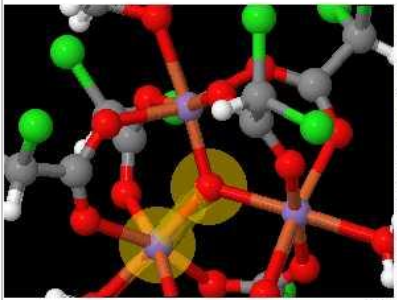
Geometric parameters (\AA , $^\circ$)

PDF of article
Article + supplement
checkCIF
Upload another CIF
Jmol

Abstract
Related literature
Experimental
Structure (I)
Computer programs
Acknowledgements
References

Supplementary materials

(I) Fe1—O13 1.84



Bond selection

Fe1—O13	1.84 (7)	C3—C4	1.541 (8)
Fe1—O7	1.82 (7)	C4—H4	0.98
Fe1—O1	1.81 (7)	C5—C6	1.516 (8)
Fe1—O5	1.84 (6)	C6—H6	0.98
Fe1—O3	1.82 (7)	C7—C8	1.542 (8)
Fe1—O1w	1.81 (7)	C8—H8	0.98
Fe2—O13	1.9 (9)	C9—C10	1.534 (8)
Fe2—O12	1.82 (8)	C10—H10	0.98
Fe2—O9	1.81 (1)	C11—C12	1.538 (8)
Fe2—O6	1.81 (1)	C12—H12a	0.98
Fe2—O4	1.81 (1)	C13—C14	1.519 (9)
Fe2—O14	1.81 (1)	C13—H13A	0.97
Fe3—O13	1.82 (8)	C13—H13B	0.97
Fe3—O2	1.81 (8)	C14—C15	1.493 (9)
Fe3—O11	2.148 (4)	C14—H14A	0.97
Fe3—O2w	2.155 (4)	C14—H14B	0.97
Fe3—O10	2.178 (4)	C15—C16	1.521 (9)
Fe3—O8	2.216 (4)	C15—H15A	0.97
Cl1—C2	1.773 (8)	C15—H15B	0.97
Cl2—C2	1.740 (9)	C16—H16A	0.97
Cl3—C4	1.725 (8)	C16—H16B	0.97
Cl4—C4	1.781 (9)		
Cl5—C6	1.740 (10)		
Cl6—C6	1.774 (10)		
Cl7—C8	1.779 (8)		
Cl8—C8	1.717 (7)		
Cl9—C10	1.736 (7)		
Cl10—C10	1.751 (7)		
Cl11—C12	1.751 (8)		
Cl12—C12	1.752 (8)		
O1—C1	1.250 (7)		
O2—C1	1.217 (7)		
O3—C3	1.240 (6)		
O4—C3	1.226 (7)		
O5—C5	1.236 (7)		
O6—C5	1.240 (7)		

