

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

(2-Chloro-8-methoxyquinolin-3-yl)-methanol monohydrate

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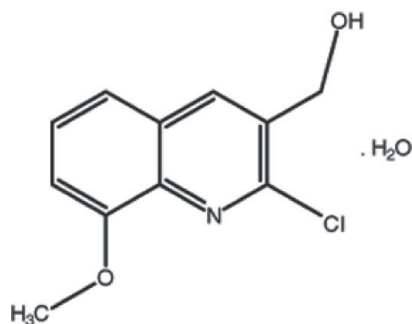
Received 25 May 2010; accepted 29 May 2010

Key indicators: single-crystal X-ray study; $T = 290$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.054; wR factor = 0.134; data-to-parameter ratio = 13.4.

In the title compound, $\text{C}_{11}\text{H}_{10}\text{ClNO}_2 \cdot \text{H}_2\text{O}$, the organic molecule is roughly planar (r.m.s. deviation = 0.074 Å). In the crystal structure, molecules are linked by $\text{O}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{N}$ hydrogen bonds and weak $\text{C}-\text{H} \cdots \pi$ and $\pi-\pi$ interactions [centroid-centroid distance = 3.578 (3) Å] consolidate the packing. A short $\text{Cl} \cdots \text{O}$ contact [3.147 (3) Å] is also observed.

Related literature

For further information on the starting material, see: Subashini *et al.* (2009). For general background to the title compound, see: Roopan *et al.* (2009). For related structures, see: Khan *et al.* (2010a,b). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_{11}\text{H}_{10}\text{ClNO}_2 \cdot \text{H}_2\text{O}$
 $M_r = 241.67$
Monoclinic, $P2_1/n$
 $a = 9.161$ (5) Å

$b = 14.246$ (5) Å
 $c = 9.464$ (5) Å
 $\beta = 116.819$ (5)°
 $V = 1102.3$ (9) Å³

$Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.34$ mm⁻¹

$T = 290$ K
 $0.31 \times 0.21 \times 0.10$ mm

Data collection

Oxford Xcalibur Eos (Nova) CCD detector diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
 $T_{\min} = 0.903$, $T_{\max} = 0.967$

8360 measured reflections
2044 independent reflections
1212 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.100$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.134$
 $S = 0.90$
2044 reflections
153 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.40$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.26$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the N1/C1–C3/C8/C9 ring.

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O1}-\text{H1O} \cdots \text{O3}^{\text{i}}$	0.82	1.90	2.705 (4)	165
$\text{O3}-\text{H1W} \cdots \text{N1}^{\text{ii}}$	0.85 (5)	2.17 (5)	2.988 (4)	163 (4)
$\text{O3}-\text{H2W} \cdots \text{O1}^{\text{iii}}$	0.83 (4)	2.02 (4)	2.836 (4)	171 (5)
$\text{C10}-\text{H10B} \cdots \text{Cg1}^{\text{i}}$	0.97	2.93	3.738 (5)	142

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

Data collection: *CrysAlis PRO CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO CCD*; data reduction: *CrysAlis PRO RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We thank the Department of Science and Technology, India, for use of the CCD facility set up under the FIST-DST program at SSCU, IISc. We also thank Professor T. N. Guru Row, IISc, Bangalore, for his help with the data collection. FNK thanks the DST for Fast Track Proposal funding.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5469).

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supplementary materials

Acta Cryst. (2010). E66, o1542 [doi:10.1107/S1600536810020489]

(2-Chloro-8-methoxyquinolin-3-yl)methanol monohydrate

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Comment

The importance and general background of the title compound is given in our earlier paper (Roopan *et al.*, 2009).

In the main molecule of the title compound (I), (Fig. 1), all the non-H atoms are roughly coplanar (r.m.s. deviation = 0.074 Å). The bond lengths and angles are comparable to the similar structures 2-chloro-3-hydroxymethyl-7,8-dimethylquinoline and 2-chloro-3-hydroxymethyl-6-methoxyquinoline (Khan *et al.*, 2010a,b), and also those in literature (Allen *et al.*, 1987).

