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Crystal structures and hydrogen bonding in the co-crystalline adducts of 3,5-dinitrobenzoic acid with 4-aminosalicylic acid and 2-hydroxy-3-(1*H*-indol-3-yl)propenoic acid

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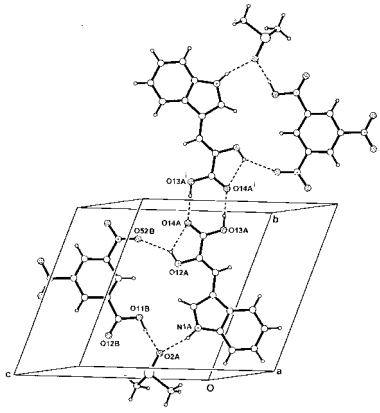
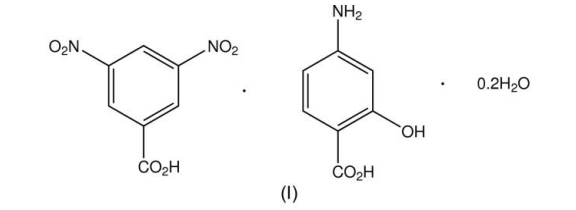
The structures of the co-crystalline adducts of 3,5-dinitrobenzoic acid (3,5-DNBA) with 4-aminosalicylic acid (PASA), the 1:1 partial hydrate, $C_7H_4N_2O_6\cdot C_7H_7NO_3\cdot 0.2H_2O$, (I), and with 2-hydroxy-3-(1*H*-indol-3-yl)propenoic acid (HIPA), the 1:1:1 d^6-dimethyl sulfoxide solvate, $C_7H_4N_2O_6\cdot C_{11}H_9NO_3\cdot C_2D_6OS$, (II), are reported. The crystal substructure of (I) comprises two centrosymmetric hydrogen-bonded $R_2^2(8)$ homodimers, one with 3,5-DNBA, the other with PASA, and an $R_2^2(8)$ 3,5-DNBA–PASA heterodimer. In the crystal, inter-unit amine N—H···O and water O—H···O hydrogen bonds generate a three-dimensional supramolecular structure. In (II), the asymmetric unit consists of the three constituent molecules, which form an essentially planar cyclic hydrogen-bonded heterotrimer unit [graph set $R_3^2(17)$] through carboxyl, hydroxy and amino groups. These units associate across a crystallographic inversion centre through the HIPA carboxylic acid group in an $R_2^2(8)$ hydrogen-bonding association, giving a zero-dimensional structure lying parallel to (100). In both structures, π–π interactions are present [minimum ring-centroid separations = 3.6471 (18) Å in (I) and 3.5819 (10) Å in (II)].

1. Chemical context

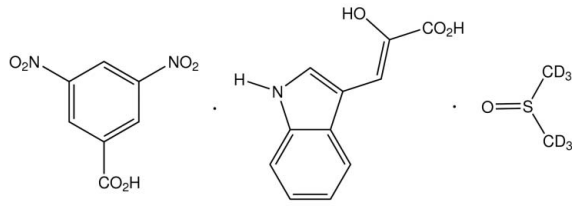
3,5-Dinitrobenzoic acid (3,5-DNBA) has been an important acid for the formation of crystalline materials, which have allowed structural characterization using single crystal X-ray methods. Most commonly proton-transfer salts are formed with organic Lewis bases, *e.g.* with 1-*H*-pyrazole (Aakeröy *et al.*, 2012) but salt–adducts are also known, *e.g.* 2-pyridyl-4′-pyridinium$^+$–3,5-DNBA$^-$–3,5-DNBA (1/1/1) (Chantrapromma *et al.*, 2002). Although co-crystalline non-transfer molecular adducts with 3,5-DNBA are now relatively common, interest was stimulated with the original reporting of non-transfer adduct formation with 4-aminobenzoic acid to form a chiral 1:1 co-crystalline material (Etter & Frankenbach, 1989), which represented one of the earliest examples of designed crystal engineering, in that case with a view to producing non-linear optical materials. In the crystalline state, carboxylic acids usually form cyclic hydrogen-bonded dimers through head-to-head carboxyl O—H···O hydrogen bonds (Leiserowitz, 1976) [graph set $R_2^2(8)$]. This is the case with 3,5-DNBA (*A*), which when co-crystallized with certain aromatic acids, *e.g.* 4-(*N*,*N*-dimethylamino)benzoic acid (*B*), gives separate mixed *AA* and *BB* homodimer pairs (Sharma *et al.*, 1993). Although uncommon with 3,5-DNBA, with other aromatic acid analogues, *AB* heterodimer formation appears more prevalent, *e.g.* the 1:1 adducts of 3,5-dinitrocinnamic acid with



4-(*N,N*-dimethylamino)benzoic acid and 2,4-dinitrocinnamic acid with 2,5-dimethoxycinnamic acid (Sharma *et al.*, 1993). In both *AA* and *BB* structure types, π–π interactions are commonly involved in stabilization, usually accompanied by enhanced colour generation. Absence of dimer pairs in 3,5-DNBA adducts is usually the result of preferential hydrogen bonding with solvent molecules, such as is found in the structure of 3,5-DNBA–phenoxyacetic acid–water (2/1/1) (Lynch *et al.*, 1991), in which a cyclic $R_3^3(10)$ interaction is found, involving two 3,5-DNBA molecules and the water molecule. The title adducts $C_7H_4N_2O_6\cdot C_7H_7NO_3\cdot 0.2H_2O$ (I) and $C_7H_4N_2O_6\cdot C_{11}H_9NO_3\cdot C_2D_6OS$ (II) were prepared from the interaction of 3,5-DNBA with 4-aminosalicylic acid (PASA) and 2-hydroxy-3-(1*H*-indol-3-yl)propenoic acid (HIPA), respectively, and the structures are reported herein. With (II), the incorporation of C_2D_6OS resulted from recrystallization from d^6-dimethylsulfoxide.



(I)



(II)

2. Structural commentary

In the co-crystal of 3,5-DNBA with 4-aminosalicylic acid, (I) (Fig. 1), the asymmetric unit consists of two PASA molecules (*A* and *B*), two 3,5-DNBA molecules (*C* and *D*) and a partially occupied water molecule of solvation (O1*W*), with site occupancy = 0.4. However, what is most unusual in this structure is

Table 1
Hydrogen-bond geometry (Å, °) for (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O11*A*—H11*A*···O12*A*^{i}	0.91 (3)	1.78 (3)	2.678 (3)	175 (3)
O11*B*—H11*B*···O11*D*	0.94 (3)	1.74 (3)	2.673 (3)	175 (3)
O11*C*—H11*C*···O12*C*^{ii}	0.91 (3)	1.73 (3)	2.640 (3)	177 (2)
O12*D*—H12*D*···O12*B*	0.90 (3)	1.71 (3)	2.610 (3)	176 (2)
N4*B*—H41*B*···O31*C*	0.86 (3)	2.58 (3)	3.350 (4)	150 (3)
N4*B*—H42*B*···O52*D*^{iii}	0.85 (2)	2.44 (3)	3.210 (4)	151 (3)
O2*A*—H2*A*···O12*A*	0.84	1.89	2.625 (3)	145
O2*B*—H2*B*···O12*B*	0.84	1.85	2.587 (3)	145
O1*W*—H11*W*···O2*B*	0.90	2.05	2.952 (6)	179
O1*W*—H12*W*···O32*C*^{iv}	0.93	2.08	3.005 (5)	179
C3*B*—H3*B*···O31*C*	0.95	2.58	3.382 (3)	142
C4*D*—H4*D*···O32*C*^{v}	0.95	2.49	3.425 (3)	170

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

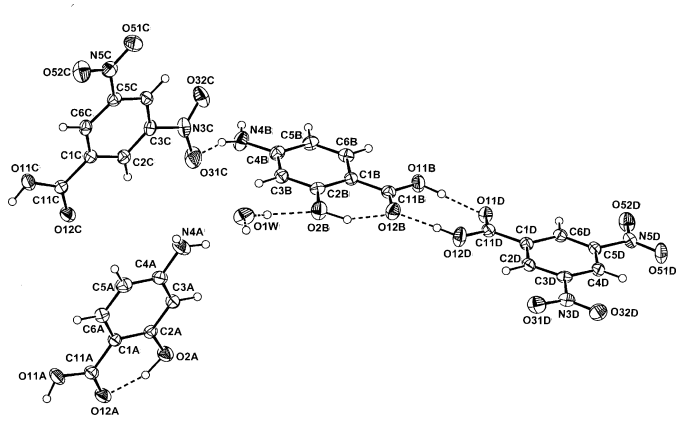


Figure 1
Molecular conformation and atom-naming scheme for the two PABA molecules (*A* and *B*), the two 3,5-DNBA molecules (*C* and *D*) and the disordered water molecule (O1*W*) in the asymmetric unit of adduct (I), with displacement ellipsoids drawn at the 40% probability level. Inter-species hydrogen bonds are shown as dashed lines.

the presence of not four homodimers in the unit cell, but two homodimers (centrosymmetric PASA *A*–*A*^{i} and 3,5-DNBA *C*–*C*^{ii} pairs), as well as two heterodimer *B*–*D* pairs (for symmetry codes, see Table 1). All dimers are formed through the common cyclic $R_2^2(8)$ ring motif. Present in the PASA molecules are the expected intramolecular salicylic acid phenolic O—H···O$_{carboxyl}$ hydrogen bonds, also present in the parent acid (Montis & Hursthouse, 2012).

