The co-crystal 4,6-diacetylresorcinol–1-aminopyrene (2/1)
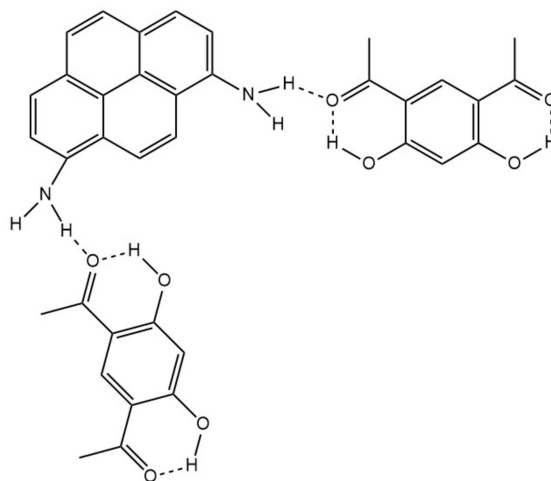
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The structure of the title molecular complex, $C_{16}H_{11}N \cdot 2C_{10}H_{10}O_4$, at 150 K has been determined. The molecules form stacks consisting of aggregates with disordered 1-aminopyrene molecule surrounded by two 4,6-diacetylresorcinol molecules. Neighbouring stacks are linked by hydrogen bonds between the amine H atoms of the 1-aminopyrene molecule with the adjacent carbonyl oxygen atom of the 4,6-diacetylresorcinol molecule.

1. Chemical context

Co-crystals are crystalline single phase materials made up of molecules of two or more compounds. They are used in a variety of fields, including paper, textiles and the chemical, photographic, and electronic industries (Golbedaghi & Fausto, 2018). However, their main uses are centered in the pharmaceutical industry, where they have been gaining importance in recent years.

Schiff bases are the products of the condensation reaction of aldehydes or ketones with amines. They have multiple uses, for example as pigments and dyes, intermediates in organic synthesis, and as catalysts and polymer stabilizers. They also exhibit a broad range of biological activities. They play an important role in coordination chemistry as they readily form stable complexes with most transition metals (Aazam *et al.*, 2006, 2008, 2010; El-Attar & Aazam, 2021). In the process of the synthesis of such compounds with 4,6-diacetylresorcinol and 1-aminopyrene as the precursors, a new co-crystal, $C_{16}H_{11}N \cdot 2C_{10}H_{10}O_4$, has been obtained.
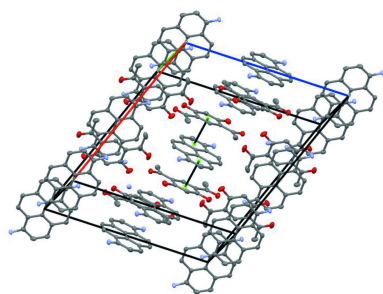
Table 1
Hydrogen-bond geometry (Å, °).

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
O2—H2$\cdots$O1	0.85 (3)	1.80 (3)	2.545 (2)	146 (2)
O3—H3$\cdots$O4	0.84 (2)	1.80 (2)	2.542 (2)	147 (2)
N1—H1$A\cdots$O3^{i}	0.88 (1)	2.30 (2)	3.131 (3)	156 (4)
N1—H1$B\cdots$O1	0.88 (1)	2.16 (2)	2.966 (3)	154 (3)
N1B—H1$C\cdots$O2^{ii}	0.88 (1)	2.05 (6)	2.902 (18)	162 (17)
N1B—H1$D\cdots$O4^{iii}	0.88 (1)	1.89 (6)	2.731 (18)	159 (14)

Symmetry codes: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z+1$; (iii) $x, y, z+1$.

2. Structural commentary

The formula unit of the title compound consists of two 4,6-diacetylresorcinol molecules and one 1-aminopyrene molecule, which lies on an inversion center. Besides this, this molecule is further disordered so that the amino N atom is distributed over four chemically equivalent positions, at the C11 and C13 atoms, with the occupancies of 0.428 (2) and 0.072 (2) for N1 and N1B, respectively (Fig. 1).

In the 4,6-diacetylresorcinol molecule, the hydroxy groups form intramolecular hydrogen bonds with the oxygen atoms of neighbouring acetyl groups, generating $S(6)$ rings (Table 1).

