Crystal structure of a trifluoromethyl benzoato quadruple-bonded dimolybdenum complex

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The study of quadruple bonds between transition metals, in particular those of dimolybdenum, has revealed much about the two-electron bond. The solid-state structure of the quadruple-bonded dimolybdenum(II) complex tetrakis[μ-4-(trifluoromethyl)benzoato-κ²O:O']bis[(tetrahydrofuran-κO)molybdenum(II)] 0.762-pentane 0.238-tetrahydrofuran solvate, [Mo₂(p-O₂CC₆H₄CF₃)₄(THF)₄]·0.762C₅H₁₂·0.238C₄H₈O or [Mo₂(C₈H₄F₃O₂)₄(C₄H₈O)₂]·0.762C₅H₁₂·0.238C₆H₄O is reported. The complex crystallizes within a triclinic cell and low symmetry (P1) results from the intercalated pentane/THF solvent molecules. The paddlewheel structure at 100 K has inversion symmetry and comprises four bridging carboxylate ligands encases the Mo₂(II,II) core that is characterized by two axially coordinated THF molecules and an Mo–Mo distance of 2.1098 (7) Å.

1. Chemical context

The σ²π⁴δ² quadruple bond has contributed prominently to the elucidation of the single most distinguishing feature of the discipline of chemistry – the two-electron bond (Lewis, 1916). As originally defined with the inception of valence and molecular orbital bonding models (Heitler & London, 1927; Pauling, 1928; Lennard-Jones, 1929; Mulliken, 1932; James & Coolidge, 1933; Coulson & Fischer, 1949), the two-electron bond forms from pairing two electrons in two orbitals. Remarkably, the four states that characterize the two-electron bond remained undefined experimentally for over 60 years owing to the dissociative nature of the σ and π antibonding orbitals. This experimental challenge was overcome with the two-electron δ bond of quadruple-bonded metal–metal complexes. Anchored by a σ²π⁴ framework and sterically locking ligands, the four states of the two-electron bond, 1δδ, 3δδ*, 1δ*δ* and 1δ*δ*, were experimentally defined for dimolybdenum quadruple-bond complexes (Engebretson et al. 1994, 1999; Cotton & Nocera, 2000). Within the group of dimolybdenum quadruple-bond complexes, the tetraacetates are exemplars. The initial structure of Mo₂O₂C₂H₄₄ by Lawton & Mason (1965) established the existence of the quadruple bond in the now familiar paddlewheel arrangement of acetates. The dimolybdenum bond distance of 2.11 Å in this structure was subsequently refined nearly a decade later to be 2.0934 (8) Å (Cotton et al., 1974). Intriguingly, many subsequent structures have shown that the inductive effect of the R group on the carboxylic acid does not perturb the Mo–Mo bond distance, indicating the robustness of the two-electron bond within a quadruple-bond architecture. It has been postulated...
that the strength of the Mo—Mo quadruple bond may be perturbed, but only in cases where R is a strong electron-withdrawing group and there is a good axial donor ligand (Cotton et al., 1978). To add further to an understanding of Mo2(II,II) quadruple bond distances, we examined a dimolybdenum core ligated by trifluoromethylbenzoate with THF axial donor ligands. We now report the synthesis and X-ray crystal structure of tetrakis(μ-4-trifluoromethylbenzoato)dimolybdenum(II) 0.762-pentane 0.238-tetrahydrofuran solvate [Mo2(p-O2CC6H4CF3)4]2THF]0.762C5H120.238C4H8O. The presence of the CF3 electron-withdrawing group on the bridging benzoate ligands, together with the donor THF axial ligands, results in a slightly elongated metal–metal bond distance as compared to its benzoate congener, Mo2(O2CC6H5)4.

2. Structural commentary

The dimolybdenum complex, [Mo2(p-O2CC6H4CF3)4]2THF] (Fig. 1), was characterized by using single-crystal X-ray diffraction. Half of the molecule (Fig. 1) resides in the asymmetric unit, with the complete molecule generated by inversion about the quadruple-bond inversion center. The fluorine atoms of the trifluoromethyl groups are rotationally disordered and the highest occupancy positions are shown in Fig. 1. The crystallization solvents, THF and pentane, are disordered (0.238:0.762) (Fig. 2).

Selected bond metrics for Mo2(p-O2CC6H4CF3)4]2THF are listed in Table 1. Complete lists of the structural metrics for the compound are presented in the Supporting information. The Mo—Mo bond length is 2.1098 (7) Å. Whereas the bond distance is within the typical range of dimolybdenum quadruple bond lengths of 2.06–2.17 Å (Cotton et al., 2002), it is slightly longer than what is observed for dimolybdenum cores bridged by carboxylates. As a comparison, the dimolybdenum bond distance in the Mo2(O2CC6H5)4 congener, is 2.096 (1) Å. Thus, with the addition of a CF3 group in the 4-position of benzoate, the Mo—Mo bond length increases by 0.014 (2) Å.

A similar trend is observed for the bond distances in the primary coordination sphere. The minimum Mo—O bond distance decreases by 0.008 (5) Å, and the maximum Mo—O bond distance decreases by 0.011 (5) Å as compared to Mo2(O2CC6H5)4. The most significant decrease in bond metrics is observed for the Mo—O1S axial ligand distance, which results in a decrease of 0.033 (4) Å for the axial coordinated oxygen atom of THF as compared to the axially coordinated oxygen in Mo2(O2CC6H5)4. However, we note for this compound that the oxygen is provided from a carboxylate ligand of a neighboring molecule as opposed to an axially coordinated solvent molecule. Consequently, as proposed by Cotton (Cotton et al., 1978), the presence of ligands about the dimolybdenum center that are electron withdrawing and donating in the axial position is needed to perturb the overall bonding within a quadruple-bond framework. To this point, the metrics of [Mo2(p-O2CC6H4CF3)4]2THF] are indistinguishable from those of Mo2(O2CC6F5)4]2THF (Han, 2011).