In the ternary co-crystal of 3,5-DNBA (*B*) with 2-hydroxy-3-(1*H*-indol-3-yl)propenoic acid (*A*) and d^6-dimethylsulfoxide (*C*), (II) the three molecules inter-associate through carboxylic acid O—H···O and N—H···O hydrogen bonds, forming a cyclic $R_3^2(17)$ heterotrimeric asymmetric unit (Fig. 2).

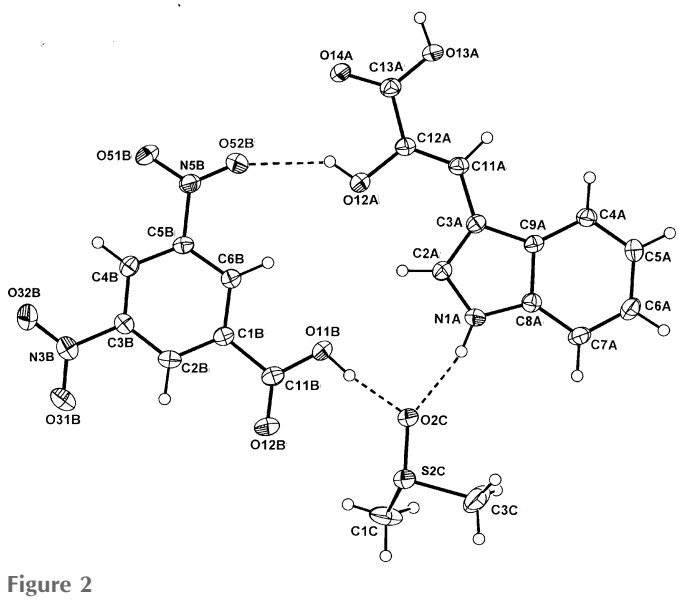


Figure 2
Molecular conformation and atom-naming scheme for adduct (II), with displacement ellipsoids drawn at the 40% probability level. Inter-species hydrogen bonds are shown as dashed lines.

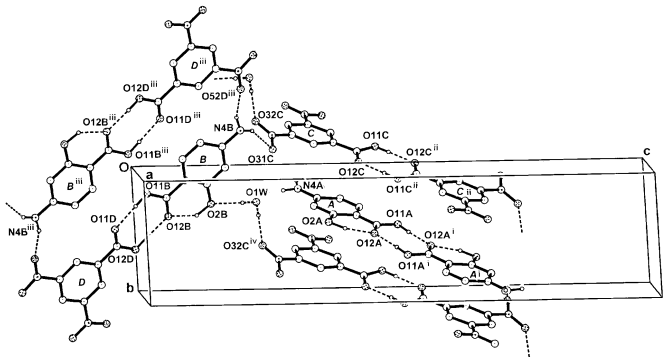


Figure 3
A partial expansion in the three-dimensional hydrogen-bonded structure of the adduct (I) in the unit cell, viewed down *a*. Non-associative H atoms have been omitted. For symmetry codes, see Table 1.

This unit is essentially planar with a dihedral angle of 4.97 (7)° between the indole ring of *A* and the benzene ring of *B*. With the HIPA molecule there is a maximum deviation from the least-squares plane of the 15-atom molecule of 0.120 (2) Å (C6*A*). The planar conformation of the acid side chain in this molecule is maintained by the presence of delocalization extending from C2*A* of the ring to O14*A* of the carboxylic acid group [torsion angle C11*A*—C12*A*—C13*A*—O14*A* = −177.43 (16)°]. This is also found in the parent acid, which has the similar *enol* configuration as in (II) [corresponding torsion angle 170.0 (3)°] with an *E* orientation and in the crystal forms a centrosymmetric homodimer with an $R_2^2(8)$ hydrogen-bond motif (Okabe & Adachi, 1998).

In the adducts (I) and (II), the 3,5-DNBA molecules are essentially planar with the exception of the C3-nitro groups of the *C* molecule in (I), and the *B* molecule in (II), where the defining C2—C3—N3—O32 torsion angles are 158.2 (3) and 168.39 (17)°, respectively. The overall torsion angle range for the remaining groups in both (I) and (II) is 170.8 (3)–179.2 (2)°. These minor deviations from planarity are consistent with conformational features of both polymorphs of the parent acid 3,5-DNBA (Prince *et al.*, 1991) and in examples both of its salts (Aakeröy *et al.*, 2012) and its adducts (Aakeröy *et al.*, 2001; Jones *et al.*, 2010; Chadwick *et al.*, 2009).

3. Supramolecular features

In the supramolecular structure of (I), the carboxylic acid dimers are extended through inter-dimer or inter-heterodimer amine N—H···O and water O—H···O hydrogen bonds (Table 1), giving a three-dimensional framework structure (Fig. 3). Within the structure there are a number of inter-ring π–π associations [ring-centroid separations: $A\cdots C^{vi}$, 3.7542 (16); $A\cdots C^{vii}$, 3.6471 (16); $B\cdots D^{viii}$, 3.6785 (14) Å] [symmetry codes: (vi) $x+1, y-1, z$; (vii) $x, y-1, z$; (viii) $-x+1, -y+1, -z$]. The $B\cdots D$ heterodimers in the π–π association are not only related by inversion but are cyclically linked by the amine N4*B*—H···O52*D*^{iii} hydrogen bond, forming an enlarged $R_2^2(32)$ ring motif. This cyclic relationship with associated π–π bonding is also found in some aromatic homodimer carboxylic acid structures (Sharma *et al.*, 1993). In (I), the disordered water molecule also provides a link between the *B* molecule [the phenolic O2*B* acceptor] and the *C* molecule [the nitro O32*C*^{iv} acceptor]. Also present in the structure are two very weak C—H···O$_{nitro}$ interactions [C3*B*—H···O31*C* 3.382 (3) and C4*D*—H···O32*C*^{v} 3.425 (3) Å; Table 1]. The H atoms of the N4*A* amine group have no acceptors with the PASA *A* homodimer unassociated in the overall structure except for the previously mentioned π–π ring interactions.

Table 2
Hydrogen-bond geometry (Å, °) for (II).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O2*C*	0.87 (2)	2.02 (2)	2.856 (2)	161 (2)
O11*B*—H11*B*···O2*C*	0.88 (2)	1.72 (2)	2.591 (2)	174 (2)
O12*A*—H12*A*···O14*A*	0.88 (2)	2.15 (2)	2.672 (2)	118 (2)
O12*A*—H12*A*···O52*B*	0.88 (2)	2.20 (2)	2.951 (2)	144 (2)
O13*A*—H13*A*···O14*A*^{i}	0.90 (2)	1.75 (2)	2.644 (2)	178 (2)
C1*C*—D12*C*···O14*A*^{ii}	0.98	2.56	3.472 (3)	155
C1*C*—D13*C*···O12*B*^{iii}	0.98	2.52	3.372 (3)	145

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

In (II) the hydrogen-bonded heterotrimer units associate across a crystallographic inversion centre through the HIPA carboxylic acid group [O13*A*—H···O14*A*]^{i} in a cyclic $R_2^2(8)$ hydrogen-bonding association, giving a zero-dimensional heterohexamer structure which is essentially planar and lies parallel to (100) (Fig. 4). Only two very weak intermolecular d^6-DMSO methyl C—H···O interactions are present between

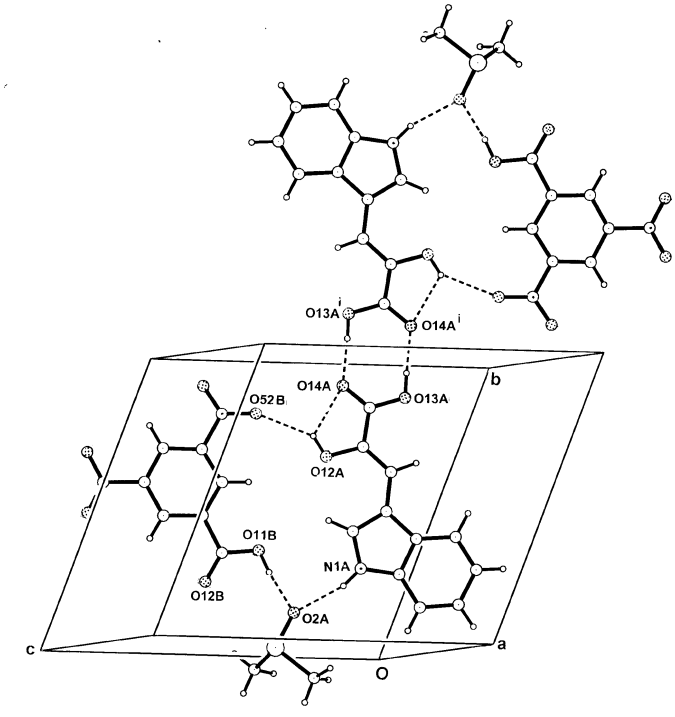


Figure 4
The centrosymmetric hydrogen-bonded heterohexameric structure of the adduct (II) in the unit cell, viewed down *a*. For symmetry code (i), see Table 2.