3. Supramolecular features

In the crystal, the molecules form centrosymmetric aggregates with two molecules of 4,6-diacetylresorcinol positioned on both sides of the 1-aminopyrene molecule (Fig. 2). The mean planes of the aromatic rings of the 4,6-diacetylresorcinol molecules are inclined at 2.91 (10)° to the mean plane of the tetracyclic core of the 1-aminopyrene molecule. A short intercentroid separation $Cg1\cdots Cg2$ of 3.492 (1) Å is observed in this aggregate, with $Cg1$ being the centroid of C3–C8 ring of diacetylresorcinol and $Cg2$ the centroid of one of the aminopyrene rings, C11–C18. These aggregates are packed into stacks by π–π stacking interactions between 4,6-diacetylresorcinol molecules. Neighbouring stacks are linked by hydrogen bonds between the amino H atom of the 1-amino-
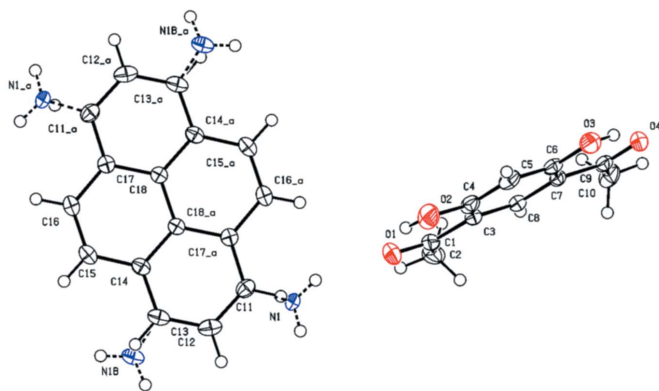

Figure 2
A view of the crystal packing showing the π–π stacking interactions.

pyrene molecule with the adjacent carbonyl oxygen atom of the 4,6-diacetylresorcinol molecule, thus forming a three-dimensional network (Fig. 3).

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.42; May 2021; Groom *et al.*, 2016) gave the structures of the individual components. In the structure of 4,6-diacetylresorcinol (Kokila *et al.*, 1992, refcode VOXPED) the molecule is almost planar, with the oxygen atoms of the acetyl groups deviating by 0.074 (1) and 0.072 (2) Å from the mean plane of the benzene ring. There are intramolecular hydrogen bonds between the oxygen atoms of the acetyl groups and the hydroxy hydrogen atoms. A search for 1-aminopyrene gave two hits for co-crystals composed of 1-aminopyrene molecules with either 7,7′,8,8′-tetracyanoquinodimethane or 3,5-dinitro-

Figure 1
The asymmetric unit of the title compound with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

