Crystal structure of 1,3-dihydroxy-2-(hydroxymethyl)propan-2-aminium 2-(4-isobutylphenyl)propanoate: a simple organic salt of racemic ibuprofen
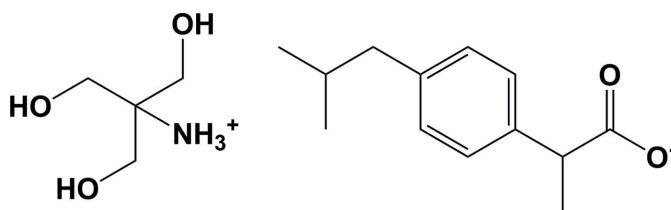
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In the title organic salt of ibuprofen with trometamol, $C_4H_{12}NO_3^+ \cdot C_{13}H_{17}O_2^-$, the carboxylic acid group of ibuprofen has transferred its proton to the amino N atom of trometamol. In the crystal, the trometamol cations are linked via N—H⋯O hydrogen bonds, forming chains along [001]. To these chains are attached the ibuprofen anions via O—H⋯O and N—H⋯O hydrogen bonds. The chains are linked via further N—H⋯O and O—H⋯O hydrogen bonds, forming sheets parallel to (100). Two C atoms of the propanoate substituent in the ibuprofen anion are disordered over two sets of sites and were refined with a fixed occupancy ratio of 0.7:0.3.
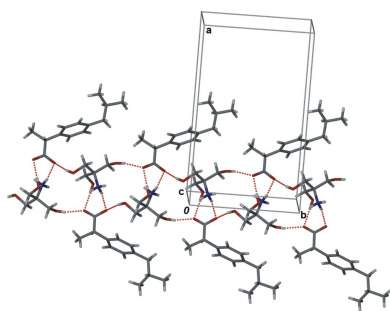
1. Chemical context

Salt formation is an effective approach for modifying the properties of active pharmaceutical ingredients (APIs) (Childs *et al.*, 2007). Tris(hydroxymethyl)amino methane, commonly called trometamol, has been successfully exploited for improving the properties of APIs such as ketoprofen (Zippel & Wagenitz, 2006). In this study, trometamol was employed to crystallize with ibuprofen, giving rise to a new crystalline form, whose crystal structure is reported on herein.

2. Structural commentary

The molecular structure of the title molecular salt is shown in Fig. 1. From difference Fourier maps, it was shown that the carboxylic group of ibuprofen has transferred its proton to the amino N atom of trometamol. This is supported by the C—O bond distances of the carboxylate group of the ibuprofen anion, which are 1.252 (2) and 1.251 (2) Å for C1—O1 and C1—O2, respectively. The carboxylate anion interacts with one hydroxyl group of the trometamol cation through a strong hydrogen bond [O5⋯O2 = 2.730 (2) Å; Table 1]. There also exist hydrogen-bonding interactions between the carboxylate anion and aminium H atoms of the cation [N1⋯O1 = 2.763 (2) Å; Table 1].
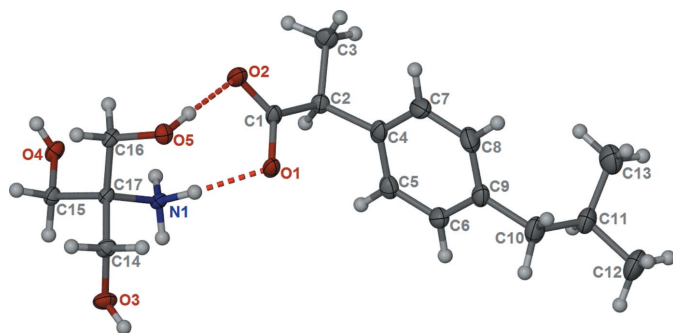
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Figure 1
The molecular structure of the title molecular salt, with atom labeling. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bonds are shown as dashed lines (see Table 1 for details). The minor components of the disordered atoms (*viz.* C2 and C3) have been omitted for clarity in all three figures.

3. Supramolecular features

In the crystal, the trometamol cations are linked *via* N—H···O hydrogen bonds, forming chains along [010]; Table 1 and Fig. 2. To these chains are attached the ibuprofen anions *via* N—H···O and O—H···O hydrogen bonds. The chains are linked *via* further O—H···O and N—H···O hydrogen bonds, forming sheets parallel to (100); Table 1 and Fig. 3.