The electron-withdrawing nature of the fluoro-substituted benzoates is established by their pKas as compared to that of benzoate (pKa = 1.75, 3.77 and 4.20 for C6F5COOH, p-CF3C6H4COOH and C6H5COOH, respectively; Rumble, 2021; Boiadjiev & Lightner, 1999). That an electron-withdrawing group alone is insufficient to perturb the dimolybdenum bond distance is indicated by a comparison of the structures for Mo2(O2CCH3)4 and Mo2(O2CCF3)4. The d(Mo—Mo) of 2.0934 (8) and 2.090 (4) Å for Mo2(O2CCH3)4 and

Table 1

| Bond | Distance (Å) |
|------|--------------|
| Mo1—O1 | 2.0996 (17) |
| Mo1—O4 | 2.1030 (17) |
| Mo1—O2′ | 2.1076 (17) |
| O1—Mo1—O1′ | 93.20 (5) |
| O4—Mo1—O1′ | 92.37 (5) |

Symmetry code: (i) −x, −y+1, −z+1.

Figure 1

Ellipsoid plot of the dimolybdenum complex. The CF3 groups are rotationally disordered, therefore the highest occupancy positions are shown for each atom. Hydrogen atoms and unbound solvent are omitted for clarity.
Mo$_2$(O$_2$CCF$_3$)$_4$, respectively (Cotton & Norman, 1971; Cotton et al., 1974), are indistinguishable despite a significant difference in electron-withdrawing properties \( pK_a(CH_3COOH) = 4.76 \), \( pK_a(CF_3COOH) = 0.52 \); Rumble, 2021. Thus, a donor ligand is needed in addition to electron-withdrawing carboxylate equatorial ligands to observe a difference in the dimolybdenum quadruple bond.

3. Supramolecular features

The structure was solved in the triclinic space group \( P\overline{1} \) with a half of an Mo-dimer per asymmetric unit and one full molecule per unit cell (Fig. 2). The low symmetry arises from the presence of disordered THF/pentane solvent molecules embedded within a solvent channel arising from the crystal packing. The disordered solvents are situated in the body-center of eight \( [\text{Mo}_2(p-O_2C_6H_4CF_3)_4]_4 \cdot \text{THF} \) complexes with two THF molecules skewed towards the pentane; the next nearest neighbors are a series of four trifluoromethyl groups from distinct \( [\text{Mo}_2(p-O_2C_6H_4CF_3)_4]_4 \cdot \text{THF} \) complexes. These four trifluoromethyl groups are oriented tangentially to the solvent channel (Fig. 2b) along the \( b \)-axis direction with a volume of 162 Å$^3$ for one void volume within the unit cell according to established methods for determining solvent-accessible voids (van der Sluis & Spek, 1990). The adjacent pairs of symmetry-related benzene rings (C10–C16) in the \( p-O_2C_6H_4CF_3 \) ligands interact through aromatic \( \pi-\pi \) stacking interactions with a face-to-face distance of 3.7856 (9) Å (Fig. 2b) and form a one-dimensional chain. In addition, the trifluoromethyl group of a \( p-O_2C_6H_4CF_3 \) ligand (C10–C16 and F4–F6) is perpendicular to the aromatic plane of a neighboring \( p-O_2C_6H_4CF_3 \) ligand (C1–C7 and F1–F3) with weak C–F⋯C interactions (Kawahara et al., 2004) [the distances between the F atoms and the C2–C8 plane are 3.024 (2)–3.430 (1) Å]. The coordinated THF molecules also have weak C–H⋯F interactions (D’Oria & Novoa, 2008) with the trifluoromethyl group of the \( p-O_2C_6H_4CF_3 \) ligands [the C–H⋯F distances are 2.568 (1)–3.045 (1) Å].

4. Database survey

In a search of the Cambridge Structural Database (WebCSD, accessed 17 December 2021; Groom et al., 2016), the CSD search fragment, C$_3$H$_8$F$_2$Mo$_2$O$_8$, for Mo$_2$(O$_2$CC$_6$H$_4$CF$_3$)$_4$ yielded no hits in the database and the CSD search fragment, C$_{40}$H$_{32}$F$_{12}$Mo$_2$O$_{10}$, for \( [\text{Mo}_2(p-O_2C_6H_4CF_3)_4]_4 \cdot \text{THF} \) also yielded no hits. The CSD reference code for Mo$_2$(O$_2$CC$_6$F$_5$)$_4$ (Han, 2011) is AYODOI, for Mo$_2$(O$_2$CCH$_3$)$_4$ (Cotton et al., 1978) is MOBZOA, for Mo$_2$(O$_2$CCF)$_4$ (Cotton et al., 1974) is MOLACE01, and for Mo$_2$(O$_2$CCF)$_4$ (Cotton & Norman, 1971) is TFACMO.

5. Purification and crystallization

The overall synthetic scheme is shown in the reaction scheme. Molybdenum hexacarbonyl, 4-(trifluoromethyl) benzoic acid, THF, and 1,2-dichlorobenzene were purchased from Sigma-Aldrich.
Aldrich. Mo(CO)₆ and 4-(trifluoromethyl)benzoic acid were combined in a flask with THF and anhydrous 1,2-dichlorobenzene. The reaction was heated under reflux for 24 h at 413 K under nitrogen (Pence et al., 1999). The reaction mixture was cooled, the solution was filtered and the collected residue was washed with dichloromethane and hexanes.

The crystallization was set up in a glove box. The crude product was dissolved in THF and recrystallized by vapor diffusion of pentane using a 6 by 50 mm borosilicate glass crystallization tube housed within a 20 mL glass vial. The assembly was allowed to stand at 238 K for 24 days. Orange rectangular crystals were observed and harvested for X-ray diffraction analysis.

6. Refinement
Crystal data, data collection and structure refinement details are included in Table 2. Hydrogen atoms on C atoms were placed at idealized positions and refined using a riding model. The isotropic displacement parameters of all hydrogen atoms were fixed to 1.2 times the atoms to which they are linked (1.5 times for methyl groups). Rotational and positional disorder for one trifluoromethyl substituent containing C1 and C13 was modeled. The overlapping solvent molecules (assigned as THF and pentane based on solvent crystallization conditions and apparent arrangement of electron-density peaks) were disordered adjacent to an inversion center (special position). The restraints on bond lengths and constraints of the atomic displacement parameters on each pair of disorder fragments (SADI/SAME and EADP instructions of SHELXL2014) were applied for the disorder refinement (Zheng et al., 2008). Crystallographic refinement details, including disorder modeling and the software employed, are given in the crystallographic information file (*.cif). To stabilize the refinement model, 713 restraints (SADI/SAME and RIGU/SIMU) were applied to accommodate the disordered trifluoromethyl group, the coordinated THF molecules, as well as the THF/pentane solvent molecules in the channel as detailed by Müller et al. (2006) to furnish a data+restraint-to-parameter ratio of 9.75. This ratio increases to 11.6 if the disordered THF/pentane solvent molecules in the channel are squeezed out of the structure.

