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The new chiral ligand 3-ethoxy-4-[(1*R*,2*S*)-(2-hydroxy-1,2-diphenylethyl)amino]-3-cyclobutene-1,2-dione

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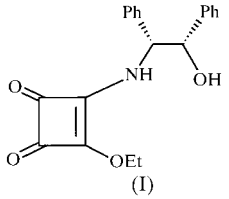
The asymmetric unit of $C_{20}H_{19}NO_4$ contains two molecules with slightly different conformations. In the crystal, the molecules are linked by O—H···O and N—H···O hydrogen bonds [O···O 2.764 (3) and 2.811 (3) Å; N···O 2.907 (3) and 2.968 (3) Å] to form a two-dimensional network.

Comment

Squaric acid and its derivatives have long been known as aromatic ring systems having unique characteristics and wide application, but reports on the synthesis of chiral derivatives of squaric acid are few and their application in asymmetric catalytic reactions is totally unprecedented. We report herein for the first time the crystal structure of the new chiral ligand 3-ethoxy-4-[(1*R*,2S*S*)-(2-hydroxy-1,2-diphenylethyl)amino]-3-cyclobutene-1,2-dione, (I), which contains two independent molecules with slightly different conformations, they are linked by O—H···O and N—H···O hydrogen bonds (see Table 1) to form a two-dimensional network. This ligand may provide an effect chiral environment for coordination of the substrate in the reactions, such as the enantioselective catalytic borane reduction of prochiral ketones and addition of dialkylzinc to aldehydes *etc*, ascribed to the combining the aromatic squaric acid moiety of rigidity tetracarbon ring and



(I)

flexible ethoxy group with the chiral aminoalcohol, that proved to be the effective catalyst for the reactions mentioned above when the new ligand was used.

Experimental

The title compound was prepared by reaction of 3,4-diethoxy-3-cyclobutene-1,2-dione and (1*S*,2*R*)-(±)-2-amino-1,2-diphenylethanol in the presence of triethylamine (molar ratio 1.1:1:1) in ethanol and recrystallized from it.

Crystal data

$C_{20}H_{19}NO_4$
M_r = 337.36
Monoclinic, $P2_1$
a = 10.758 (2) Å
b = 15.879 (3) Å
c = 11.040 (2) Å
β = 112.740 (10)°
V = 1739.3 (6) Å^3
Z = 4
D_x = 1.288 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 3.41–17.40°
μ = 0.090 mm^{-1}
T = 296 (2) K
Flake, colourless
0.48 × 0.38 × 0.36 mm

Data collection

Bruker $P4$ diffractometer
ω scans
5817 measured reflections
4871 independent reflections
3018 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 28.50°
$h = -1 \rightarrow 14$
$k = -1 \rightarrow 21$
$l = -14 \rightarrow 14$
3 standard reflections
every 97 reflections
intensity decay: 3.98%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.073
S = 0.846
4871 reflections
456 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0321P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.159 e Å^{-3}
$\Delta\rho_{min}$ = −0.141 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997*a*)
Extinction coefficient: 0.0168 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O3^{i}	0.82	2.01	2.811 (3)	165
O1′—H1′O···O3′^{ii}	0.82	1.95	2.764 (3)	173
N—H···O2′^{iii}	0.86	2.06	2.907 (3)	169
N'—H'···O2	0.86	2.15	2.968 (3)	159

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

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*b*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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supporting information

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The new chiral ligand 3-ethoxy-4-[(1*R*,2*S*)-(2-hydroxy-1,2-diphenylethyl)-amino]-3-cyclobutene-1,2-dione

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Computing details

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997b); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

3-Ethoxy-4-[(1′*R*,2′*S*)-(2′-hydroxy-1′,2′-diphenyl)ethylamino]-3-cyclobutene- 1,2-dione

