

2-(4-Iodophenoxy)acetamide

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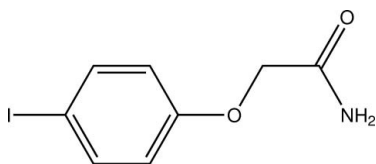
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 Key indicators: single-crystal X-ray study; $T = 200$ K; mean $\sigma(\text{C}-\text{C}) = 0.009$ Å; R factor = 0.064; wR factor = 0.120; data-to-parameter ratio = 20.9.

The molecule of the title compound, $\text{C}_8\text{H}_8\text{INO}_2$, amide-typical resonance shortens the nominal C—N single bond to 1.322 (7) Å. In the crystal, hydrogen bonds involving both nitrogen-bound H atoms as well as C—H \cdots O contacts connect the molecules into double layers approximately perpendicular to the crystallographic b axis. No π -stacking is apparent in the crystal structure.

Related literature

For the crystal structure of 2-(4-nitrophenoxy)acetamide, see: Lakshmi Rao *et al.* (1987) and of 2-(4-chloro-2-methylphenoxy)acetamide, see: Rao *et al.* (1987). For graph-set analysis of hydrogen bonds, see: Etter *et al.* (1990); Bernstein *et al.* (1995). For a description of the Cambridge Structural Database, see: Allen (2002). For the preparation, see: Glover *et al.* (1973).



Experimental

Crystal data

$\text{C}_8\text{H}_8\text{INO}_2$
 $M_r = 277.05$
 Monoclinic, $P2_1/c$
 $a = 5.1411$ (4) Å
 $b = 26.473$ (2) Å
 $c = 7.2960$ (7) Å
 $\beta = 109.564$ (3)°

$V = 935.66$ (14) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 3.38$ mm⁻¹
 $T = 200$ K
 $0.55 \times 0.18 \times 0.10$ mm

Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2008)
 $T_{\min} = 0.824$, $T_{\max} = 1.000$

8332 measured reflections
 2282 independent reflections
 2089 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.064$
 $wR(F^2) = 0.120$
 $S = 1.29$
 2282 reflections

109 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.50$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.58$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H71}\cdots\text{O1}^{\text{i}}$	0.88	2.00	2.881 (6)	178
$\text{N1}-\text{H72}\cdots\text{O1}^{\text{ii}}$	0.88	2.28	2.954 (6)	133
$\text{C2}-\text{H21}\cdots\text{O1}^{\text{iii}}$	0.99	2.48	3.422 (8)	158

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

Data collection: APEX2 (Bruker, 2010); cell refinement: SAINT (Bruker, 2010); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

The authors thank Mrs Vida Maqoko for helpful discussions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AA2016).

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supplementary materials

Acta Cryst. (2011). E67, o1928 [doi:10.1107/S1600536811025840]

2-(4-Iodophenoxy)acetamide

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Comment

Unlike carboxylic acids, their pertaining amides have not been studied extensively as ligands in coordination chemistry. Owing to their versatility in terms of denticity, keto-enol tautomerism as well as their possible use as neutral – or upon deprotonation – anionic ligands we set out to investigate the coordination behaviour of substituted acetamide derivatives to elucidate the rules guiding the formation of complex compounds. To enable comparative studies with envisioned reaction products, we determined the molecular and crystal structure of the title compound. So far, only the structures of 2-(4-nitrophenoxy)acetamide (Lakshmi Rao *et al.*, 1987) and of 2-(4-chloro-2-methylphenoxy)acetamide (Rao *et al.*, 1987) have been discussed as examples of phenoxy-substituted derivatives of acetamide.

The C–N single bond is shortened to 1.322 (7) Å due to the amide-typical resonance. This value is in good agreement with other derivatives of acetamide whose crystallographic data has been deposited with the Cambridge Structural Database (Allen, 2002) and whose ketonic oxygen atom is not involved in donor action towards transition metals. Intracyclic C–C–C angles hardly deviate from the ideal value of 120 °. The least-squares planes defined by the acetamide moiety and the oxygen atom of the phenoxy-derivative substituent on the one hand and the carbon atoms of the carbocycle on the other hand intersect at an angle of 24.91 (29) ° (Fig. 1 and Fig. 2).