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Computing details

Data collection: APEX2 (Bruker, 2015); cell refinement: SAINT (Bruker, 2015); data reduction: SAINT (Bruker, 2015); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2014 (Sheldrick, 2015b); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXL (Sheldrick, 2008) and PLATON (Spek, 2020).

Tetrakis[µ-4-(trifluoromethyl)benzoato-κ²O:O']bis[(tetrahydrofuran-κO)molybdenum(II)] 0.762-pentane 0.238-tetrahydrofuran solvate

Crystal data

$\mathrm{[Mo_2(C_8H_4F_2O_2)(C_4H_8O)_2] \cdot 0.762C_5H_{12} \cdot 0.238C_4H_8O}$

$M_r = 1164.68$

Triclinic, $P\overline{1}$

$a = 7.7687$ (17) Å

$b = 12.099$ (5) Å

$c = 12.572$ (2) Å

$α = 85.843$ (13)°

$β = 81.208$ (8)°

$γ = 83.107$ (16)°

$V = 1157.6$ (6) Å$^3$

$Z = 1$

$F(000) = 586$

$D_x = 1.671$ Mg m$^{-3}$

Mo $Kα$ radiation, $λ = 0.71073$ Å

Cell parameters from 9835 reflections

$θ = 2.5$–27.2°

$μ = 0.65$ mm$^{-1}$

$T = 100$ K

Block, orange

$0.30 \times 0.13 \times 0.06$ mm

Data collection

Bruker D8 goniometer with Photon 100 CMOS detector
diffractometer

Radiation source: $Iμ$S microfocus tube

$ω$ and phi scans

Absorption correction: multi-scan

(SADABS; Krause et al., 2015)

$T_{\text{min}} = 0.701$, $T_{\text{max}} = 0.745$

4933 measured reflections

39433 independent reflections

3814 reflections with $I > 2σ(I)$

$R_{\text{int}} = 0.033$

$θ_{\text{max}} = 25.1°$, $θ_{\text{min}} = 2.7°$

$h = −9→9$

$k = −14→14$

$l = −14→14$

Refinement

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2σ(F^2)] = 0.028$

$wR(F^2) = 0.064$

$S = 1.12$

4094 reflections

493 parameters

713 restraints

Primary atom site location: dual

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
\[ w = \frac{1}{[\sigma^2(F_o^2) + (0.0191P)^2 + 2.0296P]} \]
\[ P = (F_o^2 + 2F_c^2)/3 \]
\[ \Delta \sigma_{\text{max}} < 0.001 \]
\[ \Delta \rho_{\text{max}} = 0.67 \text{ e} \text{ Å}^{-3} \]
\[ \Delta \rho_{\text{min}} = -0.39 \text{ e} \text{ Å}^{-3} \]

**Supporting Information**

**Experimental.** A single orange plate (0.297 mm × 0.132 mm × 0.056 mm) was chosen for single crystal X-ray diffraction using a Bruker three-circle platform goniometer equipped with an Photon100 CMOS detector. Data were collected as a series of \( \phi \) and/or \( \omega \) scans. Data integration down to 0.84 Å resolution was carried out using SAINT V8.37A with reflection spot size optimization. Absorption corrections were made with the program SADABS 2016/2 (Krause et al., 2015). Space group assignments were determined by examination of systematic absences, \( E \)-statistics, and successive refinement of the structures. The structure was solved by the Intrinsic Phasing methods and refined by least squares methods also using SHELXT-2014 and SHELXL-2014 with the OLEX 2 (Dolomanov et al., 2019) interface. The program PLATON (Spek, 2020) was employed to confirm the absence of higher symmetry space groups. All non-H atoms, including the disorder fragment, were located in difference Fourier maps, and then refined anisotropically. Outlier reflections were omitted from refinement when appropriate.

**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.** 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. All non-H atoms, as well as the disordered atoms were located in difference-Fourier maps, and then refined anisotropically. The restraints on bond lengths and constraints of the atomic displacement parameters on each pair of disorder fragments (SADI/SAME and EADP instructions of SHELXL-2014) as well as the restraints of the atomic displacement parameters (SIMU/RIGU instructions of SHELXL-2014), if necessary, have been applied for the disorder refinement.