Crystal data

$C_{20}H_{19}NO_4$
M_r = 337.36
Monoclinic, $P2_1$
a = 10.758 (2) Å
b = 15.879 (3) Å
c = 11.040 (2) Å
β = 112.74 (1)°
V = 1739.3 (6) Å^3
Z = 4
F(000) = 712
D_x = 1.288 Mg m^{-3}
Melting point = 481–483 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 3.4–17.4°
μ = 0.09 mm^{-1}
T = 296 K
Flake, colourless
0.48 × 0.38 × 0.36 mm

Data collection

Bruker P4 diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
ω scans
5817 measured reflections
4871 independent reflections
3018 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 28.5°, θ_{min} = 2.0°
h = −1→14
k = −1→21
l = −14→14
3 standard reflections every 97 reflections
intensity decay: 4.0%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.073
S = 0.85
4871 reflections
456 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0321P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 1997a), $Fc^{*}=3DkFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0168 (8)

Special details

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

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}
O1	0.3818 (2)	0.06773 (12)	0.6832 (2)	0.0486 (5)
H1O	0.3916	0.0169	0.6962	0.058*
O2	0.32990 (19)	0.27491 (13)	0.32206 (16)	0.0507 (5)
O3	0.5318 (2)	0.40081 (12)	0.25040 (16)	0.0494 (5)
O4	0.77171 (18)	0.34608 (13)	0.53789 (16)	0.0474 (5)
N	0.5751 (2)	0.23305 (14)	0.60626 (18)	0.0370 (5)
H0	0.6560	0.2255	0.6625	0.044*
C1	0.6479 (3)	0.01575 (18)	0.5883 (3)	0.0474 (7)
H1	0.7156	0.0219	0.6714	0.057*
C2	0.6721 (3)	−0.0313 (2)	0.4944 (3)	0.0612 (9)
H2	0.7550	−0.0576	0.5151	0.073*
C3	0.5737 (4)	−0.0392 (2)	0.3703 (3)	0.0610 (9)
H3	0.5900	−0.0703	0.3066	0.073*
C4	0.4521 (4)	−0.0010 (2)	0.3416 (3)	0.0638 (9)
H4	0.3854	−0.0060	0.2578	0.077*
C5	0.4275 (3)	0.0449 (2)	0.4358 (2)	0.0504 (8)
H5	0.3440	0.0704	0.4150	0.061*
C6	0.5251 (2)	0.05343 (16)	0.5606 (2)	0.0347 (6)
C7	0.4963 (2)	0.10123 (16)	0.6653 (2)	0.0366 (6)
H7	0.5747	0.0967	0.7484	0.044*
C8	0.4650 (2)	0.19442 (15)	0.6333 (2)	0.0324 (5)
H8	0.3830	0.1982	0.5532	0.039*
C9	0.4399 (2)	0.24145 (15)	0.7424 (2)	0.0336 (6)
C10	0.3102 (3)	0.25466 (18)	0.7345 (2)	0.0467 (7)
H10	0.2377	0.2351	0.6618	0.056*
C11	0.2865 (3)	0.2964 (2)	0.8329 (3)	0.0574 (9)
H11	0.1986	0.3042	0.8266	0.069*
C12	0.3921 (3)	0.3265 (2)	0.9399 (3)	0.0563 (8)
H12	0.3762	0.3548	1.0063	0.068*
C13	0.5213 (3)	0.3146 (2)	0.9482 (3)	0.0551 (8)
H13	0.5933	0.3354	1.0202	0.066*
C14	0.5453 (3)	0.27216 (18)	0.8511 (2)	0.0464 (7)

