

## **Supporting Information**

### **Structural Insights into a Hexamorphic System of an Isoniazid Derivative**

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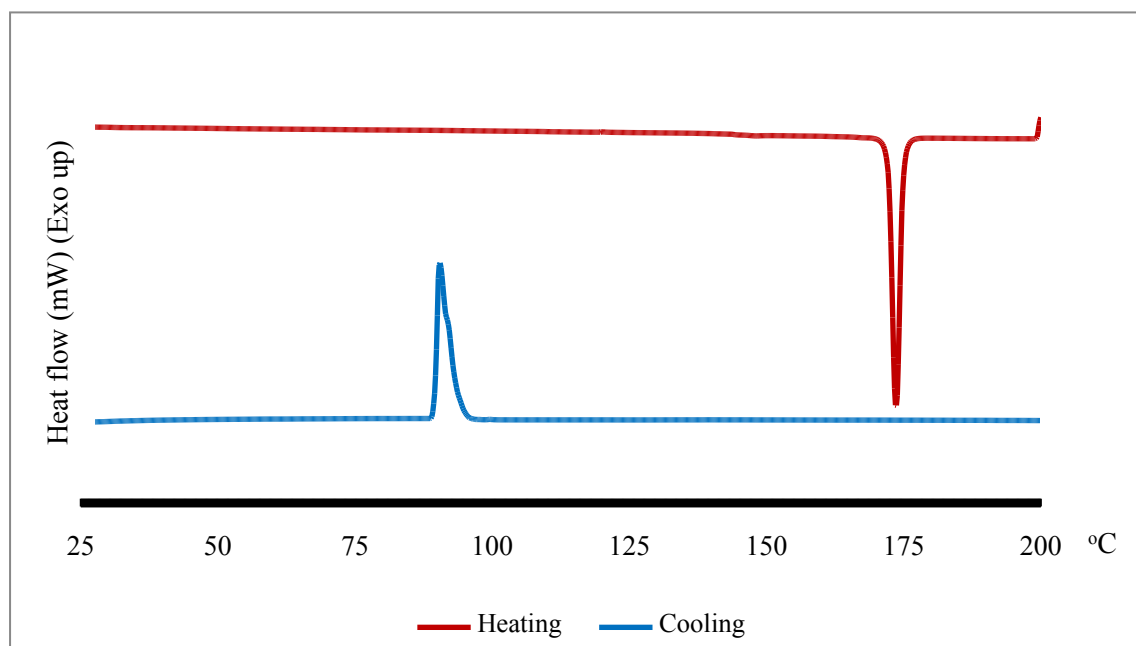
## SI1. Solution crystallization experimental details

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

IPH	Solvent system (10 ml)		
	Slow evaporation	Fast evaporation	Vapour diffusion (polar/non-polar)
II	acetonitrile	-	-
III	-	ethanol/ water (1:1)	-
V	acetone, butanol, ethanol, methanol, 1-propanol, water.  acetone/cyclohexane (1:1) butanol/cyclohexane (1:1) ethanol/cyclohexane (1:1) ethyl acetate/cyclohexane (1:1) methanol/cyclohexane (1:1)  acetone/diethyl ether (1:1) butanol/diethyl ether (1:1) ethanol/diethyl ether (1:1) ethyl acetate/diethyl ether (1:1) methanol/diethyl ether (1:1)  acetone/ <i>n</i> -hexane (1:1) butanol/ <i>n</i> -hexane (1:1) ethanol/ <i>n</i> -hexane (1:1) ethyl acetate/ <i>n</i> -hexane (1:1) methanol/ <i>n</i> -hexane (1:1)  acetone/1-propanol (1:1) butanol/1-propanol (1:1) butanone/1-propanol (1:1) ethanol/1-propanol (1:1) ethyl acetate/1-propanol (1:1) methanol/1-propanol (1:1)  acetone/toluene (1:1) butanol/toluene (1:1) ethanol/toluene (1:1) ethyl acetate/toluene (1:1) methanol/toluene (1:1)  acetone/tetrahydrofuran (1:1) butanol/tetrahydrofuran (1:1) ethanol/tetrahydrofuran (1:1) ethyl acetate/ tetrahydrofuran (1:1) methanol/ tetrahydrofuran (1:1)  chloroform/methanol (2:1) cyclohexane/methanol (2:1) diethyl ether/methanol (2:1) 1,4-dioxane/methanol (2:1) ethyl acetate/methanol (2:1) <i>n</i> -hexane/methanol (2:1) tetrahydrofuran/methanol (2:1)	ethanol/ water (1:1)	acetone/cyclohexane ethanol/cyclohexane methanol/cyclohexane  acetone/diethyl ether ethanol/diethyl ether methanol/diethyl ether  acetone/ <i>n</i> -hexane ethanol/ <i>n</i> -hexane methanol/ <i>n</i> -hexane  acetone/toluene ethanol/toluene methanol/toluene

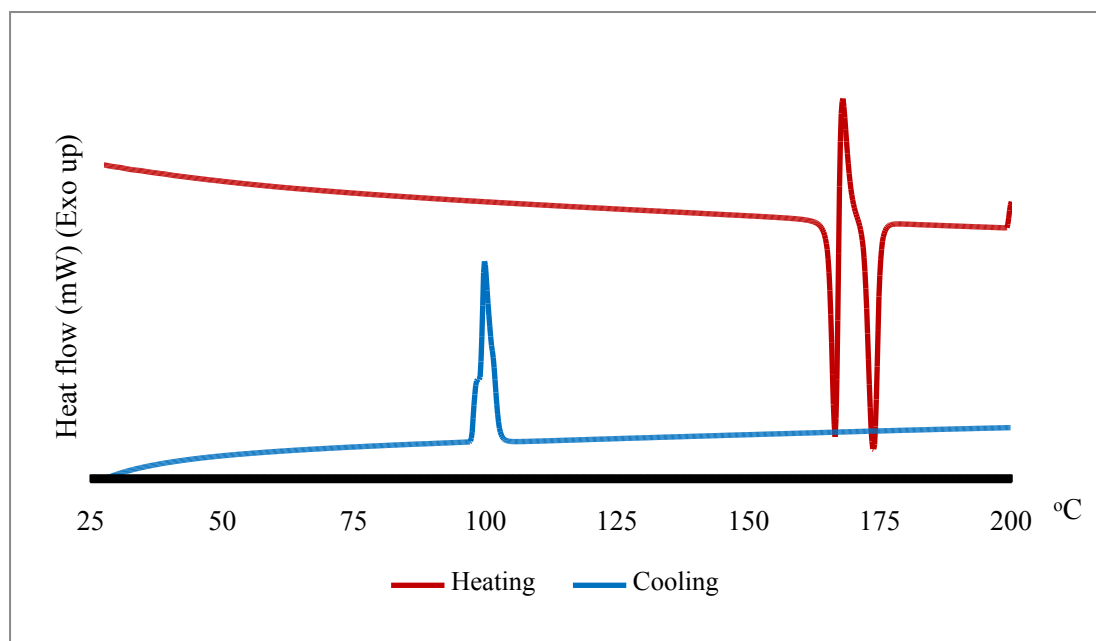
## SI2. Representative DSC traces for IPH I – III

