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# Di-*tert*-butyl 2,2′-(biphenyl-2,2′-diyldioxy)diacetate

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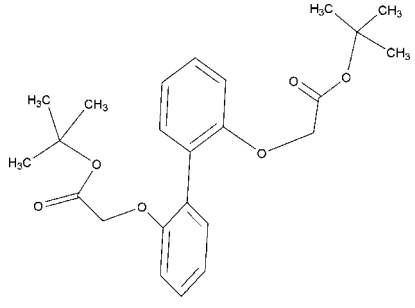
Key indicators: single-crystal X-ray study; $T$ = 153 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.043; $wR$ factor = 0.113; data-to-parameter ratio = 15.5.

The title compound, $C_{24}H_{30}O_6$, does not exhibit $\pi$–$\pi$ interactions due to the steric effect of the bulky *tert*-butyl groups present in the molecule. The presence of these groups at the 2 and 2′ positions hinders the free motion of the benzene rings relative to each other, causing them to adopt an antiperiplanar arrangement. The benzene rings are twisted by just under 50.96 (17)° with respect to each other. The carbonyl groups within the molecule are directed in different directions, one towards the biphenyl group and the other away from it. The molecules are linked together by C═O···H—C hydrogen bonds.

## Related literature

For general background on chemical and biological studies of biphenyl compounds, see: Toshiaki *et al.* (2007); Kamoda *et al.* (2006); Makarov *et al.* (2005); Weisburger *et al.* (1967); Spivey *et al.* (1999); Sisson *et al.* (2006); Litvinchuk *et al.* (2004); Baudry *et al.* (2006). For the crystal structures of related compounds, see: Ali *et al.* (2008); Ibad *et al.* (2008).



## Experimental

### Crystal data

$C_{24}H_{30}O_6$
$M_r$ = 414.48
Triclinic, $P\overline{1}$
$a$ = 7.7458 (15) Å
$b$ = 12.112 (2) Å
$c$ = 13.480 (3) Å
$\alpha$ = 67.36 (3)°
$\beta$ = 82.11 (3)°
$\gamma$ = 82.68 (3)°
$V$ = 1152.3 (4) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 153 (2) K
0.48 × 0.38 × 0.19 mm

### Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)
$T_{min}$ = 0.960, $T_{max}$ = 0.984
8742 measured reflections
4191 independent reflections
3687 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.012

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.113
$S$ = 1.06
4191 reflections
271 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C2—H2$A$···O2$^{i}$ | 0.99 | 2.51 | 3.482 (2) | 166 |
| C20—H20$C$···O5$^{ii}$ | 0.98 | 2.47 | 3.414 (2) | 162 |

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

Data collection: *CrystalClear* (Rigaku/MSC, 2006); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2127).

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# supporting information

*Acta Cryst.* (2008). E**64**, o1408 [doi:10.1107/S1600536808019764]

## Di-*tert*-butyl 2,2′-(biphenyl-2,2′-diyldioxy)diacetate

**Qamar Ali, Farooq Ibad, Muhammad Raza Shah and Donald VanDerveer**

### S1. Comment

Biphenyl moieties have been found to act as pharmacophores in many biological studies such as antimycobacterial testing (Kamoda *et al.*, 2006). Several derivatives of biphenyl are reported to be potential inhibitors of HRV-2 (Makarov *et al.*, 2005). However, they also show carcinogenic activity (Weisburger *et al.*, 1967). Furthermore, they occupy a unique place in various classes of organic compounds not only due to their prevalence as the core framework of numerous natural products, but also for their use as chiral reagents, as chiral phases for chromatography, and as chiral nucleophilic catalysts (Spivey *et al.*, 1999). Biphenyl derivatives are also used as precursors for the synthesis of oligo(*p*-phenylene)s (Sisson *et al.*, 2006). Oligo(*p*-phenylene)s have been extensively studied in the domain of artificial ion channels (Litvinchuk *et al.*, 2004). Our interest in the synthesis of biphenyl derivatives stems from the fact that we wish to attach macrocycles like porphyrins and calix[4]arenes to oligo(*p*-phenylene)s to obtain functionalized pores (Baudry *et al.*, 2006). In order to achieve these goals the synthesis of a number of biphenyl derivatives has been accomplished (Ali *et al.*, 2008; Ibad *et al.*, 2008). In this paper we report the synthesis and crystal structure of the title compound (I).