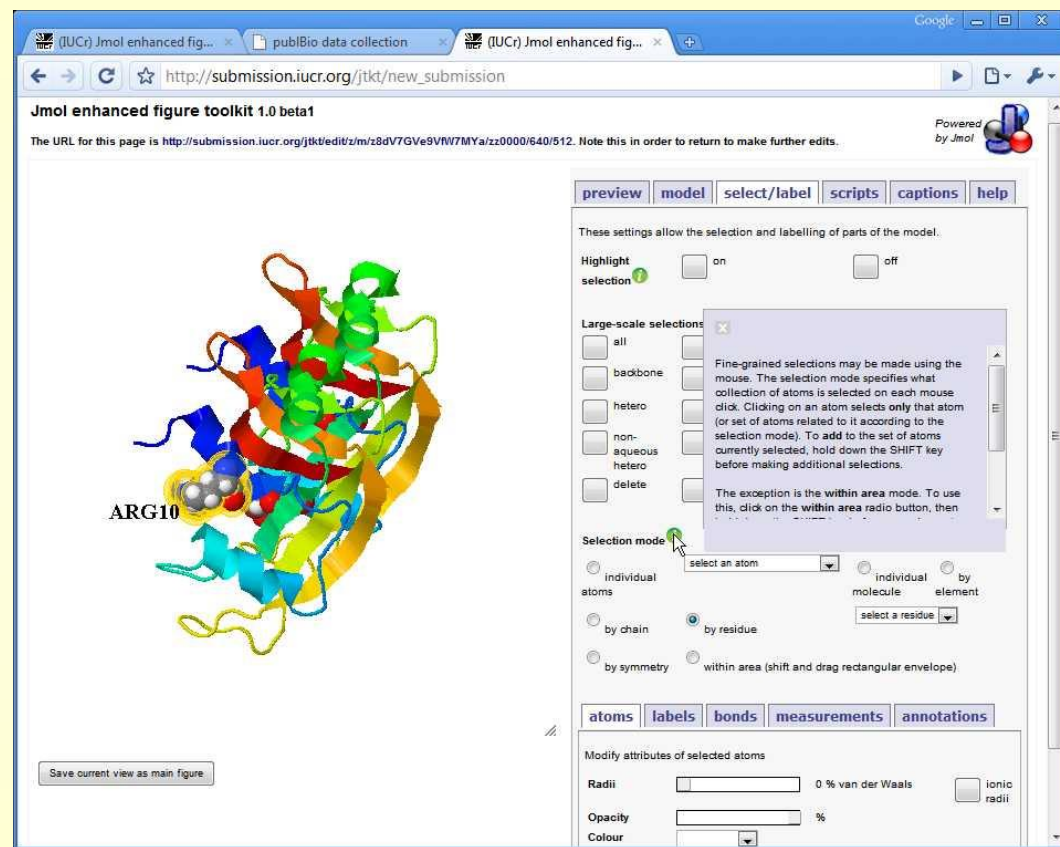
Authoring tools

(3) *Enhanced figure toolkit*

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

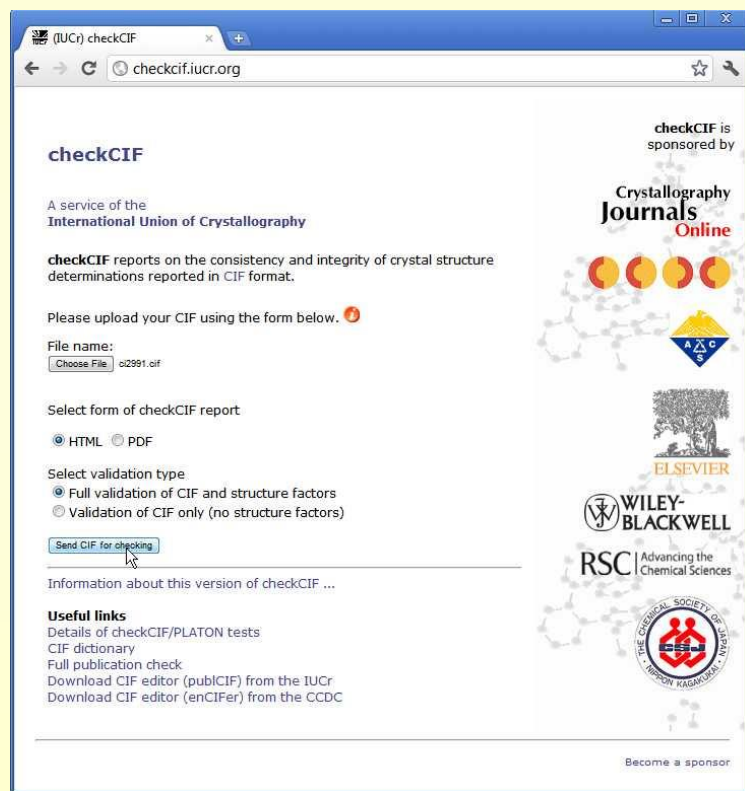
These figures form an *integral* part of the online published article

<http://submission.iucr.org/jtkit>



Authoring tools

(4) *checkCIF: validation of structural model*



<http://checkcif.iucr.org>

The screenshot shows the checkCIF/PLATON (standard) results page. It displays various crystallographic parameters and validation results.

checkCIF/PLATON (standard)

You have not supplied any structure factors. As a result the full set of tests cannot be run.