### IPH I



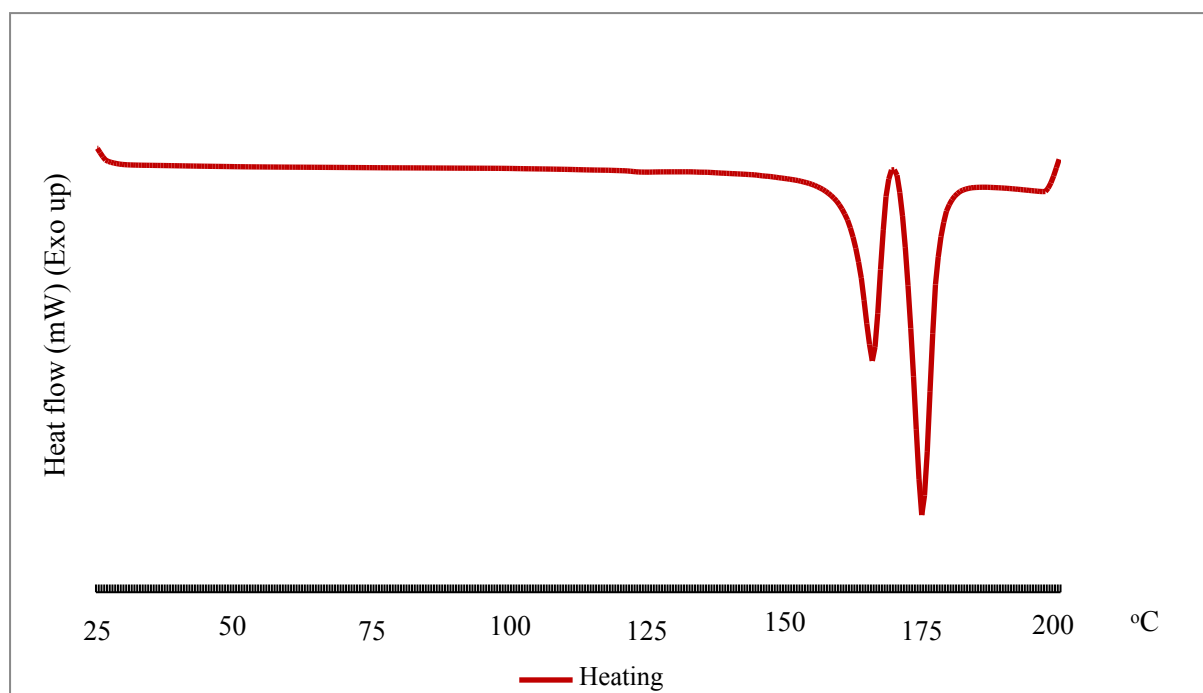
**Figure S1:** Representative DSC trace (exothermic is up) for numerous experiments for the melting and cool of IPH I. The top DSC trace (red) shows the melting endotherm of phase pure IPH I and the lower one (blue) the crystallization of this form from the melt on cooling.

### IPH II



**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.

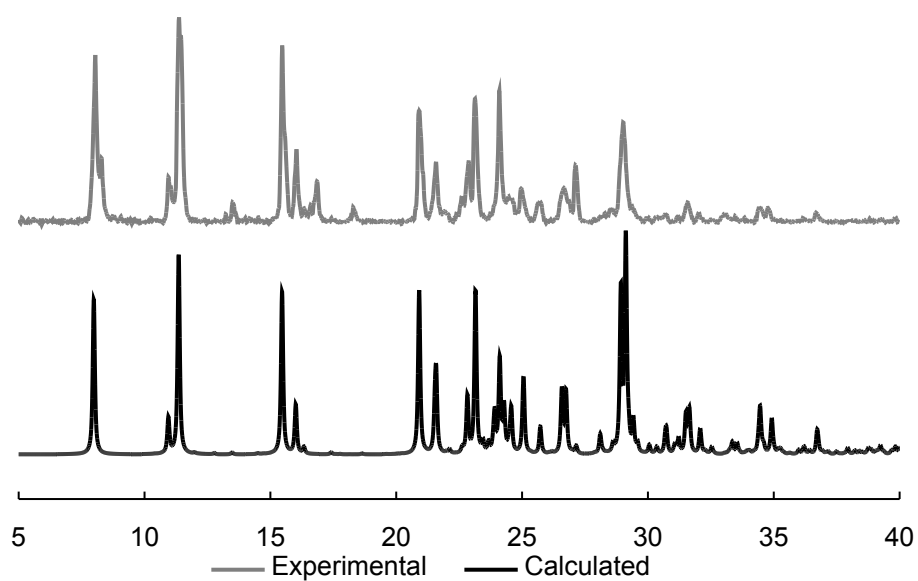
### IPH III



**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.

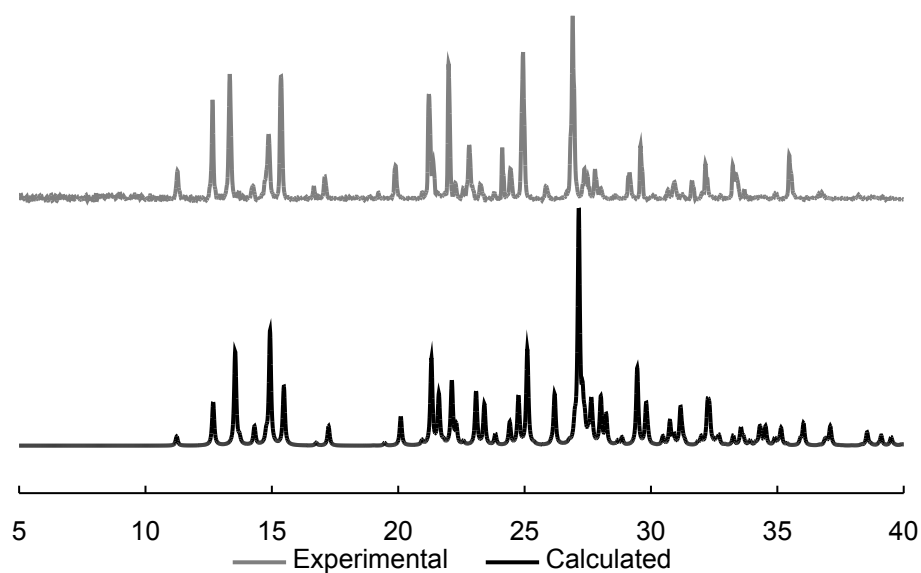
### SI3. Powder X-ray diffraction patterns for forms IPH I – III and V

#### IPH I



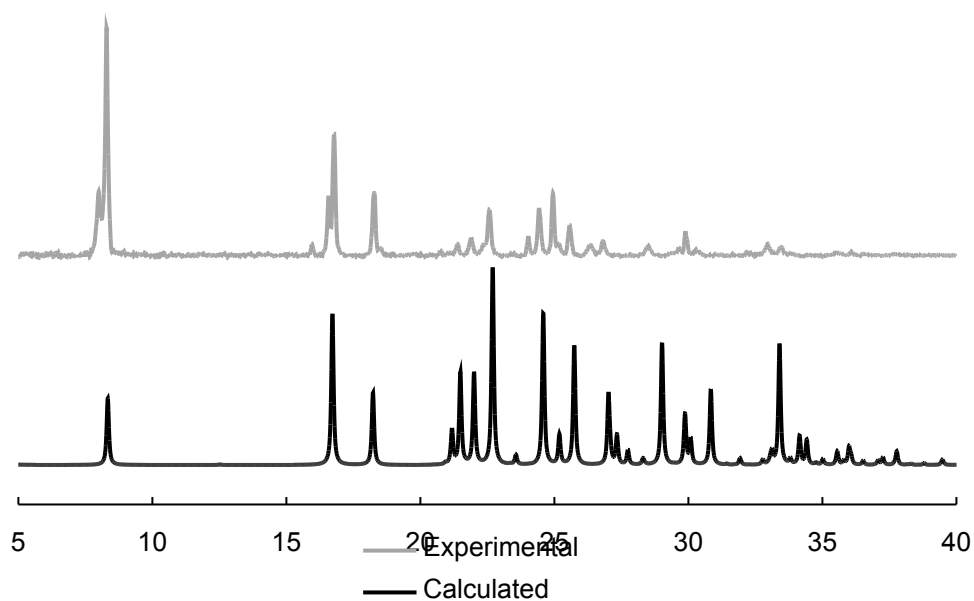
**Figure S4:** Experimental PXRD pattern of IPH I, recorded at room temperature, and the PXRD pattern calculated from single crystal structure data of IPH I (at 173 K).

#### IPH II



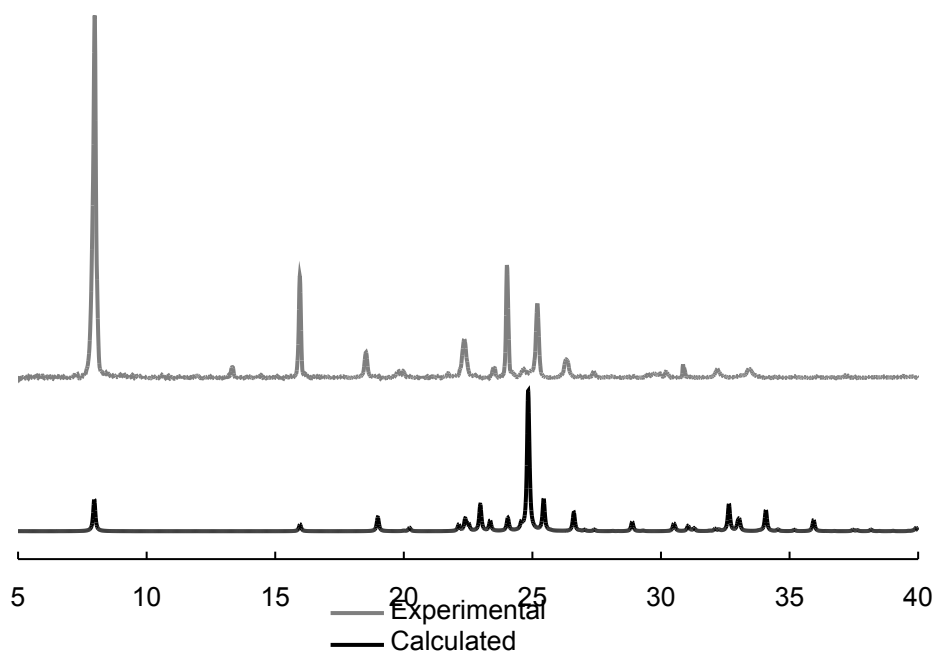
**Figure S5:** PXRD of IPH II compared to PXRD of single crystal structure determination of IPH II. The experimental pattern was calculated at room temperature and the calculated pattern at 173 K.