4. Database survey

A search of the Cambridge Structural Database (Version 5.36, May 2015; Groom & Allen, 2014) revealed only one hit for organic salts of racemic ibuprofen, *viz.* benzylammonium 2-(4-isobutylphenyl)propionate 2-(4-isobutylphenyl)propionic acid (refcode VUCHUX; Molnár *et al.*, 2009). In fact, it is a salt co-crystal based on ibuprofen and the organic salt (Sun, 2013).
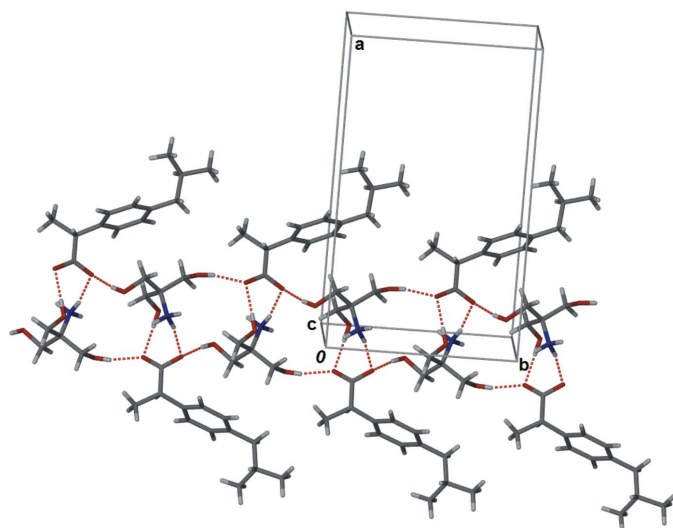

Figure 2
Part of the crystal structure of the title salt, viewed along the *c* axis, showing the hydrogen bonds (dashed lines) forming chains along [001]; see Table 1 for details.

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2^i	0.90 (3)	1.84 (3)	2.725 (2)	167 (2)
O4—H4···O1^{ii}	0.79 (2)	1.92 (3)	2.689 (2)	163.7 (18)
O5—H5A···O1	0.86 (2)	2.57 (2)	3.0825 (19)	119.5 (17)
O5—H5A···O2	0.86 (2)	1.88 (2)	2.730 (2)	168.1 (18)
N1—H1A···O1	0.94 (2)	1.85 (2)	2.763 (2)	162.9 (18)
N1—H1B···O4^{iii}	0.94 (2)	2.09 (2)	2.9224 (19)	146.7 (16)
N1—H1C···O5^{iv}	0.91 (2)	1.97 (2)	2.806 (2)	152.1 (18)

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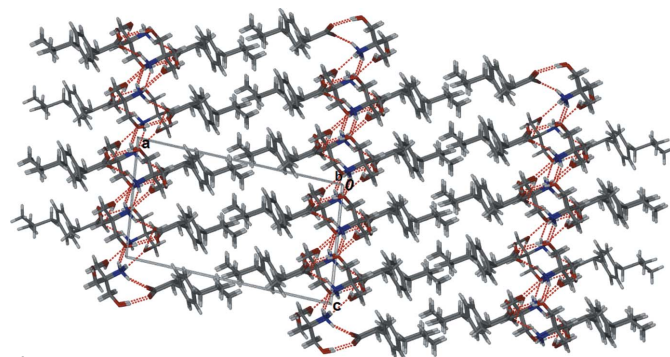

Figure 3
Part of the crystal structure of the title salt, viewed along the *b* axis, showing the sheets parallel to (100) formed by hydrogen bonding (dashed lines; see Table 1 for details).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_4H_{12}NO_3^+ \cdot C_{13}H_{17}O_2^-$
M_r	327.41
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	173
a, b, c (Å)	17.523 (7), 10.400 (4), 9.976 (4)
β (°)	97.032 (7)
V (Å^3)	1804.3 (12)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	$0.29 \times 0.22 \times 0.04$
Data collection	
Diffractometer	Rigaku Mercury CCD
Absorption correction	Multi-scan (*CrystalClear*; Rigaku, 2000)
T_{min}, T_{max}	0.914, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13747, 4096, 3391
R_{int}	0.028
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.058, 0.148, 1.11
No. of reflections	4096
No. of parameters	260
No. of restraints	6
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.22, −0.23

Computer programs: *CrystalClear* (Rigaku, 2000), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *X-SEED* (Barbour, 2001).