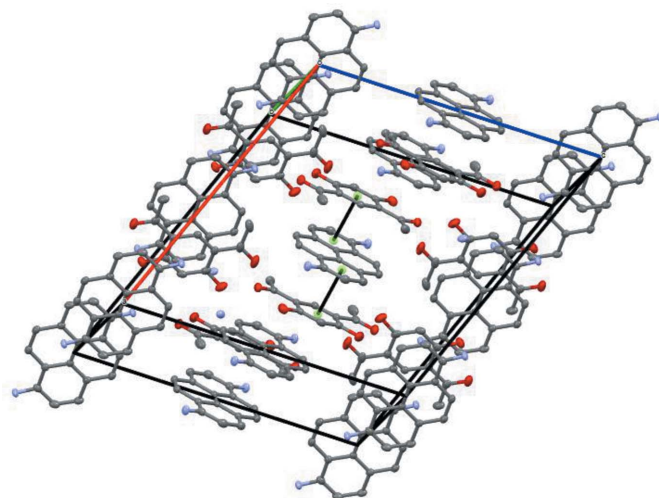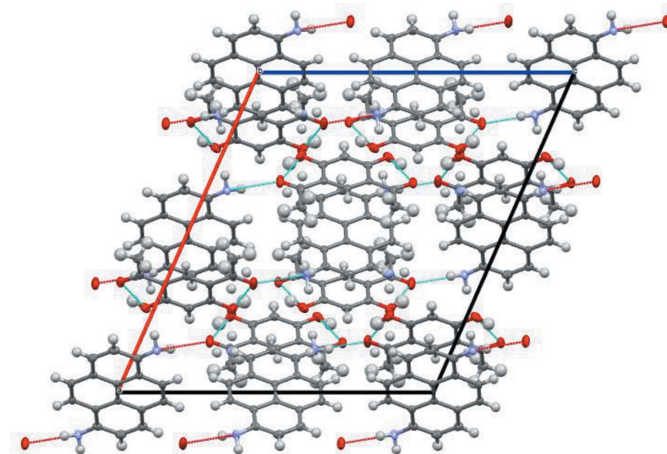

Figure 3
The crystal packing of the title compound viewed along the *b* axis, showing the N—H$\cdots$O hydrogen bonds.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{11}N\cdot2C_{10}H_{10}O_4$
M_r	605.62
Crystal system, space group	Monoclinic, $C2/c$
Temperature (K)	150
a, b, c (Å)	18.7222 (14), 9.7870 (6), 16.9398 (15)
β (°)	113.758 (4)
V (Å³)	2840.9 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm⁻¹)	0.10
Crystal size (mm)	$0.45 \times 0.26 \times 0.22$
Data collection	
Diffractometer	Bruker AXS D8 Quest diffractometer with PhotonII charge-integrating pixel array detector (CPAD)
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.673, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	33773, 4316, 3741
R_{int}	0.046
$(\sin \theta/\lambda)_{max}$ (Å⁻¹)	0.714
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.061, 0.153, 1.15
No. of reflections	4316
No. of parameters	231
No. of restraints	13
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å⁻³)	0.35, −0.26

Computer programs: *APEX4* and *SAINT* (Bruker, 2021), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2019/2* (Sheldrick, 2015*b*), *ShelXle* (Hübschle *et al.*, 2011) and *publCIF* (Westrip, 2010).

benzoic acid and showed that the NH₂ groups can act as H-donors in the intermolecular hydrogen-bonding interactions, as in the title compound. (Mandal *et al.*, 2020, refcode BOYQOY; Mandal *et al.*, 2019, refcode LORBOM).

5. Synthesis and crystallization

A solution of 1-aminopyrene (1 mmol, 0.233 g) dissolved in 10 ml of ethanol was added dropwise to 4,6-diacetylresorcinol (DAR) (0.5 mmol, 0.097 g) dissolved in 10 ml of ethanol, 3 drops of acetic acid were added, and the mixture was stirred for 15 min at room temperature and then for about 3 h under reflux. Yellow fiber-like crystals of the Schiff base ligand were separated. They were filtered off and washed with 4 ml of ethanol, weight = 0.021 g, yield = 7.12%, m.p. = 523 K, m/z = 592.7 ($C_{42}H_{28}N_2O_2$). The filtrate was left overnight upon which dark-brown rectangular co-crystals were formed, weight = 0.04 g, yield = 19.5%, m.p. = 418 K, m/z = 605.62

($C_{16}H_{11}N\cdot2C_{10}H_{10}O_4$). ¹H NMR (800 MHz, DMSO-d_6) δ 12.75 (s, br, –OH), 8.406 (s, 2H, DAR), 8.251 (d, 1H), 7.992 (d, 2H), 7.992 (d, 1H), 7.958 (d, 1H), 87.915 (d, 2H), 7.880 (m, 1H), 7.367 (d, 1H), 6.392 (s, 2H, DAR), 6.314 (s, br, NH2, 2H), 2.661 (s, Me, 12H).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The C—N bond distances for the disordered N atom were restrained to be similar. The minor occupancy N1*B* atom was constrained to have the same ADPs as the C atom to which it is bonded. N—H bond distances were restrained to a target value of 0.88 (2) Å, and the H—N—H and C—N—H bond angles were restrained to be similar to each other. Subject to these conditions the occupancy rates refined to 0.428 (2) and 0.072 (2). O-bound H atoms were refined with $U_{iso}(H) = 1.5U_{eq}(O)$. C-bound H atoms were positioned geometrically (C—H = 0.9–0.98 Å) and refined as riding on their parent atoms with $U_{iso}(H) = 1.2$–$1.5U_{eq}(C)$.

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Computing details

Data collection: *APEX4* (Bruker, 2021); cell refinement: *SAINT* (Bruker, 2021); data reduction: *SAINT* (Bruker, 2021); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2019/2* (Sheldrick, 2015b), *ShelXle* (Hübschle *et al.*, 2011); software used to prepare material for publication: *publCIF* (Westrip, 2010).