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_7H_4N_2O_6{\cdot}C_7H_7NO_3{\cdot}0.2H_2O$	$C_7H_4N_2O_6{\cdot}C_{11}H_9NO_3{\cdot}C_2D_6OS$
M_r	368.86	499.49
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
Temperature (K)	200	200
a, b, c (Å)	7.0717 (5), 7.5974 (4), 28.7175 (19)	7.6488 (6), 12.3552 (10), 13.3768 (10)
α, β, γ (°)	87.926 (5), 86.498 (6), 87.584 (5)	116.833 (8), 96.274 (6), 97.626 (7)
V (Å^3)	1537.77 (17)	1097.40 (18)
Z	4	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.14	0.21
Crystal size (mm)	0.35 × 0.35 × 0.30	0.45 × 0.40 × 0.32
Data collection		
Diffractometer	Oxford Diffraction Gemini-S CCD detector	Oxford Diffraction Gemini-S CCD detector
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2013)	Multi-scan (*CrysAlis PRO*; Agilent, 2013)
T_{min}, T_{max}	0.966, 0.990	0.94, 0.98
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10302, 6044, 4158	7457, 4310, 3490
R_{int}	0.027	0.023
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617	0.617
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.149, 1.01	0.040, 0.097, 1.02
No. of reflections	6044	4310
No. of parameters	502	319
No. of restraints	8	4
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.86, −0.28	0.26, −0.25

Computer programs: *CrysAlis PRO* (Agilent, 2013), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) within *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

these units interactions [C1*C*—D···O14*A*^{ii} 3.472 (3) and C1*C*—D···O12*B*^{iii} 3.372 (3) Å; Table 2]. In the structure, π–π interactions are also present between the benzene rings of the *A* and *B*^{viii} molecules] [minimum ring-centroid separation 3.5819 (10) Å; symmetry code: (viii) $-x, -y+2, -z+1$].

4. Synthesis and crystallization

The title co-crystalline adducts (I) and (II) were prepared by dissolving equimolar quantities of 3,5-dinitrobenzoic acid and the respective acids 4-aminosalicylic acid [for (I)] or (1*H*-indol-3-yl)propenoic acid [for (II)] in ethanol and heating under reflux for 5 min after which room-temperature evaporation of the solutions gave for (I), yellow prisms and for (II), a red powder. This latter compound was dissolved in d^6-deuterated DMSO and solvent diffusion of water into this solution gave red prisms of (II). Specimens were cleaved from both prismatic crystals for the X-ray analyses.

5. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 3. Hydrogen atoms on all potentially interactive O—H and N—H groups in all molecular species were located by difference-Fourier methods and positional and displacement parameters were refined for all but those of the phenolic O2*A* and O2*B* groups and on the disordered water molecule O1*W*, with riding restraints [O—H bond length = 0.90 (2) Å and U_{iso}(H) = 1.5U_{eq}(O) or N—H = 0.88 (2) Å, with U_{iso}(H) = 1.2U_{eq}(N)]. The phenolic and water H atoms were set invariant with U_{iso}(H) = 1.2U_{eq}(O). Other H atoms were included in the refinement at calculated positions [C—H (aromatic) = 0.95 or (methylene) 0.99 Å] , with U_{iso}(H) = 1.2U_{eq}(C), using a riding-model approximation. The site-occupancy factor for the disordered water molecule of solvation was determined as 0.403 (4) [for the (2:2) 3,5-DNBA:PASA pair in the asymmetric unit] and was subsequently fixed as 0.40. In the structure of (I), the relatively large maximum residual electron density (0.835 e Å^{-3}) was located 0.80 Å from H6*B*.

Acknowledgements

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supporting information

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Crystal structures and hydrogen bonding in the co-crystalline adducts of 3,5-dinitrobenzoic acid with 4-aminosalicylic acid and 2-hydroxy-3-(1*H*-indol-3-yl)propenoic acid

Graham Smith and Daniel E. Lynch

Computing details

For both compounds, data collection: *CrysAlis PRO* (Agilent, 2013); cell refinement: *CrysAlis PRO* (Agilent, 2013); data reduction: *CrysAlis PRO* (Agilent, 2013); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) within *WinGX* (Farrugia, 2012); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON* (Spek, 2009).

(I) 4-Amino-2-hydroxybenzoic acid–3,5-dinitrobenzoic acid–water (2/2/0.4)

Crystal data

$C_7H_4N_2O_6 \cdot C_7H_7NO_3 \cdot 0.2H_2O$
$M_r = 368.86$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.0717\ (5)$ Å
$b = 7.5974\ (4)$ Å
$c = 28.7175\ (19)$ Å
$\alpha = 87.926\ (5)°$
$\beta = 86.498\ (6)°$
$\gamma = 87.584\ (5)°$
$V = 1537.77\ (17)$ Å^3
$Z = 4$
$F(000) = 760$
$D_x = 1.593$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2357 reflections
$\theta = 3.3–27.2°$
$\mu = 0.14$ mm^{-1}
$T = 200$ K
Block, yellow
0.35 × 0.35 × 0.30 mm

Data collection

Oxford Diffraction Gemini-S CCD detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2013)
$T_{min} = 0.966$, $T_{max} = 0.990$
10302 measured reflections
6044 independent reflections
4158 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.1°$
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 8$
$l = -35 \rightarrow 31$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.149$
$S = 1.01$
6044 reflections
502 parameters
8 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0599P)^2 + 0.6456P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} < 0.001$
H atoms treated by a mixture of independent and constrained refinement	$\Delta\rho_{max} = 0.86$ e Å^{-3}
	$\Delta\rho_{min} = -0.28$ e Å^{-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 of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness 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 threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O11C	−0.1050 (3)	1.2197 (3)	0.48465 (7)	0.0453 (7)	
O12C	−0.0102 (3)	0.9810 (2)	0.44480 (6)	0.0412 (7)	
O31C	−0.1244 (4)	0.9769 (3)	0.27862 (8)	0.0629 (9)	
O32C	−0.3286 (3)	1.1671 (3)	0.25221 (7)	0.0562 (8)	
O51C	−0.3819 (3)	1.7339 (3)	0.32553 (8)	0.0560 (9)	
O52C	−0.2958 (4)	1.7528 (3)	0.39588 (8)	0.0573 (9)	
N3C	−0.2215 (4)	1.1123 (3)	0.28179 (8)	0.0409 (9)	
N5C	−0.3155 (4)	1.6703 (3)	0.36123 (9)	0.0396 (8)	
C1C	−0.1424 (4)	1.2259 (3)	0.40352 (9)	0.0284 (8)	
C2C	−0.1454 (4)	1.1313 (3)	0.36322 (9)	0.0299 (8)	
C3C	−0.2105 (4)	1.2152 (3)	0.32363 (9)	0.0302 (8)	
C4C	−0.2689 (4)	1.3908 (3)	0.32177 (9)	0.0310 (8)	
C5C	−0.2592 (4)	1.4815 (3)	0.36191 (9)	0.0299 (8)	
C6C	−0.1988 (4)	1.4038 (3)	0.40322 (9)	0.0307 (8)	
C11C	−0.0797 (4)	1.1307 (3)	0.44630 (9)	0.0305 (8)	
O11D	0.4620 (3)	0.6430 (2)	−0.04675 (6)	0.0354 (6)	
O12D	0.6448 (3)	0.8050 (3)	−0.00552 (7)	0.0467 (8)	
O31D	1.2556 (3)	0.9762 (2)	−0.06601 (7)	0.0462 (8)	
O32D	1.3586 (3)	0.9146 (3)	−0.13627 (8)	0.0477 (8)	
O51D	0.9545 (3)	0.6273 (3)	−0.23683 (7)	0.0493 (8)	
O52D	0.6769 (3)	0.5502 (3)	−0.21049 (7)	0.0479 (8)	
N3D	1.2378 (3)	0.9137 (3)	−0.10404 (9)	0.0352 (8)	
N5D	0.8272 (3)	0.6196 (3)	−0.20596 (8)	0.0337 (8)	
C1D	0.7529 (4)	0.7425 (3)	−0.08154 (9)	0.0279 (8)	
C2D	0.9234 (4)	0.8206 (3)	−0.07471 (9)	0.0291 (8)	
C3D	1.0568 (4)	0.8320 (3)	−0.11161 (9)	0.0282 (8)	
C4D	1.0307 (4)	0.7687 (3)	−0.15504 (9)	0.0292 (8)	
C5D	0.8599 (4)	0.6923 (3)	−0.16043 (8)	0.0273 (8)	
C6D	0.7212 (4)	0.6777 (3)	−0.12500 (9)	0.0284 (8)	
C11D	0.6060 (4)	0.7264 (3)	−0.04270 (9)	0.0306 (8)	