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

|       | x     | y     | z     | \( U_{eq} \) | Occ. (<1) |
|-------|-------|-------|-------|--------------|-----------|
| Mo1   | -0.08669 (3) | 0.43800 (2) | 0.51162 (2) | 0.01390 (8) | 0.796 (10) |
| C1    | 0.8554 (11)  | 0.0198 (7)  | 0.3139 (9)  | 0.0362 (17) | 0.796 (10) |
| F1    | 0.8182 (10)  | -0.0767 (5) | 0.2806 (5)  | 0.0615 (15) | 0.796 (10) |
| F2    | 0.9308 (6)   | -0.0074 (4) | 0.4035 (4)  | 0.0685 (14) | 0.796 (10) |
| F3    | 0.9798 (5)   | 0.0542 (3)  | 0.2420 (4)  | 0.0629 (14) | 0.796 (10) |
| C1A   | 0.840 (5)    | 0.013 (4)   | 0.301 (5)   | 0.054 (5)   | 0.204 (10) |
| F1A   | 0.817 (4)    | -0.083 (2)  | 0.327 (2)   | 0.066 (5)   | 0.204 (10) |
| F2A   | 0.9862 (16)  | 0.0311 (12) | 0.3439 (19) | 0.059 (4)   | 0.204 (10) |
| F3A   | 0.908 (2)    | 0.0366 (15) | 0.1933 (16) | 0.080 (5)   | 0.204 (10) |
| C14   | -0.273 (2)   | 0.6434 (15) | -0.1264 (11)| 0.044 (4)   | 0.38 (3)   |
| F4    | -0.421 (2)   | 0.7128 (18) | -0.1227 (17)| 0.052 (4)   | 0.38 (3)   |
| F5    | -0.303 (3)   | 0.5510 (13) | -0.1700 (15)| 0.055 (3)   | 0.38 (3)   |
| F6    | -0.1534 (18) | 0.692 (2)   | -0.1933 (11)| 0.059 (3)   | 0.38 (3)   |
| C14A  | -0.2779 (13) | 0.6523 (8)  | -0.1247 (6) | 0.034 (2)   | 0.62 (3)   |
| F4A   | -0.4522 (11) | 0.6827 (10) | -0.1123 (11)| 0.0398 (19) | 0.62 (3)   |
| F5A   | -0.2538 (17) | 0.5654 (8)  | -0.1889 (9) | 0.044 (2)   | 0.62 (3)   |
| F6A   | -0.1972 (17) | 0.7326 (11) | -0.1815 (8) | 0.053 (2)   | 0.62 (3)   |
| O1    | 0.1106 (2)   | 0.31186 (14)| 0.45777 (13)| 0.0156 (4)  | 0.62 (3)   |
| O2    | 0.2954 (2)   | 0.44209 (14)| 0.43387 (13)| 0.0154 (4)  | 0.62 (3)   |
|   |   |   |   |   |
|---|---|---|---|---|
| O3  | 0.0284 (2) | 0.60139 (14) | 0.32975 (13) | 0.0159 (4) |
| O4  | −0.1488 (2) | 0.46706 (14) | 0.35423 (13) | 0.0160 (4) |
| C2  | 0.6966 (4) | 0.1020 (2) | 0.3359 (2) | 0.0258 (6) |
| C3  | 0.5312 (4) | 0.0680 (2) | 0.3649 (2) | 0.0287 (6) |
| H3  | 0.5155 | −0.0086 | 0.3645 | 0.034* |
| C4  | 0.3887 (4) | 0.1452 (2) | 0.3945 (2) | 0.0238 (6) |
| H4  | 0.2752 | 0.1218 | 0.4143 | 0.029* |
| C5  | 0.4124 (3) | 0.2572 (2) | 0.39516 (19) | 0.0169 (5) |
| C6  | 0.5781 (3) | 0.2909 (2) | 0.3642 (2) | 0.0189 (5) |
| H6  | 0.5937 | 0.3677 | 0.3635 | 0.023* |
| C7  | 0.7206 (3) | 0.2141 (2) | 0.3345 (2) | 0.0231 (6) |
| H7  | 0.8337 | 0.2376 | 0.3133 | 0.028* |
| C8  | 0.2638 (3) | 0.3418 (2) | 0.43068 (19) | 0.0159 (5) |
| C9  | −0.0782 (3) | 0.5447 (2) | 0.2967 (2) | 0.0173 (5) |
| C10 | −0.1245 (3) | 0.5704 (2) | 0.1862 (2) | 0.0190 (5) |
| C11 | −0.2181 (3) | 0.4987 (2) | 0.1418 (2) | 0.0233 (6) |
| H11 | −0.2507 | 0.4327 | 0.1815 | 0.028* |
| C12 | −0.2635 (4) | 0.5235 (3) | 0.0398 (2) | 0.0284 (7) |
| H12 | −0.3263 | 0.4743 | 0.0091 | 0.034* |
| C13 | −0.2173 (4) | 0.6201 (3) | −0.0171 (2) | 0.0282 (7) |
| C15 | −0.1237 (4) | 0.6919 (3) | 0.0260 (2) | 0.0304 (7) |
| H15 | −0.0920 | 0.7580 | −0.0138 | 0.036* |
| C16 | −0.0768 (4) | 0.6667 (2) | 0.1275 (2) | 0.0250 (6) |
| H16 | −0.0117 | 0.7153 | 0.1572 | 0.030* |
| O1S | −0.3188 (2) | 0.31076 (15) | 0.58783 (14) | 0.0215 (4) |
| C1S | −0.237 (3) | 0.1999 (13) | 0.6187 (15) | 0.025 (3) |
| H1SA | −0.1085 | 0.1989 | 0.6102 | 0.030* |
| H1SB | −0.2658 | 0.1435 | 0.5728 | 0.030* |
| C2S | −0.310 (5) | 0.175 (3) | 0.735 (2) | 0.032 (4) |
| H2SA | −0.2329 | 0.1961 | 0.7844 | 0.038* |
| H2SB | −0.3254 | 0.0946 | 0.7494 | 0.038* |
| C3S | −0.4855 (15) | 0.2465 (9) | 0.7481 (8) | 0.033 (3) |
| H3SA | −0.5817 | 0.2005 | 0.7457 | 0.039* |
| H3SB | −0.5062 | 0.2823 | 0.8180 | 0.039* |
| C4S | −0.4781 (18) | 0.3292 (12) | 0.6608 (11) | 0.020 (3) |
| H4SA | −0.5790 | 0.3272 | 0.6217 | 0.024* |
| H4SB | −0.4868 | 0.4040 | 0.6896 | 0.024* |
| O1T | −0.3188 (2) | 0.31076 (15) | 0.58783 (14) | 0.0215 (4) |
| C1T | −0.2628 (19) | 0.1954 (9) | 0.6098 (9) | 0.025 (2) |
| H1TA | −0.1365 | 0.1778 | 0.5831 | 0.030* |
| H1TB | −0.3303 | 0.1473 | 0.5756 | 0.030* |
| C2T | −0.298 (3) | 0.1793 (19) | 0.7318 (12) | 0.029 (3) |
| H2TA | −0.1874 | 0.1726 | 0.7628 | 0.035* |
| H2TB | −0.3569 | 0.1113 | 0.7536 | 0.035* |
| C3T | −0.4179 (10) | 0.2836 (6) | 0.7699 (4) | 0.0307 (17) |
| H3TA | −0.5269 | 0.2626 | 0.8142 | 0.037* |
| H3TB | −0.3577 | 0.3276 | 0.8133 | 0.037* |
| C4T | −0.4562 (12) | 0.3465 (8) | 0.6735 (7) | 0.021 (2) |
| Atomic coordinates (Å) | Mo1      | C1       | F1       | F2       | F3       | C1A      | F1A      | F2A      | F3A      | C14      | F4       | F5       |
|------------------------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|
|                         | 0.01239 (11) | 0.01700 (12) | 0.01261 (12) | −0.00162 (8) | −0.00291 (8) | −0.00050 (8) |             |          |          |          |          |          |
|                         | 0.030 (3)   | 0.027 (3)   | 0.051 (3)   | 0.002 (2)   | −0.003 (2)   | −0.009 (2)   |             |          |          |          |          |          |
|                         | 0.044 (2)   | 0.036 (2)   | 0.104 (4)   | 0.0089 (15) | −0.002 (3)   | −0.040 (3)   |             |          |          |          |          |          |
|                         | 0.058 (2)   | 0.075 (3)   | 0.064 (2)   | 0.0443 (19) | −0.0200 (19) | −0.0107 (19) |             |          |          |          |          |          |
|                         | 0.035 (2)   | 0.0470 (17) | 0.092 (3)   | 0.0125 (14) | 0.0261 (19)  | −0.0070 (18) |             |          |          |          |          |          |
|                         | 0.030 (8)   | 0.048 (8)   | 0.084 (9)   | 0.013 (6)   | −0.003 (6)   | −0.033 (7)   |             |          |          |          |          |          |
|                         | 0.033 (7)   | 0.043 (6)   | 0.116 (13)  | 0.016 (5)   | 0.003 (9)    | −0.029 (8)   |             |          |          |          |          |          |
|                         | 0.028 (6)   | 0.049 (7)   | 0.101 (11)  | 0.012 (4)   | −0.011 (6)   | −0.029 (7)   |             |          |          |          |          |          |
|                         | 0.042 (8)   | 0.092 (9)   | 0.095 (8)   | 0.030 (6)   | 0.010 (6)    | −0.029 (6)   |             |          |          |          |          |          |
|                         | 0.035 (6)   | 0.070 (6)   | 0.021 (7)   | 0.018 (4)   | −0.010 (4)   | −0.003 (5)   |             |          |          |          |          |          |
|                         | 0.043 (5)   | 0.081 (7)   | 0.022 (4)   | 0.033 (5)   | −0.009 (4)   | 0.006 (6)    |             |          |          |          |          |          |
|                         | 0.058 (7)   | 0.091 (5)   | 0.015 (5)   | 0.000 (4)   | −0.008 (5)   | −0.009 (4)   |             |          |          |          |          |          |
Geometric parameters (Å, °)