4,6-Diacetylresorcinol–1-aminopyrene (2/1)

Crystal data

$C_{16}H_{11}N \cdot 2C_{10}H_{10}O_4$	$F(000) = 1272$
$M_r = 605.62$	$D_x = 1.416$ Mg m^{-3}
Monoclinic, *C2/c*	Mo *Kα* radiation, $\lambda = 0.71073$ Å
$a = 18.7222\,(14)$ Å	Cell parameters from 9850 reflections
$b = 9.7870\,(6)$ Å	$\theta = 2.4$–33.2°
$c = 16.9398\,(15)$ Å	$\mu = 0.10$ mm^{-1}
$\beta = 113.758\,(4)°$	$T = 150$ K
$V = 2840.9\,(4)$ Å^3	Block, yellow
$Z = 4$	$0.45 \times 0.26 \times 0.22$ mm

Data collection

Bruker AXS D8 Quest diffractometer with PhotonII charge-integrating pixel array detector (CPAD)	Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
	$T_{min} = 0.673$, $T_{max} = 0.747$
Radiation source: fine focus sealed tube X-ray source	33773 measured reflections
	4316 independent reflections
Triumph curved graphite crystal monochromator	3741 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.046$
Detector resolution: 7.4074 pixels mm^{-1}	$\theta_{max} = 30.5°$, $\theta_{min} = 2.5°$
ω and phi scans	$h = -26 \rightarrow 26$
	$k = -13 \rightarrow 13$
	$l = -24 \rightarrow 24$

Refinement

Refinement on F^2	Primary atom site location: dual
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.061$	
$wR(F^2) = 0.153$	Hydrogen site location: mixed
$S = 1.15$	H atoms treated by a mixture of independent and constrained refinement
4316 reflections	
231 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0356P)^2 + 4.9256P]$
13 restraints	where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.35$ e Å^{-3}

$\Delta\rho_{min} = -0.26$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. The single nitrogen atom is disordered over four chemically equivalent positions (each two are also crystallographically equivalent, by inversion). The C-N bond distances were restrained to be similar. The minor N atom was constrained to have the same ADP as the C atom it is bonded to. The N-H bond distances were restrained to a target value of 0.88 (2) Angstrom, and the H-N-H and C-N-H bond angles were each restrained to be similar to each other. Subject to these conditions the occupancy rates refined to two times 0.428 (2) and two times 0.072 (2).

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

	x	y	z	$U_{iso}*/U_{eq}$	Occ. (<1)
O1	0.65970 (9)	0.27504 (17)	0.27887 (8)	0.0435 (4)	
O2	0.73863 (8)	0.46240 (16)	0.24707 (9)	0.0406 (3)	
H2	0.7158 (15)	0.422 (3)	0.2746 (11)	0.061*	
O3	0.73548 (7)	0.49314 (13)	−0.02964 (9)	0.0318 (3)	
H3	0.7141 (13)	0.459 (2)	−0.0788 (16)	0.048*	
O4	0.65513 (7)	0.32200 (14)	−0.14377 (8)	0.0333 (3)	
N1	0.63242 (19)	0.2052 (3)	0.43483 (19)	0.0232 (6)	0.428 (2)
H1A	0.6785 (13)	0.167 (4)	0.462 (2)	0.028*	0.428 (2)
H1B	0.636 (3)	0.252 (3)	0.3924 (19)	0.028*	0.428 (2)
N1B	0.6296 (10)	0.2240 (18)	0.6962 (10)	0.0255 (3)	0.072 (2)
H1C	0.676 (5)	0.184 (17)	0.716 (6)	0.031*	0.072 (2)
H1D	0.625 (10)	0.261 (10)	0.741 (4)	0.031*	0.072 (2)
C1	0.63682 (10)	0.23098 (19)	0.20409 (11)	0.0303 (4)	
C2	0.58258 (12)	0.1108 (2)	0.17814 (13)	0.0392 (4)	
H2A	0.535960	0.134104	0.126697	0.059*	
H2B	0.567315	0.086926	0.225370	0.059*	
H2C	0.609006	0.032779	0.165398	0.059*	
C3	0.66261 (9)	0.29590 (16)	0.14205 (9)	0.0232 (3)	
C4	0.71264 (9)	0.41203 (17)	0.16646 (10)	0.0264 (3)	
C5	0.73613 (9)	0.47613 (17)	0.10833 (11)	0.0265 (3)	
H5	0.769710	0.553232	0.125827	0.032*	
C6	0.71078 (8)	0.42813 (15)	0.02447 (10)	0.0228 (3)	
C7	0.66054 (8)	0.31225 (15)	−0.00255 (9)	0.0194 (3)	
C8	0.63794 (8)	0.24981 (15)	0.05741 (9)	0.0206 (3)	
H8	0.604356	0.172691	0.039992	0.025*	
C9	0.63396 (9)	0.26350 (17)	−0.09192 (10)	0.0242 (3)	
C10	0.58093 (10)	0.1419 (2)	−0.12108 (12)	0.0339 (4)	
H10A	0.568838	0.123625	−0.182006	0.051*	
H10B	0.532509	0.160674	−0.113824	0.051*	
H10C	0.606809	0.062098	−0.086448	0.051*	
C11	0.60624 (9)	0.27031 (15)	0.48402 (10)	0.0237 (3)	
H11	0.624879	0.227360	0.445699	0.028*	0.572 (2)