In the crystal structure, both nitrogen-bonded H atoms participate in hydrogen bonds which invariably have the carbonyl oxygen atom as acceptor. While one of the H atoms of the amino group connects the molecules to centrosymmetric dimeric subunits, the other H atom of the NH₂ group connects these dimers to chains along [1 0 0]. Additionally, one of the hydrogen atoms of the methylene group forms a C–H···O contact whose range falls by more than 0.2 Å below the sum of van-der-Waals radii of the respective atoms. Again, the double-bonded O atom acts as acceptor. In terms of graph-set analysis (Etter *et al.*, 1990; Bernstein *et al.*, 1995), the hydrogen bonds stemming from the amino group can be described by a $C^1_1(4)R^2_2(8)$ descriptor on the unitary level while the C–H···O contacts necessitate a $R^2_2(4)$ descriptor on the same level. In total, the molecules are connected to double layers approximately perpendicular to the crystallographic *b*-axis (Fig. 3). No π -stacking is apparent in the crystal structure.

The packing of the compound is shown in Figure 4.

Experimental

The compound was prepared upon reacting 2-phenoxyacetamide with *tert*-butyl hypochlorite and iodine according to a published procedure (Glover *et al.*, 1973).

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.99 Å for the methylene group and C—H 0.95 Å for aromatic carbon atoms) and were included in the refinement in the riding model approximation, with $U(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$. Nitrogen-bound H-atoms were placed in calculated positions (N—H 0.88 Å) and were included in the refinement in the riding model approximation, with $U(\text{H})$ set to $1.2U_{\text{eq}}(\text{N})$.

Figures

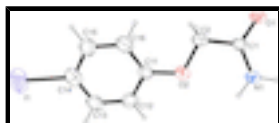


Fig. 1. The molecular structure of the title compound, with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level).

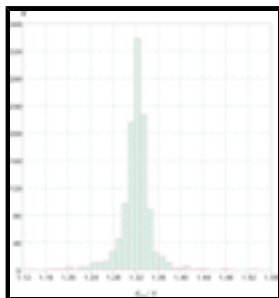


Fig. 2. Observed distribution of C—N bond lengths in the O=C—NH₂ moiety of acetamide derivatives in which the O atom is not acting as a donor atom in coordination compounds (data based on CSD search including all deposited crystal structures up to November 2010).

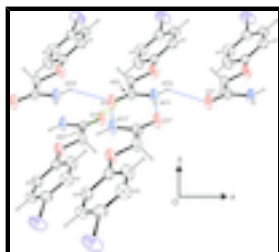


Fig. 3. Intermolecular contacts, viewed along [0 0 -1]. Symmetry operators: ⁱ $x - 1, y, z$; ⁱⁱ $-x + 1, -y, -z + 1$; ⁱⁱⁱ $-x + 2, -y, -z + 2$; ^{iv} $x + 1, y, z$.

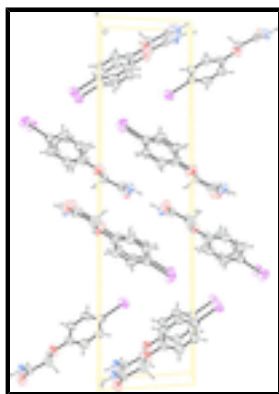


Fig. 4. Molecular packing of the title compound, viewed along [-1 0 0] (anisotropic displacement ellipsoids drawn at 50% probability level).

2-(4-Iodophenoxy)acetamide

Crystal data

C₈H₈INO₂

$F(000) = 528$

$M_r = 277.05$
 Monoclinic, $P2_1/c$
 Hall symbol: -P 2ybc
 $a = 5.1411$ (4) Å
 $b = 26.473$ (2) Å
 $c = 7.2960$ (7) Å
 $\beta = 109.564$ (3)°
 $V = 935.66$ (14) Å³
 $Z = 4$
 $D_x = 1.967$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 5293 reflections
 $\theta = 3.1$ – 28.2 °
 $\mu = 3.38$ mm⁻¹
 $T = 200$ K
 Rod, colourless
 $0.55 \times 0.18 \times 0.10$ mm

Data collection

Bruker APEXII CCD diffractometer
 Radiation source: fine-focus sealed tube graphite
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2008)
 $T_{\min} = 0.824$, $T_{\max} = 1.000$
 8332 measured reflections
 2282 independent reflections
 2089 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 $\theta_{\max} = 28.2$ °, $\theta_{\min} = 3.1$ °
 $h = -6 \rightarrow 6$
 $k = -34 \rightarrow 35$
 $l = -8 \rightarrow 9$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.064$
 $wR(F^2) = 0.120$
 $S = 1.29$
 2282 reflections
 109 parameters
 0 restraints
 Primary atom site location: structure-invariant direct methods
 Secondary atom site location: difference Fourier map
 Hydrogen site location: inferred from neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0042P)^2 + 6.0795P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 1.50$ e Å⁻³
 $\Delta\rho_{\min} = -1.58$ e Å⁻³

