Supporting Information

Structural Insights into a Hexamorphic System of an Isoniazid Derivative

D. Hean, T. Gelbrich, U. J. Griesser, J. P. Michael, A. Lemmerer
# Table of Contents

SI1. Solution crystallization experimental details .................................................................3  
SI2. Representative DSC traces for IPH I – III ..................................................................4  
SI3. Powder X-ray diffraction patterns for forms IPH I – III and V .................................6  
SI4. FT-IR and Raman spectra of IPH I – III and V .............................................................8  
SI5. XPac Studies ...........................................................................................................11  
SI6. ORTEP diagrams and hydrogen bonding tables for IPH I – VI ..............................13  
SI7. CIF check reports for IPH I – VI .............................................................................17  
References .......................................................................................................................38
### SI1. Solution crystallization experimental details

**Table S1.** Solvent and evaporation procedures for obtaining IPH II, III and V.

<table>
<thead>
<tr>
<th>IPH</th>
<th>Slow evaporation</th>
<th>Fast evaporation</th>
<th>Vapour diffusion (polar/non-polar)</th>
</tr>
</thead>
<tbody>
<tr>
<td>II</td>
<td>acetonitrile</td>
<td>-</td>
<td>acetone/cyclohexane</td>
</tr>
<tr>
<td>III</td>
<td>-</td>
<td>ethanol/water (1:1)</td>
<td>ethanol/cyclohexane</td>
</tr>
<tr>
<td>V</td>
<td>acetone, butanol, ethanol, methanol, 1-propanol, water.</td>
<td>ethanol/water (1:1)</td>
<td>acetone/cyclohexane methanol/cyclohexane</td>
</tr>
<tr>
<td></td>
<td>acetone/cyclohexane (1:1)</td>
<td></td>
<td>acetone/diethyl ether methanol/diethyl ether</td>
</tr>
<tr>
<td></td>
<td>butanol/cyclohexane (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>ethanol/cyclohexane (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>ethyl acetate/cyclohexane (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>methanol/cyclohexane (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>acetone/diethyl ether (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>butanol/diethyl ether (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>ethanol/diethyl ether (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>ethyl acetate/diethyl ether (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>methanol/diethyl ether (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>acetone/n-hexane (1:1)</td>
<td></td>
<td>acetone/toluene methanol/toluene</td>
</tr>
<tr>
<td></td>
<td>butanol/n-hexane (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>ethanol/n-hexane (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>ethyl acetate/n-hexane (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>methanol/n-hexane (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>acetone/1-propanol (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>butanol/1-propanol (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>butanone/1-propanol (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>ethanol/1-propanol (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>ethyl acetate/1-propanol (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>methanol/1-propanol (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>acetone/toluene (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>butanol/toluene (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>ethanol/toluene (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>ethyl acetate/toluene (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>methanol/toluene (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>acetone/tetrahydrofuran (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>butanol/tetrahydrofuran (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>ethanol/tetrahydrofuran (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>ethyl acetate/tetrahydrofuran (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>methanol/tetrahydrofuran (1:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>chloroform/methanol (2:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>cyclohexane/methanol (2:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>diethyl ether/methanol (2:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1,4-dioxane/methanol (2:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>ethyl acetate/methanol (2:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>n-hexane/methanol (2:1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>tetrahydrofuran/methanol (2:1)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
SI2. Representative DSC traces for IPH I – III

**IPH I**

![Representative DSC trace for IPH I](image)

**Figure S1**: Representative DSC trace (exothermic is up) for numerous experiments for the melting and cool of IPH I. The top DSC trace (red) shows the melting endotherm of phase pure IPH I and the lower one (blue) the crystallization of this form from the melt on cooling.

**IPH II**

![Representative DSC trace for IPH II](image)

**Figure S2**: Representative DSC trace (exothermic is up) of IPH II. The upper DSC trace (red) shows the inhomogeneous melting of IPH II (melting of II and simultaneous crystallization of I) followed by the melting endotherm of IPH I.
Figure S3: Representative DSC trace (exothermic is up) of IPH III. The DSC trace shows a similar behaviour as IPH II but the onset of the inhomogeneous melting process is observed at 163 °C indicating a lower melting point.
SI3. Powder X-ray diffraction patterns for forms IPH I – III and V

**IPH I**

![Experimental and Calculated PXRD Patterns for IPH I](image)

**Figure S4**: Experimental PXRD pattern of IPH I, recorded at room temperature, and the PXRD pattern calculated from single crystal structure data of IPH I (at 173 K).

**IPH II**

![Experimental and Calculated PXRD Patterns for IPH II](image)

**Figure S5**: PXRD of IPH II compared to PXRD of single crystal structure determination of IPH II. The experimental pattern was calculated at room temperature and the calculated pattern at 173 K.
Figure S6: PXRD of IPH III compared to PXRD of single crystal structure determination of IPH III. The experimental pattern was calculated at room temperature and the calculated pattern at 173 K.