O2A	0.3595 (3)	0.0813 (2)	0.35993 (7)	0.0414 (7)	
O11A	0.4058 (3)	0.2349 (3)	0.49746 (7)	0.0460 (8)	
O12A	0.4496 (3)	0.0192 (2)	0.44648 (7)	0.0413 (7)	
N4A	0.1581 (4)	0.6629 (4)	0.31680 (10)	0.0531 (10)	
C1A	0.3356 (4)	0.3003 (3)	0.41957 (9)	0.0293 (8)	
C2A	0.3186 (4)	0.2488 (3)	0.37348 (9)	0.0299 (8)	
C3A	0.2632 (4)	0.3695 (4)	0.33956 (9)	0.0339 (9)	
C4A	0.2202 (4)	0.5441 (4)	0.35021 (10)	0.0360 (9)	
C5A	0.2345 (4)	0.5972 (4)	0.39630 (10)	0.0390 (10)	
C6A	0.2913 (4)	0.4775 (3)	0.42978 (10)	0.0347 (9)	
C11A	0.4010 (4)	0.1748 (3)	0.45526 (9)	0.0321 (9)	
O2B	0.2909 (3)	0.8426 (3)	0.14868 (7)	0.0481 (8)	
O11B	0.1793 (3)	0.6474 (3)	0.01942 (7)	0.0421 (7)	
O12B	0.3803 (3)	0.7848 (2)	0.06171 (7)	0.0412 (7)	
N4B	−0.3161 (4)	0.6924 (4)	0.21044 (10)	0.0465 (9)	
C1B	0.0835 (4)	0.7114 (3)	0.09718 (9)	0.0280 (8)	
C2B	0.1231 (4)	0.7741 (3)	0.14100 (9)	0.0282 (8)	
C3B	−0.0089 (4)	0.7679 (3)	0.17821 (9)	0.0314 (8)	
C4B	−0.1867 (4)	0.6999 (3)	0.17321 (10)	0.0333 (9)	
C5B	−0.2278 (4)	0.6362 (3)	0.12966 (10)	0.0348 (9)	
C6B	−0.0968 (4)	0.6421 (3)	0.09281 (10)	0.0324 (9)	
C11B	0.2232 (4)	0.7176 (3)	0.05843 (9)	0.0307 (8)	
O1W	0.3006 (7)	0.9987 (7)	0.24093 (19)	0.0511 (19)	0.400
H2C	−0.10340	1.01090	0.36290	0.0360*	
H4C	−0.31350	1.44600	0.29410	0.0370*	
H6C	−0.19590	1.47020	0.43050	0.0370*	
H11C	−0.067 (5)	1.148 (4)	0.5085 (9)	0.0680*	
H2D	0.94710	0.86520	−0.04520	0.0350*	
H4D	1.12510	0.77710	−0.17990	0.0350*	
H6D	0.60550	0.62430	−0.13010	0.0340*	
H12D	0.552 (4)	0.793 (4)	0.0173 (10)	0.0700*	
H2A	0.39860	0.02000	0.38260	0.0620*	
H3A	0.25440	0.33300	0.30850	0.0410*	
H5A	0.20480	0.71610	0.40410	0.0470*	
H6A	0.30110	0.51490	0.46070	0.0420*	
H11A	0.460 (5)	0.148 (4)	0.5152 (11)	0.0690*	
H41A	0.167 (5)	0.636 (4)	0.2874 (7)	0.0640*	
H42A	0.173 (5)	0.773 (3)	0.3223 (12)	0.0640*	
H2B	0.36360	0.83470	0.12460	0.0720*	
H3B	0.02110	0.81010	0.20750	0.0380*	
H5B	−0.34780	0.58860	0.12590	0.0420*	
H6B	−0.12730	0.59900	0.06370	0.0390*	
H11B	0.279 (4)	0.653 (4)	−0.0035 (10)	0.0630*	
H41B	−0.304 (5)	0.751 (4)	0.2349 (8)	0.0560*	
H42B	−0.424 (3)	0.659 (4)	0.2034 (12)	0.0560*	
H11W	0.29970	0.95200	0.21260	0.0760*	0.400
H12W	0.41470	1.05100	0.24460	0.0760*	0.400

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O11C	0.0629 (15)	0.0449 (12)	0.0267 (11)	0.0126 (10)	−0.0028 (10)	0.0005 (8)
O12C	0.0515 (14)	0.0378 (11)	0.0336 (11)	0.0079 (9)	−0.0057 (10)	0.0021 (8)
O31C	0.097 (2)	0.0468 (13)	0.0441 (14)	0.0108 (13)	0.0001 (13)	−0.0157 (10)
O32C	0.0653 (16)	0.0726 (15)	0.0330 (12)	−0.0158 (12)	−0.0147 (12)	−0.0005 (11)
O51C	0.0687 (17)	0.0423 (12)	0.0548 (15)	0.0133 (11)	−0.0044 (12)	0.0108 (10)
O52C	0.0886 (19)	0.0330 (11)	0.0500 (14)	−0.0017 (11)	0.0021 (13)	−0.0088 (10)
N3C	0.0536 (17)	0.0410 (14)	0.0282 (13)	−0.0132 (12)	0.0038 (12)	−0.0015 (10)
N5C	0.0421 (15)	0.0323 (13)	0.0427 (15)	0.0006 (11)	0.0065 (12)	0.0045 (11)
C1C	0.0236 (14)	0.0330 (14)	0.0281 (14)	−0.0024 (11)	0.0015 (11)	0.0021 (10)
C2C	0.0303 (15)	0.0279 (13)	0.0309 (15)	−0.0028 (11)	0.0021 (12)	0.0014 (11)
C3C	0.0312 (15)	0.0318 (14)	0.0273 (14)	−0.0069 (11)	0.0044 (11)	−0.0024 (11)
C4C	0.0248 (14)	0.0394 (15)	0.0283 (14)	−0.0050 (11)	−0.0001 (11)	0.0062 (11)
C5C	0.0259 (14)	0.0290 (13)	0.0340 (15)	−0.0019 (11)	0.0034 (12)	0.0005 (11)
C6C	0.0288 (15)	0.0358 (14)	0.0273 (14)	−0.0065 (11)	0.0036 (11)	−0.0013 (11)
C11C	0.0269 (14)	0.0350 (15)	0.0295 (15)	−0.0011 (11)	−0.0014 (11)	0.0001 (11)
O11D	0.0368 (12)	0.0388 (10)	0.0309 (11)	−0.0069 (9)	0.0016 (9)	−0.0034 (8)
O12D	0.0429 (13)	0.0667 (14)	0.0316 (12)	−0.0123 (11)	0.0053 (9)	−0.0173 (10)
O31D	0.0454 (13)	0.0455 (12)	0.0505 (14)	−0.0058 (10)	−0.0148 (10)	−0.0141 (10)
O32D	0.0302 (12)	0.0610 (13)	0.0517 (14)	−0.0065 (10)	−0.0005 (10)	0.0018 (10)
O51D	0.0509 (14)	0.0709 (14)	0.0262 (11)	−0.0136 (11)	0.0059 (10)	−0.0057 (9)
O52D	0.0460 (14)	0.0623 (13)	0.0379 (12)	−0.0188 (11)	−0.0070 (10)	−0.0087 (10)
N3D	0.0331 (14)	0.0318 (12)	0.0410 (15)	0.0006 (10)	−0.0077 (12)	0.0012 (10)
N5D	0.0409 (15)	0.0354 (12)	0.0254 (13)	−0.0039 (10)	−0.0059 (11)	0.0015 (9)
C1D	0.0338 (15)	0.0234 (12)	0.0264 (14)	0.0014 (11)	−0.0041 (11)	−0.0008 (10)
C2D	0.0383 (16)	0.0243 (13)	0.0251 (14)	0.0048 (11)	−0.0074 (12)	−0.0040 (10)
C3D	0.0284 (14)	0.0204 (12)	0.0360 (15)	0.0015 (10)	−0.0068 (12)	−0.0001 (10)
C4D	0.0337 (15)	0.0261 (13)	0.0271 (14)	0.0022 (11)	0.0001 (12)	0.0023 (10)
C5D	0.0341 (15)	0.0252 (12)	0.0228 (13)	0.0005 (11)	−0.0051 (11)	0.0008 (10)
C6D	0.0303 (15)	0.0255 (13)	0.0296 (14)	−0.0005 (11)	−0.0041 (12)	0.0000 (10)
C11D	0.0344 (16)	0.0300 (14)	0.0273 (14)	0.0032 (12)	−0.0043 (12)	−0.0024 (11)
O2A	0.0521 (14)	0.0347 (11)	0.0376 (12)	0.0061 (9)	−0.0087 (10)	−0.0013 (8)
O11A	0.0634 (16)	0.0445 (12)	0.0300 (12)	0.0093 (10)	−0.0124 (10)	0.0038 (9)
O12A	0.0514 (13)	0.0353 (11)	0.0367 (11)	0.0072 (9)	−0.0081 (10)	0.0050 (8)
N4A	0.065 (2)	0.0487 (16)	0.0446 (16)	0.0085 (15)	−0.0121 (15)	0.0137 (14)
C1A	0.0250 (14)	0.0340 (14)	0.0285 (14)	−0.0005 (11)	−0.0007 (11)	0.0037 (10)
C2A	0.0236 (14)	0.0331 (14)	0.0322 (15)	0.0007 (11)	0.0016 (11)	0.0008 (11)
C3A	0.0293 (15)	0.0427 (16)	0.0293 (15)	0.0000 (12)	−0.0017 (12)	0.0025 (11)
C4A	0.0292 (16)	0.0401 (16)	0.0377 (16)	0.0006 (12)	−0.0008 (13)	0.0090 (12)
C5A	0.0395 (17)	0.0323 (15)	0.0446 (18)	0.0018 (12)	−0.0019 (14)	0.0009 (12)
C6A	0.0354 (16)	0.0346 (15)	0.0339 (16)	0.0004 (12)	−0.0021 (13)	−0.0011 (11)
C11A	0.0278 (15)	0.0372 (15)	0.0309 (15)	−0.0016 (11)	−0.0019 (12)	0.0053 (11)
O2B	0.0414 (13)	0.0614 (13)	0.0422 (13)	−0.0122 (10)	0.0013 (10)	−0.0081 (10)
O11B	0.0445 (13)	0.0539 (12)	0.0281 (11)	−0.0045 (10)	0.0005 (9)	−0.0058 (9)
O12B	0.0357 (12)	0.0495 (12)	0.0383 (12)	−0.0072 (9)	0.0045 (9)	−0.0053 (9)
N4B	0.0396 (16)	0.0567 (17)	0.0433 (16)	−0.0129 (13)	0.0087 (13)	−0.0106 (12)