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
11	0.81735 (14)	0.20849 (2)	-0.25506 (9)	0.0685 (2)
O1	0.6872 (8)	0.01765 (17)	0.7947 (6)	0.0390 (10)
O2	1.0024 (8)	0.08086 (17)	0.4969 (6)	0.0416 (10)
N1	1.1340 (9)	0.0300 (2)	0.8286 (7)	0.0367 (11)
H71	1.1917	0.0152	0.9432	0.044*
H72	1.2543	0.0422	0.7781	0.044*
C1	0.8667 (10)	0.0339 (2)	0.7322 (8)	0.0278 (11)
C2	0.7756 (11)	0.0587 (2)	0.5354 (9)	0.0359 (13)

supplementary materials

H21	0.6883	0.0332	0.4343	0.043*
H22	0.6365	0.0850	0.5302	0.043*
C11	0.9411 (12)	0.1079 (2)	0.3245 (9)	0.0345 (12)
C12	1.1409 (13)	0.1428 (2)	0.3187 (10)	0.0399 (14)
H12	1.3013	0.1474	0.4296	0.048*
C13	1.1073 (13)	0.1706 (2)	0.1538 (11)	0.0425 (15)
H13	1.2466	0.1936	0.1489	0.051*
C14	0.8721 (14)	0.1650 (2)	-0.0038 (10)	0.0408 (14)
C15	0.6678 (15)	0.1314 (3)	0.0021 (10)	0.0440 (15)
H15	0.5036	0.1281	-0.1069	0.053*
C16	0.7036 (13)	0.1029 (3)	0.1658 (10)	0.0435 (15)
H16	0.5647	0.0797	0.1699	0.052*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
II	0.0910 (5)	0.0588 (3)	0.0581 (3)	-0.0160 (3)	0.0282 (3)	0.0173 (3)
O1	0.0201 (19)	0.062 (3)	0.038 (2)	-0.0006 (18)	0.0138 (17)	0.007 (2)
O2	0.027 (2)	0.056 (3)	0.042 (2)	-0.0059 (19)	0.0109 (18)	0.014 (2)
N1	0.022 (2)	0.055 (3)	0.035 (3)	0.001 (2)	0.012 (2)	0.011 (2)
C1	0.022 (3)	0.035 (3)	0.029 (3)	0.001 (2)	0.013 (2)	-0.001 (2)
C2	0.022 (3)	0.049 (3)	0.038 (3)	-0.002 (2)	0.012 (2)	0.003 (3)
C11	0.030 (3)	0.037 (3)	0.041 (3)	0.007 (2)	0.018 (3)	0.010 (2)
C12	0.031 (3)	0.035 (3)	0.051 (4)	-0.002 (2)	0.012 (3)	0.005 (3)
C13	0.039 (3)	0.031 (3)	0.063 (4)	-0.004 (3)	0.024 (3)	0.008 (3)
C14	0.052 (4)	0.032 (3)	0.046 (4)	0.001 (3)	0.026 (3)	0.000 (3)
C15	0.050 (4)	0.047 (4)	0.035 (3)	-0.007 (3)	0.014 (3)	-0.001 (3)
C16	0.038 (3)	0.054 (4)	0.044 (4)	-0.010 (3)	0.020 (3)	0.000 (3)

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

II—C14	2.102 (6)	C11—C16	1.380 (9)
O1—C1	1.236 (6)	C11—C12	1.392 (8)
O2—C11	1.389 (7)	C12—C13	1.371 (9)
O2—C2	1.415 (6)	C12—H12	0.9500
N1—C1	1.322 (7)	C13—C14	1.370 (10)
N1—H71	0.8800	C13—H13	0.9500
N1—H72	0.8800	C14—C15	1.388 (9)
C1—C2	1.503 (8)	C15—C16	1.372 (9)
C2—H21	0.9900	C15—H15	0.9500
C2—H22	0.9900	C16—H16	0.9500
C11—O2—C2	116.2 (4)	C13—C12—C11	120.3 (6)
C1—N1—H71	120.0	C13—C12—H12	119.8
C1—N1—H72	120.0	C11—C12—H12	119.8
H71—N1—H72	120.0	C14—C13—C12	119.7 (6)
O1—C1—N1	123.2 (5)	C14—C13—H13	120.1
O1—C1—C2	118.2 (5)	C12—C13—H13	120.1
N1—C1—C2	118.6 (5)	C13—C14—C15	120.5 (6)

