The IUCr perspective

Workshop on New Routes to Crystallographic Data Publication

IUCr 2008 Osaka

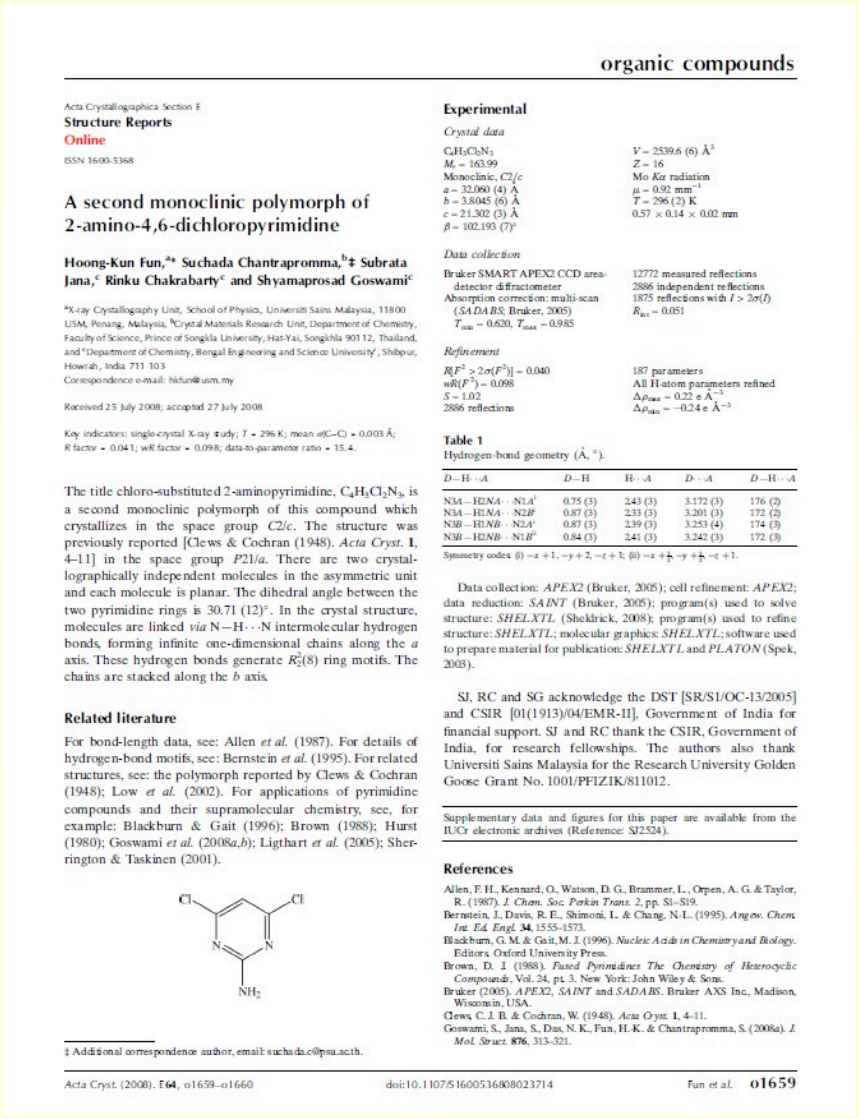
Crystallography Journals Online

The long view

- IUCr 60th anniversary
- Publication = make public / broadcast / disseminate / … / preserve
- Traditional journals = the historical record of science
- ‘Datuments’ (value-added/annotated data sets) are now also being ‘published’
- They deserve to be preserved also

Crystallography Journals Online

Structure Reports Online



organic compounds

Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

A second monoclinic polymorph of 2-amino-4,6-dichloropyrimidine

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Received 25 July 2008; accepted 27 July 2008

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.098; data-to-parameter ratio = 15.4.

The title chloro-substituted 2-aminopyrimidine, $C_4H_3Cl_2N_3$, is a second monoclinic polymorph of this compound which crystallizes in the space group $C2/c$. The structure was previously reported [Clews & Cochran (1948). *Acta Cryst.* **1**, 4–11] in the space group $P21/a$. There are two crystallographically independent molecules in the asymmetric unit and each molecule is planar. The dihedral angle between the two pyrimidine rings is 30.71 (12)°. In the crystal structure, molecules are linked *via* N—H···N intermolecular hydrogen bonds, forming infinite one-dimensional chains along the *a* axis. These hydrogen bonds generate $R_2^2(8)$ ring motifs. The chains are stacked along the *b* axis.