### IPH III



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

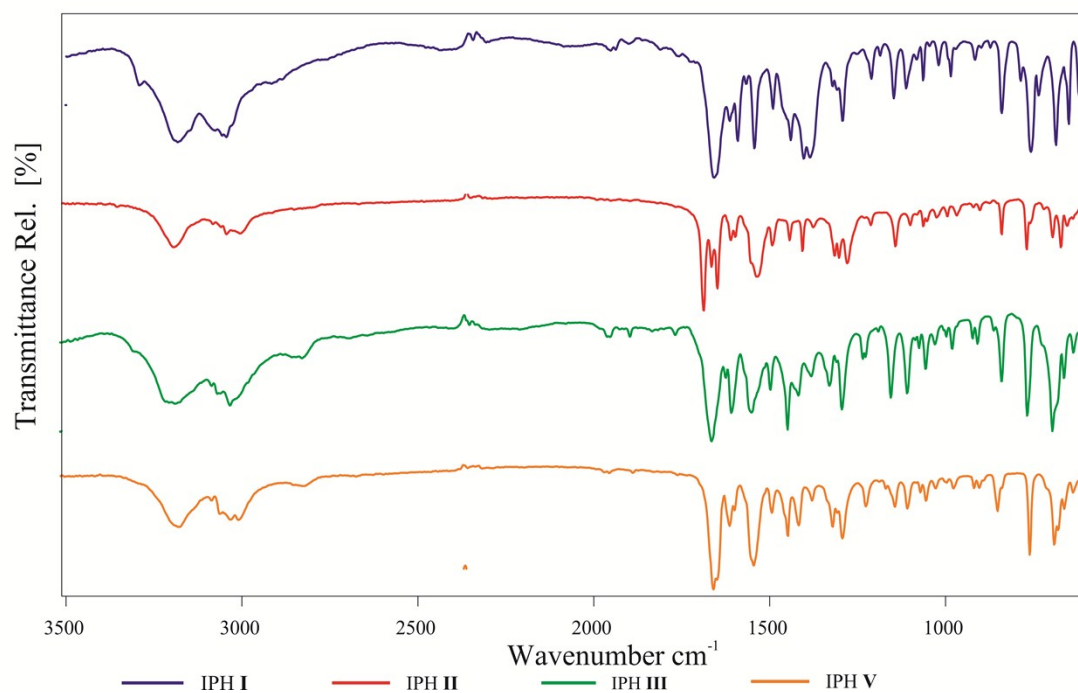
### IPH V



**Figure S7:** PXR D of IPH V compared to PXR D 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

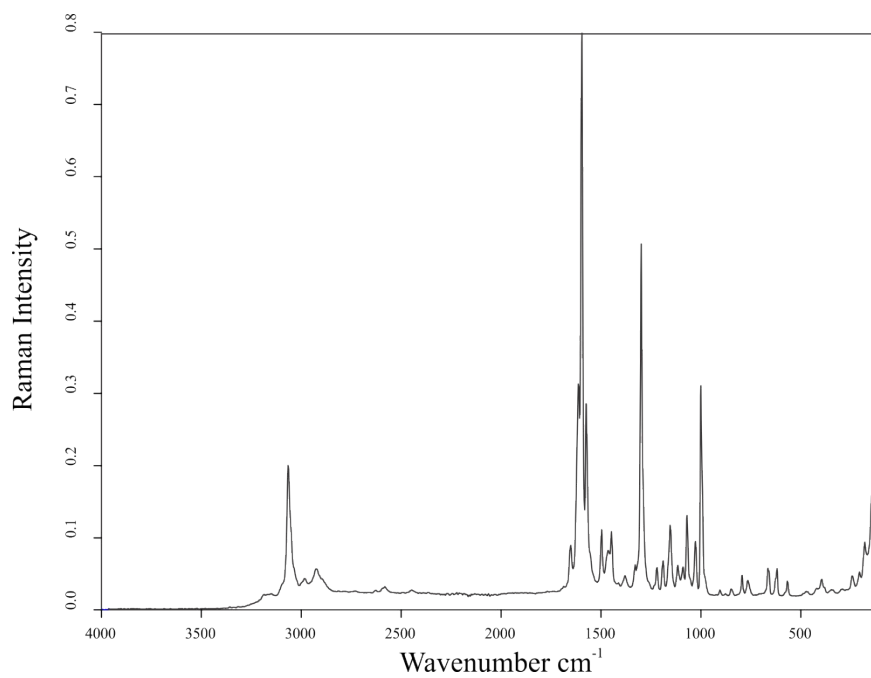
FT-IR spectra of IPH I – III and V



**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

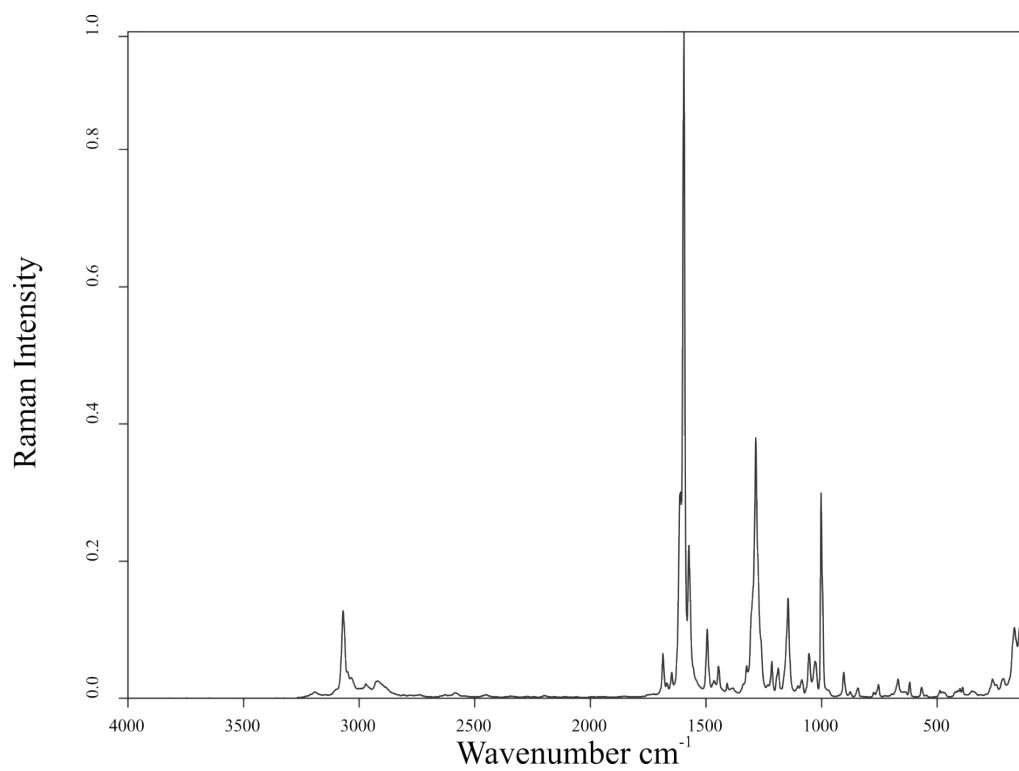
IPH I



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

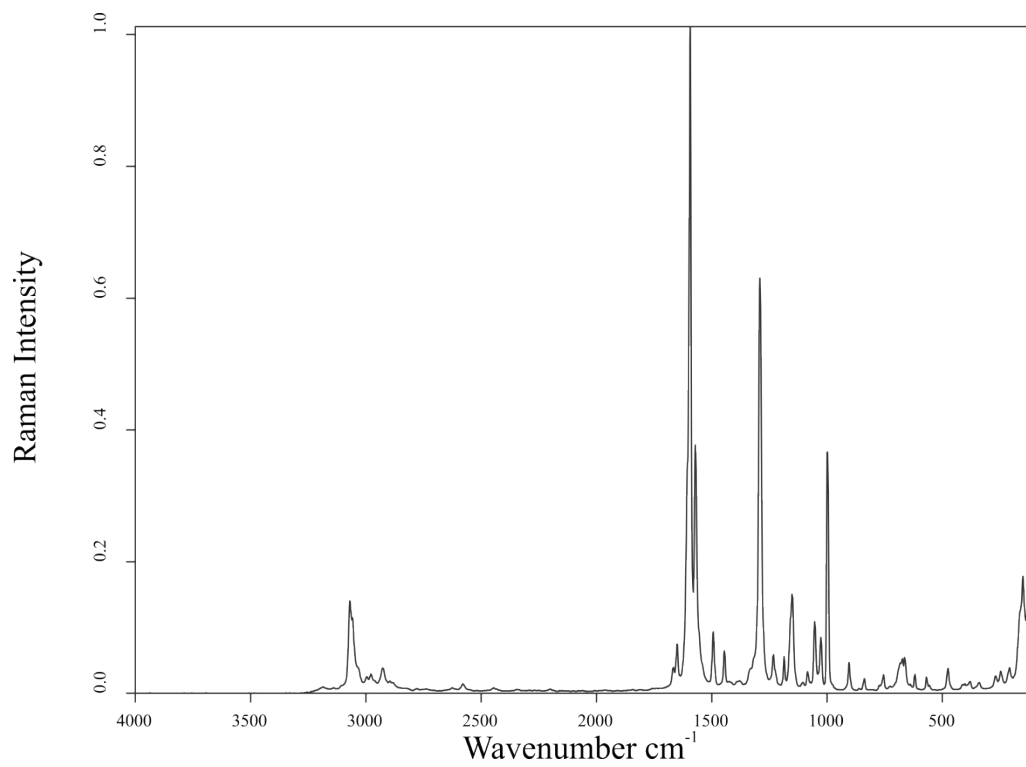


## IPH II



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

## IPH III



**Figure S11:** Raman spectrum of IPH III.

IPH V

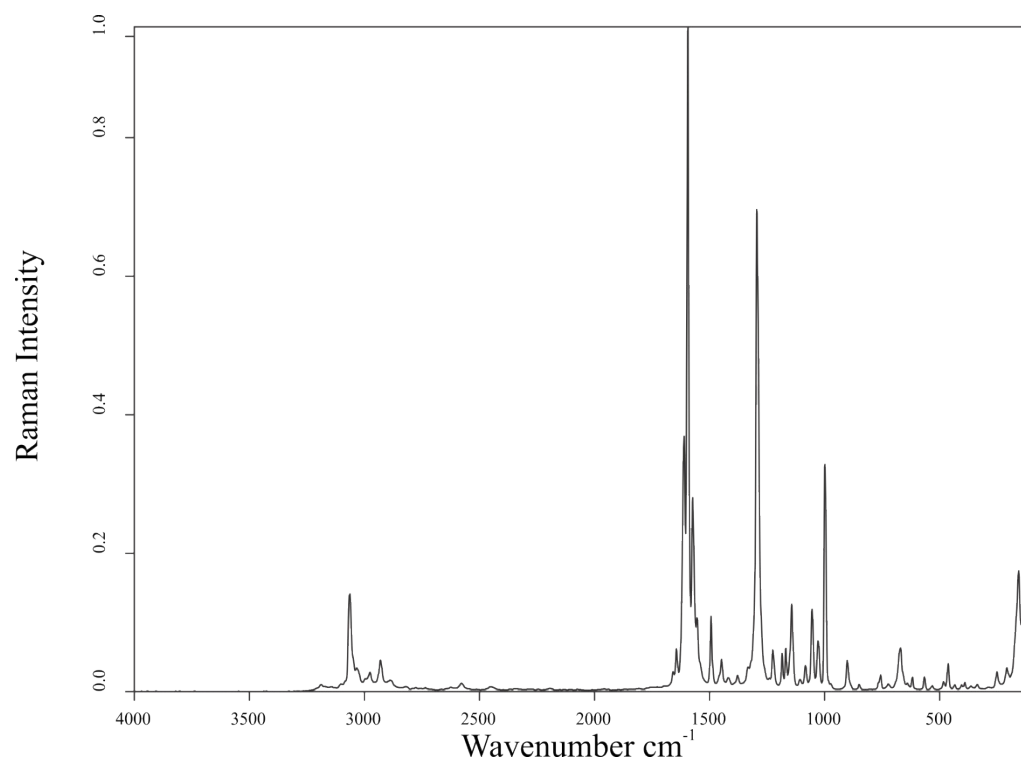


Figure S12: Raman spectrum of IPH V.

## SI5. *XPac* Studies

All comparisons were carried out with the program *XPac*.<sup>1</sup> Dissimilarity parameters were calculated in the previously described manner<sup>2</sup> (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

SC	supramolecular construct
$n$	in the representative molecular cluster of a crystal structure, the number of the surrounding molecules which together with the central molecule define a particular SC
$t_1, t_2$	basis vectors of an SC
$\angle t_{1,2}$	angle formed between two base vectors of an SC
$x_8, x_{18}$	<i>XPac</i> dissimilarity parameters calculated obtained with different sets of geometrical parameters obtained from different sets of atomic positions (see above)