Figure S7: PXRD of IPH V compared to PXRD of single crystal structure determination of IPH V. The experimental pattern was calculated at room temperature and the calculated pattern at 173 K.
SI4. FT-IR and Raman spectra of IPH I – III and V

FT-IR spectra of IPH I – III and V

Figure S8: FT-IR spectra of IPH I – III and V. A spectrum for IPH IV and VI is unavailable as result from the small quantities recovered.

Raman spectroscopy of IPH I – III and V

IPH I

Figure S9: Raman spectrum of IPH I.
**Figure S10:** Raman spectrum of IPH II.

**Figure S11:** Raman spectrum of IPH III.
Figure S12: Raman spectrum of IPH V.
**SI5. XPac Studies**

All comparisons were carried out with the program *XPac*.¹ Dissimilarity parameters were calculated in the previously described manner ² (see ref. 3 for additional reference examples). Two sets of calculations were performed. The first set was based on geometrical parameters calculated from all 18 non-H atomic positions matching the IPH template structure, and the dissimilarity indices obtained from it will be denoted $x_{18}$. For the second set of calculations, only a core molecular unit defined by the positions of eight atoms (C1, C6, O1, N1, N3, C8, C7, C9; see Scheme 1) was used to minimise the effect of variations in the rotation angles of aromatic rings about the C1–C6 and C8–C9 bonds (the dissimilarity indices from this analysis will be denoted $x_8$).

**List of abbreviations**

<table>
<thead>
<tr>
<th>SC</th>
<th>Description</th>
<th>$n$</th>
<th>Dimension</th>
<th>Occurrences</th>
</tr>
</thead>
<tbody>
<tr>
<td>X1</td>
<td>monolayer</td>
<td>8</td>
<td>2D</td>
<td>IPH III, IPH V, AHE, PEH</td>
</tr>
<tr>
<td>X2</td>
<td>bilayer</td>
<td>12</td>
<td>2D</td>
<td>IPH V, PEH</td>
</tr>
<tr>
<td>Y1</td>
<td>monolayer</td>
<td>8</td>
<td>2D</td>
<td>IPH II (A), IPH IV</td>
</tr>
<tr>
<td>D</td>
<td>dimeric unit</td>
<td>1</td>
<td>0D</td>
<td>IPH II (B), IPH VI</td>
</tr>
</tbody>
</table>

**Table S2. Overview of the identified SCs.**

<table>
<thead>
<tr>
<th>SC</th>
<th>Description</th>
<th>$n$</th>
<th>Dimension</th>
<th>Occurrences</th>
</tr>
</thead>
<tbody>
<tr>
<td>X1</td>
<td>monolayer</td>
<td>8</td>
<td>2D</td>
<td>IPH III, IPH V, AHE, PEH</td>
</tr>
<tr>
<td>X2</td>
<td>bilayer</td>
<td>12</td>
<td>2D</td>
<td>IPH V, PEH</td>
</tr>
<tr>
<td>Y1</td>
<td>monolayer</td>
<td>8</td>
<td>2D</td>
<td>IPH II (A), IPH IV</td>
</tr>
<tr>
<td>D</td>
<td>dimeric unit</td>
<td>1</td>
<td>0D</td>
<td>IPH II (B), IPH VI</td>
</tr>
</tbody>
</table>

**Table S3. Corresponding lattice parameters for SCs X1 and X2.**

<table>
<thead>
<tr>
<th>Structure</th>
<th>IPH III</th>
<th>IPH V</th>
<th>AHE</th>
<th>PEH</th>
</tr>
</thead>
<tbody>
<tr>
<td>$t_1$</td>
<td>100</td>
<td>010</td>
<td>010</td>
<td>010</td>
</tr>
<tr>
<td></td>
<td>6.354 Å</td>
<td>5.546 Å</td>
<td>5.784 Å</td>
<td>5.594 Å</td>
</tr>
<tr>
<td>$t_2$</td>
<td>010</td>
<td>001</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td></td>
<td>7.662 Å</td>
<td>8.319 Å</td>
<td>8.594 Å</td>
<td>8.224 Å</td>
</tr>
<tr>
<td>$\angle t_{1,2}$</td>
<td>90°</td>
<td>90°</td>
<td>90°</td>
<td>90°</td>
</tr>
</tbody>
</table>
**Table S4.** Corresponding lattice parameters for SC Y1.