The crystal structure is stabilized by intermolecular O—H...O and O—H...N interactions between the symmetry-related molecules (Table 1, Fig. 2). Adjacent molecules are stacked along the *b* axis through weak C—H... π interactions (Table 1) and π - π interactions [$Cg1 \cdots Cg2(-x, 1 - y, -z) = 3.578(3)$ Å, where *Cg1* and *Cg2* are centroids of the N1/C1–C3/C8/C9 and C4–C9 rings, respectively]. In addition a short C11...O2 contact of 3.15 Å is also observed.

Experimental

2-Chloro-8-methoxyquinoline-3-carbaldehyde (222 mg, 1 mmol), sodium borohydride (38 mg, 1 mmol) and catalytic amount of montmorillonite K-10 were taken in an open vessel and the resulting mixture was irradiated at 500 W for 5 min. Ethylacetate was poured into the reaction mixture and filtered off. The filtrate after removal of solvent was subjected to column chromatography packed with silica and ethyl acetate/petroleum ether was used as the eluant. Colourless slabs of (I) were grown by solvent evaporation from a solution of the compound in chloroform.

Refinement

The H atoms of the water molecule were located in difference map and its positional parameters were refined freely [O3—H1W = 0.85 (5) and O3—H2W = 0.83 (4) Å]. The remaining H atoms were positioned geometrically, with O—H = 0.82 Å (for OH) and C—H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H, respectively, and refined as riding with $U_{iso}(H) = 1.2$ or $1.5 U_{eq}(O, C)$.

Figures

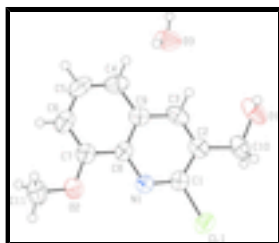


Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids.

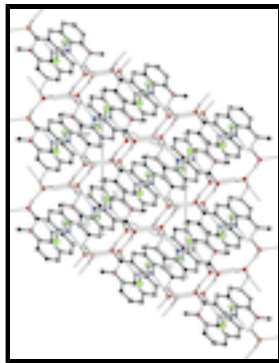


Fig. 2. Molecular packing of (I) with hydrogen bonding shown as dashed lines. The H atoms not involved in hydrogen bonds have been omitted for clarity.

(2-Chloro-8-methoxyquinolin-3-yl)methanol monohydrate

Crystal data

$C_{11}H_{10}ClNO_2 \cdot H_2O$

$M_r = 241.67$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 9.161\ (5)\ \text{\AA}$

$b = 14.246\ (5)\ \text{\AA}$

$c = 9.464\ (5)\ \text{\AA}$

$\beta = 116.819\ (5)^\circ$

$V = 1102.3\ (9)\ \text{\AA}^3$

$Z = 4$

$F(000) = 504$

$D_x = 1.456\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1021 reflections

$\theta = 1.9\text{--}20.2^\circ$

$\mu = 0.34\ \text{mm}^{-1}$

$T = 290\ \text{K}$

Slab, colourless

$0.31 \times 0.21 \times 0.10\ \text{mm}$

Data collection

Oxford Xcalibur Eos (Nova) CCD detector diffractometer

Radiation source: Enhance (Mo) X-ray Source graphite

ω scans

Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)

$T_{\min} = 0.903$, $T_{\max} = 0.967$

8360 measured reflections

2044 independent reflections

1212 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.100$

$\theta_{\max} = 25.5^\circ$, $\theta_{\min} = 2.9^\circ$

$h = -11 \rightarrow 11$

$k = -17 \rightarrow 17$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.134$

$S = 0.90$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0692P)^2]$

2044 reflections

153 parameters

0 restraints

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.40 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.26 \text{ e } \text{\AA}^{-3}$$