No syntax errors found. Please wait while processing CIF dictionary Interpreting this report

Datablock: I

Bond precision: C-C = 0.0118 Å Wavelength=0.71073

Cell: a=9.380(1) b=13.316(1) c=15.432(1)
alpha=90.131(1) beta=100.067(1) gamma=97.677(1)

Temperature: 295 K

	Calculated	Reported
Volume	1880.2(3)	1880.1(2)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C16 H18 Cl12 Fe2 Mn O16	C16 H18 Cl12 Fe2 Mn O16
Sum formula	C16 H18 Cl12 Fe2 Mn O16	C16 H18 Cl12 Fe2 Mn O16
Mr	1058.85	1058.34
Dx, g cm-3	1.870	1.869
Z	2	2
Mu (mm-1)	2.009	2.005
F000	1046.0	1046.0
F000'	1052.49	
h, k, lmax	12, 17, 20	12, 17, 20
Nref	8641	8543
Tmin, Tmax	0.704, 0.740	0.540, 0.753
Tmin'	0.491	

Correction method= MULTI-SCAN

Data completeness= 0.989 Theta(max)= 27.500

R(reflections)= 0.0626(5788) wR2(reflections)= 0.2066(8543)

S = 1.025 Npar= 440

The following ALERTS were generated. Each ALERT has the format **test-name_ALERT_alert-type_alert-level**. Click on the hyperlinks for more details of the test.

Alert level A

PLAT241_ALERT_2_A Check High Ueq as Compared to Neighbors for C16

Alert level B

PLAT220_ALERT_2_B Large Non-Solvent C Ueq(max)/Ueq(min) ... 4.5 Ratio

PLAT230_ALERT_2_B Hirshfeld Test Diff for O14 -- C13 .. 7.1 su

PLAT230_ALERT_2_B Hirshfeld Test Diff for O14 -- C16 .. 9.0 su

PLAT241_ALERT_2_B Check High Ueq as Compared to Neighbors for C13

PLAT732_ALERT_1_B Angle Calc 108(8), Rep 107.5(18) 4.44 su-Ra

H21 -O2W -H22 1.555 1.555 1.555 # 69

Alert level C

PLAT222_ALERT_3_C Large Non-Solvent H Uiso(max)/Uiso(min) .. 4.3 Ratio

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/>

CIF geometry data tables - Mozilla Firefox

Show/hide non-selected geometry Select... Change layout Download RTF document

Prepare table of experimental details Upload another CIF

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

Bonds **Angles** **Hydrogen bonds**

(I) Select... Jmol

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

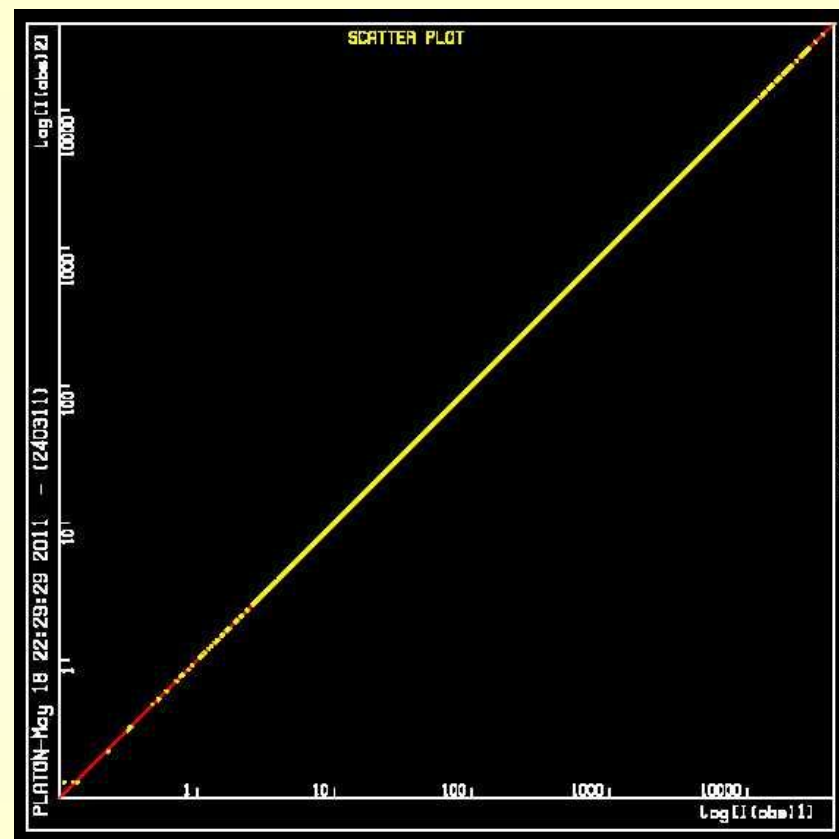
(I) Co-Re-C1 134.8

Angle selection

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.