<table>
<thead>
<tr>
<th>Structure</th>
<th>IPH II</th>
<th>IPH IV</th>
</tr>
</thead>
<tbody>
<tr>
<td>(t_1)</td>
<td>001</td>
<td>001</td>
</tr>
<tr>
<td></td>
<td>8.235 Å</td>
<td>8.259 Å</td>
</tr>
<tr>
<td>(t_2)</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td></td>
<td>10.211 Å</td>
<td>10.612 Å</td>
</tr>
<tr>
<td>(\angle t_{1,2})</td>
<td>110.2°</td>
<td>109.6°</td>
</tr>
</tbody>
</table>

**Table S5.** XPac dissimilarity parameters \(x_8\) and \(x_{18}\) for the identified SCs.

<table>
<thead>
<tr>
<th>Structure 1</th>
<th>Structure 2</th>
<th>Dim</th>
<th>SC</th>
<th>(n)</th>
<th>(x_8)</th>
<th>(x_{18})</th>
</tr>
</thead>
<tbody>
<tr>
<td>AHE</td>
<td>IPH III</td>
<td>2D</td>
<td>X1</td>
<td>8</td>
<td>11.7</td>
<td>14.2</td>
</tr>
<tr>
<td>AHE</td>
<td>IPH IV</td>
<td>2D</td>
<td>X1</td>
<td>8</td>
<td>3.0</td>
<td>5.0</td>
</tr>
<tr>
<td>AHE</td>
<td>PEH</td>
<td>2D</td>
<td>X1</td>
<td>8</td>
<td>3.3</td>
<td>3.8</td>
</tr>
<tr>
<td>IPH III</td>
<td>IPH V</td>
<td>2D</td>
<td>X1</td>
<td>8</td>
<td>10.7</td>
<td>13.6</td>
</tr>
<tr>
<td>IPH III</td>
<td>PEH</td>
<td>2D</td>
<td>X1</td>
<td>8</td>
<td>9.7</td>
<td>12.6</td>
</tr>
<tr>
<td>IPH V</td>
<td>PEH</td>
<td>2D</td>
<td>X2</td>
<td>12</td>
<td>1.1</td>
<td>2.2</td>
</tr>
<tr>
<td>IPH II</td>
<td>IPH IV</td>
<td>2D</td>
<td>Y1</td>
<td>8</td>
<td>8.7</td>
<td>15.0</td>
</tr>
<tr>
<td>IPH II</td>
<td>IPH VI</td>
<td>0D</td>
<td>D</td>
<td>1</td>
<td>5.8</td>
<td>8.2</td>
</tr>
</tbody>
</table>
SI6. ORTEP diagrams and hydrogen bonding tables for IPH I – VI

**IPH I**

![ORTEP diagram of IPH I](image)

**Figure S13:** The asymmetric unit of IPH I depicting the numbering scheme of 50% displacement ellipsoids.

**IPH II**

![ORTEP diagram of IPH II](image)

**Figure S14:** The asymmetric unit of IPH II depicting the numbering scheme of 50% displacement ellipsoids.
**Figure S15:** The asymmetric unit of IPH III depicting the numbering scheme of 50% displacement ellipsoids.

**Figure S16:** The asymmetric unit of IPH IV depicting the numbering scheme of 50% displacement ellipsoids.

**Figure S17:** The asymmetric unit of IPH V depicting the numbering scheme of 50% displacement ellipsoids.
**IPH VI**

![Figure S18](image-url): The asymmetric unit of IPH VI depicting the numbering scheme of 50% displacement ellipsoids.

**H-bond tables for IPH I – VI**

**Table S5. H-bonds for IPH I**

<table>
<thead>
<tr>
<th>D-H...A</th>
<th>d(D-H)</th>
<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N(1B)-H(1B)...O(1A)#1</td>
<td>0.90(3)</td>
<td>2.04(3)</td>
<td>2.934(3)</td>
<td>169(3)</td>
</tr>
<tr>
<td>N(1C)-H(1C)...O(1D)#2</td>
<td>1.00(3)</td>
<td>1.96(3)</td>
<td>2.937(3)</td>
<td>165(3)</td>
</tr>
<tr>
<td>N(1D)-H(1D)...O(1C)#3</td>
<td>0.89(3)</td>
<td>2.03(3)</td>
<td>2.903(3)</td>
<td>166(3)</td>
</tr>
<tr>
<td>N(1A)-H(1A)...O(1B)#4</td>
<td>0.94(3)</td>
<td>2.00(3)</td>
<td>2.926(3)</td>
<td>170(2)</td>
</tr>
</tbody>
</table>

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,-y,-z+1    #2 x,y+1,z    #3 x,y-1,z  #4 –x+2, y, -z+1

**Table S6. H-bonds for IPH II**

<table>
<thead>
<tr>
<th>D-H...A</th>
<th>d(D-H)</th>
<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N(1A)-H(1A)...O(1A)#1</td>
<td>0.911(18)</td>
<td>2.068(18)</td>
<td>2.9748(14)</td>
<td>173.3(15)</td>
</tr>
<tr>
<td>N(1B)-H(1B)...N(2B)#2</td>
<td>0.866(18)</td>
<td>2.245(18)</td>
<td>3.0930(15)</td>
<td>166.4(15)</td>
</tr>
</tbody>
</table>

Symmetry transformations used to generate equivalent atoms:

#1 x,-y+1/2,z-1/2   #2 -x+2,-y,-z+2
### Table S7. H-bonds for IPH III

<table>
<thead>
<tr>
<th>D-H...A</th>
<th>d(D-H)</th>
<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N(1)-H(1)...O(1)#1</td>
<td>0.89(2)</td>
<td>2.04(2)</td>
<td>2.911(2)</td>
<td>168(2)</td>
</tr>
</tbody>
</table>