The title compound is the first crystal structure of a simple organic salt of racemic ibuprofen.

5. Synthesis and crystallization

Ibuprofen (206 mg, 1 mmol) and trometamol (121 mg, 1 mmol) were dissolved in methanol (15 mL). The resulting solution was kept in air and after several days colorless plate-like crystals were obtained.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The hydroxyl and and aminium H atoms were located in difference Fourier maps and freely refined. Two C atoms, C3 and C2, of the propanoate substituent in the ibuprofen anion are disordered over two sets of sites (C3/C3′and C2/C2′) and were refined with a fixed occupancy ratio of 0.7:0.3. H atoms H2 and H2′ were refined with distance restraints C—H = 0.98 (2) Å with $U_{\mathrm{iso}}(\mathrm{H})$ = $1.2U_{\mathrm{eq}}(\mathrm{C})$. The remainder of the C-bound H atoms were positioned geometrically and refined as riding atoms: C—H = 0.95–1.00 Å with $U_{\mathrm{iso}}(\mathrm{H}) = 1.5U_{\mathrm{eq}}(\mathrm{C})$ for methyl H atoms and $1.2U_{\mathrm{eq}}(\mathrm{C})$ for other H atoms.

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supporting information

Crystal structure of 1,3-dihydroxy-2-(hydroxymethyl)propan-2-aminium 2-(4-isobutylphenyl)propanoate: a simple organic salt of racemic ibuprofen

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Computing details

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear* (Rigaku, 2000); data reduction: *CrystalClear* (Rigaku, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

1,3-Dihydroxy-2-(hydroxymethyl)propan-2-aminium 2-(4-isobutylphenyl)propanoate

Crystal data

$C_4H_{12}NO_3^+ \cdot C_{13}H_{17}O_2^-$	$F(000) = 712$
$M_r = 327.41$	$D_x = 1.205$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 17.523$ (7) Å	Cell parameters from 4551 reflections
$b = 10.400$ (4) Å	$\theta = 2.8–27.5°$
$c = 9.976$ (4) Å	$\mu = 0.09$ mm^{-1}
$\beta = 97.032$ (7)°	$T = 173$ K
$V = 1804.3$ (12) Å^3	Plate, colorless
$Z = 4$	$0.29 \times 0.22 \times 0.04$ mm

Data collection

Rigaku Mercury CCD diffractometer	13747 measured reflections
Radiation source: fine-focus sealed tube	4096 independent reflections
Graphite monochromator	3391 reflections with $I > 2\sigma(I)$
Detector resolution: 28.5714 pixels mm^{-1}	$R_{int} = 0.028$
CCD_Profile_fitting scans	$\theta_{max} = 27.5°$, $\theta_{min} = 2.3°$
Absorption correction: multi-scan (CrystalClear; Rigaku, 2000)	$h = -22 \rightarrow 22$
	$k = -13 \rightarrow 13$
$T_{min} = 0.914$, $T_{max} = 1.000$	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent
$R[F^2 > 2\sigma(F^2)] = 0.058$	and constrained refinement
$wR(F^2) = 0.148$	$w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 0.4122P]$
$S = 1.11$	where $P = (F_o^2 + 2F_c^2)/3$
4096 reflections	$(\Delta/\sigma)_{max} = 0.001$
260 parameters	$\Delta\rho_{max} = 0.22$ e Å^{-3}
6 restraints	$\Delta\rho_{min} = -0.23$ 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.