C1B	0.0322 (15)	0.0235 (12)	0.0279 (14)	0.0025 (10)	−0.0021 (11)	−0.0010 (10)
C2B	0.0257 (14)	0.0225 (12)	0.0366 (15)	−0.0017 (10)	−0.0033 (12)	−0.0018 (10)
C3B	0.0355 (16)	0.0282 (13)	0.0305 (15)	−0.0003 (11)	−0.0019 (12)	−0.0024 (11)
C4B	0.0324 (16)	0.0276 (13)	0.0390 (16)	0.0003 (11)	0.0028 (13)	−0.0006 (11)
C5B	0.0285 (15)	0.0323 (14)	0.0438 (17)	−0.0029 (11)	−0.0022 (13)	−0.0016 (12)
C6B	0.0335 (16)	0.0282 (13)	0.0362 (16)	−0.0010 (11)	−0.0081 (13)	−0.0021 (11)
C11B	0.0333 (16)	0.0265 (13)	0.0320 (15)	0.0023 (11)	−0.0036 (12)	0.0014 (11)
O1W	0.034 (3)	0.057 (3)	0.066 (4)	−0.010 (2)	−0.017 (3)	−0.022 (3)

Geometric parameters (Å, º)

O11C—C11C	1.312 (3)	C1C—C11C	1.485 (4)
O12C—C11C	1.221 (3)	C2C—C3C	1.378 (4)
O31C—N3C	1.216 (3)	C3C—C4C	1.380 (3)
O32C—N3C	1.223 (3)	C4C—C5C	1.371 (4)
O51C—N5C	1.228 (3)	C5C—C6C	1.389 (4)
O52C—N5C	1.213 (3)	C2C—H2C	0.9500
O11C—H11C	0.91 (3)	C4C—H4C	0.9500
O11D—C11D	1.235 (3)	C6C—H6C	0.9500
O12D—C11D	1.291 (3)	C1D—C6D	1.392 (4)
O31D—N3D	1.222 (3)	C1D—C11D	1.482 (4)
O32D—N3D	1.221 (3)	C1D—C2D	1.394 (4)
O51D—N5D	1.226 (3)	C2D—C3D	1.378 (4)
O52D—N5D	1.222 (3)	C3D—C4D	1.378 (4)
O12D—H12D	0.90 (3)	C4D—C5D	1.381 (4)
O2A—C2A	1.359 (3)	C5D—C6D	1.374 (4)
O11A—C11A	1.313 (3)	C2D—H2D	0.9500
O12A—C11A	1.247 (3)	C4D—H4D	0.9500
O2A—H2A	0.8400	C6D—H6D	0.9500
O11A—H11A	0.91 (3)	C1A—C2A	1.407 (4)
O2B—C2B	1.349 (3)	C1A—C6A	1.407 (3)
O11B—C11B	1.317 (3)	C1A—C11A	1.457 (4)
O12B—C11B	1.252 (3)	C2A—C3A	1.377 (4)
O2B—H2B	0.8400	C3A—C4A	1.389 (4)
O11B—H11B	0.94 (3)	C4A—C5A	1.408 (4)
O1W—H12W	0.9300	C5A—C6A	1.366 (4)
O1W—H11W	0.9000	C3A—H3A	0.9500
N3C—C3C	1.465 (3)	C5A—H5A	0.9500
N5C—C5C	1.472 (3)	C6A—H6A	0.9500
N3D—C3D	1.477 (3)	C1B—C11B	1.443 (4)
N5D—C5D	1.472 (3)	C1B—C6B	1.414 (4)
N4A—C4A	1.372 (4)	C1B—C2B	1.410 (4)
N4A—H41A	0.87 (2)	C2B—C3B	1.376 (4)
N4A—H42A	0.87 (2)	C3B—C4B	1.397 (4)
N4B—C4B	1.365 (4)	C4B—C5B	1.408 (4)
N4B—H41B	0.86 (3)	C5B—C6B	1.364 (4)
N4B—H42B	0.85 (2)	C3B—H3B	0.9500
C1C—C2C	1.386 (4)	C5B—H5B	0.9500

C1C—C6C	1.393 (3)	C6B—H6B	0.9500
C11C—O11C—H11C	107.4 (18)	C4D—C5D—C6D	123.0 (2)
C11D—O12D—H12D	111.4 (19)	N5D—C5D—C6D	118.6 (2)
C2A—O2A—H2A	109.00	C1D—C6D—C5D	119.0 (2)
C11A—O11A—H11A	107 (2)	O11D—C11D—C1D	121.3 (2)
C2B—O2B—H2B	110.00	O12D—C11D—C1D	113.9 (2)
C11B—O11B—H11B	111.6 (18)	O11D—C11D—O12D	124.8 (2)
H11W—O1W—H12W	111.00	C1D—C2D—H2D	121.00
O31C—N3C—O32C	123.8 (2)	C3D—C2D—H2D	121.00
O31C—N3C—C3C	118.1 (2)	C5D—C4D—H4D	122.00
O32C—N3C—C3C	118.1 (2)	C3D—C4D—H4D	122.00
O51C—N5C—C5C	117.5 (2)	C5D—C6D—H6D	121.00
O52C—N5C—C5C	118.4 (2)	C1D—C6D—H6D	121.00
O51C—N5C—O52C	124.1 (2)	C2A—C1A—C11A	121.0 (2)
O31D—N3D—C3D	117.7 (2)	C2A—C1A—C6A	117.8 (2)
O31D—N3D—O32D	124.7 (2)	C6A—C1A—C11A	121.2 (2)
O32D—N3D—C3D	117.7 (2)	C1A—C2A—C3A	120.7 (2)
O51D—N5D—C5D	118.2 (2)	O2A—C2A—C1A	122.5 (2)
O51D—N5D—O52D	123.5 (2)	O2A—C2A—C3A	116.9 (2)
O52D—N5D—C5D	118.2 (2)	C2A—C3A—C4A	120.8 (2)
C4A—N4A—H41A	120 (2)	N4A—C4A—C5A	120.0 (3)
C4A—N4A—H42A	115 (2)	C3A—C4A—C5A	119.2 (3)
H41A—N4A—H42A	116 (3)	N4A—C4A—C3A	120.8 (3)
C4B—N4B—H42B	113 (2)	C4A—C5A—C6A	119.9 (3)
H41B—N4B—H42B	121 (3)	C1A—C6A—C5A	121.6 (3)
C4B—N4B—H41B	122 (2)	O11A—C11A—C1A	116.1 (2)
C2C—C1C—C6C	120.2 (2)	O11A—C11A—O12A	121.7 (2)
C6C—C1C—C11C	122.1 (2)	O12A—C11A—C1A	122.3 (2)
C2C—C1C—C11C	117.8 (2)	C2A—C3A—H3A	120.00
C1C—C2C—C3C	118.7 (2)	C4A—C3A—H3A	120.00
C2C—C3C—C4C	123.1 (2)	C6A—C5A—H5A	120.00
N3C—C3C—C2C	118.4 (2)	C4A—C5A—H5A	120.00
N3C—C3C—C4C	118.5 (2)	C1A—C6A—H6A	119.00
C3C—C4C—C5C	116.6 (2)	C5A—C6A—H6A	119.00
N5C—C5C—C4C	118.5 (2)	C2B—C1B—C11B	120.8 (2)
C4C—C5C—C6C	123.1 (2)	C6B—C1B—C11B	121.6 (2)
N5C—C5C—C6C	118.4 (2)	C2B—C1B—C6B	117.6 (2)
C1C—C6C—C5C	118.2 (2)	O2B—C2B—C3B	116.7 (2)
O12C—C11C—C1C	121.2 (2)	C1B—C2B—C3B	121.2 (3)
O11C—C11C—O12C	124.0 (2)	O2B—C2B—C1B	122.1 (2)
O11C—C11C—C1C	114.9 (2)	C2B—C3B—C4B	120.4 (2)
C3C—C2C—H2C	121.00	N4B—C4B—C3B	120.1 (3)
C1C—C2C—H2C	121.00	C3B—C4B—C5B	118.9 (3)
C3C—C4C—H4C	122.00	N4B—C4B—C5B	121.0 (3)
C5C—C4C—H4C	122.00	C4B—C5B—C6B	120.6 (3)
C1C—C6C—H6C	121.00	C1B—C6B—C5B	121.2 (3)
C5C—C6C—H6C	121.00	O12B—C11B—C1B	121.7 (2)