Why publish data?

Some reasons:

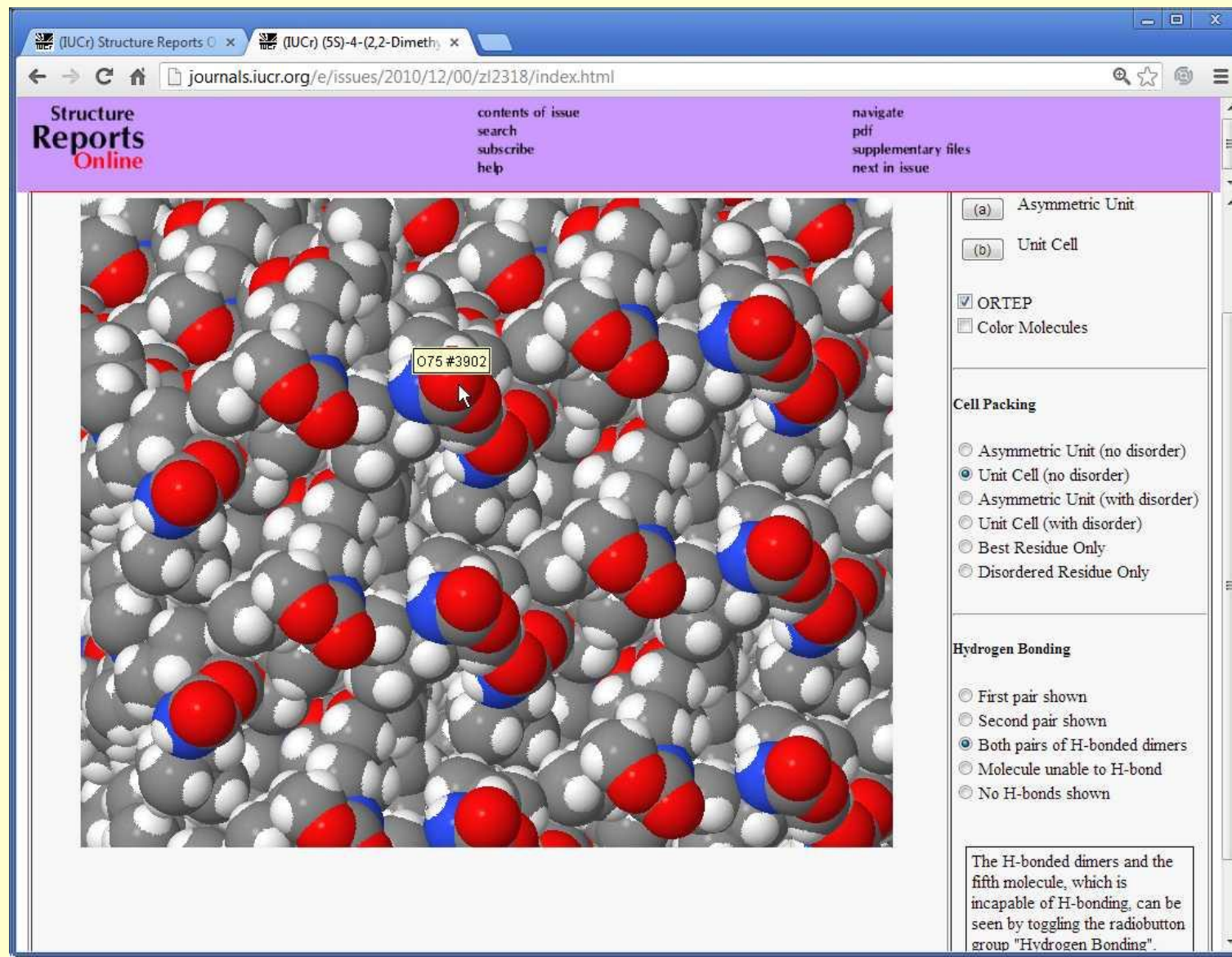
- To enhance the reproducibility of a scientific experiment
- To verify or support the validity of deductions from an experiment
- To safeguard against error
- To safeguard against fraud
- To allow other scholars to conduct further research based on experiments already conducted
- To allow reanalysis 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