H14	0.6336	0.2640	0.8585	0.056*
C15	0.5584 (2)	0.27859 (16)	0.5017 (2)	0.0315 (6)
C16	0.4473 (3)	0.29696 (17)	0.3756 (2)	0.0363 (6)
C17	0.5413 (3)	0.35431 (16)	0.3423 (2)	0.0362 (6)
C18	0.6450 (3)	0.33035 (16)	0.4681 (2)	0.0338 (6)
C19	0.8392 (3)	0.4036 (2)	0.4807 (3)	0.0526 (8)
H19A	0.8149	0.4613	0.4900	0.063*
H19B	0.8133	0.3916	0.3880	0.063*
C20	0.9872 (3)	0.3908 (2)	0.5528 (3)	0.0756 (11)
H20A	1.0117	0.4043	0.6439	0.091*
H20B	1.0354	0.4267	0.5162	0.091*
H20C	1.0094	0.3331	0.5448	0.091*
O1′	−0.12148 (19)	0.38791 (12)	0.1911 (2)	0.0507 (5)
H1′O	−0.0871	0.4343	0.2143	0.061*
O2′	−0.16835 (19)	0.19230 (14)	−0.18421 (17)	0.0558 (6)
O3′	0.0141 (2)	0.04816 (12)	−0.24695 (17)	0.0556 (6)
O4′	0.26944 (17)	0.11000 (12)	0.02442 (16)	0.0441 (5)
N′	0.0825 (2)	0.24079 (13)	0.08601 (18)	0.0358 (5)
H′	0.1540	0.2364	0.1565	0.043*
C1′	0.0554 (3)	0.20636 (18)	0.3724 (2)	0.0445 (7)
H1′	0.1442	0.2195	0.3868	0.053*
C2′	0.0295 (3)	0.1424 (2)	0.4433 (3)	0.0552 (9)
H2′	0.1007	0.1128	0.5046	0.066*
C3′	−0.1003 (4)	0.1220 (2)	0.4241 (3)	0.0607 (9)
H3′	−0.1177	0.0798	0.4738	0.073*
C4′	−0.2050 (3)	0.1646 (2)	0.3307 (3)	0.0614 (9)
H4′	−0.2936	0.1501	0.3153	0.074*
C5′	−0.1788 (3)	0.22893 (19)	0.2595 (3)	0.0503 (7)
H5′	−0.2504	0.2574	0.1968	0.060*
C6′	−0.0478 (2)	0.25167 (16)	0.2801 (2)	0.0364 (6)
C7′	−0.0186 (2)	0.32683 (16)	0.2115 (2)	0.0372 (6)
H7′	0.0685	0.3511	0.2676	0.045*
C8′	−0.0169 (2)	0.30707 (16)	0.0763 (2)	0.0352 (6)
H8′	−0.1063	0.2860	0.0201	0.042*
C9′	0.0120 (3)	0.38485 (15)	0.0112 (2)	0.0347 (6)
C10′	−0.0860 (3)	0.4190 (2)	−0.1005 (3)	0.0514 (8)
H10′	−0.1708	0.3942	−0.1364	0.062*
C11′	−0.0585 (4)	0.4891 (2)	−0.1587 (3)	0.0618 (9)
H11′	−0.1259	0.5118	−0.2327	0.074*
C12′	0.0660 (4)	0.5265 (2)	−0.1100 (3)	0.0591 (9)
H12′	0.0841	0.5732	−0.1514	0.071*
C13′	0.1627 (3)	0.4936 (2)	0.0006 (3)	0.0554 (8)
H13′	0.2473	0.5188	0.0356	0.066*
C14′	0.1371 (3)	0.42341 (18)	0.0614 (3)	0.0461 (7)
H14′	0.2044	0.4019	0.1366	0.055*
C15′	0.0633 (2)	0.18835 (16)	−0.0116 (2)	0.0315 (6)
C16′	−0.0529 (3)	0.16716 (17)	−0.1328 (3)	0.0376 (6)
C17′	0.0330 (3)	0.10166 (17)	−0.1615 (2)	0.0383 (6)