C6D—C1D—C11D	119.8 (2)	O11B—C11B—C1B	117.1 (2)
C2D—C1D—C6D	119.8 (2)	O11B—C11B—O12B	121.2 (2)
C2D—C1D—C11D	120.4 (2)	C4B—C3B—H3B	120.00
C1D—C2D—C3D	118.6 (2)	C2B—C3B—H3B	120.00
N3D—C3D—C4D	118.4 (2)	C4B—C5B—H5B	120.00
C2D—C3D—C4D	123.2 (3)	C6B—C5B—H5B	120.00
N3D—C3D—C2D	118.4 (2)	C1B—C6B—H6B	119.00
C3D—C4D—C5D	116.4 (2)	C5B—C6B—H6B	119.00
N5D—C5D—C4D	118.3 (2)		

O31C—N3C—C3C—C2C	−21.4 (4)	C1D—C2D—C3D—N3D	−179.5 (2)
O32C—N3C—C3C—C2C	158.2 (3)	C2D—C3D—C4D—C5D	0.5 (4)
O31C—N3C—C3C—C4C	159.7 (3)	N3D—C3D—C4D—C5D	179.6 (2)
O32C—N3C—C3C—C4C	−20.7 (4)	C3D—C4D—C5D—N5D	−178.8 (2)
O51C—N5C—C5C—C4C	5.3 (4)	C3D—C4D—C5D—C6D	−0.3 (4)
O52C—N5C—C5C—C4C	−175.5 (3)	N5D—C5D—C6D—C1D	178.5 (2)
O51C—N5C—C5C—C6C	−174.7 (3)	C4D—C5D—C6D—C1D	0.0 (4)
O52C—N5C—C5C—C6C	4.5 (4)	C6A—C1A—C2A—O2A	179.4 (3)
O31D—N3D—C3D—C4D	176.8 (2)	C6A—C1A—C2A—C3A	1.0 (4)
O32D—N3D—C3D—C4D	−2.9 (3)	C11A—C1A—C2A—O2A	0.7 (4)
O32D—N3D—C3D—C2D	176.2 (2)	C11A—C1A—C2A—C3A	−177.7 (3)
O31D—N3D—C3D—C2D	−4.1 (3)	C2A—C1A—C6A—C5A	−0.4 (4)
O52D—N5D—C5D—C4D	179.2 (2)	C11A—C1A—C6A—C5A	178.3 (3)
O51D—N5D—C5D—C6D	−177.3 (2)	C2A—C1A—C11A—O11A	−177.8 (3)
O51D—N5D—C5D—C4D	1.3 (3)	C2A—C1A—C11A—O12A	2.1 (4)
O52D—N5D—C5D—C6D	0.6 (3)	C6A—C1A—C11A—O11A	3.5 (4)
C2C—C1C—C6C—C5C	0.7 (4)	C6A—C1A—C11A—O12A	−176.5 (3)
C11C—C1C—C2C—C3C	177.4 (3)	O2A—C2A—C3A—C4A	−179.5 (3)
C6C—C1C—C11C—O11C	8.5 (4)	C1A—C2A—C3A—C4A	−1.0 (4)
C2C—C1C—C11C—O12C	9.2 (4)	C2A—C3A—C4A—N4A	−177.5 (3)
C2C—C1C—C11C—O11C	−170.8 (3)	C2A—C3A—C4A—C5A	0.2 (4)
C6C—C1C—C2C—C3C	−2.0 (4)	N4A—C4A—C5A—C6A	178.2 (3)
C6C—C1C—C11C—O12C	−171.4 (3)	C3A—C4A—C5A—C6A	0.4 (4)
C11C—C1C—C6C—C5C	−178.7 (3)	C4A—C5A—C6A—C1A	−0.3 (4)
C1C—C2C—C3C—N3C	−177.2 (3)	C6B—C1B—C2B—O2B	−180.0 (2)
C1C—C2C—C3C—C4C	1.6 (4)	C6B—C1B—C2B—C3B	0.1 (4)
C2C—C3C—C4C—C5C	0.1 (4)	C11B—C1B—C2B—O2B	0.3 (4)
N3C—C3C—C4C—C5C	179.0 (3)	C11B—C1B—C2B—C3B	−179.7 (2)
C3C—C4C—C5C—C6C	−1.5 (4)	C2B—C1B—C6B—C5B	0.0 (4)
C3C—C4C—C5C—N5C	178.5 (3)	C11B—C1B—C6B—C5B	179.8 (2)
C4C—C5C—C6C—C1C	1.1 (4)	C2B—C1B—C11B—O11B	176.3 (2)
N5C—C5C—C6C—C1C	−178.9 (3)	C2B—C1B—C11B—O12B	−3.0 (4)
C2D—C1D—C6D—C5D	0.0 (4)	C6B—C1B—C11B—O11B	−3.5 (4)
C6D—C1D—C2D—C3D	0.2 (4)	C6B—C1B—C11B—O12B	177.3 (2)
C11D—C1D—C2D—C3D	179.6 (2)	O2B—C2B—C3B—C4B	179.6 (2)
C6D—C1D—C11D—O11D	6.3 (4)	C1B—C2B—C3B—C4B	−0.4 (4)
C11D—C1D—C6D—C5D	−179.4 (2)	C2B—C3B—C4B—N4B	179.3 (2)
C6D—C1D—C11D—O12D	−174.2 (2)	C2B—C3B—C4B—C5B	0.7 (4)

C2D—C1D—C11D—O12D	6.4 (3)	N4B—C4B—C5B—C6B	−179.1 (3)
C2D—C1D—C11D—O11D	−173.1 (2)	C3B—C4B—C5B—C6B	−0.6 (4)
C1D—C2D—C3D—C4D	−0.5 (4)	C4B—C5B—C6B—C1B	0.2 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O11*A*—H11*A*···O12*A*^{i}	0.91 (3)	1.78 (3)	2.678 (3)	175 (3)
O11*B*—H11*B*···O11*D*	0.94 (3)	1.74 (3)	2.673 (3)	175 (3)
O11*C*—H11*C*···O12*C*^{ii}	0.91 (3)	1.73 (3)	2.640 (3)	177 (2)
O12*D*—H12*D*···O12*B*	0.90 (3)	1.71 (3)	2.610 (3)	176 (2)
N4*B*—H41*B*···O31*C*	0.86 (3)	2.58 (3)	3.350 (4)	150 (3)
N4*B*—H42*B*···O52*D*^{iii}	0.85 (2)	2.44 (3)	3.210 (4)	151 (3)
O2*A*—H2*A*···O12*A*	0.84	1.89	2.625 (3)	145
O2*B*—H2*B*···O12*B*	0.84	1.85	2.587 (3)	145
O1*W*—H11*W*···O2*B*	0.90	2.05	2.952 (6)	179
O1*W*—H12*W*···O32*C*^{iv}	0.93	2.08	3.005 (5)	179
C3*B*—H3*B*···O31*C*	0.95	2.58	3.382 (3)	142
C4*D*—H4*D*···O32*C*^{v}	0.95	2.49	3.425 (3)	170

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1; (ii) −*x*, −*y*+2, −*z*+1; (iii) −*x*, −*y*+1, −*z*; (iv) *x*+1, *y*, *z*; (v) −*x*+1, −*y*+2, −*z*.

(II) 3,5-Dinitrobenzoic acid–2-hydroxy-3-(1*H*-indol-3-yl)propenoic acid–*d*^{6}-dimethyl sulfoxide (1/1/1)

Crystal data

$C_7H_4N_2O_6·C_{11}H_9NO_3·C_2D_6OS$
M_r = 499.49
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.6488 (6) Å
b = 12.3552 (10) Å
c = 13.3768 (10) Å
α = 116.833 (8)°
β = 96.274 (6)°
γ = 97.626 (7)°
V = 1097.40 (18) Å^3
Z = 2
F(000) = 512
D_x = 1.511 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 2343 reflections
θ = 3.3–28.4°
μ = 0.21 mm^{-1}
T = 200 K
Block, red
0.45 × 0.40 × 0.32 mm