Related literature

For bond-length data, see: Allen *et al.* (1987). For details of hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related structures, see: the polymorph reported by Clews & Cochran (1948); Low *et al.* (2002). For applications of pyrimidine compounds and their supramolecular chemistry, see, for example: Blackburn & Gait (1996); Brown (1988); Hurst (1980); Goswami *et al.* (2008*a*,*b*); Ligthart *et al.* (2005); Sherrington & Taskinen (2001).

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Experimental

Crystal data

$C_4H_3Cl_2N_3$
M_r = 163.99
Monoclinic, $C2/c$
a = 32.060 (4) Å
b = 3.8045 (6) Å
c = 21.302 (3) Å
β = 102.193 (7)°
V = 2539.6 (6) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.92 mm^{-1}
T = 296 (2) K
0.57 × 0.14 × 0.02 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.620, T_{max} = 0.985
12772 measured reflections
2886 independent reflections
1875 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.098
S = 1.02
2886 reflections
187 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3*A*—H2*NA*···N1*A*^{i}	0.75 (3)	2.43 (3)	3.172 (3)	176 (2)
N3*A*—H1*NA*···N2*B*	0.87 (3)	2.33 (3)	3.201 (3)	172 (2)
N3*B*—H1*NB*···N2*A*^{ii}	0.87 (3)	2.39 (3)	3.253 (4)	174 (3)
N3*B*—H2*NB*···N1*B*^{iii}	0.84 (3)	2.41 (3)	3.242 (3)	172 (3)

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

SJ, RC and SG acknowledge the DST [SR/S1/OC-13/2005] and CSIR [01(1913)/04/EMR-II], Government of India for financial support. SJ and RC thank the CSIR, Government of India, for research fellowships. The authors also thank Universiti Sains Malaysia for the Research University Golden Goose Grant No. 1001/PFIZIK/811012.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2524).

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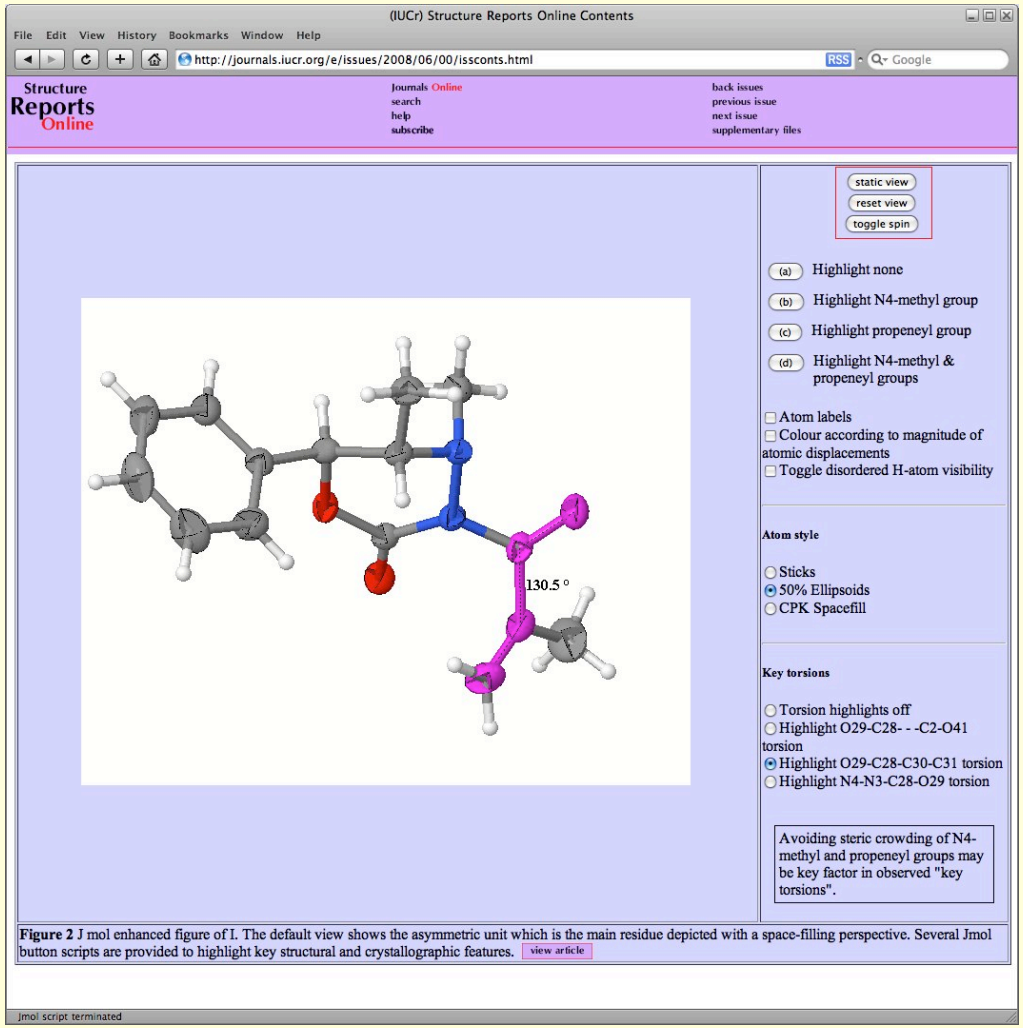
Acta Crystallographica Section E

