Synthesis and structure of a new bulky bis(alkoxide) ligand on a terphenyl platform

Sudheer S. Kurup,³ Sandra Nasser,³ Cassandra L. Ward⁴* and Stanislav Groysman⁴*

*Department of Chemistry, Wayne State University, 5101 Cass Avenue, Detroit, Michigan 48202, USA, and ³Lumigen Instrument Center, Wayne State University, 5101 Cass Avenue, Detroit, Michigan 48202, USA. *Correspondence e-mail: ward@wayne.edu, groysman@wayne.edu

A new sterically bulky chelating bis(alkoxide) ligand 3,30-(1,10:40,100-terphenyl)-2,200-diylbis(2,2,4,4-tetramethylpentan-3-ol), (H2[OO]tBu), was prepared in a two-step process as the dichloromethane monosolvate, C36H50O2/C1CH2Cl2. The first step is a Suzuki–Miyaura coupling reaction between 2-bromophenylboronic acid and 1,4-diiodobenzene. The resulting 2,200-dibromo-1,10:40,100-terphenyl was reacted with tBuLi and hexamethylacetone to obtain the desired product. The crystal structure of H2[OO]tBu revealed an anti conformation of the [CPh2(OH)] fragments relative to the central phenyl. Furthermore, the hydroxyl groups point away from each other. Likely because of this anti–anti conformation, the attempts to synthesize first-row transition-metal complexes with H2[OO]tBu were not successful.

1. Chemical context

Bulky alkoxides are becoming increasingly used as ancillary ligands in group-transfer chemistry and catalysis (Brazeau & Doerrer, 2019; Chua & Duong, 2014; Hannigan et al., 2017; Jayasundara et al., 2018; Wannipurage et al., 2020). As a result of their stereoelectronic properties, profoundly weak-field bulky alkoxides enable formation of reactive low-coordinate high-spin middle and late transition-metal centers (Bellow et al., 2016b; Grass et al., 2019b). We have previously reported bulky monodentate alkoxides that led to reactive chromium and iron nitrene-transfer catalysts, (Bellow et al., 2015; Wannipurage et al., 2021; Yousif et al., 2015, 2018) and a series of low-coordinate cobalt carbene complexes capable of carbene transfer to isocyanides (Bellow et al., 2016a; Grass et al., 2019a, 2020). However, the lability of monodentate alkoxides affected catalyst stability and the substrate scope. To remediate the problem of lability of monodentate alkoxides, we have designed and synthesized a new chelating bis-(alkoxide) ligand [1,10:40,100'-terphenyl]-2,200-diylbis(diphenylmethanol) (H2[OO]Ph) (Fig. 1) (Kurup et al., 2019). The H2[OO]Ph ligand employs a 1,10:40,100'-terphenyl platform, which increases the bite angle between the alkoxide donors to form approximately seesaw transition-metal centers. While the isolated ligand precursor H2[OO]Ph exhibits an anti conformation of the [CPh2(OH)] fragments relative to the central phenyl in the solid state (crystals obtained at 238 K), the hydroxyl groups point towards the central phenyl, exhibiting overall an anti–syn conformation (Fig. 1) (Kurup et al., 2019). Furthermore, while two different isomers were observed by 1H NMR spectroscopy at low temperatures, a single species was observed at room temperature, suggesting...
facile equilibration of anti and syn conformers. As a result, H$_2$[OO]$^{\text{Ph}}$ led to the formation of the desired bis(alkoxide) complexes with iron and chromium (Fig. 1) (Kurup et al., 2019, 2020). The resulting iron complex exhibited broader range of nitrene transfer reactivity, forming a variety of symmetric azoarenes.