Data collection

Oxford Diffraction Gemini-S CCD detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2013)
T_{min} = 0.94, T_{max} = 0.98
7457 measured reflections
4310 independent reflections
3490 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 26.0°, θ_{min} = 3.3°
h = −9→9
k = −15→15
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.097$
$S = 1.02$
4310 reflections
319 parameters
4 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2 + 0.3315P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-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 esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness 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 threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O11B	0.11950 (19)	0.35376 (12)	0.50949 (11)	0.0386 (5)
O12B	0.0099 (2)	0.24989 (12)	0.59702 (12)	0.0458 (5)
O31B	−0.19578 (19)	0.48461 (14)	0.95269 (12)	0.0438 (5)
O32B	−0.0994 (2)	0.68345 (14)	1.05107 (12)	0.0483 (5)
O51B	0.1446 (2)	0.90405 (13)	0.86272 (12)	0.0504 (5)
O52B	0.2503 (2)	0.80830 (13)	0.71029 (12)	0.0443 (5)
N3B	−0.1163 (2)	0.58414 (16)	0.96484 (14)	0.0353 (6)
N5B	0.1697 (2)	0.80906 (14)	0.78469 (13)	0.0331 (5)
C1B	0.0493 (2)	0.46984 (16)	0.69011 (14)	0.0272 (5)
C2B	−0.0251 (2)	0.47096 (17)	0.78086 (15)	0.0291 (6)
C3B	−0.0359 (2)	0.58338 (17)	0.86952 (14)	0.0282 (5)
C4B	0.0267 (2)	0.69546 (17)	0.87299 (15)	0.0289 (5)
C5B	0.1009 (2)	0.69054 (16)	0.78195 (14)	0.0268 (5)
C6B	0.1133 (2)	0.58021 (16)	0.68980 (15)	0.0273 (5)
C11B	0.0569 (2)	0.34589 (17)	0.59463 (16)	0.0312 (6)
O12A	0.36207 (19)	0.66095 (12)	0.49243 (11)	0.0372 (4)
O13A	0.5239 (2)	0.85805 (12)	0.37886 (12)	0.0423 (5)
O14A	0.42998 (17)	0.89974 (12)	0.54337 (11)	0.0365 (4)
N1A	0.3546 (2)	0.28647 (14)	0.26491 (13)	0.0311 (5)
C2A	0.3735 (2)	0.41030 (16)	0.33443 (15)	0.0293 (6)
C3A	0.4425 (2)	0.47577 (16)	0.28182 (14)	0.0259 (5)
C4A	0.5334 (2)	0.39011 (17)	0.08035 (15)	0.0298 (6)
C5A	0.5337 (3)	0.28088 (18)	−0.01547 (16)	0.0369 (7)
C6A	0.4707 (3)	0.16552 (18)	−0.02214 (17)	0.0397 (7)
C7A	0.4090 (3)	0.15632 (17)	0.06754 (16)	0.0351 (6)

C8A	0.4094 (2)	0.26675 (16)	0.16438 (15)	0.0280 (6)
C9A	0.4681 (2)	0.38401 (15)	0.17227 (14)	0.0247 (5)
C11A	0.4720 (2)	0.60721 (16)	0.32128 (15)	0.0273 (5)
C12A	0.4337 (2)	0.69302 (16)	0.41764 (15)	0.0281 (5)
C13A	0.4624 (2)	0.82503 (17)	0.45126 (15)	0.0309 (6)
S2C	0.13671 (7)	0.02349 (4)	0.35794 (4)	0.0359 (2)
O2C	0.16071 (19)	0.14384 (12)	0.35128 (11)	0.0422 (5)
C1C	−0.0974 (3)	−0.0384 (2)	0.3210 (2)	0.0547 (9)
C3C	0.2017 (4)	−0.0805 (2)	0.2321 (2)	0.0615 (9)
H2B	−0.06770	0.39570	0.78180	0.0350*
H4B	0.01890	0.77200	0.93490	0.0350*
H6B	0.16420	0.58030	0.62820	0.0330*
H11B	0.130 (3)	0.2799 (17)	0.4585 (17)	0.0580*
H1A	0.308 (3)	0.2298 (16)	0.2818 (16)	0.0370*
H2A	0.34380	0.44630	0.40820	0.0350*
H4A	0.57650	0.46790	0.08410	0.0360*
H5A	0.57740	0.28390	−0.07810	0.0440*
H6A	0.47050	0.09200	−0.08990	0.0480*
H7A	0.36810	0.07810	0.06340	0.0420*
H11A	0.52400	0.63630	0.27430	0.0330*
H12A	0.353 (3)	0.7300 (17)	0.5506 (16)	0.0560*
H13A	0.540 (3)	0.9407 (15)	0.4071 (19)	0.0630*
D11C	−0.14460	−0.04780	0.24570	0.0820*
D12C	−0.15770	0.01790	0.37790	0.0820*
D13C	−0.11950	−0.11940	0.31880	0.0820*
D31C	0.13170	−0.08140	0.16570	0.0920*
D32C	0.17950	−0.16400	0.22480	0.0920*
D33C	0.32990	−0.05370	0.23610	0.0920*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O11B	0.0554 (9)	0.0241 (7)	0.0344 (8)	0.0102 (6)	0.0154 (6)	0.0102 (6)
O12B	0.0661 (10)	0.0244 (8)	0.0504 (9)	0.0104 (7)	0.0179 (7)	0.0190 (7)
O31B	0.0418 (8)	0.0501 (9)	0.0519 (9)	0.0064 (7)	0.0145 (6)	0.0342 (7)
O32B	0.0582 (10)	0.0481 (10)	0.0336 (8)	0.0091 (7)	0.0151 (7)	0.0143 (7)
O51B	0.0814 (11)	0.0227 (8)	0.0397 (8)	0.0078 (7)	0.0175 (7)	0.0080 (6)
O52B	0.0587 (9)	0.0350 (8)	0.0480 (9)	0.0107 (7)	0.0234 (7)	0.0240 (7)
N3B	0.0309 (9)	0.0450 (11)	0.0361 (9)	0.0098 (7)	0.0075 (7)	0.0235 (8)
N5B	0.0404 (9)	0.0248 (9)	0.0323 (9)	0.0047 (7)	0.0051 (7)	0.0128 (7)
C1B	0.0256 (9)	0.0259 (10)	0.0301 (9)	0.0068 (7)	0.0024 (7)	0.0135 (8)
C2B	0.0256 (9)	0.0266 (10)	0.0362 (10)	0.0019 (7)	0.0006 (7)	0.0179 (8)
C3B	0.0240 (9)	0.0338 (10)	0.0274 (9)	0.0039 (7)	0.0024 (7)	0.0161 (8)
C4B	0.0288 (9)	0.0285 (10)	0.0254 (9)	0.0068 (7)	0.0013 (7)	0.0099 (7)
C5B	0.0279 (9)	0.0227 (9)	0.0292 (9)	0.0039 (7)	0.0012 (7)	0.0131 (7)
C6B	0.0266 (9)	0.0278 (10)	0.0280 (9)	0.0066 (7)	0.0036 (7)	0.0137 (7)
C11B	0.0314 (10)	0.0263 (10)	0.0352 (10)	0.0071 (7)	0.0032 (8)	0.0144 (8)
O12A	0.0537 (9)	0.0254 (7)	0.0322 (7)	0.0090 (6)	0.0146 (6)	0.0118 (6)

O13A	0.0594 (9)	0.0207 (7)	0.0456 (8)	0.0053 (6)	0.0244 (7)	0.0124 (6)
O14A	0.0424 (8)	0.0231 (7)	0.0390 (8)	0.0053 (6)	0.0160 (6)	0.0091 (6)
N1A	0.0373 (9)	0.0255 (9)	0.0350 (9)	0.0069 (7)	0.0112 (7)	0.0170 (7)
C2A	0.0317 (10)	0.0265 (10)	0.0295 (10)	0.0083 (7)	0.0075 (7)	0.0121 (8)
C3A	0.0245 (9)	0.0241 (9)	0.0271 (9)	0.0057 (7)	0.0036 (7)	0.0106 (7)
C4A	0.0287 (9)	0.0263 (10)	0.0353 (10)	0.0052 (7)	0.0091 (7)	0.0149 (8)
C5A	0.0392 (11)	0.0383 (12)	0.0359 (11)	0.0114 (9)	0.0176 (8)	0.0166 (9)
C6A	0.0452 (12)	0.0289 (11)	0.0378 (11)	0.0109 (9)	0.0148 (9)	0.0074 (8)
C7A	0.0388 (11)	0.0218 (10)	0.0410 (11)	0.0051 (8)	0.0094 (8)	0.0116 (8)
C8A	0.0263 (9)	0.0275 (10)	0.0315 (10)	0.0074 (7)	0.0062 (7)	0.0145 (8)
C9A	0.0206 (8)	0.0221 (9)	0.0302 (9)	0.0053 (7)	0.0036 (7)	0.0115 (7)
C11A	0.0249 (9)	0.0242 (9)	0.0316 (9)	0.0032 (7)	0.0051 (7)	0.0128 (7)
C12A	0.0272 (9)	0.0241 (9)	0.0312 (9)	0.0031 (7)	0.0041 (7)	0.0125 (8)
C13A	0.0265 (9)	0.0260 (10)	0.0353 (10)	0.0021 (7)	0.0064 (7)	0.0111 (8)
S2C	0.0396 (3)	0.0279 (3)	0.0398 (3)	0.0052 (2)	0.0107 (2)	0.0155 (2)
O2C	0.0567 (9)	0.0236 (7)	0.0450 (8)	0.0049 (6)	0.0255 (7)	0.0127 (6)
C1C	0.0390 (12)	0.0443 (14)	0.0970 (19)	0.0062 (10)	0.0194 (12)	0.0464 (14)
C3C	0.0669 (16)	0.0361 (14)	0.0623 (15)	0.0159 (11)	0.0213 (12)	0.0036 (11)

Geometric parameters (Å, º)