|       | Mo1—O1   | C1S—C2S   | C1—H1SA   | C1S—C2S   | C1S—H1SA   |
|-------|----------|-----------|-----------|-----------|------------|
| Mo1   | 2.0996 (17) | 1.512 (13) | 0.9900    |           |            |
| O1    | 0.0200 (9)  | 0.0255 (10) | 0.0013 (7) | 0.0011 (7) | 0.0022 (7) |
| O2T   | 0.0200 (9)  | 0.0255 (10) | 0.0013 (7) | 0.0011 (7) | 0.0022 (7) |
| C1T   | 0.021 (4)   | 0.037 (5)   | 0.004 (4)  | 0.001 (4)  | 0.003 (3)  |
| C2T   | 0.035 (6)   | 0.032 (5)   | 0.003 (5)  | 0.003 (4)  | 0.004 (4)  |
| C3T   | 0.034 (4)   | 0.031 (4)   | 0.002 (3)  | 0.009 (3)  | 0.002 (3)  |
| C4T   | 0.010 (4)   | 0.024 (4)   | 0.008 (3)  | 0.006 (3)  | 0.005 (3)  |
| C5S   | 0.080 (8)   | 0.064 (8)   | 0.009 (6)  | 0.005 (6)  | 0.015 (7)  |
| C6S   | 0.082 (8)   | 0.042 (6)   | 0.015 (6)  | 0.005 (6)  | 0.027 (5)  |
| C7S   | 0.086 (8)   | 0.056 (6)   | 0.013 (7)  | 0.002 (6)  | 0.025 (6)  |
| C8S   | 0.101 (9)   | 0.076 (11)  | 0.012 (7)  | 0.011 (7)  | 0.013 (7)  |
| C9S   | 0.109 (10)  | 0.079 (11)  | 0.002 (9)  | 0.019 (8)  | 0.015 (12) |
| O2S   | 0.087 (11)  | 0.049 (11)  | 0.025 (9)  | 0.011 (10) | 0.020 (9)  |
| C10S  | 0.094 (12)  | 0.052 (11)  | 0.022 (10) | 0.005 (11) | 0.026 (10) |
| C11S  | 0.092 (12)  | 0.051 (11)  | 0.026 (10) | 0.012 (10) | 0.031 (10) |
| C12S  | 0.101 (13)  | 0.067 (12)  | 0.013 (11) | 0.008 (11) | 0.019 (11) |
| C13S  | 0.087 (12)  | 0.072 (11)  | 0.014 (11) | 0.020 (11) | 0.025 (10) |
| Bond               | Length (Å) | Bond               | Length (Å) |
|--------------------|------------|--------------------|------------|
| Mo1—O2i            | 2.1076 (17) | C1S—H1SB          | 0.9900     |
| Mo1—Mo1i           | 2.1098 (7)  | C2S—C3S           | 1.519 (17) |
| Mo1—O3i            | 2.1204 (17)| C2S—H2SA          | 0.9900     |
| Mo1—O1T            | 2.5422 (19)| C2S—H2SB          | 0.9900     |
| Mo1—O1S            | 2.5422 (19)| C3S—C4S           | 1.430 (12) |
| C1—F3              | 1.304 (12)  | C3S—H3SA          | 0.9900     |
| C1—F1              | 1.345 (11)  | C3S—H3SB          | 0.9900     |
| C1—F2              | 1.349 (11)  | C4S—H4SA          | 0.9900     |
| C1—C2              | 1.493 (6)   | C4S—H4SB          | 0.9900     |
| C1A—F1A            | 1.21 (7)    | O1T—C1T           | 1.431 (9)  |
| C1A—F2A            | 1.38 (5)    | O1T—C4T           | 1.447 (7)  |
| C1A—F3A            | 1.40 (7)    | C1T—C2T           | 1.518 (11) |
| C1A—C2             | 1.494 (18)  | C1T—H1TA          | 0.9900     |
| C14—F6             | 1.314 (14)  | C1T—H1TB          | 0.9900     |
| C14—F4             | 1.341 (13)  | C2T—C3T           | 1.535 (13) |
| C14—F5             | 1.341 (14)  | C2T—H2TA          | 0.9900     |
| C14—C13            | 1.500 (13)  | C2T—H2TB          | 0.9900     |
| C14A—F6A           | 1.326 (8)   | C3T—C4T           | 1.434 (8)  |
| C14A—F4A           | 1.348 (9)   | C3T—H3TA          | 0.9900     |
| C14A—F5A           | 1.349 (8)   | C3T—H3TB          | 0.9900     |
| C14A—C13           | 1.509 (8)   | C4T—H4TA          | 0.9900     |
| O1—C8              | 1.275 (3)   | C4T—H4TB          | 0.9900     |
| O2—C8              | 1.271 (3)   | C5S—C6S           | 1.605 (14) |
| O2—Mo1i            | 2.1076 (17) | C5S—H5SA          | 0.9800     |
| O3—C9              | 1.269 (3)   | C5S—H5SB          | 0.9800     |
| O3—Mo1i            | 2.1204 (17)| C5S—H5SC          | 0.9800     |
| O4—C9              | 1.270 (3)   | C6S—C7S           | 1.602 (14) |
| C2—C3              | 1.384 (4)   | C6S—H6SA          | 0.9900     |
| C2—C7              | 1.389 (4)   | C6S—H6SB          | 0.9900     |
| C3—C4              | 1.382 (4)   | C7S—C8S           | 1.590 (15) |
| C3—H3              | 0.9500      | C7S—H7SA          | 0.9900     |
| C4—C5              | 1.390 (4)   | C7S—H7SB          | 0.9900     |