The success of this strategy led us to design a new, even bulkier ligand (H$_2$[OO]$^{\text{tBu}}$). The ligand was synthesized in a two-step procedure as described in Fig. 2. Previously reported 2,2’-dibromo-1,1’:4’,1’-terphenyl was synthesized through a Suzuki–Miyaura coupling reaction between 2-bromophenylboronic acid and 1,4-diiodobenzene following a literature procedure (Velian et al., 2010). Next, 2,2’-dibromo-1,1’:4’,1’-terphenyl was treated with tBuLi followed by hexamethylacetone. The formation of the desired product H$_2$[OO]$^{\text{tBu}}$ (35% isolated yield) was accompanied by the formation of significant amounts of p-terphenyl by-product (38% isolated yield). H$_2$[OO]$^{\text{tBu}}$ was characterized by $^1$H and $^{13}$C NMR spectroscopy, high-resolution mass spectrometry, and X-ray crystallography. $^1$H NMR spectroscopy demonstrates the presence of two isomers at room temperature in an approximately 2:1 ratio, as manifested by two tert-butyli resonances (1.05 and 1.03 ppm) and two OH resonances (2.09 and 2.07 ppm). This observation suggests that, in contrast to H$_2$[OO]$^{\text{Ph}}$, various isomers of H$_2$[OO]$^{\text{tBu}}$ do not readily interconvert at room temperature, possibly due to the more significant steric hindrance of the tert-butyl groups. An X-ray crystallography study (see below) suggests that in at least one of these isomers the hydroxyl groups are pointing away from each other; such an isomer is unlikely to coordinate a single metal in a chelating fashion. Accordingly, the reaction of H$_2$[OO]$^{\text{tBu}}$ with several representative transition-metal amides (M = Cr, Mn, Fe) failed to produce isolable complexes.

2. Structural commentary

The crystals of H$_2$[OO]$^{\text{tBu}}$ were obtained from dichloromethane at 238 K. The structure crystallized in space group $\text{P1}$ and is presented in Fig. 3. Selected bond distances and angles are given in Table 1. H$_2$[OO]$^{\text{tBu}}$ exhibits a crystallographic inversion center, with only half of the molecule occupying the asymmetric unit. In addition to H$_2$[OO]$^{\text{tBu}}$, the structure contains one solvent molecule (CH$_2$Cl$_2$) disordered by symmetry over two positions. Selected bond distances, angles, and torsion angles appear in Table 1. The lateral phenyls of the terphenyl unit are approximately perpendicular to the central phenyl ring, as indicated by the corresponding torsion angles close to 90° (see Table 1). Similar to the structure of H$_2$[OO]$^{\text{Ph}}$, H$_2$[OO]$^{\text{tBu}}$ manifests an anti conformation of the two [CtBu$_2$(OH)]$^+$ donors relative to the central phenyl ring.

Figure 1
Schematic representation of the ‘anti–syn’ structure of the previously synthesized H$_2$[OO]$^{\text{Ph}}$ ligand and its reactivity with transition-metal precursors.

Figure 2
Synthesis of H$_2$[OO]$^{\text{tBu}}$, its schematic structure, and the lack of well-defined reactivity with transition-metal amide precursors.

Figure 3
The structure of H$_2$[OO]$^{\text{tBu}}$ (50% probability ellipsoids) is shown with the co-crystallized dichloromethane solvent molecule. The dichloromethane carbon atom was found to be disordered about an inversion center; only one orientation is shown, which is not the one belonging to the asymmetric unit. Hydroxyl H atoms are disordered over two positions, both positions are shown above.

Acta Cryst. (2022). E78, 92–96
In contrast to the structure of H$_2$[OO]$^{\text{Ph}}$, the hydroxyls point away from each other in the structure of H$_2$[OO]$^{\text{tBu}}$, leading to an overall *anti*–*anti* conformation. This disposition results in the placement of the tert-butyl groups above and below the central phenyl ring. The presence of bulky groups on both sides of the central phenyl is likely responsible for the distortion of the terphenyl fragment, which is indicated by the C10—C15—C16 angle of 130.70 (15)$^\circ$ and the C14—C15—C16 angle of 110.28 (15)$^\circ$. Same distortion is likely responsible for the slight variation in (lateral) phenyl bond distances (Table 1).