C18′	0.1416 (3)	0.12883 (16)	−0.0418 (2)	0.0335 (6)
C19′	0.3314 (3)	0.0519 (2)	−0.0384 (3)	0.0544 (8)
H19C	0.2973	0.0626	−0.1321	0.065*
H19D	0.3097	−0.0057	−0.0250	0.065*
C20′	0.4790 (3)	0.0644 (2)	0.0201 (3)	0.0657 (9)
H20D	0.5002	0.1201	0.0000	0.079*
H20E	0.5217	0.0236	−0.0153	0.079*
H20F	0.5110	0.0576	0.1137	0.079*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0624 (14)	0.0358 (11)	0.0616 (12)	−0.0064 (11)	0.0391 (11)	−0.0014 (11)
O2	0.0331 (11)	0.0647 (14)	0.0441 (10)	−0.0087 (10)	0.0036 (9)	0.0040 (10)
O3	0.0676 (13)	0.0447 (11)	0.0352 (9)	−0.0033 (11)	0.0191 (9)	0.0067 (9)
O4	0.0344 (11)	0.0594 (13)	0.0448 (10)	−0.0109 (10)	0.0114 (8)	0.0041 (10)
N	0.0245 (11)	0.0487 (13)	0.0304 (10)	−0.0016 (11)	0.0025 (9)	0.0082 (10)
C1	0.0414 (16)	0.0514 (18)	0.0519 (16)	0.0042 (15)	0.0207 (13)	0.0054 (15)
C2	0.064 (2)	0.054 (2)	0.082 (2)	0.0116 (18)	0.047 (2)	0.0060 (19)
C3	0.089 (3)	0.0481 (19)	0.069 (2)	−0.0051 (19)	0.055 (2)	−0.0104 (17)
C4	0.072 (2)	0.075 (2)	0.0468 (18)	−0.009 (2)	0.0248 (16)	−0.0136 (17)
C5	0.0466 (18)	0.062 (2)	0.0416 (15)	0.0029 (16)	0.0161 (14)	−0.0070 (15)
C6	0.0382 (15)	0.0333 (14)	0.0367 (12)	−0.0055 (12)	0.0188 (12)	0.0008 (11)
C7	0.0348 (14)	0.0392 (15)	0.0335 (12)	−0.0006 (12)	0.0106 (11)	0.0004 (11)
C8	0.0270 (12)	0.0373 (14)	0.0296 (11)	−0.0049 (11)	0.0072 (10)	0.0019 (11)
C9	0.0367 (14)	0.0276 (13)	0.0336 (12)	−0.0008 (12)	0.0106 (11)	0.0046 (11)
C10	0.0405 (16)	0.0516 (18)	0.0464 (14)	−0.0039 (15)	0.0150 (12)	−0.0028 (14)
C11	0.058 (2)	0.062 (2)	0.0619 (18)	0.0059 (18)	0.0338 (17)	−0.0062 (18)
C12	0.073 (2)	0.0517 (19)	0.0445 (15)	0.0154 (18)	0.0232 (16)	−0.0043 (15)
C13	0.062 (2)	0.0523 (19)	0.0399 (15)	0.0071 (18)	0.0070 (14)	−0.0076 (15)
C14	0.0424 (16)	0.0468 (17)	0.0425 (14)	0.0039 (14)	0.0081 (13)	−0.0055 (13)
C15	0.0277 (13)	0.0336 (14)	0.0307 (12)	−0.0008 (11)	0.0086 (10)	−0.0033 (11)
C16	0.0356 (16)	0.0396 (15)	0.0316 (12)	−0.0009 (14)	0.0108 (11)	−0.0023 (12)
C17	0.0466 (17)	0.0318 (14)	0.0308 (13)	0.0016 (13)	0.0157 (12)	−0.0037 (12)
C18	0.0326 (15)	0.0357 (14)	0.0323 (12)	0.0000 (13)	0.0118 (11)	−0.0027 (12)
C19	0.0468 (19)	0.0526 (18)	0.0672 (18)	−0.0105 (16)	0.0317 (16)	−0.0011 (17)
C20	0.0472 (19)	0.086 (3)	0.104 (3)	−0.014 (2)	0.0407 (19)	−0.029 (2)
O1′	0.0600 (14)	0.0360 (11)	0.0675 (13)	0.0087 (11)	0.0373 (11)	0.0045 (11)
O2′	0.0351 (12)	0.0644 (14)	0.0525 (11)	0.0074 (11)	−0.0002 (9)	−0.0066 (11)
O3′	0.0629 (14)	0.0490 (12)	0.0447 (10)	0.0041 (11)	0.0094 (10)	−0.0123 (10)
O4′	0.0351 (11)	0.0540 (12)	0.0408 (9)	0.0130 (10)	0.0120 (8)	−0.0052 (9)
N′	0.0315 (12)	0.0382 (12)	0.0303 (10)	0.0083 (10)	0.0039 (9)	−0.0029 (10)
C1′	0.0411 (16)	0.0423 (16)	0.0473 (14)	0.0002 (14)	0.0139 (13)	0.0008 (13)
C2′	0.060 (2)	0.0472 (18)	0.0484 (17)	0.0053 (17)	0.0105 (15)	0.0091 (16)
C3′	0.079 (3)	0.0433 (18)	0.067 (2)	−0.0036 (18)	0.0367 (18)	0.0083 (17)
C4′	0.051 (2)	0.053 (2)	0.086 (2)	−0.0125 (18)	0.0333 (18)	0.003 (2)
C5′	0.0438 (17)	0.0481 (17)	0.0558 (16)	−0.0005 (15)	0.0155 (13)	0.0056 (15)
C6′	0.0401 (15)	0.0338 (14)	0.0371 (13)	0.0002 (13)	0.0168 (12)	−0.0038 (11)