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating $-R$ -factor-obs *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	-0.12994 (10)	0.21700 (5)	-0.05478 (9)	0.0525 (3)
O1	0.0858 (3)	0.2656 (2)	-0.3822 (3)	0.0713 (10)
O2	0.1487 (3)	0.46372 (14)	0.3536 (2)	0.0515 (8)
N1	0.0521 (3)	0.35801 (15)	0.0948 (3)	0.0371 (8)
C1	0.0114 (4)	0.30580 (19)	-0.0297 (3)	0.0399 (10)
C2	0.0664 (4)	0.3140 (2)	-0.1453 (3)	0.0435 (10)
C3	0.1715 (4)	0.3870 (2)	-0.1243 (3)	0.0493 (11)
C4	0.3261 (4)	0.5254 (2)	0.0313 (4)	0.0558 (12)
C5	0.3720 (4)	0.5792 (2)	0.1620 (5)	0.0603 (14)
C6	0.3158 (4)	0.5601 (2)	0.2743 (4)	0.0545 (12)
C7	0.2105 (4)	0.4875 (2)	0.2525 (3)	0.0436 (10)
C8	0.1595 (3)	0.43029 (18)	0.1160 (3)	0.0379 (9)
C9	0.2198 (4)	0.4484 (2)	0.0056 (3)	0.0444 (11)
C10	0.0147 (4)	0.2459 (3)	-0.2813 (4)	0.0573 (11)
C11	0.2035 (5)	0.5135 (3)	0.4973 (4)	0.0678 (16)
O3	0.8868 (3)	0.3074 (2)	0.2937 (3)	0.0716 (10)
H1O	0.18120	0.24780	-0.34070	0.1070*
H3	0.21180	0.39630	-0.19750	0.0590*
H4	0.36470	0.53900	-0.04200	0.0670*
H5	0.44190	0.62970	0.17760	0.0730*
H6	0.35070	0.59720	0.36460	0.0660*
H10A	0.04510	0.18290	-0.23950	0.0690*
H10B	-0.10340	0.24780	-0.34210	0.0690*
H11A	0.32050	0.50900	0.55460	0.1020*
H11B	0.15520	0.48700	0.55960	0.1020*
H11C	0.17230	0.57820	0.47550	0.1020*
H1W	0.943 (5)	0.331 (3)	0.252 (5)	0.1080*
H2W	0.949 (6)	0.301 (3)	0.389 (5)	0.1080*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0537 (5)	0.0487 (5)	0.0489 (5)	-0.0099 (4)	0.0176 (4)	-0.0054 (4)
O1	0.0644 (17)	0.112 (2)	0.0369 (13)	0.0262 (15)	0.0223 (13)	0.0030 (12)
O2	0.0555 (15)	0.0550 (14)	0.0428 (12)	-0.0026 (11)	0.0212 (11)	-0.0056 (10)
N1	0.0369 (14)	0.0372 (14)	0.0321 (13)	0.0029 (11)	0.0112 (11)	0.0046 (11)
C1	0.0379 (17)	0.0400 (17)	0.0342 (17)	0.0072 (14)	0.0096 (14)	0.0066 (13)
C2	0.0369 (17)	0.054 (2)	0.0324 (17)	0.0087 (15)	0.0094 (14)	0.0063 (13)
C3	0.047 (2)	0.067 (2)	0.0379 (18)	0.0167 (18)	0.0228 (16)	0.0161 (16)
C4	0.042 (2)	0.056 (2)	0.072 (2)	0.0051 (17)	0.0280 (18)	0.0169 (19)
C5	0.043 (2)	0.040 (2)	0.090 (3)	-0.0063 (16)	0.023 (2)	0.0051 (19)
C6	0.047 (2)	0.044 (2)	0.061 (2)	-0.0025 (16)	0.0141 (18)	-0.0045 (16)
C7	0.0358 (17)	0.0434 (18)	0.0448 (19)	0.0081 (15)	0.0122 (15)	0.0033 (14)
C8	0.0317 (16)	0.0343 (16)	0.0396 (16)	0.0055 (14)	0.0089 (14)	0.0054 (13)
C9	0.0380 (18)	0.0480 (19)	0.0458 (19)	0.0053 (15)	0.0176 (16)	0.0110 (15)
C10	0.056 (2)	0.076 (2)	0.0358 (19)	0.0121 (18)	0.0170 (17)	-0.0025 (16)
C11	0.071 (3)	0.078 (3)	0.046 (2)	0.000 (2)	0.019 (2)	-0.0121 (17)
O3	0.0575 (17)	0.108 (2)	0.0480 (15)	-0.0196 (15)	0.0226 (13)	0.0007 (15)