O2—C2—C1	110.9 (4)	C13—C14—I1	119.7 (5)
O2—C2—H21	109.5	C15—C14—I1	119.8 (5)
C1—C2—H21	109.5	C16—C15—C14	119.8 (6)
O2—C2—H22	109.5	C16—C15—H15	120.1
C1—C2—H22	109.5	C14—C15—H15	120.1
H21—C2—H22	108.0	C15—C16—C11	120.0 (6)
C16—C11—O2	125.4 (5)	C15—C16—H16	120.0
C16—C11—C12	119.6 (6)	C11—C16—H16	120.0
O2—C11—C12	115.1 (5)		
C11—O2—C2—C1	-175.7 (5)	C12—C13—C14—C15	-0.1 (10)
O1—C1—C2—O2	172.4 (5)	C12—C13—C14—I1	179.0 (5)
N1—C1—C2—O2	-8.5 (8)	C13—C14—C15—C16	-1.1 (10)
C2—O2—C11—C16	-20.4 (9)	I1—C14—C15—C16	179.8 (5)
C2—O2—C11—C12	159.1 (5)	C14—C15—C16—C11	0.5 (10)
C16—C11—C12—C13	-2.6 (9)	O2—C11—C16—C15	-179.2 (6)
O2—C11—C12—C13	177.9 (6)	C12—C11—C16—C15	1.3 (10)
C11—C12—C13—C14	2.0 (10)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H71 \cdots O1 ⁱ	0.88	2.00	2.881 (6)	178
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Fig. 1