| C4—H4              | 0.9500      | C8S—C9S           | 1.607 (15) |
| C5—C6              | 1.387 (4)   | C8S—H8SA          | 0.9900     |
| C5—C8              | 1.485 (3)   | C8S—H8SB          | 0.9900     |
| C6—C7              | 1.381 (4)   | C9S—H9SA          | 0.9800     |
| C6—H6              | 0.9500      | C9S—H9SB          | 0.9800     |
| C7—H7              | 0.9500      | C9S—H9SC          | 0.9800     |
| C9—C10             | 1.490 (3)   | O2S—C13S          | 1.425 (18) |
| C10—C16            | 1.391 (4)   | O2S—C10S          | 1.442 (18) |
| C10—C11            | 1.392 (4)   | C10S—C11S         | 1.505 (18) |
| C11—C12            | 1.385 (4)   | C10S—H10A         | 0.9900     |
| C11—H11            | 0.9500      | C10S—H10B         | 0.9900     |
| C12—C13            | 1.382 (4)   | C11S—C12S         | 1.53 (2)   |
| C12—H12            | 0.9500      | C11S—H11A         | 0.9900     |
| C13—C15            | 1.385 (4)   | C11S—H11B         | 0.9900     |
| C15—C16            | 1.383 (4)   | C12S—C13S         | 1.436 (18) |
| C15—H15            | 0.9500      | C12S—H12A         | 0.9900     |
| Bond          | Length (Å) | Bond          | Length (Å) | Bond          | Length (Å) |
|---------------|------------|---------------|------------|---------------|------------|
| C16—H16      | 0.9500     | C12S—H12B     | 0.9900     | C13S—H13A     | 0.9900     |
| O1S—C4S      | 1.428 (11) | O1S—C1S       | 1.465 (12) |               |            |
|               |            |               |            |               |            |
| O1—Mo1—O4    | 89.95 (7)  | H1SA—C1S—H1SB | 108.6      |               |            |
| O1—Mo1—O2i   | 176.69 (6) | C1S—C2S—C3S  | 102.6 (12) |               |            |
| O4—Mo1—O2i   | 89.57 (7)  | C1S—C2S—H2SA | 111.3      |               |            |
| O1—Mo1—Mo1i  | 93.20 (5)  | C3S—C2S—H2SA | 111.3      |               |            |
| O4—Mo1—Mo1i  | 92.37 (5)  | C1S—C2S—H2SB | 111.3      |               |            |
| O2i—Mo1—O3i  | 88.41 (7)  | H2SA—C2S—H2SB| 109.2      |               |            |
| O4—Mo1—O3i   | 176.47 (7) | C4S—C3S—C2S  | 106.9 (10) |               |            |
| O2i—Mo1—O3i  | 91.89 (6)  | C4S—C3S—H3SA | 110.3      |               |            |
| Mo1i—Mo1—O3i | 90.84 (5)  | C2S—C3S—H3SA | 110.3      |               |            |
| O1—Mo1—O1T   | 96.83 (6)  | C4S—C3S—H3SB | 110.3      |               |            |
| O4—Mo1—O1T   | 98.97 (6)  | C2S—C3S—H3SB | 110.3      |               |            |
| O2i—Mo1—O1T  | 80.01 (6)  | H3SA—C3S—H3SB| 108.6      |               |            |
| Mo1—Mo1—O1T  | 164.83 (4) | O1S—C4S—C3S  | 111.1 (8)  |               |            |
| O3i—Mo1—O1T  | 78.13 (6)  | O1S—C4S—H4SA | 109.4      |               |            |
| O1—Mo1—O1S   | 96.83 (6)  | C3S—C4S—H4SA | 109.4      |               |            |
| O4—Mo1—O1S   | 98.97 (6)  | O1S—C4S—H4SB | 109.4      |               |            |
| O2i—Mo1—O1S  | 80.01 (6)  | C3S—C4S—H4SB | 109.4      |               |            |
| Mo1—Mo1—O1S  | 164.83 (4) | H4SA—C4S—H4SB| 108.0      |               |            |
| O3i—Mo1—O1S  | 78.13 (6)  | C1T—O1T—C4T  | 108.1 (6)  |               |            |
| F3—C1—F1     | 106.4 (6)  | C1T—O1T—Mo1  | 118.0 (6)  |               |            |
| F3—C1—F2     | 105.0 (7)  | C4T—O1T—Mo1  | 120.2 (5)  |               |            |
| F1—C1—F2     | 106.0 (8)  | O1T—C1T—C2T  | 104.5 (9)  |               |            |
| F3—C1—C2     | 114.9 (8)  | O1T—C1T—H1TA | 110.8      |               |            |
| F1—C1—C2     | 112.5 (7)  | C2T—C1T—H1TA | 110.8      |               |            |
| F2—C1—C2     | 111.3 (6)  | O1T—C1T—H1TB | 110.8      |               |            |
| F1A—C1A—F2A  | 105 (4)    | C2T—C1T—H1TB | 110.8      |               |            |