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

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.11773 (7)	0.77640 (11)	0.78429 (13)	0.0439 (3)	
O2	0.13430 (7)	0.58002 (12)	0.71039 (15)	0.0544 (4)	
C1	0.15768 (10)	0.67741 (15)	0.7767 (2)	0.0422 (4)	
C2	0.23375 (17)	0.6782 (3)	0.8754 (4)	0.0408 (6)	0.7
H2	0.2182 (17)	0.670 (3)	0.966 (2)	0.049*	0.7
C3	0.28440 (19)	0.5630 (3)	0.8537 (4)	0.0610 (9)	0.7
H3A	0.2992	0.5660	0.7622	0.091*	0.7
H3B	0.2559	0.4835	0.8652	0.091*	0.7
H3C	0.3307	0.5654	0.9197	0.091*	0.7
C2′	0.2464 (4)	0.6735 (7)	0.8061 (9)	0.0417 (16)	0.3
H2′	0.271 (3)	0.616 (2)	0.746 (6)	0.050*	0.3
C3′	0.2585 (5)	0.5894 (9)	0.9312 (11)	0.070 (2)	0.3
H3′1	0.3115	0.5989	0.9744	0.105*	0.3
H3′2	0.2490	0.4993	0.9055	0.105*	0.3
H3′3	0.2228	0.6156	0.9945	0.105*	0.3
C4	0.27435 (10)	0.80792 (18)	0.8575 (2)	0.0542 (5)	
C5	0.26039 (11)	0.9010 (2)	0.9485 (2)	0.0540 (5)	
H5	0.2281	0.8817	1.0157	0.065*	
C6	0.29208 (11)	1.0220 (2)	0.9450 (2)	0.0548 (5)	
H6	0.2825	1.0830	1.0118	0.066*	
C7	0.32041 (12)	0.8410 (2)	0.7599 (3)	0.0667 (6)	
H7	0.3315	0.7785	0.6955	0.080*	
C8	0.35065 (13)	0.9638 (2)	0.7543 (3)	0.0682 (6)	
H8	0.3813	0.9843	0.6850	0.082*	
C9	0.33732 (11)	1.05711 (19)	0.8470 (2)	0.0549 (5)	
C10	0.37079 (14)	1.1906 (2)	0.8433 (3)	0.0768 (7)	
H10A	0.3344	1.2522	0.8768	0.092*	
H10B	0.3755	1.2131	0.7482	0.092*	
C11	0.44861 (14)	1.2067 (2)	0.9256 (3)	0.0768 (7)	
H11	0.4438	1.1754	1.0190	0.092*	
C12	0.4706 (2)	1.3480 (3)	0.9369 (4)	0.1136 (12)	
H12A	0.4710	1.3842	0.8463	0.170*	
H12B	0.5219	1.3565	0.9878	0.170*	
H12C	0.4331	1.3945	0.9839	0.170*	
C13	0.51137 (16)	1.1303 (3)	0.8743 (4)	0.0991 (10)	
H13A	0.5186	1.1600	0.7834	0.149*	
H13B	0.4973	1.0391	0.8708	0.149*	
H13C	0.5593	1.1419	0.9348	0.149*	
O3	−0.15157 (8)	0.82574 (12)	0.84440 (14)	0.0511 (4)	

