Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(isobutylammonium) phthalate monohydrate

Zakaria Norzalida, Azhar Ariffin and Seik Weng Ng*

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

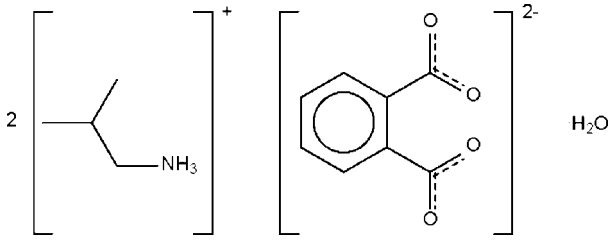
Received 14 May 2008; accepted 20 May 2008

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.062; wR factor = 0.156; data-to-parameter ratio = 18.0.

N-Isobutylphthalimic acid hydrolyzes to the title salt, $2C_4H_{12}N^+\cdot C_8H_4O_4^-\cdot H_2O$, which adopts a hydrogen-bonded layer structure. In the anion, the carboxylate groups are twisted with respect to the benzene ring [dihedral angles = 43.8 (1) and 50.9 (1)°].

Related literature

For kinetic studies relating to the hydrolysis of *N*-isobutylphthalimic acid, see: Ariffin & Khan (2005); Khan & Ariffin (2003).



Experimental

Crystal data

$2C_4H_{12}N^+\cdot C_8H_4O_4^{2-}\cdot H_2O$
M_r = 330.42
Triclinic, $P\overline{1}$
a = 8.8647 (4) Å
b = 9.4340 (5) Å
c = 12.9119 (6) Å
α = 72.298 (3)°
β = 79.449 (3)°
γ = 69.059 (3)°
V = 957.37 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 100 (2) K
0.32 × 0.08 × 0.08 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: none
8057 measured reflections
4343 independent reflections
2454 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.156
S = 0.97
4343 reflections
241 parameters
15 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1w—H1w1···O2	0.86 (1)	1.97 (2)	2.780 (2)	156 (3)
O1w—H1w2···O4^{i}	0.86 (1)	1.97 (2)	2.780 (2)	157 (3)
N1—H1n1···O1	0.85 (1)	1.94 (1)	2.788 (2)	172 (2)
N1—H1n2···O3^{ii}	0.87 (1)	1.91 (1)	2.755 (2)	167 (2)
N1—H1n3···O1w^{iii}	0.86 (1)	1.99 (1)	2.823 (3)	164 (2)
N2—H2n1···O2	0.86 (1)	2.35 (2)	2.996 (2)	132 (2)
N2—H2n1···O4^{i}	0.86 (1)	2.42 (2)	2.995 (3)	124 (2)
N2—H2n2···O2^{i}	0.87 (1)	1.91 (1)	2.781 (2)	172 (2)
N2—H2n3···O3	0.87 (1)	1.89 (1)	2.741 (2)	166 (2)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

We acknowledge the SAGA grant (06–02-03–0147) for supporting this study, and the University of Malaya for the purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2629).

References

Ariffin, A. & Khan, M. N. (2005). *Bull. Kor. Chem. Soc.* **26**, 1037–1043.
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Khan, M. N. & Ariffin, A. (2003). *Org. Biomol. Chem.* **1**, 1404–1408.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2008). *publCIF*. In preparation.

supplementary materials

Acta Cryst. (2008). E**64**, o1152 [**doi:10.1107/S1600536808015201**]

Bis(isobutylammonium) phthalate monohydrate

Z. Norzalida, A. Ariffin and S. W. Ng

Comment

The title salt (Fig. 1) was obtained as a wet crystalline compound when *N*-isobutylphthalimic acid was left aside for several years. The acid has been shown by kinetic studies to be converted to phthalic acid and isobutylamine under neutral and acidic conditions (Ariffin & Khan, 2005; Khan Ariffin, 2003). In the anion, the carboxyl $–CO_2$ groups are twisted with respect to the phenylene ring [dihedral angles 43.8 (1) and 50.9 (1) °]. Hydrogen bonds which involve the ammonium cations and water molecules link the components of the salt into a layer motif (Table 1).