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

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

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

Structure	IPH III		IPH V		AHE		PEH	
$t_1$	100	6.354 Å	010	5.546 Å	010	5.784 Å	010	5.594 Å
$t_2$	010	7.662 Å	001	8.319 Å	100	8.594 Å	100	8.224 Å
$\angle t_{1,2}$	90°		90°		90°		90°	

**Table S4.** Corresponding lattice parameters for SC Y1.

Structure	IPH II		IPH IV	
$t_1$	001	8.235 Å	00 $\bar{1}$	8.259 Å
$t_2$	100	10.211 Å	$\bar{1}00$	10.612 Å
$\angle t_{1,2}$	110.2°		109.6°	

**Table S5.** *XPac* dissimilarity parameters  $x_\delta$  and  $x_{I\delta}$  for the identified SCs.

Structure 1	Structure 2	Dim	SC	$n$	$x_\delta$	$x_{I\delta}$
<b>AHE</b>	<b>IPH III</b>	2D	<b>X1</b>	8	11.7	14.2
<b>AHE</b>	<b>IPH V</b>	2D	<b>X1</b>	8	3.0	5.0
<b>AHE</b>	<b>PEH</b>	2D	<b>X1</b>	8	3.3	3.8
<b>IPH III</b>	<b>IPH V</b>	2D	<b>X1</b>	8	10.7	13.6
<b>IPH III</b>	<b>PEH</b>	2D	<b>X1</b>	8	9.7	12.6
<b>IPH V</b>	<b>PEH</b>	2D	<b>X2</b>	12	1.1	2.2
<b>IPH II</b>	<b>IPH IV</b>	2D	<b>Y1</b>	8	8.7	15.0
<b>IPH II</b>	<b>IPH VI</b>	0D	<b>D</b>	1	5.8	8.2

## SI6. ORTEP diagrams and hydrogen bonding tables for IPH I – VI

### IPH I

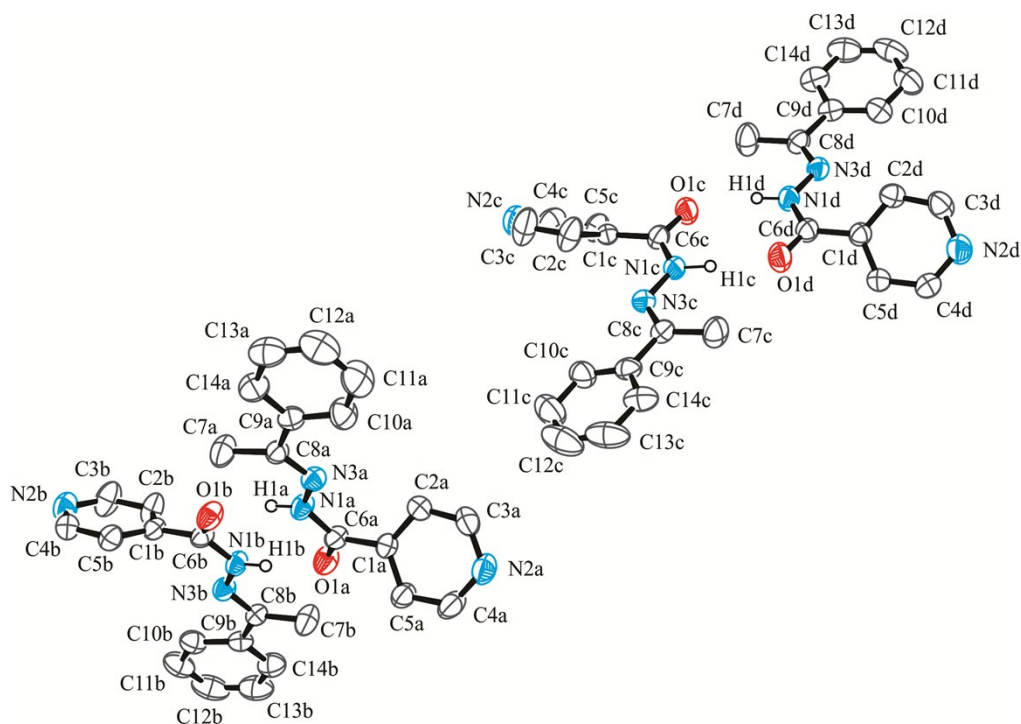


Figure S13: The asymmetric unit of IPH I depicting the numbering scheme of 50% displacement ellipsoids.