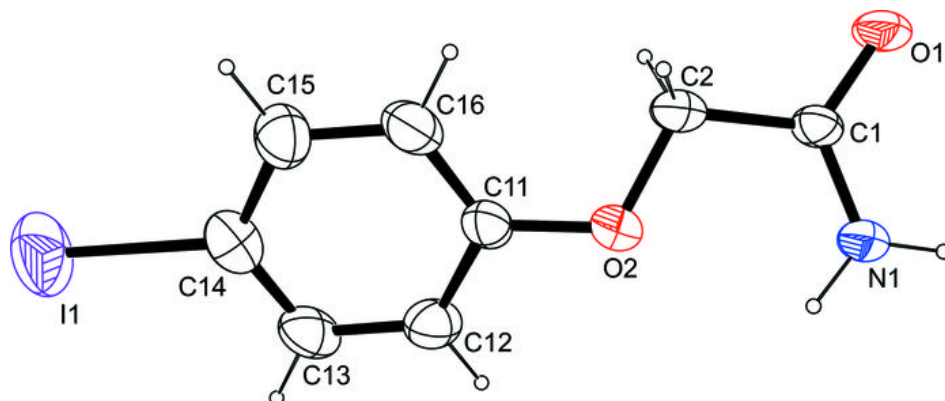


Fig. 2

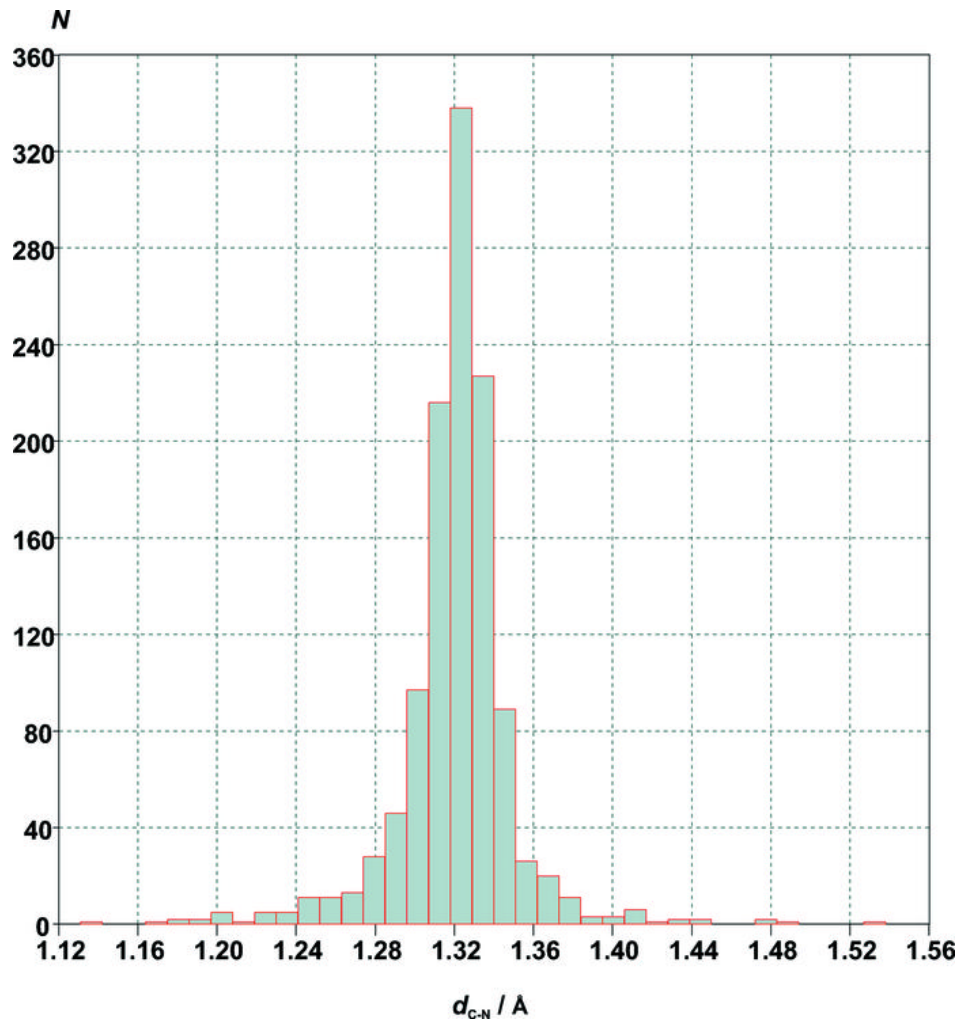


Fig. 3

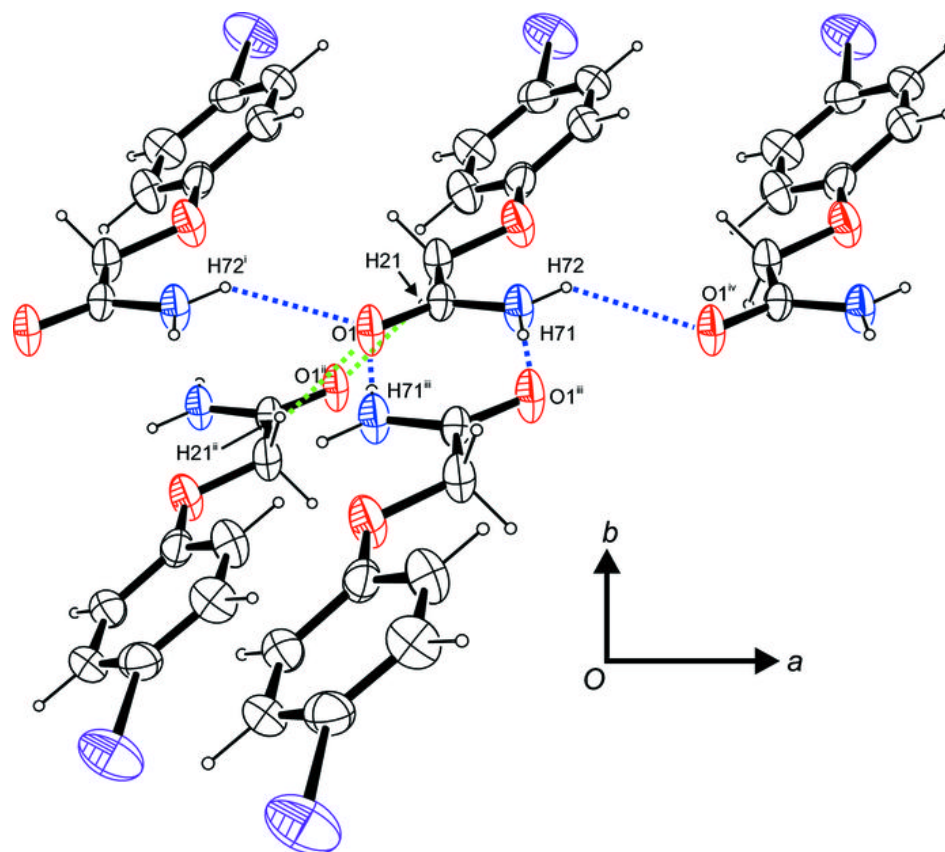


Fig. 4