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

C11—C1	1.748 (4)	C4—C9	1.413 (5)
O1—C10	1.406 (5)	C5—C6	1.401 (6)
O2—C7	1.356 (4)	C6—C7	1.365 (5)
O2—C11	1.410 (4)	C7—C8	1.417 (4)
O1—H10	0.8200	C8—C9	1.409 (4)
O3—H1W	0.85 (5)	C3—H3	0.9300
O3—H2W	0.83 (4)	C4—H4	0.9300
N1—C1	1.298 (4)	C5—H5	0.9300
N1—C8	1.375 (4)	C6—H6	0.9300
C1—C2	1.401 (5)	C10—H10B	0.9700
C2—C3	1.369 (5)	C10—H10A	0.9700
C2—C10	1.507 (5)	C11—H11C	0.9600
C3—C9	1.408 (4)	C11—H11A	0.9600
C4—C5	1.351 (5)	C11—H11B	0.9600
C11...O2 ⁱ	3.147 (3)		
C7—O2—C11	118.3 (3)	C3—C9—C8	117.4 (3)
C10—O1—H10	109.00	O1—C10—C2	112.8 (3)
H1W—O3—H2W	107 (5)	C2—C3—H3	119.00
C1—N1—C8	117.1 (3)	C9—C3—H3	119.00
C11—C1—N1	115.5 (3)	C5—C4—H4	120.00
C11—C1—C2	117.4 (2)	C9—C4—H4	120.00
N1—C1—C2	127.2 (3)	C6—C5—H5	120.00
C1—C2—C10	121.9 (3)	C4—C5—H5	119.00
C1—C2—C3	115.3 (3)	C7—C6—H6	120.00
C3—C2—C10	122.8 (3)	C5—C6—H6	120.00

C2—C3—C9	121.5 (3)	O1—C10—H10A	109.00
C5—C4—C9	120.2 (3)	C2—C10—H10A	109.00
C4—C5—C6	121.0 (3)	C2—C10—H10B	109.00
C5—C6—C7	120.6 (3)	O1—C10—H10B	109.00
O2—C7—C8	115.4 (3)	H10A—C10—H10B	108.00
C6—C7—C8	119.6 (3)	O2—C11—H11B	109.00
O2—C7—C6	125.0 (3)	O2—C11—H11C	110.00
N1—C8—C9	121.6 (2)	O2—C11—H11A	109.00
N1—C8—C7	118.9 (3)	H11A—C11—H11C	109.00
C7—C8—C9	119.5 (3)	H11B—C11—H11C	110.00
C3—C9—C4	123.6 (3)	H11A—C11—H11B	109.00
C4—C9—C8	119.0 (3)		
C11—O2—C7—C6	-4.0 (5)	C2—C3—C9—C8	1.8 (5)
C11—O2—C7—C8	175.5 (3)	C9—C4—C5—C6	0.0 (6)
C8—N1—C1—C11	-178.2 (2)	C5—C4—C9—C3	-177.5 (3)
C8—N1—C1—C2	0.8 (5)	C5—C4—C9—C8	1.6 (5)
C1—N1—C8—C7	-178.4 (3)	C4—C5—C6—C7	-1.3 (6)
C1—N1—C8—C9	1.5 (4)	C5—C6—C7—O2	-179.7 (3)
C11—C1—C2—C3	177.3 (2)	C5—C6—C7—C8	0.9 (5)
C11—C1—C2—C10	-3.6 (4)	O2—C7—C8—N1	1.1 (4)
N1—C1—C2—C3	-1.7 (5)	O2—C7—C8—C9	-178.8 (3)
N1—C1—C2—C10	177.4 (3)	C6—C7—C8—N1	-179.3 (3)
C1—C2—C3—C9	0.3 (5)	C6—C7—C8—C9	0.8 (5)
C10—C2—C3—C9	-178.8 (3)	N1—C8—C9—C3	-2.7 (4)
C1—C2—C10—O1	-179.0 (3)	N1—C8—C9—C4	178.1 (3)
C3—C2—C10—O1	0.1 (5)	C7—C8—C9—C3	177.1 (3)
C2—C3—C9—C4	-179.1 (3)	C7—C8—C9—C4	-2.0 (4)