H3	−0.1538 (14)	0.910 (2)	0.825 (2)	0.081 (8)*
O4	−0.08544 (7)	0.44727 (12)	0.91701 (13)	0.0419 (3)
H4	−0.0903 (12)	0.387 (2)	0.868 (2)	0.066 (7)*
O5	−0.00842 (7)	0.64538 (11)	0.58848 (12)	0.0397 (3)
H5A	0.0387 (12)	0.6268 (18)	0.616 (2)	0.059 (6)*
N1	−0.01557 (8)	0.68283 (13)	0.87045 (14)	0.0313 (3)
H1A	0.0234 (12)	0.7199 (19)	0.826 (2)	0.049 (5)*
H1B	0.0080 (11)	0.6126 (19)	0.9193 (19)	0.044 (5)*
H1C	−0.0290 (11)	0.745 (2)	0.927 (2)	0.046 (5)*
C14	−0.13080 (10)	0.75903 (15)	0.73082 (18)	0.0411 (4)
H14A	−0.0997	0.8157	0.6790	0.049*
H14B	−0.1776	0.7335	0.6710	0.049*
C15	−0.13077 (9)	0.55121 (15)	0.85901 (17)	0.0367 (4)
H15A	−0.1507	0.6012	0.9317	0.044*
H15B	−0.1753	0.5168	0.7991	0.044*
C16	−0.05760 (10)	0.56874 (15)	0.65824 (17)	0.0370 (4)
H16A	−0.0300	0.4895	0.6910	0.044*
H16B	−0.1030	0.5430	0.5950	0.044*
C17	−0.08440 (9)	0.63973 (14)	0.77797 (16)	0.0310 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0357 (6)	0.0295 (6)	0.0647 (8)	−0.0003 (5)	−0.0012 (6)	0.0038 (6)
O2	0.0452 (7)	0.0361 (7)	0.0795 (10)	0.0024 (5)	−0.0018 (7)	−0.0067 (6)
C1	0.0332 (8)	0.0296 (8)	0.0625 (12)	−0.0022 (6)	0.0010 (8)	0.0099 (8)
C2	0.0370 (15)	0.0368 (14)	0.047 (2)	0.0024 (11)	0.0000 (14)	0.0053 (15)
C3	0.0481 (18)	0.0458 (16)	0.084 (3)	0.0132 (13)	−0.0108 (17)	0.0029 (18)
C2′	0.038 (3)	0.042 (3)	0.046 (4)	0.001 (2)	0.007 (3)	0.006 (3)
C3′	0.054 (5)	0.063 (5)	0.087 (7)	0.001 (4)	−0.009 (4)	0.022 (5)
C4	0.0293 (8)	0.0450 (10)	0.0838 (15)	−0.0027 (7)	−0.0115 (9)	0.0023 (10)
C5	0.0395 (10)	0.0578 (12)	0.0636 (13)	−0.0034 (8)	0.0025 (9)	0.0059 (10)
C6	0.0416 (10)	0.0551 (11)	0.0667 (14)	−0.0019 (9)	0.0028 (9)	−0.0108 (10)
C7	0.0480 (12)	0.0656 (13)	0.0859 (17)	−0.0039 (10)	0.0058 (11)	−0.0294 (12)
C8	0.0493 (12)	0.0800 (16)	0.0777 (16)	−0.0129 (11)	0.0180 (11)	−0.0067 (13)
C9	0.0368 (9)	0.0496 (11)	0.0763 (15)	−0.0067 (8)	−0.0007 (9)	−0.0016 (10)
C10	0.0570 (13)	0.0523 (13)	0.119 (2)	−0.0110 (10)	0.0007 (13)	0.0100 (13)
C11	0.0639 (15)	0.0552 (13)	0.112 (2)	−0.0196 (11)	0.0132 (14)	−0.0071 (13)
C12	0.097 (2)	0.0659 (17)	0.177 (4)	−0.0342 (16)	0.015 (2)	−0.016 (2)
C13	0.0588 (16)	0.094 (2)	0.143 (3)	−0.0092 (14)	0.0058 (17)	−0.006 (2)
O3	0.0624 (9)	0.0323 (6)	0.0598 (9)	0.0120 (6)	0.0124 (7)	0.0012 (6)
O4	0.0542 (8)	0.0286 (6)	0.0411 (7)	−0.0050 (5)	−0.0013 (6)	0.0064 (5)
O5	0.0401 (7)	0.0417 (6)	0.0372 (7)	−0.0026 (5)	0.0048 (5)	0.0080 (5)
N1	0.0359 (7)	0.0243 (6)	0.0330 (8)	−0.0003 (5)	0.0014 (6)	−0.0004 (6)
C14	0.0453 (10)	0.0324 (8)	0.0439 (10)	0.0046 (7)	−0.0021 (8)	0.0023 (7)
C15	0.0387 (8)	0.0306 (8)	0.0406 (10)	−0.0046 (6)	0.0043 (7)	0.0025 (7)
C16	0.0453 (9)	0.0310 (8)	0.0344 (9)	−0.0065 (7)	0.0041 (7)	−0.0010 (7)
C17	0.0331 (8)	0.0258 (7)	0.0331 (8)	−0.0017 (6)	−0.0006 (6)	0.0005 (6)

Geometric parameters (Å, º)