Experimental

N-Isobutylphthalimidic acid was synthesized as described earlier (Ariffin & Khan, 2005; Khan & Ariffin, 2003). The crystalline compound was left aside for several years. The hydrolyzed title salt was obtained as a wet crystalline compound.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.99 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2–1.5*U*(C). The oxygen- and nitrogen-bound H-atoms were located in a difference Fourier map, and were refined with restraints of O–H = N–H = 0.85±0.01 Å; H···H = 1.39±0.01 Å; their temperature factors were freely refined.

Figures

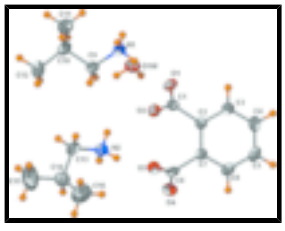


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $[C_4H_{12}N]_2[C_8H_4O_4]\cdot H_2O$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radiius.

Bis(isobutylammonium) phthalate monohydrate

Crystal data

$2C_4H_{12}N^+\cdot C_8H_4O_4^{2-}\cdot H_2O$	$Z = 2$
$M_r = 330.42$	$F_{000} = 360$
Triclinic, $P\overline{1}$	$D_x = 1.146$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å

a = 8.8647 (4) Å	Cell parameters from 941 reflections
b = 9.4340 (5) Å	θ = 2.5–22.7º
c = 12.9119 (6) Å	μ = 0.08 mm^{-1}
α = 72.298 (3)º	T = 100 (2) K
β = 79.449 (3)º	Prism, colorless
γ = 69.059 (3)º	0.32 × 0.08 × 0.08 mm
V = 957.37 (8) Å^3	

Data collection

Bruker SMART APEX diffractometer	2454 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.050
Monochromator: graphite	θ_{max} = 27.5º
T = 100(2) K	θ_{min} = 2.5º
ω scans	$h = -11 \rightarrow 11$
Absorption correction: None	$k = -10 \rightarrow 12$
8057 measured reflections	$l = -16 \rightarrow 16$
4343 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.061	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.156	$w = 1/[\sigma^2(F_o^2) + (0.0703P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.97	$(\Delta/\sigma)_{max}$ = 0.001
4343 reflections	$\Delta\rho_{max}$ = 0.33 e Å^{-3}
241 parameters	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
15 restraints	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.049 (5)

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.49468 (17)	0.58257 (19)	0.62388 (12)	0.0271 (4)
O2	0.75147 (17)	0.58034 (18)	0.58411 (12)	0.0250 (4)
O3	0.86510 (16)	0.23828 (19)	0.58460 (12)	0.0254 (4)
O4	1.08162 (16)	0.21636 (19)	0.66133 (12)	0.0285 (4)
O1w	0.7147 (2)	0.8989 (2)	0.50641 (15)	0.0342 (4)
H1w1	0.696 (3)	0.811 (2)	0.535 (2)	0.078 (12)*
H1w2	0.785 (3)	0.887 (3)	0.4520 (18)	0.096 (14)*
N1	0.4142 (2)	0.8164 (2)	0.43079 (16)	0.0230 (5)