Symmetry transformations used to generate equivalent atoms:

#1 -x+3/2,y+1/2,z

### Table S8. H-bonds for IPH IV

<table>
<thead>
<tr>
<th>D-H...A</th>
<th>d(D-H)</th>
<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N(1)-H(1)...O(1)#1</td>
<td>0.89(3)</td>
<td>2.17(4)</td>
<td>3.032(3)</td>
<td>162(3)</td>
</tr>
</tbody>
</table>

Symmetry transformations used to generate equivalent atoms:

#1 x,-y+1/2,z+1/2

### Table S9. H-bonds for IPH V

<table>
<thead>
<tr>
<th>D-H...A</th>
<th>d(D-H)</th>
<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N(1)-H(1)...O(1)#1</td>
<td>0.90(3)</td>
<td>1.95(3)</td>
<td>2.845(2)</td>
<td>176(2)</td>
</tr>
</tbody>
</table>

Symmetry transformations used to generate equivalent atoms:

#1 x,-y+1/2,z+1/2

### Table S10. H-bonds for IPH VI

<table>
<thead>
<tr>
<th>D-H...A</th>
<th>d(D-H)</th>
<th>d(H...A)</th>
<th>d(D...A)</th>
<th>&lt;(DHA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N(1)-H(1)...O(1)#1</td>
<td>0.94(3)</td>
<td>1.95(3)</td>
<td>2.887(3)</td>
<td>175(3)</td>
</tr>
</tbody>
</table>

Symmetry transformations used to generate equivalent atoms:

#1 x,-y+1/2,z+1/2
SI7. CIF check reports for IPH I – VI

**checkCIF/PLATON report**

Structure factors have been supplied for datablock(s) IPH_I

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

**Datablock: IPH_I**

<table>
<thead>
<tr>
<th>Bond precision: C-C = 0.0044 Å</th>
<th>Wavelength=0.71073</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell:</td>
<td></td>
</tr>
<tr>
<td>a=9.7360(6)</td>
<td>b=9.8752(6)</td>
</tr>
<tr>
<td>alpha=92.856(4)</td>
<td>beta=100.295(4)</td>
</tr>
<tr>
<td>c=26.1543(16)</td>
<td>gamma=91.291(4)</td>
</tr>
<tr>
<td>Temperature:</td>
<td></td>
</tr>
<tr>
<td>173 K</td>
<td></td>
</tr>
<tr>
<td>Calculated</td>
<td>Reported</td>
</tr>
<tr>
<td>Volume 2469.8(3)</td>
<td>2469.8(3)</td>
</tr>
<tr>
<td>Space group P-1</td>
<td>P-1</td>
</tr>
<tr>
<td>Hall group -P 1</td>
<td>-P 1</td>
</tr>
<tr>
<td>Moiety formula C14 H13 N3 O</td>
<td>C14 H13 N3 O</td>
</tr>
<tr>
<td>Sum formula C14 H13 N3 O</td>
<td>C14 H13 N3 O</td>
</tr>
<tr>
<td>Mr 239.27</td>
<td>239.27</td>
</tr>
<tr>
<td>Dx,g cm-3 1.287</td>
<td>1.287</td>
</tr>
<tr>
<td>Z 8</td>
<td>8</td>
</tr>
<tr>
<td>Mu (mm-1) 0.084</td>
<td>0.084</td>
</tr>
<tr>
<td>F000 1008.0</td>
<td>1008.0</td>
</tr>
<tr>
<td>F000' 1008.37</td>
<td></td>
</tr>
<tr>
<td>h,k,lmax 11,11,31</td>
<td>11,11,31</td>
</tr>
<tr>
<td>Nref 9178</td>
<td>9177</td>
</tr>
<tr>
<td>Tmin,Tmax 0.994,0.998</td>
<td></td>
</tr>
<tr>
<td>Tmin' 0.967</td>
<td></td>
</tr>
</tbody>
</table>

Correction method= Not given

Data completeness= 1.000 Theta(max) = 25.500

\( R(\text{reflections}) = 0.0559(4183) \) \( \text{wR2(reflections)} = 0.1600(9177) \)

\( S = 0.995 \quad \text{Npar= 669} \)

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 B

PLAT414_ALERT_2_B Short Intra D-H..H-X H1D .. H7D1 .. 1.87 Ang.