O1—C1	1.252 (2)	C10—H10B	0.9900
O2—C1	1.251 (2)	C11—C13	1.497 (4)
C1—C2′	1.547 (7)	C11—C12	1.520 (3)
C1—C2	1.558 (4)	C11—H11	1.0000
C2—C3	1.522 (4)	C12—H12A	0.9800
C2—C4	1.546 (4)	C12—H12B	0.9800
C2—H2	0.983 (17)	C12—H12C	0.9800
C3—H3A	0.9800	C13—H13A	0.9800
C3—H3B	0.9800	C13—H13B	0.9800
C3—H3C	0.9800	C13—H13C	0.9800
C2′—C3′	1.518 (11)	O3—C14	1.414 (2)
C2′—C4	1.548 (8)	O3—H3	0.90 (3)
C2′—H2′	0.98 (2)	O4—C15	1.422 (2)
C3′—H3′1	0.9800	O4—H4	0.79 (2)
C3′—H3′2	0.9800	O5—C16	1.4174 (19)
C3′—H3′3	0.9800	O5—H5A	0.86 (2)
C4—C5	1.369 (3)	N1—C17	1.495 (2)
C4—C7	1.383 (3)	N1—H1A	0.94 (2)
C5—C6	1.377 (3)	N1—H1B	0.94 (2)
C5—H5	0.9500	N1—H1C	0.91 (2)
C6—C9	1.381 (3)	C14—C17	1.526 (2)
C6—H6	0.9500	C14—H14A	0.9900
C7—C8	1.387 (3)	C14—H14B	0.9900
C7—H7	0.9500	C15—C17	1.524 (2)
C8—C9	1.380 (3)	C15—H15A	0.9900
C8—H8	0.9500	C15—H15B	0.9900
C9—C10	1.509 (3)	C16—C17	1.526 (2)
C10—C11	1.513 (4)	C16—H16A	0.9900
C10—H10A	0.9900	C16—H16B	0.9900
O2—C1—O1	123.26 (16)	C11—C10—H10B	108.6
O2—C1—C2′	109.6 (3)	H10A—C10—H10B	107.6
O1—C1—C2′	124.5 (3)	C13—C11—C10	114.0 (2)
O2—C1—C2	122.54 (17)	C13—C11—C12	110.4 (2)
O1—C1—C2	113.51 (18)	C10—C11—C12	110.5 (2)
C3—C2—C4	112.7 (3)	C13—C11—H11	107.2
C3—C2—C1	112.0 (3)	C10—C11—H11	107.2
C4—C2—C1	107.6 (2)	C12—C11—H11	107.2
C3—C2—H2	107.2 (18)	C11—C12—H12A	109.5
C4—C2—H2	111.5 (17)	C11—C12—H12B	109.5
C1—C2—H2	105.7 (18)	H12A—C12—H12B	109.5
C2—C3—H3A	109.5	C11—C12—H12C	109.5
C2—C3—H3B	109.5	H12A—C12—H12C	109.5
H3A—C3—H3B	109.5	H12B—C12—H12C	109.5
C2—C3—H3C	109.5	C11—C13—H13A	109.5
H3A—C3—H3C	109.5	C11—C13—H13B	109.5