The $OCH_2C(=O)OC(CH_3)_3$ residues are twisted away fom the biphenyl, as seen in the value of the C14—O4—C15—C16 torsion angle of 67.83 (14). The crystal packing diagram (Fig. 2) shows that there are fairly strong C—H···O interactions that are 0.2 Å less than the sum of the van der Waals radii, which results in the molecules forming chains in the c-direction.

### S2. Experimental

$K_2CO_3$ (414 mg, 3 mmol) and 2,2′-dihydroxybiphenyl (186 mg, 1 mmol) in 15 ml of acetone were stirred for 10 minutes, followed by addition of tertiary butyl bromoacetate (371 mg, 3 mmol). The reaction mixture was stirred at room temperature for three hours. Solvent was evaporated under reduced pressure and the residue was dissolved in a mixture of water (50 ml) and dichloromethane (50 ml). The aqueous layer was extracted three times with dichloromethane.The combined organic phases were evaporated under reduced pressure and the solid residue was dissolved in hot hexane. Slow evaporation of hot hexane gave colorless crystals (736 mg) in 80% yield.

### S3. Refinement

All H atoms were geometrically fixed and allowed to ride on the corresponding non-H atom with C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ of the attached C atom for methyl H atoms and $1.2U_{eq}(C)$ for other H atoms.

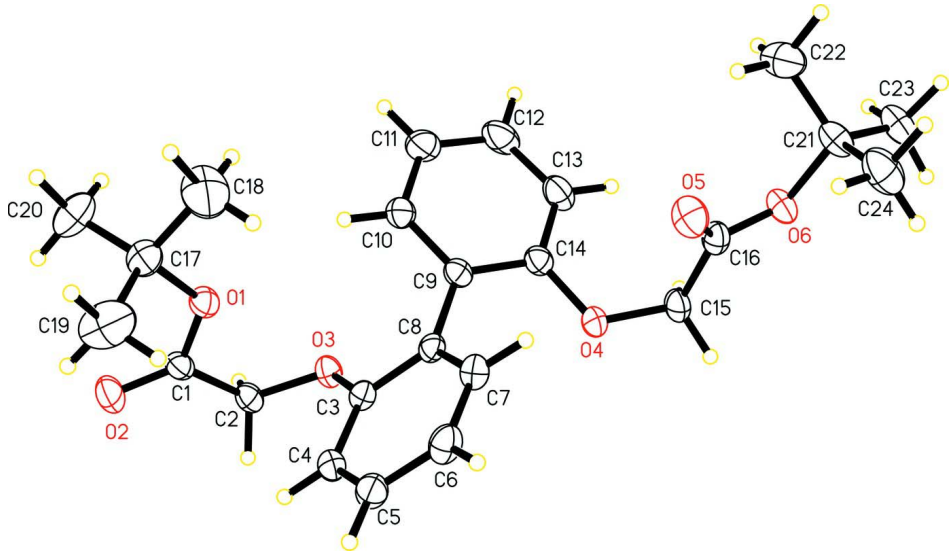


**Figure 1**

Crystal Structure of (I) showing the atom labelling scheme (50% ellipsoids).