Alert level C

PLAT242_ALERT_2_C Low Ueq as Compared to Neighbors for ... C1C Check
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ................. 0.0044 Ang.
PLAT353_ALERT_3_C Long N-H (N0.87,N1.01A) N1C - H1C ... 1.02 Ang.
PLAT414_ALERT_2_C Short Intra D-H..H-X H1A .. H7A3 .. 1.91 Ang.
PLAT906_ALERT_3_C Large K value in the Analysis of Variance ...... 5.956 Check

Alert level G

PLAT154_ALERT_1_G The su’s on the Cell Angles are Equal ............... 0.0000 Degree
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels .......... 12 Note
PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd. # C14 H13 N3 O 2 Note
PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd. # C14 H13 N3 O 4 Note
PLAT910_ALERT_3_G Missing # of FCF Reflections Below Th(Min) ...... 1 Report

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

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

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

# start Validation Reply Form
_vrf_PLAT2026_IPH_1
;
PROBLEM: Ratio Observed / Unique Reflections too Low .... 46 %
RESPONSE: ...
;
_vrf_PLAT242_IPH_1
;
PROBLEM: Low Ueq as Compared to Neighbors for ...... C1C Check
RESPONSE: ...
;
_vrf_PLAT340_IPH_1
;
PROBLEM: Low Bond Precision on C-C Bonds ................. 0.0044 Ang.
RESPONSE: ...
;
_vrf_PLAT353_IPH_1
;
PROBLEM: Long N-H (N0.87,N1.01A) N1C - H1C ... 1.02 Ang.
RESPONSE: ...

18
It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

**Publication of your CIF in IUCr journals**

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

**Publication of your CIF in other journals**

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

---

**PLATON version of 20/08/2014; check.def file version of 18/08/2014**
checkCIF/PLATON report

Structure factors have been supplied for datablock(s) IPH_II

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: IPH_II

Bond precision: C-C = 0.0018 A  Wavelength=0.71073

Cell:
- a=10.2114(2)  b=30.3315(7)  c=8.2353(2)
- alpha=90  beta=110.193(1)  gamma=90

Temperature: 173 K

Volume: 2393.92(9)  2393.92(9)
Space group: P 21/c  P 21/c
Hall group: -P 2ybc  -P 2ybc

MoIety formula: C14 H13 N3 O  C14 H13 N3 O

Sum formula: C14 H13 N3 O  C14 H13 N3 O

Mr: 239.27  239.27

Dx,g cm-3: 1.328  1.328
Z: 8  8

Mu (mm-1): 0.087  0.087

F000: 1008.0  1008.0

F000': 1008.37

h,k,lmax: 13,40,10  13,40,10

Nref: 5787  5778

Tmin,Tmax: 0.971,0.991  0.954,0.991

Twin': 0.953

Correction method= MULTI-SCAN

Data completeness= 0.998  Theta(max)= 28.000

R(reflections)= 0.0398( 4579)  wR2(reflections)= 0.1117( 5778)

S = 1.038  Npar= 335

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 C
PLAT414_ALERT_2_C Short Intra D-H..H-X   H1A .. H14C ..  1.96 Ang.
PLAT911_ALERT_3_C Missing # PFC Refl Between TMin & STh/L= 0.600  5 Report
PLAT922_ALERT_1_C WR2 in the CIF and FCF Differ by ................. -0.0013 Check
PLAT923_ALERT_1_C S values in the CIF and FCF Differ by ........... -0.015 Check

Alert level G
PLAT432_ALERT_2_G Short Inter X...Y Contact   O1A .. C2A ..  2.99 Ang.
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600  4 Note