H1n1	0.448 (3)	0.7460 (19)	0.4886 (14)	0.062 (10)*
H1n2	0.3266 (18)	0.809 (2)	0.4159 (16)	0.034 (7)*
H1n3	0.391 (2)	0.9076 (13)	0.4421 (17)	0.028 (7)*
N2	0.9312 (2)	0.4473 (3)	0.39672 (16)	0.0259 (5)
H2n1	0.873 (2)	0.5321 (15)	0.4154 (19)	0.054 (9)*
H2n2	1.0323 (12)	0.440 (2)	0.3961 (17)	0.042 (8)*
H2n3	0.917 (2)	0.3689 (16)	0.4499 (14)	0.037 (8)*
C1	0.6391 (3)	0.5332 (3)	0.64401 (17)	0.0216 (5)
C2	0.6832 (2)	0.4119 (3)	0.75120 (17)	0.0212 (5)
C3	0.5823 (3)	0.4347 (3)	0.84480 (18)	0.0265 (5)
H3	0.4850	0.5215	0.8393	0.032*
C4	0.6217 (3)	0.3327 (3)	0.94606 (19)	0.0338 (6)
H4	0.5509	0.3489	1.0092	0.041*
C5	0.7644 (3)	0.2070 (3)	0.95501 (19)	0.0326 (6)
H5	0.7928	0.1378	1.0244	0.039*
C6	0.8653 (3)	0.1828 (3)	0.86233 (18)	0.0268 (6)
H6	0.9630	0.0964	0.8688	0.032*
C7	0.8264 (2)	0.2824 (3)	0.76022 (17)	0.0207 (5)
C8	0.9328 (2)	0.2442 (3)	0.66050 (18)	0.0217 (5)
C9	0.5386 (2)	0.7900 (3)	0.33858 (17)	0.0231 (5)
H9A	0.5535	0.6865	0.3273	0.028*
H9B	0.6430	0.7861	0.3584	0.028*
C10	0.4971 (3)	0.9152 (3)	0.23207 (19)	0.0309 (6)
H10	0.3883	0.9236	0.2148	0.037*
C11	0.4909 (3)	1.0755 (3)	0.2390 (2)	0.0464 (8)
H11A	0.4091	1.1074	0.2970	0.070*
H11B	0.4626	1.1529	0.1692	0.070*
H11C	0.5972	1.0693	0.2553	0.070*
C12	0.6223 (3)	0.8638 (3)	0.14157 (19)	0.0369 (7)
H12A	0.6251	0.7605	0.1383	0.055*
H12B	0.7293	0.8575	0.1565	0.055*
H12C	0.5931	0.9405	0.0716	0.055*
C13	0.8993 (3)	0.4525 (3)	0.28710 (19)	0.0308 (6)
H13A	0.9174	0.5469	0.2340	0.037*
H13B	0.7845	0.4611	0.2877	0.037*
C14	1.0072 (3)	0.3079 (3)	0.2514 (2)	0.0397 (7)
H14	1.1214	0.2921	0.2627	0.048*
C15	0.9981 (5)	0.3334 (5)	0.1306 (3)	0.0870 (13)
H15A	1.0264	0.4277	0.0893	0.130*
H15B	0.8878	0.3469	0.1174	0.130*
H15C	1.0743	0.2421	0.1069	0.130*
C16	0.9674 (5)	0.1631 (4)	0.3167 (3)	0.0663 (10)
H16A	0.9732	0.1493	0.3944	0.100*
H16B	1.0452	0.0715	0.2941	0.100*
H16C	0.8576	0.1736	0.3043	0.100*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0211 (8)	0.0247 (10)	0.0298 (9)	−0.0054 (7)	−0.0062 (7)	0.0010 (7)
O2	0.0246 (8)	0.0226 (9)	0.0271 (8)	−0.0104 (7)	−0.0012 (7)	−0.0027 (7)
O3	0.0224 (8)	0.0283 (10)	0.0282 (9)	−0.0094 (7)	−0.0021 (7)	−0.0094 (7)
O4	0.0175 (8)	0.0308 (10)	0.0379 (10)	−0.0074 (7)	−0.0028 (7)	−0.0102 (8)
O1w	0.0366 (10)	0.0226 (11)	0.0420 (11)	−0.0109 (8)	0.0042 (9)	−0.0093 (9)
N1	0.0209 (10)	0.0173 (12)	0.0286 (12)	−0.0047 (8)	−0.0045 (9)	−0.0031 (10)
N2	0.0240 (10)	0.0219 (12)	0.0325 (12)	−0.0080 (9)	−0.0040 (9)	−0.0063 (10)
C1	0.0250 (11)	0.0155 (12)	0.0251 (12)	−0.0066 (10)	−0.0013 (10)	−0.0068 (10)
C2	0.0227 (11)	0.0195 (13)	0.0227 (12)	−0.0086 (10)	−0.0027 (9)	−0.0047 (10)
C3	0.0275 (12)	0.0233 (14)	0.0256 (13)	−0.0045 (10)	−0.0033 (10)	−0.0060 (10)
C4	0.0405 (14)	0.0363 (17)	0.0227 (13)	−0.0132 (12)	0.0010 (11)	−0.0059 (12)
C5	0.0423 (14)	0.0301 (15)	0.0227 (13)	−0.0113 (12)	−0.0097 (11)	0.0005 (11)
C6	0.0286 (12)	0.0230 (14)	0.0292 (13)	−0.0081 (10)	−0.0092 (10)	−0.0034 (11)
C7	0.0203 (11)	0.0199 (13)	0.0235 (12)	−0.0093 (10)	−0.0032 (9)	−0.0039 (10)
C8	0.0200 (11)	0.0136 (12)	0.0295 (13)	−0.0051 (9)	−0.0045 (9)	−0.0015 (10)
C9	0.0207 (11)	0.0201 (13)	0.0270 (12)	−0.0065 (9)	−0.0023 (9)	−0.0038 (10)
C10	0.0253 (12)	0.0320 (15)	0.0301 (13)	−0.0098 (11)	−0.0068 (10)	0.0027 (11)
C11	0.0577 (17)	0.0253 (16)	0.0409 (16)	−0.0109 (13)	0.0083 (14)	0.0027 (13)
C12	0.0443 (15)	0.0433 (18)	0.0267 (13)	−0.0213 (13)	−0.0027 (12)	−0.0059 (12)
C13	0.0396 (14)	0.0233 (14)	0.0286 (13)	−0.0081 (11)	−0.0115 (11)	−0.0029 (11)
C14	0.0430 (15)	0.0404 (18)	0.0359 (15)	−0.0071 (13)	−0.0066 (12)	−0.0156 (13)
C15	0.139 (4)	0.091 (3)	0.0396 (19)	−0.038 (3)	−0.001 (2)	−0.031 (2)
C16	0.116 (3)	0.0258 (18)	0.060 (2)	−0.0150 (18)	−0.023 (2)	−0.0156 (16)