### IPH II

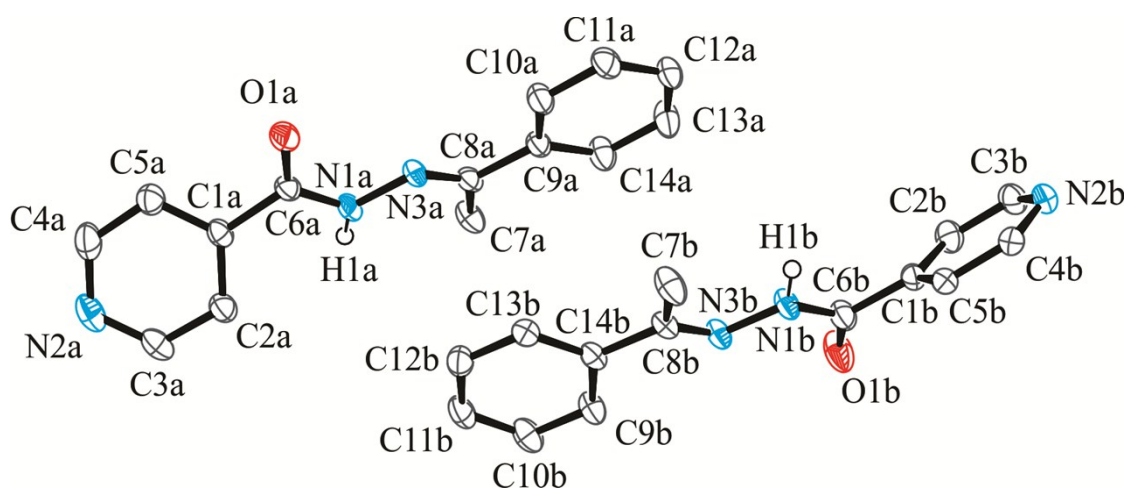
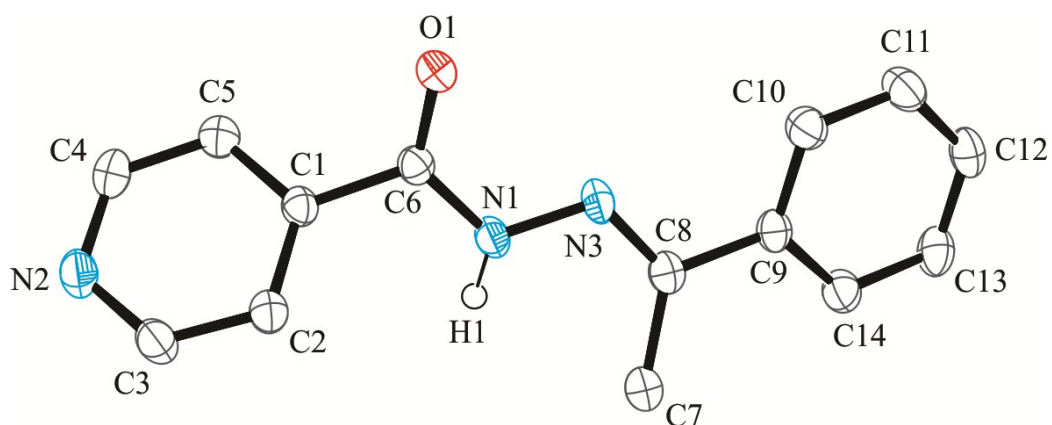


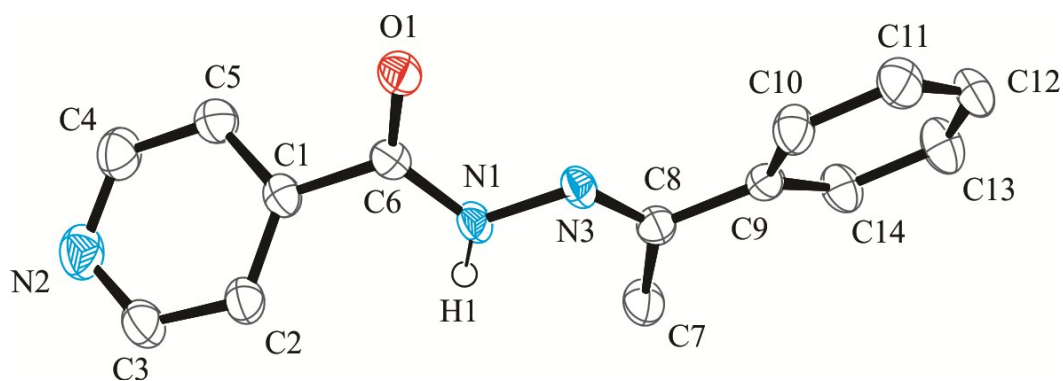
Figure S14: The asymmetric unit of IPH II depicting the numbering scheme of 50% displacement ellipsoids.

### IPH III



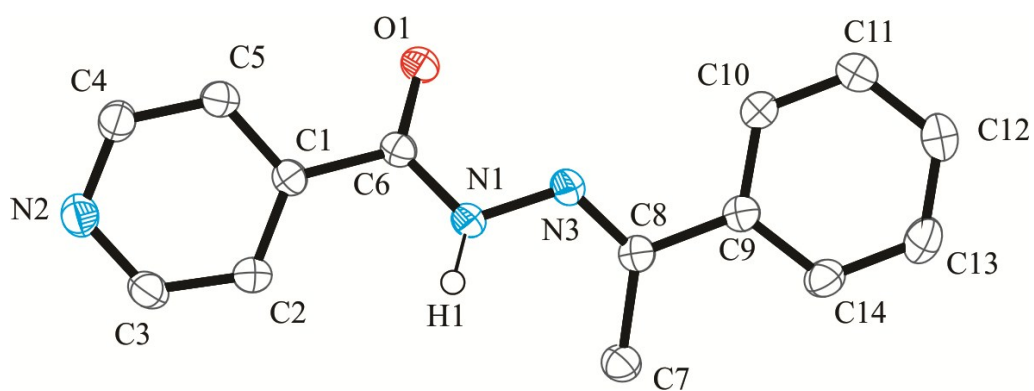
**Figure S15:** The asymmetric unit of IPH III depicting the numbering scheme of 50% displacement ellipsoids.

### IPH IV



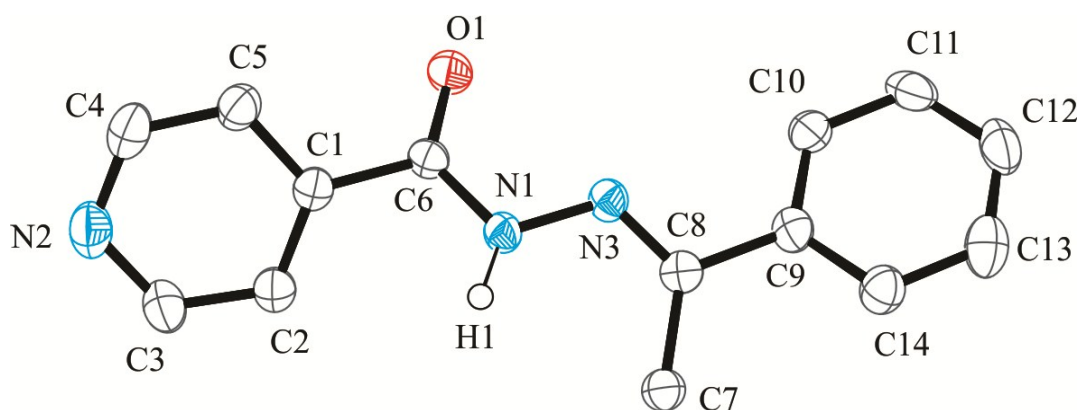
**Figure S16:** The asymmetric unit of IPH IV depicting the numbering scheme of 50% displacement ellipsoids.