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

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

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```
# start Validation Reply Form
_vrf_PLAT414_IPH_II
;
PROBLEM: Short Intra D-H..H-X   H1A .. H14C ..  1.96 Ang.
RESPONSE: ...
;
_vrf_PLAT911_IPH_II
;
PROBLEM: Missing # PFC Refl Between TMin & STh/L= 0.600  5 Report
RESPONSE: ...
;
_vrf_PLAT922_IPH_II
;
PROBLEM: WR2 in the CIF and FCF Differ by ................. -0.0013 Check
RESPONSE: ...
;
_vrf_PLAT923_IPH_II
;
PROBLEM: S values in the CIF and FCF Differ by ........... -0.015 Check
RESPONSE: ...
;
# end Validation Reply Form
```
It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

**Publication of your CIF in IUCr journals**

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C or E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

**Publication of your CIF in other journals**

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 20/08/2014; check.def file version of 18/08/2014
checkCIF/PLATON report

Structure factors have been supplied for datablock(s) IPH_III

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

**Datablock: IPH_III**

<table>
<thead>
<tr>
<th>Bond precision: C-C=0.0028 Å</th>
<th>Wavelength=0.71073</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell:</td>
<td></td>
</tr>
<tr>
<td>a=6.3542(4)</td>
<td>b=7.6624(6)</td>
</tr>
<tr>
<td>alpha=90</td>
<td>beta=90</td>
</tr>
<tr>
<td>c=49.231(3)</td>
<td>gamma=90</td>
</tr>
<tr>
<td>Temperature:</td>
<td></td>
</tr>
<tr>
<td>173 K</td>
<td></td>
</tr>
<tr>
<td>Volume</td>
<td></td>
</tr>
<tr>
<td>2397.0(3)</td>
<td>2397.0(3)</td>
</tr>
<tr>
<td>Space group:</td>
<td></td>
</tr>
<tr>
<td>P b c a</td>
<td>Pbca</td>
</tr>
<tr>
<td>Hall group:</td>
<td></td>
</tr>
<tr>
<td>-P 2ac 2ab</td>
<td>-P 2ac 2ab</td>
</tr>
<tr>
<td>Moiety formula:</td>
<td></td>
</tr>
<tr>
<td>C14 H13 N3 O</td>
<td>C14 H13 N3 O</td>
</tr>
<tr>
<td>Sum formula:</td>
<td></td>
</tr>
<tr>
<td>C14 H13 N3 O</td>
<td>C14 H13 N3 O</td>
</tr>
<tr>
<td>Mr</td>
<td></td>
</tr>
<tr>
<td>239.27</td>
<td>239.27</td>
</tr>
<tr>
<td>Dx,g cm^{-3}</td>
<td></td>
</tr>
<tr>
<td>1.326</td>
<td>1.326</td>
</tr>
<tr>
<td>Z</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td>Mu (mm^{-1})</td>
<td></td>
</tr>
<tr>
<td>0.087</td>
<td>0.087</td>
</tr>
<tr>
<td>F000</td>
<td></td>
</tr>
<tr>
<td>1008.0</td>
<td>1008.0</td>
</tr>
<tr>
<td>F000’</td>
<td></td>
</tr>
<tr>
<td>1008.37</td>
<td></td>
</tr>
<tr>
<td>h,k,lmax</td>
<td></td>
</tr>
<tr>
<td>7,9,59</td>
<td>7,9,59</td>
</tr>
<tr>
<td>Nref</td>
<td></td>
</tr>
<tr>
<td>2222</td>
<td>2124</td>
</tr>
<tr>
<td>Tmin,Tmax</td>
<td></td>
</tr>
<tr>
<td>0.989,0.996</td>
<td>0.964,0.996</td>
</tr>
<tr>
<td>Tmin’</td>
<td></td>
</tr>
<tr>
<td>0.964</td>
<td></td>
</tr>
</tbody>
</table>

Correction method= MULTI-SCAN

Data completeness= 0.956 Theta(max)= 25.500

R(reflections)= 0.0425( 1777) wR2(reflections)= 0.1317( 2124)

S = 1.194 Npar= 168

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 B

PLAT029_ALERT_3_B_diffred_measured_fraction_theta_full Low ........ 0.956 Note

Alert level C

PLAT906_ALERT_3_C_Large K value in the Analysis of Variance ........ 5.528 Check
PLAT911_ALERT_3_C_Missing # FCF Refl Between Tmin & STh/L= 0.600 80 Report
PLAT918_ALERT_3_C_Reflection{s} with I(obs) much smaller I(calc) . 1 Check
PLAT994_ALERT_3_C_Number of (Iobs-Icalc)/SigmaW > 10 Outliers .... 1 Check
PLAT993_ALERT_3_C_Large Value of Not (SHELXL) Weight Optimized S . 38.26

Alert level G

PLAT910_ALERT_3_G_Missing # of FCF Reflections Below Th(Min) ...... 3 Report
PLAT912_ALERT_4_G_Missing # of FCF Reflections Above STh/L= 0.600 15 Note
PLAT995_ALERT_1_G_Reported (CIF) and Actual (FCF) Lmax Differ by . 1 Units

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

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

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

# start Validation Reply Form
_vrf_PLAT906_IPH_III
;
PROBLEM: Large K value in the Analysis of Variance ........ 5.528 Check
RESPONSE: ... ;
_vrf_PLAT911_IPH_III
;
PROBLEM: Missing # FCF Refl Between Tmin & STh/L= 0.600 80 Report
RESPONSE: ... ;
_vrf_PLAT918_IPH_III
;
PROBLEM: Reflection{s} with I(obs) much smaller I(calc) . 1 Check
RESPONSE: ... ;
_vrf_PLAT994_IPH_III
;
PROBLEM: Number of (Iobs-Icalc)/SigmaW > 10 Outliers .... 1 Check
RESPONSE: ... ;
_vrf_PLAT993_IPH_III
;
25
It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation); however, if you intend to submit to Acta Crystallographica Section C or E, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the Notes for Authors of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 20/08/2014; check.def file version of 18/08/2014
checkCIF/PLATON report

Structure factors have been supplied for datablock(s) IPH_IV

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: IPH_IV

Bond precision:  C-C = 0.0044 Å  Wavelength=0.71073 Å

Cell:  a=10.6217(16)  b=14.442(2)  c=8.2589(12)
       alpha=90  beta=109.623(5)  gamma=90

Temperature:  173 K

Volume  Calculated  Reported
        1193.3(3)  1193.3(3)
Space group  P 21/c  P2(1)/c
Hall group  -P 2ybc  -P 2ybc
Moiety formula  C14 H13 N3 O  C14 H13 N3 O
Sum formula  C14 H13 N3 O  C14 H13 N3 O
Mr  239.27  239.27
Dx, g cm^-3  1.332  1.332
Z  4  4
Mu (mm^-1)  0.087  0.087
F000  504.0  504.0
F000’  504.19
h,k,lmax  11,16,9  11,16,9
Nref  1730  1723
Tmin,Tmax  0.995,0.997  0.969,0.989
Tmin’  0.952

Correction method= MULTI-SCAN

Data completeness= 0.996  Theta(max)= 23.310

R(reflections)= 0.0532( 1183)  wR2(reflections)= 0.1241( 1723)

S = 1.039  Npar= 167

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 B

THETM01_ALERT_3_B The value of \( \frac{\sin(\theta_{\text{max}})}{\text{wavelength}} \) is less than 0.575

Calculated \( \frac{\sin(\theta_{\text{max}})}{\text{wavelength}} = 0.5568 \)

PLAT414_ALERT_2_B Short Intra D-H..H-X H1 .. H7A .. 1.81 Ang.

Alert level C

PLAT140_ALERT_3_C Low Bond Precision on C-C Bonds ................. 0.0044 Ang.

PLAT906_ALERT_3_C Large K value in the Analysis of Variance ...... 6.100 Check

PLAT911_ALERT_3_C Missing # PFC Refl Between TMin & STh/L= 0.557 4 Report

Alert level G

PLAT909_ALERT_3_G Percentage of Observed Data at Theta(Max) still 52 %

PLAT910_ALERT_3_G Missing # of PFC Reflections Below Th(Min) ..... 3 Report

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

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

Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```plaintext
# start Validation Reply Form
  _vrf_PLAT340_IPH_IV
  ;
  PROBLEM: Low Bond Precision on C-C Bonds ................. 0.0044 Ang.
  RESPONSE: ...
  ;
  _vrf_PLAT906_IPH_IV
  ;
  PROBLEM: Large K value in the Analysis of Variance ...... 6.100 Check
  RESPONSE: ...
  ;
  _vrf_PLAT911_IPH_IV
  ;
  PROBLEM: Missing # PFC Refl Between TMin & STh/L= 0.557 4 Report
  RESPONSE: ...
  ;
# end Validation Reply Form
```