Symmetry codes: (i) $x-1/2, -y+1/2, z-1/2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

Cg1 is the centroid of the N1/C1—C3/C8/C9 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H10 \cdots O3 ⁱ	0.82	1.90	2.705 (4)	165
O3—H1W \cdots N1 ⁱⁱ	0.85 (5)	2.17 (5)	2.988 (4)	163 (4)
O3—H2W \cdots O1 ⁱⁱⁱ	0.83 (4)	2.02 (4)	2.836 (4)	171 (5)
C10—H10B \cdots Cg1 ⁱ	0.97	2.93	3.738 (5)	142

Symmetry codes: (i) $x-1/2, -y+1/2, z-1/2$; (ii) $x+1, y, z$; (iii) $x+1, y, z+1$.

Fig. 1

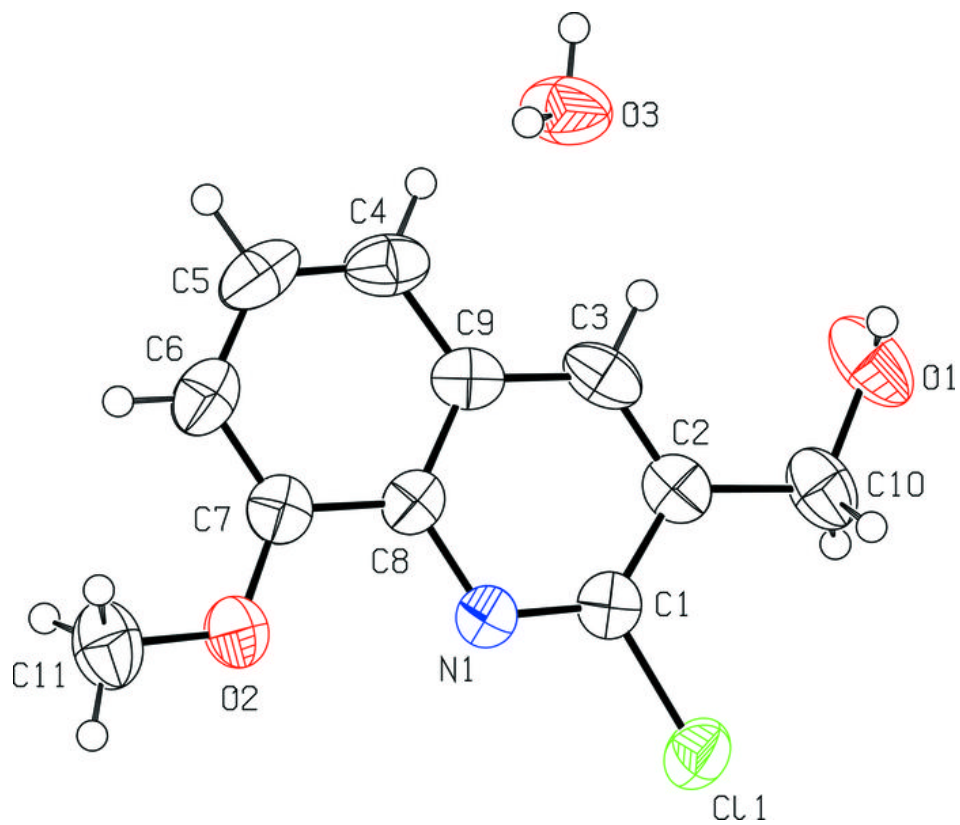
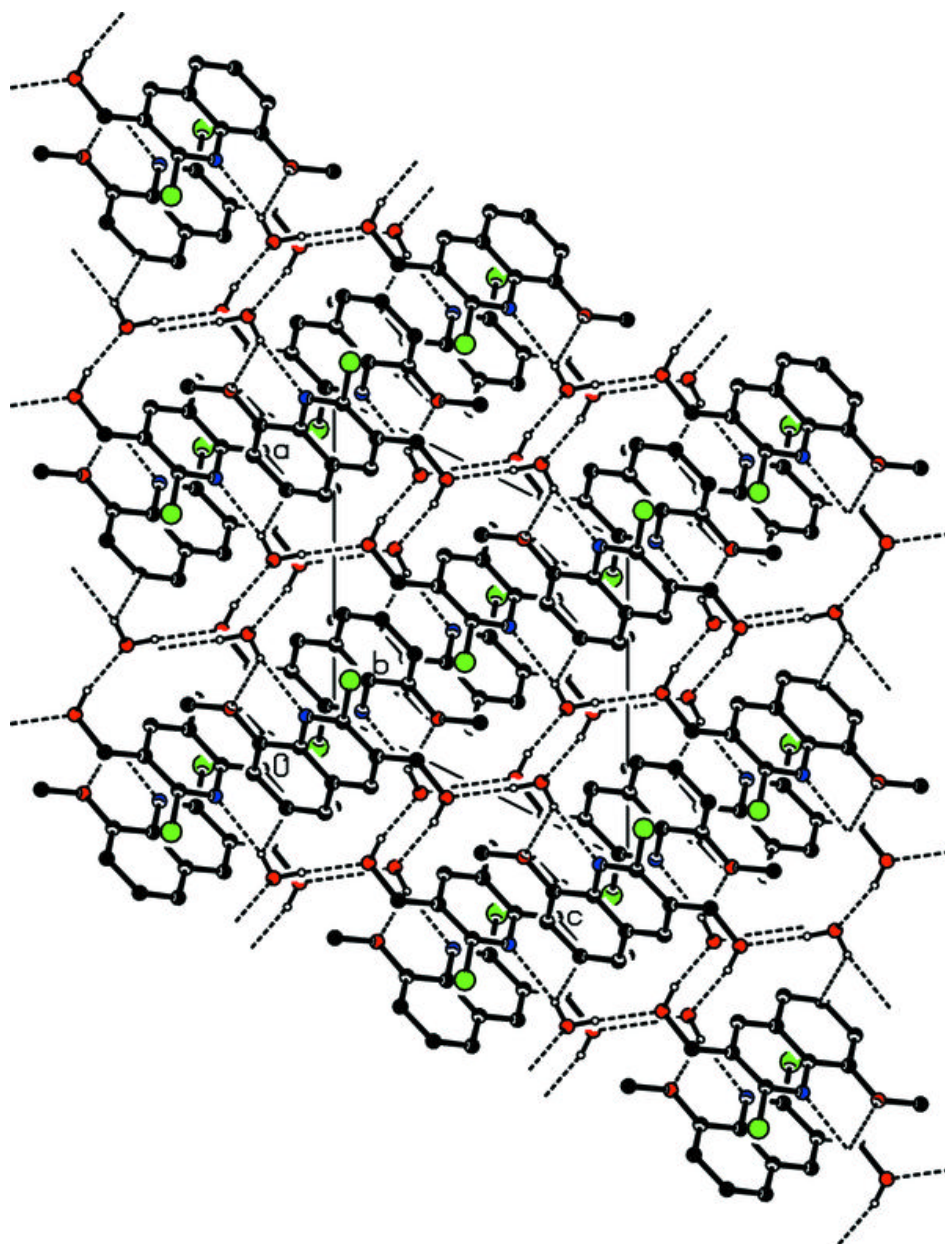


Fig. 2



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