### IPH V



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

## IPH VI



**Figure S18:** 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

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1B)-H(1B)...O(1A)#1	0.90(3)	2.04(3)	2.934(3)	169(3)
N(1C)-H(1C)...O(1D)#2	1.00(3)	1.96(3)	2.937(3)	165(3)
N(1D)-H(1D)...O(1C)#3	0.89(3)	2.03(3)	2.903(3)	166(3)
N(1A)-H(1A)...O(1B)#4	0.94(3)	2.00(3)	2.926(3)	170(2)

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

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1A)-H(1A)...O(1A)#1	0.911(18)	2.068(18)	2.9748(14)	173.3(15)
N(1B)-H(1B)...N(2B)#2	0.866(18)	2.245(18)	3.0930(15)	166.4(15)

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

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(1)#1	0.89(2)	2.04(2)	2.911(2)	168(2)

Symmetry transformations used to generate equivalent atoms:

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

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

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(1)#1	0.89(3)	2.17(4)	3.032(3)	162(3)

Symmetry transformations used to generate equivalent atoms:

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

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

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(1)#1	0.90(3)	1.95(3)	2.845(2)	176(2)

Symmetry transformations used to generate equivalent atoms:

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

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

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(1)#1	0.94(3)	1.95(3)	2.887(3)	175(3)

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

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No syntax errors found.      CIF dictionary      Interpreting this report

### Datablock: IPH\_I

---

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

Cell:                a=9.7360(6)            b=9.8752(6)            c=26.1543(16)  
                      alpha=92.856(4)      beta=100.295(4)      gamma=91.291(4)

Temperature:    173 K

	Calculated	Reported
Volume	2469.8(3)	2469.8(3)
Space group	P -1	P-1
Hall group	-P 1	-P 1
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	1.287	1.287
Z	8	8
Mu (mm-1)	0.084	0.084
F000	1008.0	1008.0
F000'	1008.37	
h, k, lmax	11, 11, 31	11, 11, 31
Nref	9178	9177
Tmin, Tmax	0.994, 0.998	
Tmin'	0.967	

Correction method= Not given

Data completeness= 1.000                      Theta(max)= 25.500

R(reflections)= 0.0559( 4183)                wR2(reflections)= 0.1600( 9177)

S = 0.995    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**

PLAT026\_ALERT\_3\_C Ratio Observed / Unique Reflections too Low .... 46 %  
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.  
PLAT414\_ALERT\_2\_C Short Intra D-H..H-X H1B .. H7B1 .. 1.96 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 ..... 0.00400 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. # 2 Note  
C14 H13 N3 O  
PLAT790\_ALERT\_4\_G Centre of Gravity not Within Unit Cell: Resd. # 4 Note  
C14 H13 N3 O  
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_PLAT026_IPH_I
;
PROBLEM: Ratio Observed / Unique Reflections too Low .... 46 %
RESPONSE: ...
;
_vrf_PLAT242_IPH_I
;
PROBLEM: Low Ueq as Compared to Neighbors for ..... C1C Check
RESPONSE: ...
;
_vrf_PLAT340_IPH_I
;
PROBLEM: Low Bond Precision on C-C Bonds ..... 0.0044 Ang.
RESPONSE: ...
;
_vrf_PLAT353_IPH_I
;
PROBLEM: Long N-H (N0.87,N1.01A) N1C - H1C ... 1.02 Ang.
RESPONSE: ...
```

```
;
_vrf_PLAT414_IPH_I
;
PROBLEM: Short Intra D-H..H-X      H1A   ..  H7A3   ..      1.91 Ang.
RESPONSE: ...
;
_vrf_PLAT906_IPH_I
;
PROBLEM: Large K value in the Analysis of Variance ..... 5.956 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**

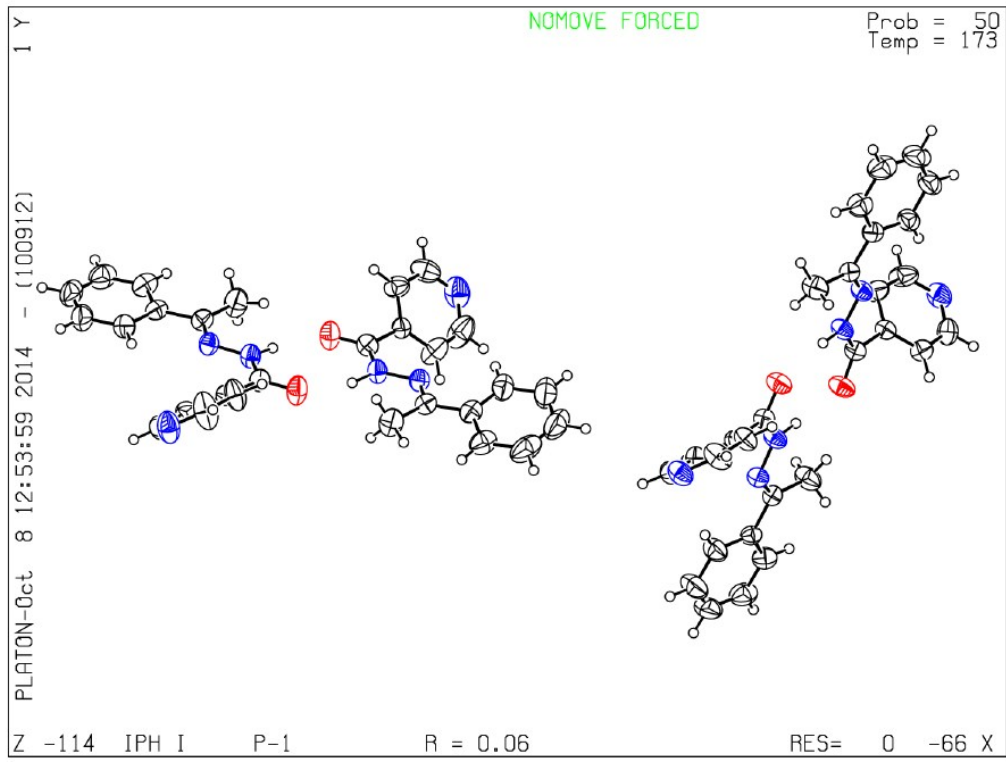
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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**





## IPH II

### checkCIF/PLATON report

Structure factors have been supplied for datablock(s) IPH\_II

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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		
	Calculated	Reported	
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	
Tmin'	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 # FCF Refl Between THmin & 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 # FCF Refl Between THmin & 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

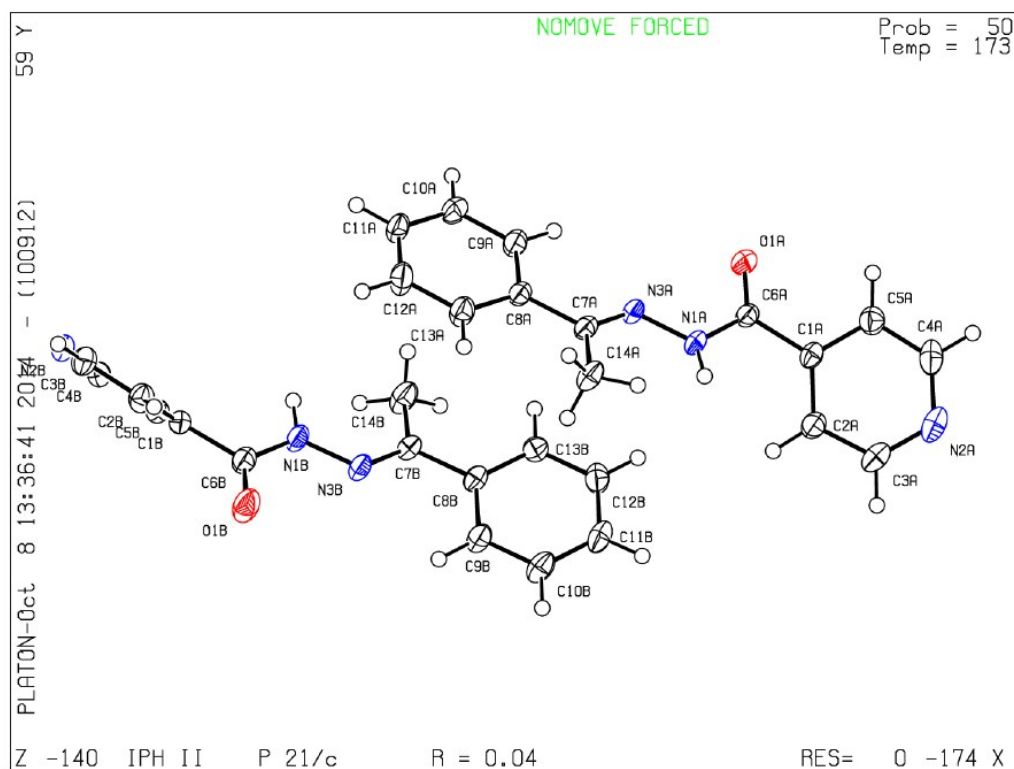
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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