29
It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation); however, if you intend to submit to Acta Crystallographica Section C or E, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the Notes for Authors of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 20/08/2014; check.def file version of 18/08/2014

Dubbelt: IFH IV - ellipsoid plot
checkCIF/PLATON report

Structure factors have been supplied for datablock(s) IPH_V

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

**Datablock: IPH_V**

<table>
<thead>
<tr>
<th>Bond precision: C-C = 0.0030 A</th>
<th>Wavelength=0.71073</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell:</td>
<td></td>
</tr>
<tr>
<td>a=25.8998(13)</td>
<td>b=5.5463(3)</td>
</tr>
<tr>
<td>alpha=90</td>
<td>c=8.3187(4)</td>
</tr>
<tr>
<td>beta=95.876(4)</td>
<td>gamma=90</td>
</tr>
<tr>
<td>Temperature:</td>
<td>173 K</td>
</tr>
<tr>
<td>Volume</td>
<td>Calculated</td>
</tr>
<tr>
<td>1188.69(11)</td>
<td>Reported</td>
</tr>
<tr>
<td>Space group</td>
<td>P 21/c</td>
</tr>
<tr>
<td>Hall group</td>
<td>-P 2ybc</td>
</tr>
<tr>
<td>Moiety formula</td>
<td>C14 H13 N3 O</td>
</tr>
<tr>
<td>Sum formula</td>
<td>C14 H13 N3 O</td>
</tr>
<tr>
<td>Mr</td>
<td>239.27</td>
</tr>
<tr>
<td>Dx,g cm^{-3}</td>
<td>1.337</td>
</tr>
<tr>
<td>Z</td>
<td>4</td>
</tr>
<tr>
<td>Mu (mm^{-1})</td>
<td>0.088</td>
</tr>
<tr>
<td>F000</td>
<td>504.0</td>
</tr>
<tr>
<td>F000’</td>
<td>504.19</td>
</tr>
<tr>
<td>h,k,lmax</td>
<td>34,7,10</td>
</tr>
<tr>
<td>Nref</td>
<td>2877</td>
</tr>
<tr>
<td>Tmin,Tmax</td>
<td>0.979, 0.995</td>
</tr>
<tr>
<td>Tmin’</td>
<td>0.961</td>
</tr>
</tbody>
</table>

Correction method= MULTI-SCAN

Data completeness= 0.996 θ(max)= 28.000

R(reflections)= 0.0605(2245) wR2(reflections)= 0.1587(2866)

S = 1.020 Npar= 168

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 C**
PLAT906_ALERT_3_C Large K value in the Analysis of Variance ...... 2.436 Check
PLAT911_ALERT_3_C Missing # PFC Refl Between THmin & Sth/L= 0.600 9 Report