C7′	0.0360 (14)	0.0369 (15)	0.0380 (13)	0.0030 (12)	0.0134 (11)	0.0012 (12)
C8′	0.0291 (13)	0.0383 (14)	0.0333 (12)	0.0050 (12)	0.0068 (10)	−0.0024 (11)
C9′	0.0389 (14)	0.0348 (15)	0.0327 (12)	0.0056 (13)	0.0162 (11)	−0.0029 (11)
C10′	0.0495 (19)	0.0546 (19)	0.0428 (15)	0.0051 (16)	0.0100 (14)	0.0051 (15)
C11′	0.074 (2)	0.059 (2)	0.0468 (17)	0.0112 (19)	0.0180 (16)	0.0192 (16)
C12′	0.088 (3)	0.0454 (19)	0.0613 (19)	0.0021 (19)	0.0487 (19)	0.0081 (16)
C13′	0.054 (2)	0.0554 (19)	0.065 (2)	−0.0053 (17)	0.0322 (17)	0.0021 (18)
C14′	0.0436 (17)	0.0484 (17)	0.0459 (15)	0.0035 (14)	0.0168 (13)	0.0064 (14)
C15′	0.0328 (14)	0.0324 (14)	0.0286 (12)	−0.0005 (12)	0.0113 (11)	0.0016 (11)
C16′	0.0334 (17)	0.0389 (16)	0.0373 (13)	−0.0008 (13)	0.0101 (12)	0.0003 (12)
C17′	0.0422 (17)	0.0374 (15)	0.0319 (12)	0.0003 (14)	0.0107 (12)	−0.0014 (13)
C18′	0.0323 (15)	0.0357 (15)	0.0324 (12)	0.0043 (13)	0.0125 (11)	0.0028 (12)
C19′	0.0486 (19)	0.0586 (19)	0.0619 (17)	0.0151 (17)	0.0280 (15)	−0.0040 (17)
C20′	0.0481 (19)	0.078 (2)	0.078 (2)	0.0199 (19)	0.0313 (16)	0.014 (2)

Geometric parameters (Å, º)