Reading the data

Interactive figures as an *integral* part of the article

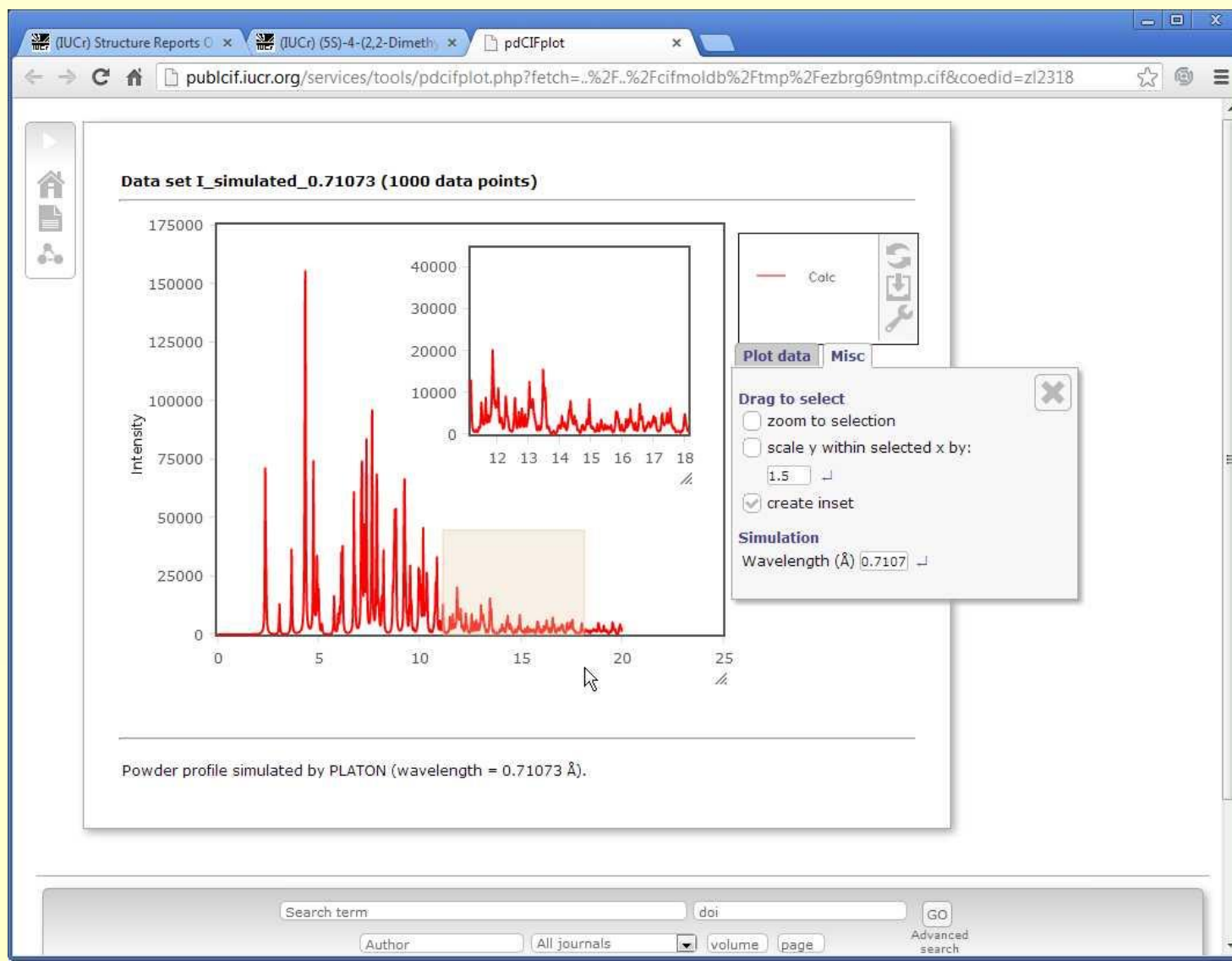
Author views

Reader freedom to explore



Working with the data