**Alert level G**
PLAT910_ALERT_3_G Missing # of PFC Reflections Below Th(MIN) ...... 1 Report

- 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

**Validation response form**

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```plaintext
# start Validation Reply Form
_vrf_PLAT906_IPH_V
;
PROBLEM: Large K value in the Analysis of Variance ...... 2.436 Check
RESPONSE: ...
;
_vrf_PLAT911_IPH_V
;
PROBLEM: Missing # PFC Refl Between THmin & Sth/L= 0.600 9 Report
RESPONSE: ...
;
# end Validation Reply Form
```
It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation); however, if you intend to submit to Acta Crystallographica Section C or E, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the Notes for Authors of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 20/08/2014; check.def file version of 18/08/2014
checkCIF/PLATON report

Structure factors have been supplied for datablock(s) IPH_VI

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: IPH_VI

Bond precision: C-C = 0.0040 Å

Wavelength=0.71073

Cell:

a=13.488(2) b=9.6611(15) c=9.3604(13)

alpha=90 beta=90.183(5) gamma=90

Temperature: 173 K

Calculated

Reported

Volume 1219.7(3) 1219.7(3)

Space group P 21/c P2(1)/c

Hall group -P 2ybc -P 2ybc

Moiety formula C14 H13 N3 O C14 H13 N3 O

Sum formula C14 H13 N3 O C14 H13 N3 O

Mr 239.27 239.27

Dx,g cm-3 1.303 1.303

Z 4 4

Mu (mm-1) 0.085 0.085

F000 504.0 504.0

F000’ 504.19

h,k,lmax 17,12,12 17,12,12

Nref 2940 2900

Tmin,Tmax 0.965,0.997 0.965,0.997

Tmin’ 0.965

Correction method= MULTI-SCAN

Data completeness= 0.986 Theta(max)= 28.000

R(reflections)= 0.0729( 1992) wR2(reflections)= 0.2468( 2900)

S = 1.087 Npar= 168

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 C**

<table>
<thead>
<tr>
<th>Alert</th>
<th>Description</th>
<th>Level</th>
</tr>
</thead>
<tbody>
<tr>
<td>PLAT911_ALERT_3_C</td>
<td>Missing # FCP Refl Between THmin &amp; STh/L= 0.600</td>
<td>33 Report</td>
</tr>
<tr>
<td>PLAT913_ALERT_3_C</td>
<td>Missing # of Very Strong Reflections in FCP</td>
<td>5 Note</td>
</tr>
<tr>
<td>PLAT918_ALERT_3_C</td>
<td>Reflection(s) with I(obs) much smaller I(calc)</td>
<td>1 Check</td>
</tr>
<tr>
<td>PLAT934_ALERT_3_C</td>
<td>Number of (Iobs-Icalc)/SigmaW &gt; 10 Outliers</td>
<td>1 Check</td>
</tr>
<tr>
<td>PLAT939_ALERT_3_C</td>
<td>Large Value of Not (SHELXL) Weight Optimized S</td>
<td>37.29</td>
</tr>
</tbody>
</table>

**Alert level G**

<table>
<thead>
<tr>
<th>Alert</th>
<th>Description</th>
<th>Level</th>
</tr>
</thead>
<tbody>
<tr>
<td>PLAT066_ALERT_1_G</td>
<td>Predicted and Reported Tmin/Tmax Range Identical</td>
<td>? Check</td>
</tr>
<tr>
<td>PLAT072_ALERT_2_G</td>
<td>SHELXL First Parameter in WHT Unusually Large</td>
<td>0.14 Report</td>
</tr>
<tr>
<td>PLAT910_ALERT_3_G</td>
<td>Missing # of FCP Reflections Below Th(Min)</td>
<td>3 Report</td>
</tr>
<tr>
<td>PLAT912_ALERT_4_G</td>
<td>Missing # of FCP Reflections Above STh/L= 0.600</td>
<td>4 Note</td>
</tr>
</tbody>
</table>

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

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

**Validation response form**

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```plaintext
# start Validation Reply Form  
_vrf_PLAT911_IPH_VI  
;  
PROBLEM: Missing # FCP Refl Between THmin & STh/L= 0.600  
RESPONSE: ...  
;  
_vrf_PLAT913_IPH_VI  
;  
PROBLEM: Missing # of Very Strong Reflections in FCP  
RESPONSE: ...  
;  
_vrf_PLAT918_IPH_VI  
;  
PROBLEM: Reflection(s) with I(obs) much smaller I(calc)  
RESPONSE: ...  
;  
_vrf_PLAT934_IPH_VI  
;  
PROBLEM: Number of (Iobs-Icalc)/SigmaW > 10 Outliers  
RESPONSE: ...  
;  
_vrf_PLAT939_IPH_VI  
;  
PROBLEM: Large Value of Not (SHELXL) Weight Optimized S  
RESPONSE: ...  
;  
# end Validation Reply Form
```

35
It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the “special_details” fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

**Publication of your CIF in IUCr journals**

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

**Publication of your CIF in other journals**

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

---

PLATON version of 20/08/2014; check.def file version of 18/08/2014
References