O1—C7	1.423 (3)	O2′—C16′	1.216 (3)
O2—C16	1.221 (3)	O3′—C17′	1.227 (3)
O3—C17	1.226 (3)	O4′—C18′	1.319 (3)
O4—C18	1.305 (3)	O4′—C19′	1.461 (3)
O4—C19	1.453 (3)	N′—C15′	1.314 (3)
N—C15	1.315 (3)	N′—C8′	1.475 (3)
N—C8	1.463 (3)	C1′—C2′	1.375 (4)
C1—C6	1.372 (3)	C1′—C6′	1.383 (3)
C1—C2	1.382 (4)	C2′—C3′	1.368 (4)
C2—C3	1.376 (4)	C3′—C4′	1.376 (4)
C3—C4	1.363 (5)	C4′—C5′	1.382 (4)
C4—C5	1.377 (4)	C5′—C6′	1.387 (4)
C5—C6	1.379 (3)	C6′—C7′	1.510 (3)
C6—C7	1.512 (3)	C7′—C8′	1.532 (3)
C7—C8	1.528 (3)	C8′—C9′	1.521 (3)
C8—C9	1.527 (3)	C9′—C14′	1.384 (4)
C9—C10	1.381 (4)	C9′—C10′	1.386 (3)
C9—C14	1.383 (3)	C10′—C11′	1.374 (4)
C10—C11	1.378 (4)	C11′—C12′	1.370 (4)
C11—C12	1.370 (4)	C12′—C13′	1.366 (4)
C12—C13	1.371 (4)	C13′—C14′	1.383 (4)
C13—C14	1.373 (4)	C15′—C18′	1.390 (3)
C15—C18	1.396 (3)	C15′—C16′	1.474 (4)
C15—C16	1.471 (3)	C16′—C17′	1.505 (4)
C16—C17	1.508 (4)	C16′—C18′	2.033 (4)
C17—C18	1.456 (3)	C17′—C18′	1.450 (3)
C19—C20	1.493 (4)	C19′—C20′	1.478 (4)
O1′—C7′	1.423 (3)		
C18—O4—C19	116.5 (2)	C3′—C2′—C1′	120.4 (3)
C15—N—C8	124.3 (2)	C2′—C3′—C4′	119.4 (3)

C6—C1—C2	120.8 (3)	C3′—C4′—C5′	120.1 (3)
C3—C2—C1	120.1 (3)	C4′—C5′—C6′	121.1 (3)
C4—C3—C2	119.4 (3)	C1′—C6′—C5′	117.4 (3)
C3—C4—C5	120.5 (3)	C1′—C6′—C7′	121.2 (2)
C4—C5—C6	120.8 (3)	C5′—C6′—C7′	121.3 (2)
C1—C6—C5	118.4 (2)	O1′—C7′—C6′	108.58 (19)
C1—C6—C7	120.8 (2)	O1′—C7′—C8′	106.67 (18)
C5—C6—C7	120.7 (2)	C6′—C7′—C8′	114.2 (2)
O1—C7—C6	111.3 (2)	N′—C8′—C9′	109.79 (19)
O1—C7—C8	105.63 (19)	N′—C8′—C7′	110.92 (18)
C6—C7—C8	113.6 (2)	C9′—C8′—C7′	112.1 (2)
N—C8—C9	111.07 (19)	C14′—C9′—C10′	118.1 (3)
N—C8—C7	109.3 (2)	C14′—C9′—C8′	121.0 (2)
C9—C8—C7	112.38 (19)	C10′—C9′—C8′	120.9 (2)
C10—C9—C14	118.1 (2)	C11′—C10′—C9′	120.4 (3)
C10—C9—C8	120.5 (2)	C12′—C11′—C10′	121.5 (3)
C14—C9—C8	121.4 (2)	C13′—C12′—C11′	118.5 (3)
C11—C10—C9	120.9 (3)	C12′—C13′—C14′	121.1 (3)
C12—C11—C10	120.3 (3)	C13′—C14′—C9′	120.5 (3)
C11—C12—C13	119.4 (3)	N′—C15′—C18′	135.2 (2)
C12—C13—C14	120.5 (3)	N′—C15′—C16′	134.3 (2)
C13—C14—C9	120.8 (3)	C18′—C15′—C16′	90.4 (2)
N—C15—C18	132.8 (2)	O2′—C16′—C15′	134.7 (3)
N—C15—C16	136.5 (2)	O2′—C16′—C17′	136.7 (3)
C18—C15—C16	90.8 (2)	C15′—C16′—C17′	88.5 (2)
O2—C16—C15	135.3 (2)	O2′—C16′—C18′	177.8 (2)
O2—C16—C17	136.1 (2)	C15′—C16′—C18′	43.13 (14)
C15—C16—C17	88.6 (2)	C17′—C16′—C18′	45.41 (15)
O3—C17—C18	137.3 (3)	O3′—C17′—C18′	138.0 (3)
O3—C17—C16	135.7 (2)	O3′—C17′—C16′	135.0 (3)
C18—C17—C16	87.02 (19)	C18′—C17′—C16′	86.9 (2)
O4—C18—C15	127.4 (2)	O4′—C18′—C15′	128.3 (2)
O4—C18—C17	138.9 (2)	O4′—C18′—C17′	137.6 (2)
C15—C18—C17	93.6 (2)	C15′—C18′—C17′	94.1 (2)
O4—C19—C20	106.9 (2)	O4′—C18′—C16′	174.7 (2)
C18′—O4′—C19′	116.72 (19)	C15′—C18′—C16′	46.46 (15)
C15′—N′—C8′	120.86 (19)	C17′—C18′—C16′	47.66 (15)
C2′—C1′—C6′	121.5 (3)	O4′—C19′—C20′	108.5 (3)