Datablock IPH\_II - ellipsoid plot



## IPH III

### 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

---

Bond precision:    C-C = 0.0028 A                      Wavelength=0.71073  
Cell:                      a=6.3542(4)              b=7.6624(6)              c=49.231(3)  
                                    alpha=90              beta=90              gamma=90  
Temperature:              173 K

	Calculated	Reported
Volume	2397.0(3)	2397.0(3)
Space group	P b c a	Pbca
Hall group	-P 2ac 2ab	-P 2ac 2ab
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	1.326	1.326
Z	8	8
Mu (mm-1)	0.087	0.087
F000	1008.0	1008.0
F000'	1008.37	
h,k,lmax	7,9,59	7,9,59
Nref	2222	2124
Tmin,Tmax	0.989,0.996	0.964,0.996
Tmin'	0.964	

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 \_diffn\_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 THmin & STh/L= 0.600 80 Report  
PLAT918\_ALERT\_3\_C Reflection(s) with I(obs) much smaller I(calc) . 1 Check  
PLAT934\_ALERT\_3\_C Number of (Iobs-Icalc)/SigmaW > 10 Outliers .... 1 Check  
PLAT939\_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  
PLAT955\_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 THmin & 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_PLAT934_IPH_III
;
PROBLEM: Number of (Iobs-Icalc)/SigmaW > 10 Outliers .... 1 Check
RESPONSE: ...
;
_vrf_PLAT939_IPH_III
;
```

PROBLEM: Large Value of Not (SHELXL) Weight Optimized S . 38.26  
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**

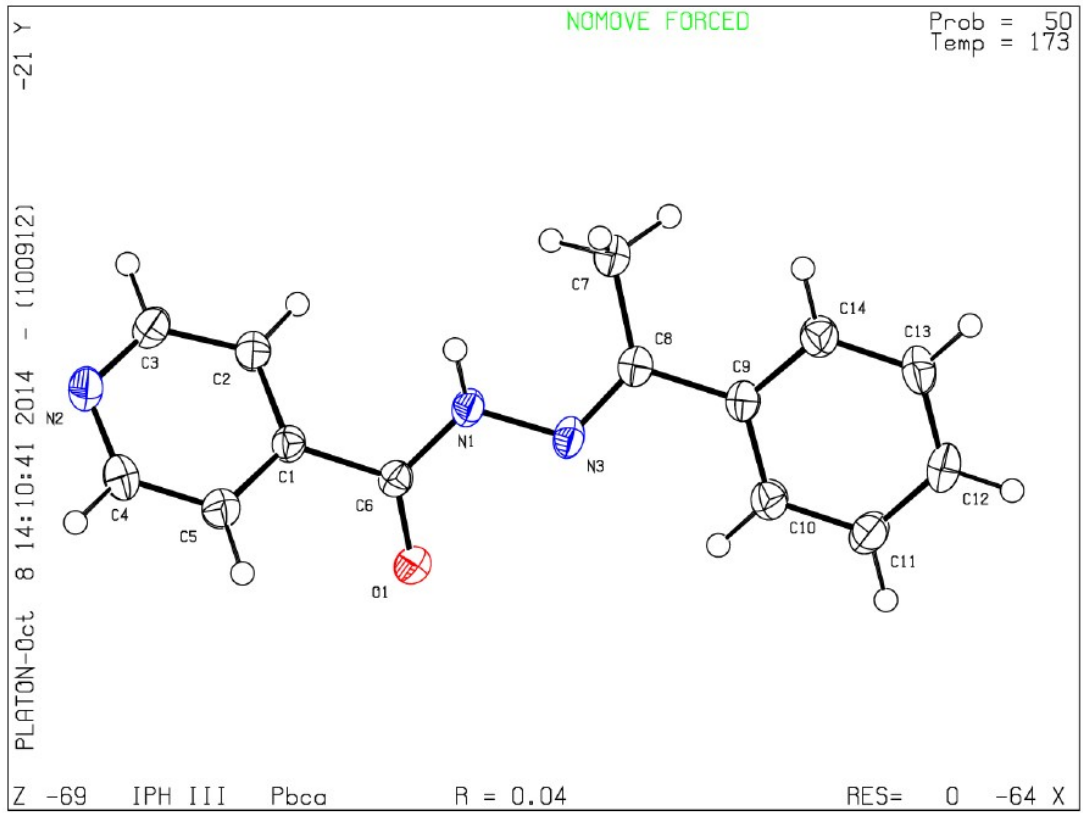
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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**



## IPH IV

### 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 A                      Wavelength=0.71073

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

Temperature:            173 K

	Calculated	Reported
Volume	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 sine(theta\_max)/wavelength is less than 0.575  
                   Calculated sin(theta\_max)/wavelength =     0.5568  
 PLAT414\_ALERT\_2\_B Short Intra D-H..H-X            H1     .. H7A     ..        1.81 Ang.

**Alert level C**  
 PLAT340\_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 # FCF Refl Between THmin & 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 FCF 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.

```
# 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 # FCF Refl Between THmin & STh/L= 0.557        4 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.

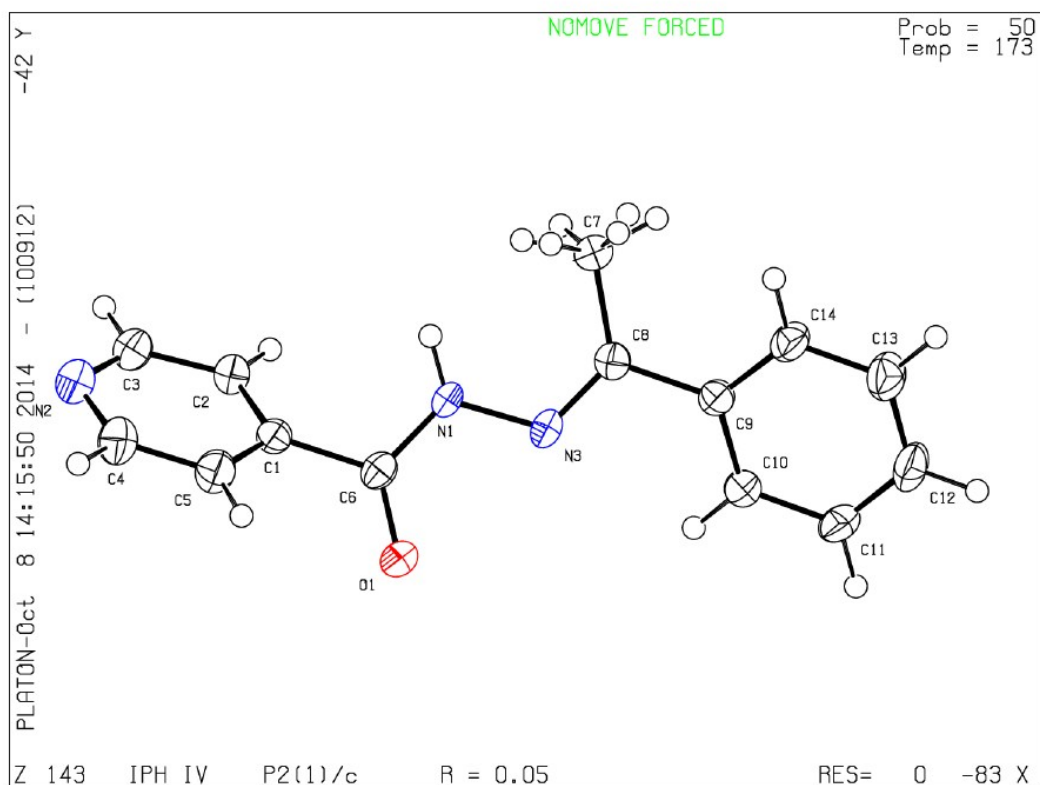
#### Publication of your CIF in other journals

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

---

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Datablock IPH\_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**