H3B—C3—H3C	109.5	H13A—C13—H13B	109.5
C3′—C2′—C1	101.9 (6)	C11—C13—H13C	109.5
C3′—C2′—C4	103.9 (7)	H13A—C13—H13C	109.5
C1—C2′—C4	108.0 (4)	H13B—C13—H13C	109.5
C3′—C2′—H2′	97 (3)	C14—O3—H3	108.7 (12)
C1—C2′—H2′	114 (4)	C15—O4—H4	109.7 (13)
C4—C2′—H2′	127.3 (18)	C16—O5—H5A	109.7 (11)
C2′—C3′—H3′1	109.5	C17—N1—H1A	114.1 (12)
C2′—C3′—H3′2	109.5	C17—N1—H1B	110.8 (12)
H3′1—C3′—H3′2	109.5	H1A—N1—H1B	105.3 (16)
C2′—C3′—H3′3	109.5	C17—N1—H1C	110.3 (13)
H3′1—C3′—H3′3	109.5	H1A—N1—H1C	104.9 (17)
H3′2—C3′—H3′3	109.5	H1B—N1—H1C	111.3 (17)
C5—C4—C7	117.28 (18)	O3—C14—C17	109.37 (14)
C5—C4—C2	114.7 (2)	O3—C14—H14A	109.8
C7—C4—C2	128.0 (2)	C17—C14—H14A	109.8
C5—C4—C2′	141.6 (3)	O3—C14—H14B	109.8
C7—C4—C2′	100.3 (3)	C17—C14—H14B	109.8
C4—C5—C6	121.6 (2)	H14A—C14—H14B	108.2
C4—C5—H5	119.2	O4—C15—C17	111.56 (13)
C6—C5—H5	119.2	O4—C15—H15A	109.3
C5—C6—C9	121.9 (2)	C17—C15—H15A	109.3
C5—C6—H6	119.1	O4—C15—H15B	109.3
C9—C6—H6	119.1	C17—C15—H15B	109.3
C4—C7—C8	121.1 (2)	H15A—C15—H15B	108.0
C4—C7—H7	119.5	O5—C16—C17	112.09 (12)
C8—C7—H7	119.5	O5—C16—H16A	109.2
C9—C8—C7	121.6 (2)	C17—C16—H16A	109.2
C9—C8—H8	119.2	O5—C16—H16B	109.2
C7—C8—H8	119.2	C17—C16—H16B	109.2
C8—C9—C6	116.57 (19)	H16A—C16—H16B	107.9
C8—C9—C10	122.1 (2)	N1—C17—C15	107.22 (13)
C6—C9—C10	121.3 (2)	N1—C17—C14	107.80 (12)
C9—C10—C11	114.6 (2)	C15—C17—C14	110.87 (13)
C9—C10—H10A	108.6	N1—C17—C16	109.01 (13)
C11—C10—H10A	108.6	C15—C17—C16	110.98 (12)
C9—C10—H10B	108.6	C14—C17—C16	110.83 (14)
O2—C1—C2—C3	14.9 (4)	C2—C4—C7—C8	177.6 (2)
O1—C1—C2—C3	−174.3 (3)	C2′—C4—C7—C8	171.4 (3)
O2—C1—C2—C4	139.3 (2)	C4—C7—C8—C9	1.2 (4)
O1—C1—C2—C4	−50.0 (3)	C7—C8—C9—C6	−0.3 (3)
O2—C1—C2′—C3′	−83.9 (6)	C7—C8—C9—C10	179.1 (2)
O1—C1—C2′—C3′	114.1 (6)	C5—C6—C9—C8	−1.4 (3)
O2—C1—C2′—C4	167.0 (4)	C5—C6—C9—C10	179.2 (2)
O1—C1—C2′—C4	5.0 (7)	C8—C9—C10—C11	−90.4 (3)
C3—C2—C4—C5	−140.2 (3)	C6—C9—C10—C11	89.0 (3)
C1—C2—C4—C5	95.8 (3)	C9—C10—C11—C13	65.5 (3)

C3—C2—C4—C7	41.7 (4)	C9—C10—C11—C12	−169.5 (3)
C1—C2—C4—C7	−82.2 (3)	O4—C15—C17—N1	−55.72 (17)
C3′—C2′—C4—C5	−59.3 (7)	O4—C15—C17—C14	−173.15 (14)
C1—C2′—C4—C5	48.5 (8)	O4—C15—C17—C16	63.24 (18)
C3′—C2′—C4—C7	132.5 (5)	O3—C14—C17—N1	−58.01 (17)
C1—C2′—C4—C7	−119.7 (4)	O3—C14—C17—C15	59.06 (18)
C7—C4—C5—C6	−1.3 (3)	O3—C14—C17—C16	−177.23 (13)
C2—C4—C5—C6	−179.6 (2)	O5—C16—C17—N1	−56.68 (17)
C2′—C4—C5—C6	−168.2 (5)	O5—C16—C17—C15	−174.56 (13)
C4—C5—C6—C9	2.3 (3)	O5—C16—C17—C14	61.80 (18)
C5—C4—C7—C8	−0.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O3—H3⋯O2[i]	0.90 (3)	1.84 (3)	2.725 (2)	167 (2)
O4—H4⋯O1[ii]	0.79 (2)	1.92 (3)	2.689 (2)	163.7 (18)
O5—H5A⋯O1	0.86 (2)	2.57 (2)	3.0825 (19)	119.5 (17)
O5—H5A⋯O2	0.86 (2)	1.88 (2)	2.730 (2)	168.1 (18)
N1—H1A⋯O1	0.94 (2)	1.85 (2)	2.763 (2)	162.9 (18)
N1—H1B⋯O4[iii]	0.94 (2)	2.09 (2)	2.9224 (19)	146.7 (16)
N1—H1C⋯O5[iv]	0.91 (2)	1.97 (2)	2.806 (2)	152.1 (18)

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