C6—C1—C2—C3	1.5 (5)	C4′—C5′—C6′—C1′	1.4 (4)
C1—C2—C3—C4	−0.6 (5)	C4′—C5′—C6′—C7′	−174.4 (2)
C2—C3—C4—C5	−0.2 (5)	C1′—C6′—C7′—O1′	−144.5 (2)
C3—C4—C5—C6	0.3 (5)	C5′—C6′—C7′—O1′	31.2 (3)
C2—C1—C6—C5	−1.5 (4)	C1′—C6′—C7′—C8′	96.7 (3)
C2—C1—C6—C7	176.9 (3)	C5′—C6′—C7′—C8′	−87.7 (3)
C4—C5—C6—C1	0.6 (4)	C15′—N′—C8′—C9′	−86.4 (3)
C4—C5—C6—C7	−177.8 (3)	C15′—N′—C8′—C7′	149.2 (2)
C1—C6—C7—O1	−122.5 (3)	O1′—C7′—C8′—N′	−177.2 (2)

C5—C6—C7—O1	55.9 (3)	C6′—C7′—C8′—N′	−57.2 (3)
C1—C6—C7—C8	118.5 (3)	O1′—C7′—C8′—C9′	59.7 (2)
C5—C6—C7—C8	−63.2 (3)	C6′—C7′—C8′—C9′	179.7 (2)
C15—N—C8—C9	−106.2 (3)	N′—C8′—C9′—C14′	−55.8 (3)
C15—N—C8—C7	129.2 (2)	C7′—C8′—C9′—C14′	67.9 (3)
O1—C7—C8—N	−176.97 (19)	N′—C8′—C9′—C10′	123.9 (2)
C6—C7—C8—N	−54.7 (2)	C7′—C8′—C9′—C10′	−112.4 (2)
O1—C7—C8—C9	59.3 (2)	C14′—C9′—C10′—C11′	−0.1 (4)
C6—C7—C8—C9	−178.5 (2)	C8′—C9′—C10′—C11′	−179.8 (3)
N—C8—C9—C10	140.4 (2)	C9′—C10′—C11′—C12′	1.1 (5)
C7—C8—C9—C10	−96.8 (3)	C10′—C11′—C12′—C13′	−1.6 (5)
N—C8—C9—C14	−39.4 (3)	C11′—C12′—C13′—C14′	1.0 (4)
C7—C8—C9—C14	83.4 (3)	C12′—C13′—C14′—C9′	−0.1 (4)
C14—C9—C10—C11	−0.7 (4)	C10′—C9′—C14′—C13′	−0.4 (4)
C8—C9—C10—C11	179.5 (2)	C8′—C9′—C14′—C13′	179.3 (2)
C9—C10—C11—C12	0.8 (5)	C8′—N′—C15′—C18′	169.2 (3)
C10—C11—C12—C13	−0.1 (5)	C8′—N′—C15′—C16′	−13.0 (4)
C11—C12—C13—C14	−0.7 (5)	N′—C15′—C16′—O2′	2.1 (5)
C12—C13—C14—C9	0.7 (5)	C18′—C15′—C16′—O2′	−179.5 (3)
C10—C9—C14—C13	0.0 (4)	N′—C15′—C16′—C17′	−177.2 (3)
C8—C9—C14—C13	179.8 (3)	C18′—C15′—C16′—C17′	1.3 (2)
C8—N—C15—C18	168.6 (3)	N′—C15′—C16′—C18′	−178.5 (4)
C8—N—C15—C16	−11.3 (5)	O2′—C16′—C17′—O3′	−2.7 (6)