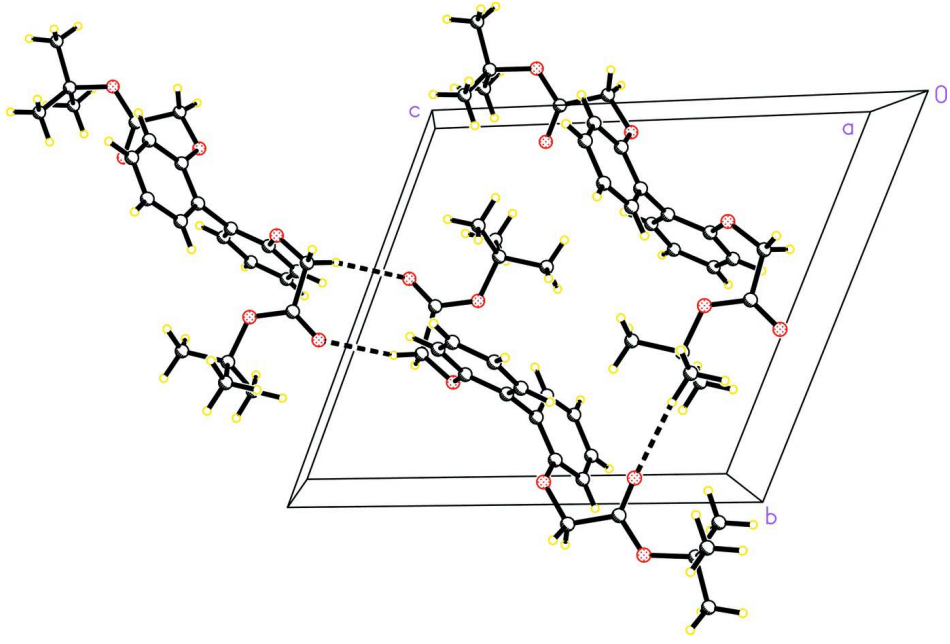


**Figure 2**

Partial packing diagram viewed along the *a* axis.

### Di-tert-butyl 2,2′-(biphenyl-2,2′-diyldioxy)diacetate

*Crystal data*

$C_{24}H_{30}O_6$
$M_r = 414.48$
Triclinic, $P\overline{1}$
$a = 7.7458\ (15)$ Å
$b = 12.112\ (2)$ Å
$c = 13.480\ (3)$ Å
$\alpha = 67.36\ (3)°$
$\beta = 82.11\ (3)°$
$\gamma = 82.68\ (3)°$
$V = 1152.3\ (4)$ Å$^3$
$Z = 2$
$F(000) = 444$

$D_x$ = 1.195 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 3800 reflections
$\theta$ = 3.0–26.4°
$\mu$ = 0.09 mm$^{-1}$
$T$ = 153 K
Chip, colorless
0.48 × 0.38 × 0.19 mm

*Data collection*

Rigaku Mercury CCD (2x2 bin mode) diffractometer
Radiation source: Sealed Tube
Graphite Monochromator monochromator
Detector resolution: 14.6306 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (REQAB; Jacobson, 1998)
$T_{min}$ = 0.960, $T_{max}$ = 0.984
8742 measured reflections
4191 independent reflections
3687 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.012
$\theta_{max}$ = 25.4°, $\theta_{min}$ = 2.9°
$h$ = −9→9
$k$ = −14→14
$l$ = −13→16

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.113
$S$ = 1.06
4191 reflections
271 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2 + 0.3008P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$

*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 | 0.63964 (12) | 0.48792 (8) | 0.74773 (7) | 0.0256 (2) |
| O2 | 0.67544 (15) | 0.42265 (9) | 0.92572 (8) | 0.0407 (3) |
| O3 | 0.51537 (11) | 0.70530 (8) | 0.73639 (7) | 0.0242 (2) |
| O4 | 0.22493 (12) | 0.95936 (8) | 0.46008 (7) | 0.0279 (2) |
| O5 | 0.13650 (14) | 0.94540 (8) | 0.27408 (8) | 0.0351 (2) |
| O6 | 0.14967 (12) | 1.14648 (8) | 0.18911 (7) | 0.0285 (2) |
| C1 | 0.63803 (16) | 0.49982 (12) | 0.84152 (10) | 0.0240 (3) |
| C2 | 0.58898 (17) | 0.62861 (11) | 0.83262 (10) | 0.0249 (3) |
| H2A | 0.5043 | 0.6279 | 0.8949 | 0.030* |
| H2B | 0.6951 | 0.6628 | 0.8381 | 0.030* |
| C3 | 0.35750 (16) | 0.67899 (11) | 0.71761 (10) | 0.0217 (3) |
| C4 | 0.24448 (17) | 0.60667 (11) | 0.79977 (11) | 0.0264 (3) |