### 3. Supramolecular features

H$_2$[OO]$^{\text{tBu}}$ forms one-dimensional polymer chains held together by hydrogen bonding between two neighboring molecules (Table 2). One polymer chain is shown in Fig. 4. This chain-like structure results from the *anti*–*anti* conformation of H$_2$[OO]$^{\text{tBu}}$ in which both hydroxyl groups are pointing outward and thus can hydrogen bond with neighboring molecules. The hydrogen-bond distance (indicated by the light-blue dashed lines in Fig. 4) is 2.13 (3) Å. It is also noted that, due to the inversion center present within the molecule, the hydroxyl hydrogen atoms are disordered over two positions. As the diffraction data was of adequate quality, we were able to locate both hydrogen positions in the difference map. The corresponding O—H bonds are very similar, 0.93 (2) and 0.94 (2) Å. Only one of these hydrogen atoms participates in the hydrogen-bonding network (alternating conformations for consecutive molecules). The solvent molecules are positioned above and below the chains.

### 4. Database survey

H$_2$[OO]$^{\text{tBu}}$ is a new compound that has not been previously synthesized and structurally characterized. As described above, the synthesis, structure, and coordination chemistry of the related compound H$_2$[OO]$^{\text{Ph}}$ has been previously reported by us (Kurup *et al.*, 2019) and reported in the Cambridge Structural Database (Groom *et al.*, 2016). We note that Agapie and coworkers have previously investigated structurally related 2,2$^{\text{00}}$-diphosphine-1,1$^{\text{00}}$:4$^{\text{00}}$,1$^{\text{00}}$-terphenyl ligands (Bailey & Agapie, 2021; Buss *et al.*, 2017) and Fortier and coworkers have investigated structurally related 2,2$^{\text{00}}$-diamide-1,1$^{\text{00}}$:4$^{\text{00}}$,1$^{\text{00}}$-terphenyl ligands (Fortier *et al.*, 2017; Yadav *et al.*, 2020). In contrast to H$_2$[OO]$^{\text{tBu}}$, both the diphosphine and the diamide terphenyl ligands serve as chelates for transition metals, adopting a syn geometry for the phospine/amide donors relative to the central phenyl ring.

### 5. Synthesis and crystallization

2,2$^{\text{00}}$-Dibromo-1,1$^{\text{00}}$:4$^{\text{00}}$,1$^{\text{00}}$-terphenyl (Velian *et al.*, 2010) (1.00 g, 2.5 mmol) was dissolved in 30 mL THF and cooled under...
238 K. To the cold solution ¹BuLi (1.7 M in pentane, 6.4 mL, 10.8 mmol) was added dropwise and the resulting solution was stirred for 4 h. This reaction mixture was then transferred to a round-bottom flask containing hexamethyacetone (8.7 mL, 5 mmol) in 20 mL of hexane and stirred for 24 h. The organic contents were extracted using a dichloromethane–water solvent system. The organic phase was dried over MgSO₄ and filtered. The filtrate was concentrated using a rotatory evaporator. The desired product H₂[OO]tBu was separated in filtered. The filtrate was concentrated using a rotatory evaporator. The desired product H₂[OO]tBu was separated in filtered. The filtrate was concentrated using a rotatory evaporator.

Fourier maps and refined to 50% occupancy. The CH₂Cl₂ solvent was also disordered by symmetry over two positions (due to the inversion center located at the hydrogen bond to the adjacent H₂[OO]tBu) over two positions. Two alternating positions were identified from the difference-Fourier maps and refined with 50% occupancy. The CH₂Cl₂ solvent was also disordered by symmetry over two positions and refined with 50% occupancy.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Data were acquired at 100 K with an Oxford 800 Cryostream low-temperature apparatus. Hydrogen atoms were placed in calculated positions using a standard riding model and refined isotropically (with the exception of hydroxyl hydrogens); all other atoms were refined anisotropically. The hydroxyl hydrogens were found to be disordered (due to the inversion center located at the hydrogen bond to the adjacent H₂[OO]tBu) over two positions. Two alternating positions were identified from the difference-Fourier maps and refined to 50% occupancy. The CH₂Cl₂ solvent was also disordered by symmetry over two positions and refined with 50% occupancy.

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Computing details
Data collection: APEX2 (Bruker, 2016); cell refinement: SAINT (Bruker, 2016); data reduction: SAINT (Bruker, 2016); program(s) used to solve structure: SHELXT2018/2 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2018/3 (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009).