C12	0.63162 (9)	0.22506 (16)	0.56916 (11)	0.0283 (3)	
H12	0.666448	0.149723	0.587700	0.034*	
C13	0.60710 (9)	0.28760 (16)	0.62685 (10)	0.0255 (3)	
H13	0.625984	0.256168	0.684761	0.031*	0.928 (2)
C14	0.55427 (8)	0.39772 (15)	0.60056 (9)	0.0207 (3)	
C15	0.52761 (9)	0.46518 (16)	0.65871 (9)	0.0240 (3)	
H15	0.546435	0.435794	0.717036	0.029*	
C16	0.47590 (9)	0.57043 (16)	0.63217 (9)	0.0232 (3)	
H16	0.458483	0.611875	0.671966	0.028*	
C17	0.44718 (8)	0.61998 (15)	0.54519 (9)	0.0196 (3)	
C18	0.47321 (8)	0.55597 (14)	0.48616 (9)	0.0174 (3)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0494 (8)	0.0610 (10)	0.0232 (6)	0.0200 (7)	0.0178 (6)	0.0096 (6)
O2	0.0380 (7)	0.0502 (9)	0.0258 (6)	0.0040 (6)	0.0048 (5)	−0.0143 (6)
O3	0.0326 (6)	0.0283 (6)	0.0415 (7)	−0.0002 (5)	0.0223 (6)	0.0058 (5)
O4	0.0363 (6)	0.0425 (7)	0.0241 (6)	0.0130 (6)	0.0153 (5)	0.0058 (5)
N1	0.0294 (15)	0.0217 (14)	0.0224 (14)	0.0077 (11)	0.0147 (12)	0.0002 (11)
N1B	0.0237 (7)	0.0230 (7)	0.0254 (7)	−0.0008 (5)	0.0052 (6)	0.0077 (6)
C1	0.0299 (8)	0.0382 (9)	0.0254 (7)	0.0158 (7)	0.0137 (6)	0.0107 (7)
C2	0.0421 (10)	0.0399 (10)	0.0448 (10)	0.0109 (8)	0.0270 (9)	0.0204 (8)
C3	0.0221 (6)	0.0265 (7)	0.0211 (6)	0.0074 (5)	0.0087 (5)	0.0025 (5)
C4	0.0207 (7)	0.0296 (8)	0.0242 (7)	0.0067 (6)	0.0039 (5)	−0.0056 (6)
C5	0.0188 (6)	0.0233 (7)	0.0346 (8)	−0.0001 (5)	0.0078 (6)	−0.0063 (6)
C6	0.0176 (6)	0.0205 (6)	0.0314 (7)	0.0043 (5)	0.0111 (5)	0.0021 (6)
C7	0.0170 (6)	0.0205 (6)	0.0202 (6)	0.0038 (5)	0.0071 (5)	−0.0006 (5)
C8	0.0189 (6)	0.0194 (6)	0.0236 (7)	0.0027 (5)	0.0086 (5)	0.0001 (5)
C9	0.0211 (6)	0.0274 (7)	0.0221 (7)	0.0090 (5)	0.0067 (5)	−0.0008 (6)
C10	0.0278 (8)	0.0369 (9)	0.0321 (8)	0.0013 (7)	0.0067 (6)	−0.0151 (7)
C11	0.0222 (6)	0.0194 (6)	0.0305 (7)	−0.0009 (5)	0.0117 (6)	−0.0020 (6)
C12	0.0236 (7)	0.0205 (7)	0.0372 (9)	0.0021 (5)	0.0084 (6)	0.0054 (6)
C13	0.0237 (7)	0.0230 (7)	0.0254 (7)	−0.0008 (5)	0.0052 (6)	0.0077 (6)
C14	0.0184 (6)	0.0217 (7)	0.0205 (6)	−0.0035 (5)	0.0065 (5)	0.0032 (5)
C15	0.0240 (7)	0.0291 (8)	0.0178 (6)	−0.0041 (6)	0.0074 (5)	0.0021 (5)
C16	0.0241 (7)	0.0275 (7)	0.0190 (6)	−0.0045 (6)	0.0100 (5)	−0.0016 (5)
C17	0.0188 (6)	0.0187 (6)	0.0213 (6)	−0.0036 (5)	0.0081 (5)	−0.0010 (5)
C18	0.0157 (6)	0.0171 (6)	0.0186 (6)	−0.0036 (5)	0.0060 (5)	−0.0002 (5)