| | | | | |
|---|---|---|---|---|
| H4A | 0.2749 | 0.5724 | 0.8720 | 0.032* |
| C5 | 0.08664 (17) | 0.58520 (12) | 0.77493 (12) | 0.0307 (3) |
| H5A | 0.0099 | 0.5348 | 0.8302 | 0.037* |
| C6 | 0.04097 (17) | 0.63678 (12) | 0.67037 (12) | 0.0306 (3) |
| H6A | −0.0676 | 0.6228 | 0.6539 | 0.037* |
| C7 | 0.15448 (16) | 0.70935 (11) | 0.58920 (11) | 0.0258 (3) |
| H7A | 0.1216 | 0.7451 | 0.5175 | 0.031* |
| C8 | 0.31533 (15) | 0.73087 (10) | 0.61059 (10) | 0.0211 (3) |
| C9 | 0.44218 (16) | 0.80053 (11) | 0.52109 (10) | 0.0219 (3) |
| C10 | 0.61274 (17) | 0.75052 (13) | 0.50921 (11) | 0.0285 (3) |
| H10A | 0.6473 | 0.6736 | 0.5598 | 0.034* |
| C11 | 0.73297 (18) | 0.81058 (15) | 0.42515 (12) | 0.0372 (4) |
| H11A | 0.8482 | 0.7749 | 0.4181 | 0.045* |
| C12 | 0.68340 (19) | 0.92246 (16) | 0.35221 (12) | 0.0403 (4) |
| H12A | 0.7657 | 0.9645 | 0.2952 | 0.048* |
| C13 | 0.51479 (18) | 0.97446 (14) | 0.36101 (11) | 0.0335 (3) |
| H13A | 0.4815 | 1.0514 | 0.3100 | 0.040* |
| C14 | 0.39452 (16) | 0.91327 (12) | 0.44496 (10) | 0.0245 (3) |
| C15 | 0.16606 (19) | 1.06562 (11) | 0.37635 (11) | 0.0284 (3) |
| H15A | 0.2491 | 1.1275 | 0.3596 | 0.034* |
| H15B | 0.0507 | 1.0966 | 0.4011 | 0.034* |
| C16 | 0.15050 (16) | 1.04284 (11) | 0.27501 (11) | 0.0255 (3) |
| C17 | 0.67465 (19) | 0.36897 (12) | 0.73667 (12) | 0.0309 (3) |
| C18 | 0.6458 (3) | 0.40082 (18) | 0.61993 (16) | 0.0636 (6) |
| H18A | 0.5237 | 0.4314 | 0.6089 | 0.095* |
| H18B | 0.6720 | 0.3291 | 0.6017 | 0.095* |
| H18C | 0.7230 | 0.4626 | 0.5734 | 0.095* |
| C19 | 0.5431 (3) | 0.28453 (16) | 0.81140 (19) | 0.0589 (5) |
| H19A | 0.5651 | 0.2644 | 0.8865 | 0.088* |
| H19B | 0.5544 | 0.2110 | 0.7960 | 0.088* |
| H19C | 0.4247 | 0.3235 | 0.8001 | 0.088* |
| C20 | 0.8607 (2) | 0.32095 (15) | 0.75818 (16) | 0.0461 (4) |
| H20A | 0.8746 | 0.3010 | 0.8345 | 0.069* |
| H20B | 0.9400 | 0.3820 | 0.7131 | 0.069* |
| H20C | 0.8886 | 0.2487 | 0.7410 | 0.069* |
| C21 | 0.12658 (19) | 1.15005 (13) | 0.08041 (11) | 0.0313 (3) |
| C22 | 0.2762 (2) | 1.07515 (15) | 0.04516 (14) | 0.0449 (4) |
| H22A | 0.3876 | 1.1053 | 0.0455 | 0.067* |
| H22B | 0.2633 | 1.0805 | −0.0279 | 0.067* |
| H22C | 0.2744 | 0.9913 | 0.0951 | 0.067* |
| C23 | 0.1343 (2) | 1.28210 (13) | 0.01004 (12) | 0.0386 (4) |
| H23A | 0.2499 | 1.3070 | 0.0098 | 0.058* |
| H23B | 0.0444 | 1.3296 | 0.0387 | 0.058* |
| H23C | 0.1136 | 1.2950 | −0.0639 | 0.058* |
| C24 | −0.0517 (2) | 1.10931 (17) | 0.08184 (13) | 0.0458 (4) |
| H24A | −0.1431 | 1.1605 | 0.1051 | 0.069* |