3,3′-((1,1′:4′,1′′-Terphenyl)-2,2′′-diyl)bis(2,2,4,4-tetramethylpentan-3-ol) dichloromethane monosolvate

Crystal data
C₃₆H₅₀O₂·CH₂Cl₂
Mr = 599.68
Triclinic, P
a = 8.2449 (4) Å
b = 9.1248 (4) Å
c = 12.1825 (6) Å
α = 101.530 (2)°
β = 102.729 (3)°
γ = 109.200 (2)°
V = 806.53 (7) Å³

Z = 1
F(000) = 324
Dₐ = 1.235 Mg m⁻³
Mo Kα radiation, λ = 0.71073 Å
Cell parameters from 9980 reflections
θ = 2.5–27.2°
µ = 0.23 mm⁻¹
T = 100 K
Prism, colourless
0.15 × 0.1 × 0.04 mm

Data collection
Bruker APEXII CCD
diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8 pixels mm⁻¹
ω and φ scans
Absorption correction: multi-scan
(SADABS; Krause et al., 2015)
Tₘᵡᵣᵦ = 0.722, Tₘᵢₙᵦ = 0.746

Refinement
Refinement on F²
Least-squares matrix: full
R[F² > 2σ(F²)] = 0.051
wR(F²) = 0.136
S = 1.05
3559 reflections
211 parameters

Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
w = 1/[σ²(Fo²) + (0.0594P)² + 0.6108P]
where P = (Fo² + 2Fc²)/3

27 restraints

Acta Cryst. (2022). E78, 92-96
Supporting Information

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

$\Delta \rho_{\text{max}} = 0.57 \text{ e} \cdot \text{Å}^{-3}$

Special details

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

**Refinement.** Used Part -1 on the dichloromethane (DCM) and hydroxyl hydrogens because they were disordered by inversion symmetry (sof=0.5). In addition, RIGU/DFIX/SIMU were employed to model the disorder of the DCM solvent.

The structure of H2[OO]tBu was collected on a Bruker X8 APEX-II diffractometer with MoKα radiation and a graphite monochromator. The diffraction intensities were measured using a Bruker APEX-II CCD detector. Data were acquired at 100?K with an Oxford 800 Cryostream low-temperature apparatus. The data were processed using APEX3 software supplied by Bruker AXS. The structures were solved by Intrinsic Phasing using SHELXT (Sheldrick, 2015a) and refined with SHELXL-2018 (Sheldrick 2015b) using Olex2 (Dolomanov, 2009).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