S2C—C3C	1.770 (3)	C2B—H2B	0.9500
S2C—C1C	1.771 (2)	C4B—H4B	0.9500
S2C—O2C	1.5177 (17)	C6B—H6B	0.9500
O11B—C11B	1.320 (2)	C2A—C3A	1.382 (3)
O12B—C11B	1.209 (3)	C3A—C11A	1.439 (3)
O31B—N3B	1.228 (3)	C3A—C9A	1.447 (2)
O32B—N3B	1.225 (2)	C4A—C9A	1.405 (3)
O51B—N5B	1.225 (2)	C4A—C5A	1.380 (3)
O52B—N5B	1.224 (2)	C5A—C6A	1.402 (3)
O11B—H11B	0.88 (2)	C6A—C7A	1.381 (3)
O12A—C12A	1.371 (2)	C7A—C8A	1.394 (3)
O13A—C13A	1.316 (3)	C8A—C9A	1.411 (3)
O14A—C13A	1.238 (2)	C11A—C12A	1.343 (3)
O12A—H12A	0.88 (2)	C12A—C13A	1.460 (3)
O13A—H13A	0.90 (2)	C2A—H2A	0.9500
N3B—C3B	1.472 (2)	C4A—H4A	0.9500
N5B—C5B	1.472 (3)	C5A—H5A	0.9500
N1A—C8A	1.378 (2)	C6A—H6A	0.9500
N1A—C2A	1.362 (3)	C7A—H7A	0.9500
N1A—H1A	0.87 (2)	C11A—H11A	0.9500
C1B—C6B	1.388 (3)	C1C—D11C	0.9800
C1B—C2B	1.391 (2)	C1C—D12C	0.9800
C1B—C11B	1.501 (3)	C1C—D13C	0.9800
C2B—C3B	1.381 (3)	C3C—D31C	0.9800
C3B—C4B	1.382 (3)	C3C—D32C	0.9800
C4B—C5B	1.379 (2)	C3C—D33C	0.9800
C5B—C6B	1.389 (3)		

O2C—S2C—C1C	106.34 (11)	C4A—C5A—C6A	121.28 (19)
O2C—S2C—C3C	103.33 (11)	C5A—C6A—C7A	121.5 (2)
C1C—S2C—C3C	99.20 (13)	C6A—C7A—C8A	117.1 (2)
C11B—O11B—H11B	109.6 (15)	N1A—C8A—C9A	107.34 (16)
C12A—O12A—H12A	106.7 (15)	N1A—C8A—C7A	130.1 (2)
C13A—O13A—H13A	110.2 (14)	C7A—C8A—C9A	122.53 (17)
O32B—N3B—C3B	118.17 (19)	C4A—C9A—C8A	118.90 (17)
O31B—N3B—C3B	117.62 (17)	C3A—C9A—C8A	107.02 (16)
O31B—N3B—O32B	124.21 (18)	C3A—C9A—C4A	134.08 (19)
O51B—N5B—C5B	117.86 (16)	C3A—C11A—C12A	126.73 (18)
O52B—N5B—C5B	118.81 (16)	O12A—C12A—C11A	121.18 (19)
O51B—N5B—O52B	123.33 (19)	C11A—C12A—C13A	124.01 (18)
C2A—N1A—C8A	109.74 (17)	O12A—C12A—C13A	114.81 (15)
C2A—N1A—H1A	123.5 (13)	O14A—C13A—C12A	120.60 (18)
C8A—N1A—H1A	126.7 (13)	O13A—C13A—C12A	116.25 (16)
C2B—C1B—C6B	120.32 (17)	O13A—C13A—O14A	123.2 (2)
C6B—C1B—C11B	122.31 (16)	C3A—C2A—H2A	125.00
C2B—C1B—C11B	117.38 (19)	N1A—C2A—H2A	125.00
C1B—C2B—C3B	118.9 (2)	C5A—C4A—H4A	121.00
N3B—C3B—C4B	118.50 (17)	C9A—C4A—H4A	121.00
N3B—C3B—C2B	118.71 (19)	C4A—C5A—H5A	119.00
C2B—C3B—C4B	122.79 (17)	C6A—C5A—H5A	119.00
C3B—C4B—C5B	116.62 (18)	C7A—C6A—H6A	119.00
C4B—C5B—C6B	123.1 (2)	C5A—C6A—H6A	119.00
N5B—C5B—C6B	119.51 (16)	C6A—C7A—H7A	121.00
N5B—C5B—C4B	117.36 (17)	C8A—C7A—H7A	121.00
C1B—C6B—C5B	118.28 (17)	C3A—C11A—H11A	117.00
O11B—C11B—O12B	124.40 (19)	C12A—C11A—H11A	117.00
O12B—C11B—C1B	122.64 (17)	S2C—C1C—D11C	109.00
O11B—C11B—C1B	112.96 (19)	S2C—C1C—D12C	109.00
C1B—C2B—H2B	121.00	S2C—C1C—D13C	110.00
C3B—C2B—H2B	121.00	D11C—C1C—D12C	109.00
C3B—C4B—H4B	122.00	D11C—C1C—D13C	109.00
C5B—C4B—H4B	122.00	D12C—C1C—D13C	110.00
C5B—C6B—H6B	121.00	S2C—C3C—D31C	109.00
C1B—C6B—H6B	121.00	S2C—C3C—D32C	109.00
N1A—C2A—C3A	109.91 (16)	S2C—C3C—D33C	109.00
C2A—C3A—C11A	128.34 (16)	D31C—C3C—D32C	109.00
C9A—C3A—C11A	125.53 (17)	D31C—C3C—D33C	109.00
C2A—C3A—C9A	105.98 (17)	D32C—C3C—D33C	109.00
C5A—C4A—C9A	118.7 (2)		

O31B—N3B—C3B—C2B	−11.4 (2)	C4B—C5B—C6B—C1B	−0.6 (2)
O31B—N3B—C3B—C4B	169.03 (16)	N1A—C2A—C3A—C9A	−0.36 (19)
O32B—N3B—C3B—C2B	168.39 (17)	N1A—C2A—C3A—C11A	175.38 (16)
O32B—N3B—C3B—C4B	−11.2 (2)	C2A—C3A—C9A—C4A	−179.69 (18)
O51B—N5B—C5B—C4B	−5.6 (2)	C2A—C3A—C9A—C8A	0.95 (18)

O51B—N5B—C5B—C6B	174.61 (16)	C11A—C3A—C9A—C4A	4.4 (3)
O52B—N5B—C5B—C4B	173.68 (16)	C11A—C3A—C9A—C8A	−174.95 (15)
O52B—N5B—C5B—C6B	−6.2 (2)	C2A—C3A—C11A—C12A	−1.6 (3)
C2A—N1A—C8A—C9A	0.98 (19)	C9A—C3A—C11A—C12A	173.37 (17)
C8A—N1A—C2A—C3A	−0.4 (2)	C9A—C4A—C5A—C6A	−0.1 (3)
C2A—N1A—C8A—C7A	−177.76 (19)	C5A—C4A—C9A—C3A	−177.79 (19)
C6B—C1B—C2B—C3B	0.8 (2)	C5A—C4A—C9A—C8A	1.5 (2)
C2B—C1B—C11B—O11B	176.16 (15)	C4A—C5A—C6A—C7A	−1.3 (3)
C2B—C1B—C11B—O12B	−3.8 (2)	C5A—C6A—C7A—C8A	1.0 (3)
C6B—C1B—C11B—O11B	−3.9 (2)	C6A—C7A—C8A—N1A	179.11 (19)
C6B—C1B—C11B—O12B	176.13 (17)	C6A—C7A—C8A—C9A	0.5 (3)
C11B—C1B—C6B—C5B	−179.91 (15)	N1A—C8A—C9A—C3A	−1.18 (18)
C11B—C1B—C2B—C3B	−179.31 (15)	N1A—C8A—C9A—C4A	179.34 (15)
C2B—C1B—C6B—C5B	0.0 (2)	C7A—C8A—C9A—C3A	177.68 (17)
C1B—C2B—C3B—N3B	179.41 (15)	C7A—C8A—C9A—C4A	−1.8 (3)
C1B—C2B—C3B—C4B	−1.0 (3)	C3A—C11A—C12A—O12A	1.4 (3)
C2B—C3B—C4B—C5B	0.5 (2)	C3A—C11A—C12A—C13A	−178.19 (16)
N3B—C3B—C4B—C5B	−179.96 (15)	O12A—C12A—C13A—O13A	−177.22 (15)
C3B—C4B—C5B—N5B	−179.48 (15)	O12A—C12A—C13A—O14A	3.0 (2)
C3B—C4B—C5B—C6B	0.4 (2)	C11A—C12A—C13A—O13A	2.4 (2)
N5B—C5B—C6B—C1B	179.23 (15)	C11A—C12A—C13A—O14A	−177.43 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O2*C*	0.87 (2)	2.02 (2)	2.856 (2)	161 (2)
O11*B*—H11*B*···S2*C*	0.88 (2)	2.84 (2)	3.6757 (17)	160 (2)
O11*B*—H11*B*···O2*C*	0.88 (2)	1.72 (2)	2.591 (2)	174 (2)
O12*A*—H12*A*···O14*A*	0.88 (2)	2.15 (2)	2.672 (2)	118 (2)
O12*A*—H12*A*···O52*B*	0.88 (2)	2.20 (2)	2.951 (2)	144 (2)
O13*A*—H13*A*···O14*A*^{i}	0.90 (2)	1.75 (2)	2.644 (2)	178 (2)
C2*A*—H2*A*···O12*A*	0.95	2.34	2.876 (2)	115
C11*A*—H11*A*···O13*A*	0.95	2.45	2.794 (3)	101
C1*C*—D12*C*···O14*A*^{ii}	0.98	2.56	3.472 (3)	155
C1*C*—D13*C*···O12*B*^{iii}	0.98	2.52	3.372 (3)	145

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