| F1A—C1A—F3A  | 117 (3)    | H1TA—C1T—H1TB| 108.9      |               |            |
| F2A—C1A—F3A  | 97 (4)     | C1T—C2T—C3T  | 105.4 (6)  |               |            |
| F1A—C1A—C2   | 118 (5)    | C1T—C2T—H2TA | 110.7      |               |            |
| F2A—C1A—C2   | 108 (3)    | C3T—C2T—H2TA | 110.7      |               |            |
| F3A—C1A—C2   | 109 (4)    | C1T—C2T—H2TB | 110.7      |               |            |
| F6—C14—F4    | 106.6 (13) | C3T—C2T—H2TB | 110.7      |               |            |
| F6—C14—F5    | 108.3 (13) | H2TA—C2T—H2TB| 108.8      |               |            |
| F4—C14—F5    | 106.3 (15) | C4T—C3T—C2T  | 105.6 (7)  |               |            |
| F6—C14—C13   | 111.0 (12) | C4T—C3T—H3TA | 110.6      |               |            |
| F4—C14—C13   | 111.7 (14) | C2T—C3T—H3TA | 110.6      |               |            |
| F5—C14—C13   | 112.6 (14) | C4T—C3T—H3TB | 110.6      |               |            |
| F6A—C14A—F4A | 108.9 (7)  | C2T—C3T—H3TB | 110.6      |               |            |
| F6A—C14A—F5A | 105.8 (7)  | H3TA—C3T—H3TB| 108.7      |               |            |
| F4A—C14A—F5A | 104.3 (8)  | C3T—C4T—O1T  | 106.8 (5)  |               |            |
| F6A—C14A—C13 | 114.5 (7)  | C3T—C4T—H4TA | 110.4      |               |            |
| F4A—C14A—C13 | 110.8 (8)  | O1T—C4T—H4TA | 110.4      |               |            |
| F5A—C14A—C13 | 112.0 (8)  | C3T—C4T—H4TB | 110.4      |               |            |
C8—O1—Mo1 115.86 (15)  O1T—C4T—H4TB 110.4
C8—O2—Mo1 118.49 (15)  H4TA—C4T—H4TB 108.6
C9—O3—Mo1 117.28 (15)  C6S—C5S—H5SA 109.5
C9—O4—Mo1 116.71 (15)  C6S—C5S—H5SB 109.5
C3—C2—C7  120.3 (3)  H5SA—C5S—H5SC 109.5
C3—C2—C1  121.5 (5)  C6S—C5S—H5SC 109.5
C7—C2—C1  118.0 (5)  H5SA—C5S—H5SC 109.5
C3—C2—C1A  116 (2)  H5SB—C5S—H5SC 109.5
C7—C2—C1A  124 (3)  C7S—C6S—C5S 106.0 (10)
C4—C3—C2  120.2 (3)  C7S—C6S—H6SA 110.5
C4—C3—H3  119.9  C5S—C6S—H6SA 110.5
C2—C3—H3  119.9  C7S—C6S—H6SB 110.5
C3—C4—C5  119.7 (3)  C5S—C6S—H6SB 110.5
C3—C4—H4  120.2  H6SA—C6S—H6SB 108.7
C5—C4—H4  120.2  C8S—C7S—C6S 105.2 (12)
C6—C5—C4  119.8 (2)  C8S—C7S—H7SA 110.7
C6—C5—C8  119.0 (2)  C6S—C7S—H7SA 110.7
C4—C5—C8  121.2 (2)  C8S—C7S—H7SB 110.7
C7—C6—C5  120.7 (2)  C6S—C7S—H7SB 110.7
C7—C6—H6  119.7  H7SA—C7S—H7SB 108.8
C5—C6—H6  119.7  C7S—C8S—C9S 104.7 (14)
C6—C7—C2  119.3 (3)  C7S—C8S—H8SA 110.8
C6—C7—H7  120.4  C9S—C8S—H8SA 110.8
C2—C7—H7  120.4  C7S—C8S—H8SB 110.8
O2—C8—O1  122.3 (2)  C9S—C8S—H8SB 110.8
O2—C8—C5  118.2 (2)  H8SA—C8S—H8SB 108.9
O1—C8—C5  119.5 (2)  C8S—C9S—H9SA 109.5
O3—C9—O4  122.8 (2)  C8S—C9S—H9SB 109.5
O3—C9—C10  119.1 (2)  H9SA—C9S—H9SB 109.5
O4—C9—C10  118.1 (2)  C8S—C9S—H9SC 109.5
C16—C10—C11  119.7 (2)  H9SA—C9S—H9SC 109.5
C16—C10—C9  120.3 (2)  C8S—C9S—H9SC 109.5
C11—C10—C9  120.0 (2)  H9SB—C9S—H9SC 109.5
C12—C11—C10  120.0 (3)  C13S—O2S—C10S 108.7 (18)
C12—C11—H11  120.0  O2S—C10S—C11S 107.7 (16)
C10—C11—H11  120.0  O2S—C10S—H10A 110.2
C13—C12—C11  119.8 (3)  C11S—C10S—H10A 110.2
C13—C12—H12  120.1  O2S—C10S—H10B 110.2
C11—C12—H12  120.1  C11S—C10S—H10B 110.2
C12—C13—C15  120.8 (3)  C10S—C11S—C12S 103.5 (16)
C12—C13—C14  117.3 (7)  C10S—C11S—H11A 111.1
C15—C13—C14  121.9 (7)  C12S—C11S—H11A 111.1
C12—C13—C14A  120.1 (5)  C10S—C11S—H11B 111.1
C15—C13—C14A  119.0 (5)  C12S—C11S—H11B 111.1
C16—C15—C13  119.5 (3)  H11A—C11S—H11B 109.0
C16—C15—H15  120.2  C13S—C12S—C11S 105.2 (19)
C13—C15—H15  120.2  C13S—C12S—H12A 110.7
C15—C16—C10  120.3 (3)  C11S—C12S—H12A 110.7
| Bond                  | Length (Å) | Bond                  | Length (Å) |
|----------------------|------------|----------------------|------------|
| C15—C16—H16         | 119.9      | C13S—C12S—H12B      | 110.7      |
| C10—C16—H16         | 119.9      | C11S—C12S—H12B      | 110.7      |
| C4S—O1S—C1S         | 106.0 (9)  | H12A—C12S—H12B      | 108.8      |