Geometric parameters (Å, º)

O1—C1	1.240 (2)	C7—C8	1.389 (2)
O2—C4	1.3450 (19)	C7—C9	1.470 (2)
O2—H2	0.84 (3)	C8—H8	0.9500
O3—C6	1.3418 (19)	C9—C10	1.500 (2)
O3—H3	0.83 (3)	C10—H10A	0.9800
O4—C9	1.240 (2)	C10—H10B	0.9800

N1—C11	1.292 (3)	C10—H10C	0.9800
N1—H1A	0.882 (10)	C11—C12	1.396 (2)
N1—H1B	0.875 (10)	C11—C17ⁱ	1.414 (2)
N1B—C13	1.243 (14)	C11—H11	0.9500
N1B—H1C	0.882 (10)	C12—C13	1.379 (2)
N1B—H1D	0.880 (10)	C12—H12	0.9500
C1—C3	1.467 (2)	C13—C14	1.408 (2)
C1—C2	1.500 (3)	C13—H13	0.9500
C2—H2A	0.9800	C14—C18ⁱ	1.4210 (19)
C2—H2B	0.9800	C14—C15	1.432 (2)
C2—H2C	0.9800	C15—C16	1.360 (2)
C3—C8	1.392 (2)	C15—H15	0.9500
C3—C4	1.424 (2)	C16—C17	1.434 (2)
C4—C5	1.380 (2)	C16—H16	0.9500
C5—C6	1.386 (2)	C17—C18	1.4220 (19)
C5—H5	0.9500	C18—C18ⁱ	1.431 (3)
C6—C7	1.426 (2)		

C4—O2—H2	109.5	O4—C9—C10	119.42 (15)
C6—O3—H3	109.5	C7—C9—C10	120.08 (14)
C11—N1—H1A	114 (2)	C9—C10—H10A	109.5
C11—N1—H1B	116 (2)	C9—C10—H10B	109.5
H1A—N1—H1B	106 (4)	H10A—C10—H10B	109.5
C13—N1B—H1C	120 (3)	C9—C10—H10C	109.5
C13—N1B—H1D	120 (3)	H10A—C10—H10C	109.5
H1C—N1B—H1D	106 (5)	H10B—C10—H10C	109.5
O1—C1—C3	120.23 (18)	N1—C11—C12	116.75 (19)
O1—C1—C2	119.14 (16)	N1—C11—C17ⁱ	123.18 (19)
C3—C1—C2	120.63 (15)	C12—C11—C17ⁱ	120.05 (14)
C1—C2—H2A	109.5	C12—C11—H11	120.0
C1—C2—H2B	109.5	C17ⁱ—C11—H11	120.0
H2A—C2—H2B	109.5	C13—C12—C11	121.28 (14)
C1—C2—H2C	109.5	C13—C12—H12	119.4
H2A—C2—H2C	109.5	C11—C12—H12	119.4
H2B—C2—H2C	109.5	N1B—C13—C12	111.3 (10)
C8—C3—C4	117.66 (14)	N1B—C13—C14	127.6 (10)
C8—C3—C1	121.95 (15)	C12—C13—C14	120.49 (14)
C4—C3—C1	120.37 (15)	C12—C13—H13	119.8
O2—C4—C5	117.95 (16)	C14—C13—H13	119.8
O2—C4—C3	120.93 (16)	C13—C14—C18ⁱ	119.12 (14)
C5—C4—C3	121.12 (14)	C13—C14—C15	122.02 (14)
C4—C5—C6	120.08 (15)	C18ⁱ—C14—C15	118.86 (13)
C4—C5—H5	120.0	C16—C15—C14	121.37 (14)
C6—C5—H5	120.0	C16—C15—H15	119.3
O3—C6—C5	118.00 (15)	C14—C15—H15	119.3
O3—C6—C7	121.48 (14)	C15—C16—C17	121.20 (14)
C5—C6—C7	120.52 (14)	C15—C16—H16	119.4
C8—C7—C6	118.10 (13)	C17—C16—H16	119.4