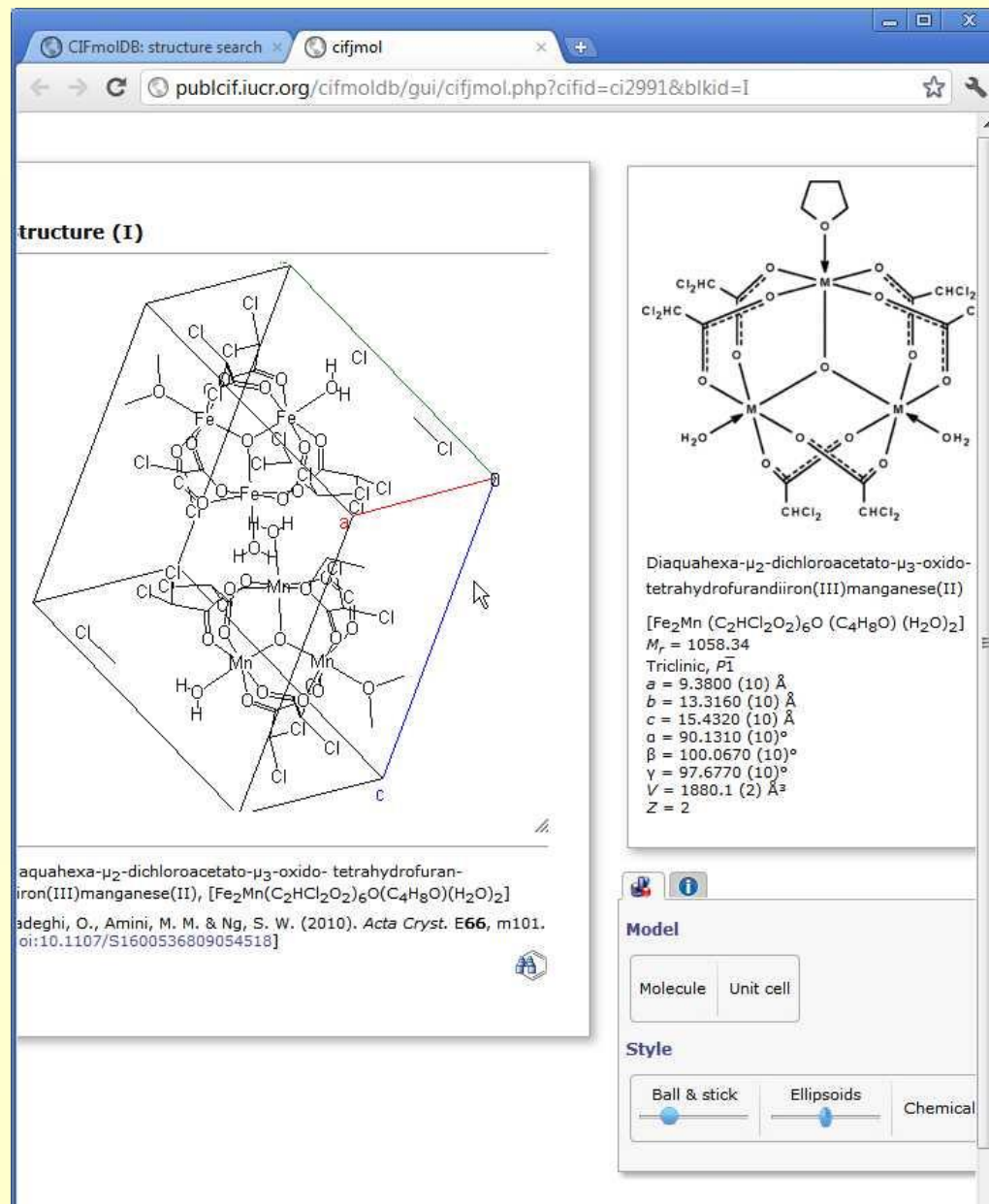
For *any* published structure, the reader can *generate* a predicted powder diffraction pattern



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