- Presentation of structural model
- Data rich
- Limited discussion
- Routine science
- Open access
- Public ‘quality’ assessment report (checkcif)

Crystallography Journals Online

Enhanced figures: reader as investigator



- Data visualization and interaction as integral part of a research article.

- IUCr journals archive and provide access (small-unit-cell structures) to
  - CIF
  - Structure factors
  - Rietveld profiles

Crystallography Journals Online

Why archive data?

- Build up knowledge base
- Allow reanalysis of experiment
- Provide ‘disaster recovery’ backup storage
- Re-analyse for new science
- Education/development material
- Detection of systematic error
- Guard against fraud

Crystallography Journals Online

What to archive?

- Completeness
- Quality
- Relative value in the processing chain
- Reproducibility
- Retrievability
- Cost

Crystallography Journals Online

‘Towards the Crystallographic Archive’

- White Paper
  - ➢ Functional specification of a crystallographic ‘meta-archive’
- Call for proposals

Drivers for crystallography:

- eBank/eCrystals initiative (UK National Crystallography Service/U. Southampton)
- 2nd CrystalGrid Workshop April 26-28, 2007
- imgCIF workshops (ACA Honolulu, July 2006; BNL, May 2007; BSR Manchester/ Diamond, August 2007; BNL, May 2008)
- *J. Appl. Cryst.* (2008). **41**, 659 [doi:10.1107/S0021889808008832] *Of crystals, structure factors and diffraction images* Jovine, L., Morgunova E. and Ladenstein, R.

Crystallography Journals Online

Major considerations

- Purpose
- Policy
- Architecture
- Protocols and standards
- Economics

Crystallography Journals Online

Purpose

- Preserve and provide long-term access to
  - Research articles (“record of science”)
  - Unpublished research (theses)
  - Structural data
    - Refined coordinates, a.d.p.’s
    - Crystallization information
    - Processed data sets (structure factors)
    - Raw data sets (*e.g.* synchrotron images)
  - Computer software (algorithms)
  - Physical samples

Crystallography Journals Online

Policy

- Define community of interest
- Selection criteria
  - What to preserve?
  - For how long?
  - Quality criteria
  - Completeness
- Intellectual property and access rights
- Governance

Crystallography Journals Online

Architecture

- Federation
- Redundancy
- Cataloguing/acquisition
- Service provision

Crystallography Journals Online

Protocols and standards

- Open Archiving Systems Reference Model (OAIS)
- Open Archives Initiative protocols for metadata exchange and object reuse (OAI-PMH, OAI-ORE)
- RSS, ATOM
- Data definition/exchange standards
  - Crystallographic Information Framework (CIF)
  - HDF/NeXuS
  - nmrSTAR
  - InChI, CML, JCAMP-DX
- Publication standards (PubMed XML DTD)
- Metadata standards
- Metadata Encoding and Transmission Standard (METS)
- Identifiers

Crystallography Journals Online

Economics

- Non-profit publishers
- Commercial publishers
- Commercial databases
- Publicly funded databases
- Public research infrastructure budgets
- Public research grant proposals
- Voluntary effort
- Industry
- Paid service provision
- Payment-in-kind arrangements

Crystallography Journals Online