C8—C7—C9	122.26 (14)	C11^i—C17—C18	118.88 (13)
C6—C7—C9	119.65 (14)	C11^i—C17—C16	122.40 (14)
C7—C8—C3	122.53 (14)	C18—C17—C16	118.72 (13)
C7—C8—H8	118.7	C14^i—C18—C17	120.17 (13)
C3—C8—H8	118.7	C14^i—C18—C18^i	119.80 (16)
O4—C9—C7	120.50 (15)	C17—C18—C18^i	120.04 (16)
O1—C1—C3—C8	−179.71 (15)	C6—C7—C9—O4	0.3 (2)
C2—C1—C3—C8	0.3 (2)	C8—C7—C9—C10	−0.3 (2)
O1—C1—C3—C4	−1.5 (2)	C6—C7—C9—C10	−179.83 (13)
C2—C1—C3—C4	178.49 (14)	N1—C11—C12—C13	180.0 (2)
C8—C3—C4—O2	179.86 (14)	C17^i—C11—C12—C13	−1.6 (2)
C1—C3—C4—O2	1.6 (2)	C11—C12—C13—N1B	173.1 (9)
C8—C3—C4—C5	−0.3 (2)	C11—C12—C13—C14	1.3 (2)
C1—C3—C4—C5	−178.61 (14)	N1B—C13—C14—C18^i	−170.7 (11)
O2—C4—C5—C6	−179.90 (14)	C12—C13—C14—C18^i	−0.3 (2)
C3—C4—C5—C6	0.3 (2)	N1B—C13—C14—C15	9.6 (11)
C4—C5—C6—O3	−179.61 (14)	C12—C13—C14—C15	−179.94 (14)
C4—C5—C6—C7	−0.2 (2)	C13—C14—C15—C16	−179.26 (14)
O3—C6—C7—C8	179.53 (13)	C18^i—C14—C15—C16	1.1 (2)
C5—C6—C7—C8	0.1 (2)	C14—C15—C16—C17	−1.3 (2)
O3—C6—C7—C9	−1.0 (2)	C15—C16—C17—C11^i	−179.63 (14)
C5—C6—C7—C9	179.64 (13)	C15—C16—C17—C18	0.6 (2)
C6—C7—C8—C3	−0.2 (2)	C11^i—C17—C18—C14^i	0.1 (2)
C9—C7—C8—C3	−179.68 (13)	C16—C17—C18—C14^i	179.82 (13)
C4—C3—C8—C7	0.3 (2)	C11^i—C17—C18—C18^i	−179.52 (15)
C1—C3—C8—C7	178.53 (13)	C16—C17—C18—C18^i	0.2 (2)
C8—C7—C9—O4	179.78 (14)		

Symmetry code: (i) −x+1, −y+1, −z+1.

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1	0.85 (3)	1.80 (3)	2.545 (2)	146 (2)
O3—H3···O4	0.84 (2)	1.80 (2)	2.542 (2)	147 (2)
N1—H1*A*···O3^{ii}	0.88 (1)	2.30 (2)	3.131 (3)	156 (4)
N1—H1*B*···O1	0.88 (1)	2.16 (2)	2.966 (3)	154 (3)
N1*B*—H1*C*···O2^{iii}	0.88 (1)	2.05 (6)	2.902 (18)	162 (17)
N1*B*—H1*D*···O4^{iv}	0.88 (1)	1.89 (6)	2.731 (18)	159 (14)

Symmetry codes: (ii) −x+3/2, y−1/2, −z+1/2; (iii) −x+3/2, −y+1/2, −z+1; (iv) x, y, z+1.