|   | x       | y       | z       | Uiso*/Ueq | Occ. (<1) |
|---|---------|---------|---------|-----------|-----------|
| O1 | 0.55932 | 0.63933 | 0.10947 | 0.0224 (3)|           |
| H1A| 0.556   | 0.555   | 0.050   | 0.027*    | 0.5       |
| H1B| 0.594   | 0.737   | 0.088   | 0.027*    | 0.5       |
| C17| 0.8826  | 0.5620  | 0.53594 | 0.0176 (4)|           |
| H17| 0.801780| 0.603330| 0.562488| 0.021*    |           |
| C1 | 0.6733  | 0.6559  | 0.22428 | 0.0174 (4)|           |
| C15| 0.6438  | 0.4210  | 0.33668 | 0.0166 (4)|           |
| C14| 0.5253  | 0.2805  | 0.34915 | 0.0200 (4)|           |
| H14| 0.57142 | 0.23510 | 0.406337| 0.024*    |           |
| C16| 0.8316  | 0.4698  | 0.41871 | 0.0168 (4)|           |
| C18| 1.0488  | 0.5946  | 0.61443 | 0.0179 (4)|           |
| H18| 1.081840| 0.662282| 0.692598| 0.021*    |           |
| C10| 0.5770  | 0.4932  | 0.25311 | 0.0164 (4)|           |
| C12| 0.2780  | 0.2706  | 0.20251 | 0.0218 (4)|           |
| H12| 0.154405| 0.221280| 0.156261| 0.026*    |           |
| C8 | 0.8541  | 0.5146  | 0.13697 | 0.0228 (4)|           |
| H8A| 0.823600| 0.436332| 0.181305| 0.034*    |           |
| H8B| 0.971115| 0.526787| 0.125134| 0.034*    |           |
| H8C| 0.760907| 0.475262| 0.060292| 0.034*    |           |
| C13| 0.3453  | 0.2034  | 0.28338 | 0.0233 (4)|           |
| H13| 0.270041| 0.106475| 0.293847| 0.028*    |           |
| C2 | 0.6571  | 0.8019  | 0.31128 | 0.0196 (4)|           |
| C11| 0.3922  | 0.4115  | 0.18886 | 0.0201 (4)|           |
| H11| 0.342427| 0.455795| 0.132331| 0.024*    |           |
| C9 | 1.0251  | 0.7558  | 0.32004 | 0.0230 (4)|           |
| H9A| 1.046663| 0.870258| 0.352693| 0.034*    |           |
| H9B| 1.133028| 0.748183| 0.302648| 0.034*    |           |
| H9C| 0.997886| 0.697588| 0.377407| 0.034*    |           |
| C6 | 0.8648  | 0.6799  | 0.20637 | 0.0205 (4)|           |
| C7 | 0.9138  | 0.7914  | 0.12807 | 0.0242 (4)|           |
|       |  \(U_{11}\)   |  \(U_{22}\)   |  \(U_{33}\)   |  \(U_{12}\)   |  \(U_{13}\)   |  \(U_{23}\)   |
|-------|---------------|---------------|---------------|---------------|---------------|---------------|
| O1    | 0.0208 (7)    | 0.0255 (7)    | 0.0181 (7)    | 0.0073 (6)    | 0.0002 (5)    | 0.0097 (6)    |