Bond precision: C-C = 0.0030 A Wavelength=0.71073  
 Cell: a=25.8998 (13) b=5.5463 (3) c=8.3187 (4)  
 alpha=90 beta=95.876 (4) gamma=90  
 Temperature: 173 K

	Calculated	Reported
Volume	1188.69 (11)	1188.69 (10)
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	1.337	1.337
Z	4	4
Mu (mm-1)	0.088	0.088
F000	504.0	504.0
F000'	504.19	
h,k,lmax	34,7,10	34,7,10
Nref	2877	2866
Tmin,Tmax	0.979,0.995	0.962,0.995
Tmin'	0.961	

Correction method= MULTI-SCAN

Data completeness= 0.996 Theta(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 # FCF Refl Between THmin & STh/L= 0.600 9 Report

---

● **Alert level G**  
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  
0 **ALERT level B** = A potentially serious problem, consider carefully  
2 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight  
1 **ALERT level G** = General information/check it is not something unexpected

0 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  
3 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.

```
# 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 # FCF 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.

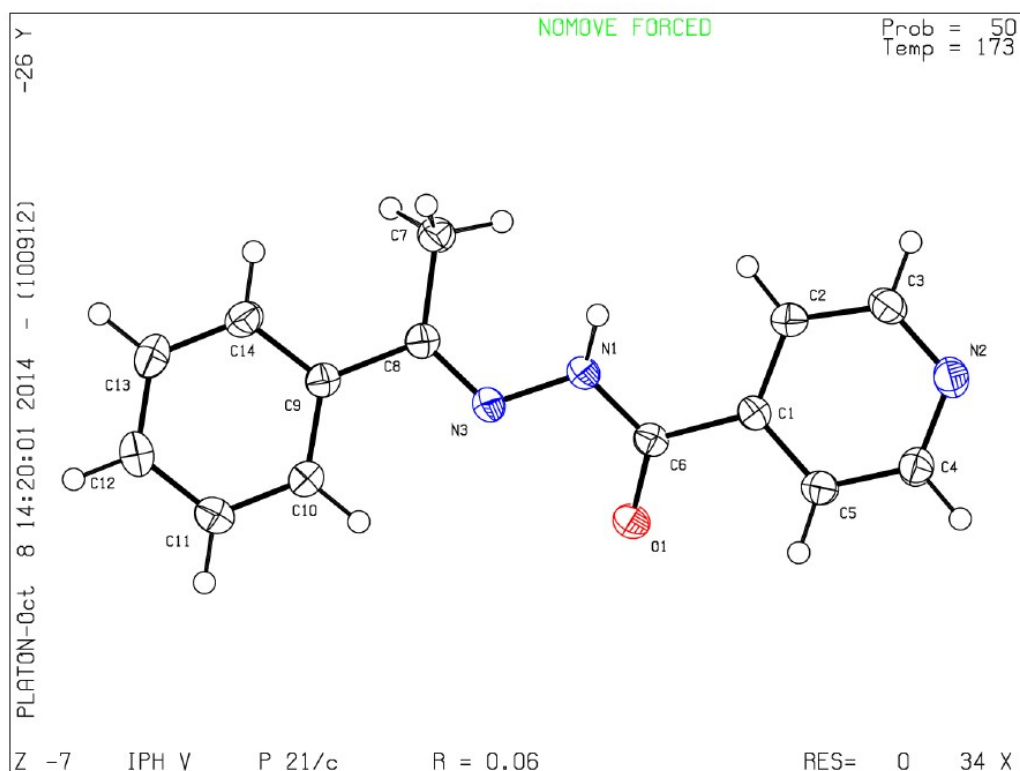
### Publication of your CIF in other journals

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

Datablock IPH\_V - ellipsoid plot



## IPH VI

### 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 A                      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**

PLAT911_ALERT_3_C	Missing # FCF Refl Between THmin & STh/L= 0.600	33	Report
PLAT913_ALERT_3_C	Missing # of Very Strong Reflections in FCF ....	5	Note
PLAT918_ALERT_3_C	Reflection(s) with I(obs) much smaller I(calc) .	1	Check
PLAT934_ALERT_3_C	Number of (Iobs-Icalc)/SigmaW > 10 Outliers ....	1	Check
PLAT939_ALERT_3_C	Large Value of Not (SHELXL) Weight Optimized S .	37.29	

---

● **Alert level G**

PLAT066_ALERT_1_G	Predicted and Reported Tmin&Tmax Range Identical	?	Check
PLAT072_ALERT_2_G	SHELXL First Parameter in WGHT Unusually Large.	0.14	Report
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	4	Note

---

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 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_PLAT911_IPH_VI
;
PROBLEM: Missing # FCF Refl Between THmin & STh/L= 0.600          33 Report
RESPONSE: ...
;
_vrf_PLAT913_IPH_VI
;
PROBLEM: Missing # of Very Strong Reflections in FCF ....          5 Note
RESPONSE: ...
;
_vrf_PLAT918_IPH_VI
;
PROBLEM: Reflection(s) with I(obs) much smaller I(calc) .          1 Check
RESPONSE: ...
;
_vrf_PLAT934_IPH_VI
;
PROBLEM: Number of (Iobs-Icalc)/SigmaW > 10 Outliers ....          1 Check
RESPONSE: ...
;
_vrf_PLAT939_IPH_VI
;
PROBLEM: Large Value of Not (SHELXL) Weight Optimized S .          37.29
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**

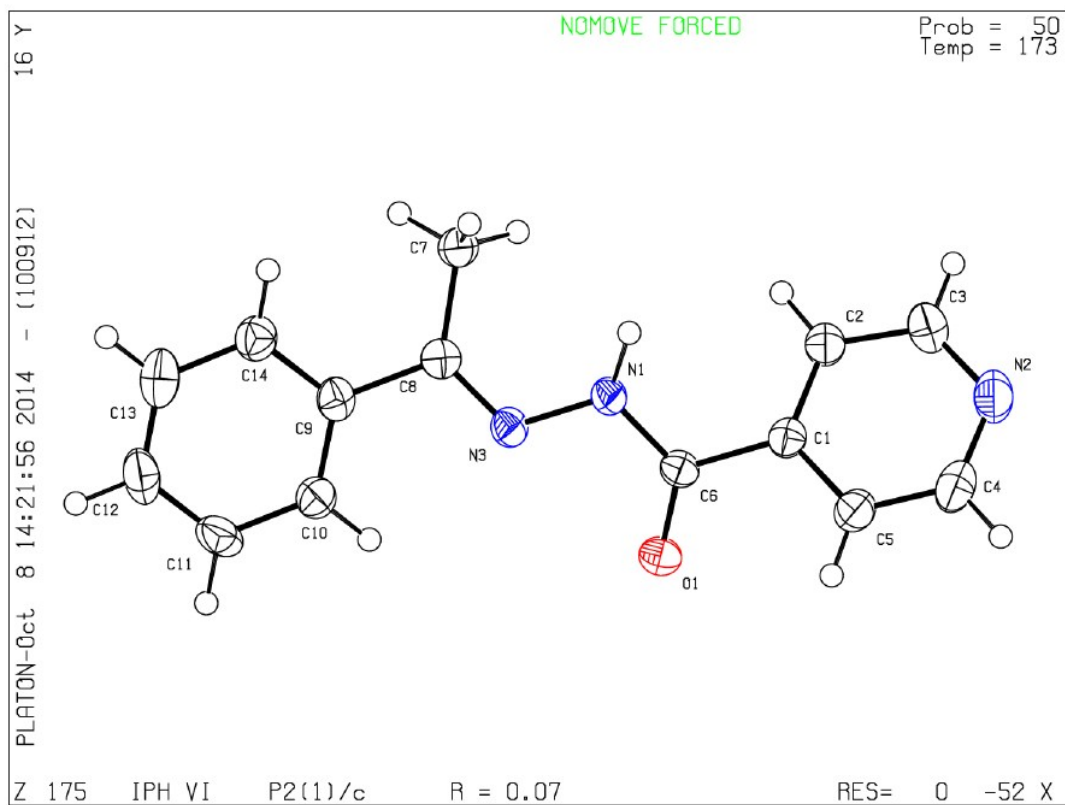
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section 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**



## References

1. T. Gelbrich and M. B. Hursthouse, *CrystEngComm*, 2005, **7**, 324-336.
2. T. Gelbrich, T. L. Threlfall and M. B. Hursthouse, *CrystEngComm*, 2012, **14**, 5454-5464.
3. T. Gelbrich, T. L. Threlfall and M. B. Hursthouse, *Acta Crystallogr., Sect. C: Cryst. Struct. Commun.*, 2012, **68**, o421-o426.