Geometric parameters (Å, °)

O1—C1	1.243 (2)	C7—C8	1.507 (3)
O2—C1	1.270 (2)	C9—C10	1.519 (3)
O3—C8	1.263 (3)	C9—H9A	0.9900
O4—C8	1.251 (2)	C9—H9B	0.9900
O1w—H1w1	0.861 (10)	C10—C12	1.522 (3)
O1w—H1w2	0.858 (10)	C10—C11	1.523 (4)
N1—C9	1.483 (3)	C10—H10	1.0000
N1—H1n1	0.852 (9)	C11—H11A	0.9800
N1—H1n2	0.865 (9)	C11—H11B	0.9800
N1—H1n3	0.861 (9)	C11—H11C	0.9800
N2—C13	1.477 (3)	C12—H12A	0.9800
N2—H2n1	0.860 (9)	C12—H12B	0.9800
N2—H2n2	0.874 (9)	C12—H12C	0.9800
N2—H2n3	0.873 (9)	C13—C14	1.508 (4)
C1—C2	1.514 (3)	C13—H13A	0.9900
C2—C3	1.389 (3)	C13—H13B	0.9900
C2—C7	1.406 (3)	C14—C16	1.496 (4)
C3—C4	1.386 (3)	C14—C15	1.518 (4)
C3—H3	0.9500	C14—H14	1.0000