| C4S—O1S—Mo1         | 131.9 (7)  | O2S—C13S—C12S       | 108 (2)    |
| C1S—O1S—Mo1         | 110.8 (10) | O2S—C13S—H13A       | 110.1      |
| O1S—C1S—C2S         | 107.0 (10) | C12S—C13S—H13A      | 110.1      |
| O1S—C1S—H1SA        | 110.3      | O2S—C13S—H13B       | 110.1      |
| C2S—C1S—H1SA        | 110.3      | C12S—C13S—H13B      | 110.1      |
| O1S—C1S—H1SB        | 110.3      | H13A—C13S—H13B      | 108.4      |
| C2S—C1S—H1SB        | 110.3      |                       |            |
| F3—C1—C2—C3         | 148.4 (6)  | C11—C12—C13—C15     | 0.9 (4)    |
| F1—C1—C2—C3         | 26.5 (11)  | C11—C12—C13—C14     | −179.0 (8) |
| F2—C1—C2—C3         | −92.4 (8)  | C11—C12—C13—C14A    | −175.4 (5) |
| F3—C1—C2—C7         | −36.4 (9)  | F6—C14—C13—C12      | −144.3 (13)|
| F1—C1—C2—C7         | −158.3 (6) | F4—C14—C13—C12      | 96.8 (18)  |
| F2—C1—C2—C7         | 82.8 (9)   | F5—C14—C13—C12      | −22.7 (17) |
| F1A—C1A—C2—C3       | −20 (6)    | F6—C14—C13—C15      | 35.7 (17)  |
| F2A—C1A—C2—C3       | −139 (3)   | F4—C14—C13—C15      | −83.1 (18) |
| F3A—C1A—C2—C3       | 117 (3)    | F5—C14—C13—C15      | 157.3 (12) |
| F1A—C1A—C2—C7       | 164 (4)    | F6A—C14A—C13—C12    | −166.7 (8) |
| F2A—C1A—C2—C7       | 44 (6)     | F4A—C14A—C13—C12    | 69.7 (10)  |
| F3A—C1A—C2—C7       | −60 (4)    | F5A—C14A—C13—C12    | −46.3 (11) |
| C7—C2—C3—C4         | −1.1 (4)   | F6A—C14A—C13—C15    | 16.9 (10)  |
| C1—C2—C3—C4         | 174.0 (5)  | C13—C15—C16—C10     | −0.6 (4)   |
| C1A—C2—C3—C4        | −178 (3)   | C14A—C13—C15—C16    | 176.0 (5)  |
| C2—C3—C4—C5         | −0.1 (4)   | C9—C10—C16—C15      | −178.4 (2) |
| C3—C4—C5—C6         | 1.2 (4)    | C12—C13—C15—C16     | 179.6 (8)  |
| C3—C4—C5—C8         | −177.5 (2) | C14A—C13—C15—C16    | 176.0 (5)  |
| C4—C5—C6—C7         | −1.1 (4)   | C13—C15—C16—C10     | 0.6 (4)    |
| C8—C5—C6—C7         | 177.6 (2)  | C14—C13—C15—C16     | 176.0 (5)  |
| C5—C6—C7—C2         | −0.1 (4)   | C10—C16—C15         | 0.8 (4)    |
| C3—C2—C7—C6         | 1.2 (4)    | C14—C13—C15—C16     | 179.6 (8)  |
| C1—C2—C7—C6         | −174.1 (5) | Mo1—O1S—C1S—C2S     | 125 (3)    |
| C1A—C2—C7—C6        | 177 (3)    | O1S—C1S—C2S—C3S     | 25 (4)     |
| Mo1′—O2—C8—O1       | 1.0 (3)    | C1S—C2S—C3S—C4S     | −18 (4)    |
| Mo1′—O2—C8—C5       | −178.29 (15) | C1S—O1S—C4S—C3S    | 11.2 (18)  |
| Mo1—O1—C8—O2        | −1.3 (3)   | Mo1—O1S—C4S—C3S     | −127.7 (9) |
| Mo1—O1—C8—C5        | 177.94 (16)| C2S—C3S—C4S—O1S     | 5 (3)      |
| C6—C5—C8—O2         | −1.7 (3)   | C4T—O1T—C1T—C2T     | −28.3 (17) |
| C4—C5—C8—O2         | 177.0 (2)  | Mo1—O1T—C1T—C2T     | 112.4 (15) |
| C6—C5—C8—O1         | 179.0 (2)  | O1T—C1T—C2T—C3T     | 14 (2)     |
| C4—C5—C8—O1         | −2.3 (4)   | C1T—C2T—C3T—C4T     | 5 (2)      |
| Mo1′—O3—C9—O4       | −0.1 (3)   | C2T—C3T—C4T—O1T     | −21.8 (15) |
| Mo1′—O3—C9—C10      | 179.30 (16)| C1T—O1T—C4T—C3T     | 32.4 (11)  |
| Mo1—O4—C9—O3        | 1.7 (3)    | Mo1—O1T—C4T—C3T     | −107.3 (6) |
| Mo1—O4—C9—C10       | −177.75 (16)| C5S—C6S—C7S—C8S    | −170.7 (15)|
| O3—C9—C10—C16       | −11.2 (4)  | C6S—C7S—C8S—C9S     | −172.8 (17)|
| Bond                  | Angle (°) (e) | | Bond                  | Angle (°) (e) |
|----------------------|--------------|---|----------------------|--------------|
| O4—C9—C10—C16       | 168.3 (2)    |   | C13S—O2S—C10S—C11S  | −6 (6)       |
| O3—C9—C10—C11       | 169.7 (2)    |   | O2S—C10S—C11S—C12S  | −10 (5)      |
| O4—C9—C10—C11       | −10.8 (3)    |   | C10S—C11S—C12S—C13S | 22 (6)       |
| C16—C10—C11—C12     | −0.2 (4)     |   | C10S—O2S—C13S—C12S  | 21 (7)       |
| C9—C10—C11—C12      | 179.0 (2)    |   | C11S—C12S—C13S—O2S  | −27 (7)      |
| C10—C11—C12—C13     | −0.7 (4)     |   |                       |              |

Symmetry code: (i) −x, y+1, z+1.