N—C15—C16—O2	−1.4 (6)	C15′—C16′—C17′—O3′	176.5 (3)
C18—C15—C16—O2	178.7 (3)	C18′—C16′—C17′—O3′	177.7 (4)
N—C15—C16—C17	178.6 (3)	O2′—C16′—C17′—C18′	179.6 (3)
C18—C15—C16—C17	−1.3 (2)	C15′—C16′—C17′—C18′	−1.2 (2)
O2—C16—C17—O3	2.2 (6)	C19′—O4′—C18′—C15′	−172.6 (3)
C15—C16—C17—O3	−177.8 (3)	C19′—O4′—C18′—C17′	7.5 (4)
O2—C16—C17—C18	−178.8 (3)	C19′—O4′—C18′—C16′	−162 (2)
C15—C16—C17—C18	1.2 (2)	N′—C15′—C18′—O4′	−2.8 (5)
C19—O4—C18—C15	−179.2 (2)	C16′—C15′—C18′—O4′	178.7 (3)
C19—O4—C18—C17	−0.8 (5)	N′—C15′—C18′—C17′	177.1 (3)
N—C15—C18—O4	0.4 (5)	C16′—C15′—C18′—C17′	−1.4 (2)
C16—C15—C18—O4	−179.7 (3)	N′—C15′—C18′—C16′	178.5 (4)
N—C15—C18—C17	−178.5 (3)	O3′—C17′—C18′—O4′	3.7 (6)
C16—C15—C18—C17	1.3 (2)	C16′—C17′—C18′—O4′	−178.7 (3)
O3—C17—C18—O4	−1.0 (6)	O3′—C17′—C18′—C15′	−176.3 (3)
C16—C17—C18—O4	180.0 (3)	C16′—C17′—C18′—C15′	1.3 (2)
O3—C17—C18—C15	177.7 (3)	O3′—C17′—C18′—C16′	−177.6 (4)
C16—C17—C18—C15	−1.3 (2)	O2′—C16′—C18′—O4′	−1 (8)
C18—O4—C19—C20	−162.0 (2)	C15′—C16′—C18′—O4′	−11 (2)
C6′—C1′—C2′—C3′	−0.2 (4)	C17′—C16′—C18′—O4′	171 (2)
C1′—C2′—C3′—C4′	1.8 (5)	O2′—C16′—C18′—C15′	10 (6)
C2′—C3′—C4′—C5′	−1.8 (5)	C17′—C16′—C18′—C15′	−178.2 (3)
C3′—C4′—C5′—C6′	0.2 (5)	O2′—C16′—C18′—C17′	−172 (6)
C2′—C1′—C6′—C5′	−1.4 (4)	C15′—C16′—C18′—C17′	178.2 (3)
C2′—C1′—C6′—C7′	174.4 (2)	C18′—O4′—C19′—C20′	157.6 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*···O3^{i}	0.82	2.01	2.811 (3)	165
O1′—H1′*O*···O3′^{ii}	0.82	1.95	2.764 (3)	173
N—H···O2′^{iii}	0.86	2.06	2.907 (3)	169
N′—H′···O2	0.86	2.15	2.968 (3)	159

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