C4—C5	1.384 (3)	C15—H15A	0.9800
C4—H4	0.9500	C15—H15B	0.9800
C5—C6	1.383 (3)	C15—H15C	0.9800
C5—H5	0.9500	C16—H16A	0.9800
C6—C7	1.386 (3)	C16—H16B	0.9800
C6—H6	0.9500	C16—H16C	0.9800
H1w1—O1w—H1w2	107.2 (15)	C9—C10—C12	108.7 (2)
C9—N1—H1n1	109.2 (17)	C9—C10—C11	112.0 (2)
C9—N1—H1n2	108.1 (14)	C12—C10—C11	110.4 (2)
H1n1—N1—H1n2	109.4 (13)	C9—C10—H10	108.5
C9—N1—H1n3	113.4 (15)	C12—C10—H10	108.5
H1n1—N1—H1n3	108.9 (13)	C11—C10—H10	108.5
H1n2—N1—H1n3	107.9 (12)	C10—C11—H11A	109.5
C13—N2—H2n1	111.6 (16)	C10—C11—H11B	109.5
C13—N2—H2n2	109.3 (15)	H11A—C11—H11B	109.5
H2n1—N2—H2n2	106.4 (13)	C10—C11—H11C	109.5
C13—N2—H2n3	116.3 (15)	H11A—C11—H11C	109.5
H2n1—N2—H2n3	107.0 (13)	H11B—C11—H11C	109.5
H2n2—N2—H2n3	105.7 (13)	C10—C12—H12A	109.5
O1—C1—O2	125.4 (2)	C10—C12—H12B	109.5
O1—C1—C2	117.22 (19)	H12A—C12—H12B	109.5
O2—C1—C2	117.30 (18)	C10—C12—H12C	109.5
C3—C2—C7	119.0 (2)	H12A—C12—H12C	109.5
C3—C2—C1	118.49 (19)	H12B—C12—H12C	109.5
C7—C2—C1	122.38 (19)	N2—C13—C14	111.78 (19)
C4—C3—C2	121.0 (2)	N2—C13—H13A	109.3
C4—C3—H3	119.5	C14—C13—H13A	109.3
C2—C3—H3	119.5	N2—C13—H13B	109.3
C5—C4—C3	119.9 (2)	C14—C13—H13B	109.3
C5—C4—H4	120.1	H13A—C13—H13B	107.9
C3—C4—H4	120.1	C16—C14—C13	112.4 (2)
C6—C5—C4	119.6 (2)	C16—C14—C15	110.8 (3)
C6—C5—H5	120.2	C13—C14—C15	110.0 (3)
C4—C5—H5	120.2	C16—C14—H14	107.8
C5—C6—C7	121.2 (2)	C13—C14—H14	107.8
C5—C6—H6	119.4	C15—C14—H14	107.8
C7—C6—H6	119.4	C14—C15—H15A	109.5
C6—C7—C2	119.3 (2)	C14—C15—H15B	109.5
C6—C7—C8	119.4 (2)	H15A—C15—H15B	109.5
C2—C7—C8	121.22 (19)	C14—C15—H15C	109.5
O4—C8—O3	125.5 (2)	H15A—C15—H15C	109.5
O4—C8—C7	117.10 (19)	H15B—C15—H15C	109.5
O3—C8—C7	117.40 (18)	C14—C16—H16A	109.5
N1—C9—C10	113.95 (18)	C14—C16—H16B	109.5
N1—C9—H9A	108.8	H16A—C16—H16B	109.5
C10—C9—H9A	108.8	C14—C16—H16C	109.5
N1—C9—H9B	108.8	H16A—C16—H16C	109.5
C10—C9—H9B	108.8	H16B—C16—H16C	109.5
H9A—C9—H9B	107.7		