| C17   | 0.0209 (9)    | 0.0168 (9)    | 0.0187 (9)    | 0.0109 (7)    | 0.0066 (7)    | 0.0063 (7)    |
| C1    | 0.0182 (9)    | 0.0162 (9)    | 0.0145 (8)    | 0.0053 (7)    | 0.0007 (7)    | 0.0045 (7)    |
| C15   | 0.0183 (9)    | 0.0159 (8)    | 0.0151 (8)    | 0.0078 (7)    | 0.0039 (7)    | 0.0028 (7)    |
| C14   | 0.0243 (9)    | 0.0202 (9)    | 0.0181 (9)    | 0.0105 (8)    | 0.0067 (7)    | 0.0079 (7)    |
| C16   | 0.0186 (9)    | 0.0149 (8)    | 0.0175 (8)    | 0.0065 (7)    | 0.0042 (7)    | 0.0077 (7)    |
| C18   | 0.0224 (9)    | 0.0155 (8)    | 0.0150 (8)    | 0.0081 (7)    | 0.0036 (7)    | 0.0044 (7)    |
| C10   | 0.0169 (8)    | 0.0143 (8)    | 0.0166 (8)    | 0.0058 (7)    | 0.0040 (7)    | 0.0036 (7)    |
| C12   | 0.0172 (9)    | 0.0220 (9)    | 0.0183 (9)    | 0.0030 (7)    | 0.0014 (7)    | 0.0018 (7)    |
| C8    | 0.0234 (9)    | 0.0247 (10)   | 0.0207 (9)    | 0.0093 (8)    | 0.0073 (8)    | 0.0070 (8)    |
| C13   | 0.0248 (10)   | 0.0181 (9)    | 0.0237 (10)   | 0.0028 (8)    | 0.0093 (8)    | 0.0069 (8)    |
| C2    | 0.0205 (9)    | 0.0171 (9)    | 0.0202 (9)    | 0.0082 (7)    | 0.0027 (7)    | 0.0058 (7)    |
| C11   | 0.0198 (9)    | 0.0216 (9)    | 0.0176 (9)    | 0.0077 (7)    | 0.0030 (7)    | 0.0065 (7)    |
| C9    | 0.0181 (9)    | 0.0232 (10)   | 0.0225 (9)    | 0.0041 (8)    | 0.0035 (7)    | 0.0060 (8)    |
| C6    | 0.0178 (9)    | 0.0224 (9)    | 0.0200 (9)    | 0.0057 (7)    | 0.0050 (7)    | 0.0077 (7)    |
| C7    | 0.0212 (9)    | 0.0246 (10)   | 0.0217 (9)    | 0.0022 (8)    | 0.0051 (7)    | 0.0092 (8)    |
| C5    | 0.0271 (10)   | 0.0201 (9)    | 0.0198 (9)    | 0.0099 (8)    | 0.0042 (8)    | 0.0037 (7)    |
| C3    | 0.0308 (11)   | 0.0181 (9)    | 0.0281 (10)   | 0.0092 (8)    | 0.0059 (8)    | 0.0071 (8)    |
| C4    | 0.0260 (10)   | 0.0268 (10)   | 0.0287 (10)   | 0.0153 (8)    | 0.0065 (8)    | 0.0066 (8)    |
| C11   | 0.103 (2)     | 0.0143 (6)    | 0.0325 (10)   | −0.0108 (9)   | 0.0021 (11)   | 0.0079 (6)    |
| C12   | 0.120 (2)     | 0.104 (2)     | 0.0643 (16)   | 0.093 (2)     | 0.0660 (15)   | 0.0541 (14)   |
| C19   | 0.057 (3)     | 0.029 (2)     | 0.0190 (19)   | 0.030 (2)     | 0.0121 (19)   | 0.0086 (17)   |
Geometric parameters (Å, °)