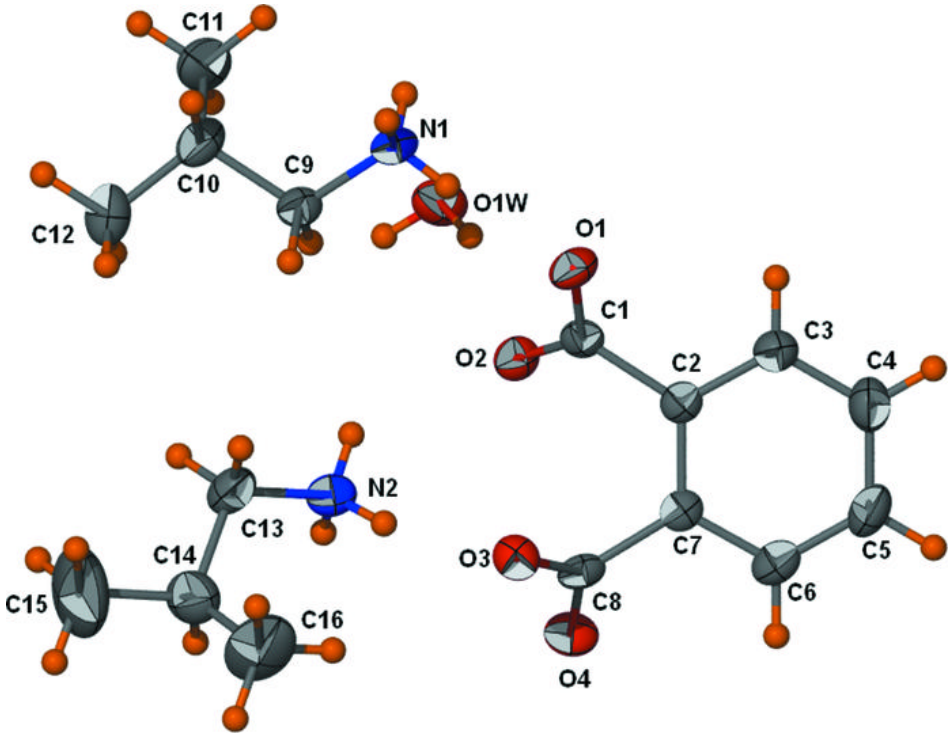
O1—C1—C2—C3	−43.9 (3)	C1—C2—C7—C6	174.98 (19)
O2—C1—C2—C3	133.5 (2)	C3—C2—C7—C8	174.7 (2)
O1—C1—C2—C7	139.7 (2)	C1—C2—C7—C8	−8.9 (3)
O2—C1—C2—C7	−42.9 (3)	C6—C7—C8—O4	−51.4 (3)
C7—C2—C3—C4	0.4 (3)	C2—C7—C8—O4	132.5 (2)
C1—C2—C3—C4	−176.1 (2)	C6—C7—C8—O3	126.5 (2)
C2—C3—C4—C5	0.8 (4)	C2—C7—C8—O3	−49.7 (3)
C3—C4—C5—C6	−1.1 (4)	N1—C9—C10—C12	−172.76 (19)
C4—C5—C6—C7	0.1 (4)	N1—C9—C10—C11	65.0 (3)
C5—C6—C7—C2	1.2 (3)	N2—C13—C14—C16	68.6 (3)
C5—C6—C7—C8	−175.0 (2)	N2—C13—C14—C15	−167.4 (2)
C3—C2—C7—C6	−1.4 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*w*—H1*w*1···O2	0.86 (1)	1.97 (2)	2.780 (2)	156 (3)
O1*w*—H1*w*2···O4^{i}	0.86 (1)	1.97 (2)	2.780 (2)	157 (3)
N1—H1*n*1···O1	0.85 (1)	1.94 (1)	2.788 (2)	172 (2)
N1—H1*n*2···O3^{ii}	0.87 (1)	1.91 (1)	2.755 (2)	167 (2)
N1—H1*n*3···O1*w*^{iii}	0.86 (1)	1.99 (1)	2.823 (3)	164 (2)
N2—H2*n*1···O2	0.86 (1)	2.35 (2)	2.996 (2)	132 (2)
N2—H2*n*1···O4^{i}	0.86 (1)	2.42 (2)	2.995 (3)	124 (2)
N2—H2*n*2···O2^{i}	0.87 (1)	1.91 (1)	2.781 (2)	172 (2)
N2—H2*n*3···O3	0.87 (1)	1.89 (1)	2.741 (2)	166 (2)

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

Fig. 1


C11
N1
C9
C10
O1W
C12
O1
C1
O2
C2
C3
C4
N2
C13
C14
C15
C16
O3
C7
C5
C8
C6
O4