| Bond/Angle | Distance/Angle |
|------------|----------------|
| O1—H1A     | 0.933 (19)     |
| O1—H1B     | 0.944 (19)     |
| O1—C1      | 1.451 (2)      |
| C17—H17    | 0.9500         |
| C17—C16    | 1.395 (2)      |
| C17—C18    | 1.386 (2)      |
| C1—C10     | 1.586 (2)      |
| C1—C2      | 1.591 (3)      |
| C1—C6      | 1.591 (3)      |
| C15—C14    | 1.393 (2)      |
| C15—C16    | 1.511 (2)      |
| C15—C10    | 1.426 (2)      |
| C14—H14    | 0.9500         |
| C14—C13    | 1.380 (3)      |
| C16—C18i   | 1.393 (3)      |
| C18—H18    | 0.9500         |
| C10—C11    | 1.408 (2)      |
| C12—H12    | 0.9500         |
| C12—C13    | 1.371 (3)      |
| C12—C11    | 1.387 (3)      |
| C8—H8A     | 0.9800         |
| C8—H8B     | 0.9800         |
| C8—H8C     | 0.9800         |
| C8—C6      | 1.539 (3)      |
| C13—H13    | 0.9500         |

Bond/Angle:

| Bond/Angle | Distance/Angle |
|------------|----------------|
| H1A—O1     | 1.09 (4)       |
| C1—O1—H1A  | 1.13 (3)       |
| C1—O1—C1   | 1.12 (3)       |
| C16—C17—H17| 1.193          |
| C18—C17—H17| 1.193          |
| C18—C17—C16| 1.2135 (16)    |
| O1—C1—C10  | 1.0449 (13)    |
| O1—C1—C2   | 1.0430 (13)    |
| O1—C1—C6   | 1.0428 (13)    |
| C10—C1—C2  | 1.0849 (14)    |
| C10—C1—C6  | 1.1507 (14)    |
| C6—C1—C2   | 1.1818 (14)    |
| C14—C15—C16| 1.1028 (15)    |
| C14—C15—C10| 1.1901 (16)    |
| C10—C15—C16| 1.5070 (15)    |
| C15—C14—C14| 1.1811         |
| C13—C14—C15| 1.2388 (17)    |
| C17—C16—C15| 1.1205 (15)    |
| C18—C16—C17| 1.1709 (16)    |
C18 — C16 — C15 121.23 (15) H7B — C7 — H7C 109.5
C17 — C18 — C16i 121.49 (16) C2 — C5 — H5A 109.5
C17 — C18 — H18 119.3 C2 — C5 — H5B 109.5
C16i — C18 — H18 119.3 C2 — C5 — H5C 109.5
C15 — C10 — C1 130.84 (15) H5A — C5 — H5B 109.5
C11 — C10 — C1 113.90 (15) H5A — C5 — H5C 109.5
C11 — C10 — C15 115.18 (15) H5B — C5 — H5C 109.5
C13 — C12 — H12 120.3 C2 — C3 — H3A 109.5
C13 — C12 — C11 119.34 (17) C2 — C3 — H3B 109.5
C11 — C12 — H12 120.3 C2 — C3 — H3C 109.5
H8A — C8 — H8B 109.5 H3A — C3 — H3B 109.5
H8A — C8 — H8C 109.5 H3A — C3 — H3C 109.5
H8B — C8 — H8C 109.5 H3B — C3 — H3C 109.5
C6 — C8 — H8A 109.5 C2 — C4 — H4A 109.5
C6 — C8 — H8B 109.5 C2 — C4 — H4B 109.5
C6 — C8 — H8C 109.5 C2 — C4 — H4C 109.5
C14 — C13 — H13 120.9 H4A — C4 — H4B 109.5
C12 — C13 — C14 118.13 (17) H4A — C4 — H4C 109.5
C12 — C13 — H13 120.9 H4B — C4 — H4C 109.5
C5 — C2 — C1 113.25 (14) C11 — C19 — C12 110.9 (2)
C5 — C2 — C3 107.88 (15) C11 — C19 — H19A 109.5
C5 — C2 — C4 105.36 (15) C11 — C19 — H19B 109.5
C3 — C2 — C1 113.32 (15) C12 — C19 — H19A 109.5
C3 — C2 — C4 106.35 (15) C12 — C19 — H19B 109.5
C4 — C2 — C1 110.16 (15) H19A — C19 — H19B 108.1
O1 — C1 — C10 — C15 162.41 (17) C10 — C1 — C2 — C5 54.70 (19)
O1 — C1 — C10 — C11 −21.10 (19) C10 — C1 — C2 — C3 178.00 (14)
O1 — C1 — C2 — C5 166.21 (14) C10 — C1 — C2 — C4 −63.00 (18)
O1 — C1 — C2 — C3 −70.49 (18) C10 — C1 — C6 — C8 39.81 (19)
O1 — C1 — C2 — C4 48.51 (18) C10 — C1 — C6 — C9 −85.03 (19)
O1 — C1 — C6 — C8 −74.63 (16) C10 — C1 — C6 — C7 154.74 (15)
O1 — C1 — C6 — C9 160.53 (15) C10 — C15 — C14 — C13 1.3 (3)
O1 — C1 — C6 — C7 40.31 (19) C10 — C15 — C16 — C17 96.7 (2)
C1 — C10 — C11 — C12 −177.15 (17) C10 — C15 — C16 — C18i −92.7 (2)
C15 — C14 — C13 — C12 −1.3 (3) C13 — C12 — C11 — C10 0.1 (3)
C15 — C10 — C11 — C12 −0.1 (3) C2 — C1 — C10 — C15 −86.5 (2)
C14 — C15 — C16 — C17 −84.3 (2) C2 — C1 — C10 — C11 89.94 (17)
C14 — C15 — C16 — C18i 86.4 (2) C2 — C1 — C6 — C8 170.19 (14)
C14 — C15 — C10 — C1 175.90 (17) C2 — C1 — C6 — C9 45.3 (2)
C14 — C15 — C10 — C11 −0.6 (2) C2 — C1 — C6 — C7 −74.88 (19)
C16 — C17 — C18 — C16i −3.1 (3) C11 — C12 — C13 — C14 0.6 (3)
C16 — C15 — C14 — C13 −177.91 (17) C6 — C1 — C10 — C15 48.4 (2)
C16 — C15 — C10 — C1 −5.1 (3) C6 — C1 — C10 — C11 −135.13 (16)
C16 — C15 — C10 — C11 178.46 (17) C6 — C1 — C2 — C5 −78.62 (19)
C18—C17—C16—C15  174.02 (16)  C6—C1—C2—C3  44.7 (2)
C18—C17—C16—C18^i  3.0 (3)  C6—C1—C2—C4  163.68 (15)

Symmetry code: (i) −x+2, −y+1, −z+1.

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| O1—H1A···O1^ii | 0.93 (4) | 2.13 (3) | 3.0066 (19) | 157 (4) |

Symmetry code: (ii) −x+1, −y+1, −z.