| H24B | −0.0581 | 1.0258 | 0.1321 | 0.069* |
| H24C | −0.0689 | 1.1153 | 0.0092 | 0.069* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0307 (5) | 0.0248 (5) | 0.0223 (5) | −0.0003 (4) | −0.0047 (4) | −0.0096 (4) |
| O2 | 0.0582 (7) | 0.0344 (6) | 0.0223 (5) | 0.0101 (5) | −0.0090 (5) | −0.0052 (5) |
| O3 | 0.0245 (4) | 0.0238 (4) | 0.0226 (5) | −0.0039 (3) | −0.0061 (4) | −0.0049 (4) |
| O4 | 0.0285 (5) | 0.0244 (5) | 0.0218 (5) | 0.0031 (4) | −0.0014 (4) | −0.0005 (4) |
| O5 | 0.0451 (6) | 0.0234 (5) | 0.0360 (6) | −0.0064 (4) | −0.0066 (4) | −0.0080 (4) |
| O6 | 0.0397 (5) | 0.0235 (5) | 0.0207 (5) | −0.0042 (4) | −0.0067 (4) | −0.0047 (4) |
| C1 | 0.0232 (6) | 0.0285 (7) | 0.0181 (6) | −0.0020 (5) | −0.0023 (5) | −0.0064 (5) |
| C2 | 0.0276 (6) | 0.0280 (7) | 0.0189 (6) | −0.0018 (5) | −0.0060 (5) | −0.0075 (5) |
| C3 | 0.0206 (6) | 0.0192 (6) | 0.0246 (6) | 0.0003 (5) | −0.0034 (5) | −0.0077 (5) |
| C4 | 0.0255 (6) | 0.0241 (6) | 0.0239 (7) | 0.0006 (5) | −0.0018 (5) | −0.0036 (5) |
| C5 | 0.0238 (6) | 0.0252 (7) | 0.0341 (8) | −0.0028 (5) | 0.0012 (5) | −0.0024 (6) |
| C6 | 0.0214 (6) | 0.0272 (7) | 0.0407 (8) | −0.0036 (5) | −0.0055 (6) | −0.0088 (6) |
| C7 | 0.0248 (6) | 0.0235 (6) | 0.0281 (7) | 0.0001 (5) | −0.0068 (5) | −0.0078 (5) |
| C8 | 0.0212 (6) | 0.0173 (6) | 0.0236 (6) | 0.0008 (4) | −0.0023 (5) | −0.0071 (5) |
| C9 | 0.0229 (6) | 0.0245 (6) | 0.0196 (6) | −0.0039 (5) | −0.0037 (5) | −0.0085 (5) |
| C10 | 0.0243 (6) | 0.0354 (7) | 0.0244 (7) | 0.0010 (5) | −0.0050 (5) | −0.0097 (6) |
| C11 | 0.0215 (6) | 0.0561 (9) | 0.0304 (7) | −0.0012 (6) | −0.0013 (5) | −0.0131 (7) |
| C12 | 0.0288 (7) | 0.0581 (10) | 0.0264 (7) | −0.0125 (7) | 0.0020 (6) | −0.0061 (7) |
| C13 | 0.0331 (7) | 0.0362 (8) | 0.0245 (7) | −0.0080 (6) | −0.0037 (6) | −0.0021 (6) |
| C14 | 0.0250 (6) | 0.0270 (6) | 0.0215 (6) | −0.0027 (5) | −0.0040 (5) | −0.0082 (5) |
| C15 | 0.0355 (7) | 0.0203 (6) | 0.0234 (7) | 0.0032 (5) | −0.0056 (5) | −0.0024 (5) |
| C16 | 0.0244 (6) | 0.0211 (6) | 0.0268 (7) | −0.0015 (5) | −0.0040 (5) | −0.0040 (5) |
| C17 | 0.0370 (7) | 0.0250 (7) | 0.0348 (8) | −0.0033 (6) | −0.0037 (6) | −0.0155 (6) |
| C18 | 0.1049 (17) | 0.0525 (11) | 0.0492 (11) | 0.0072 (11) | −0.0259 (11) | −0.0343 (10) |
| C19 | 0.0529 (10) | 0.0426 (9) | 0.0859 (15) | −0.0215 (8) | 0.0172 (10) | −0.0317 (10) |
| C20 | 0.0403 (9) | 0.0373 (8) | 0.0676 (12) | 0.0044 (7) | −0.0056 (8) | −0.0292 (8) |
| C21 | 0.0412 (8) | 0.0321 (7) | 0.0215 (7) | −0.0085 (6) | −0.0057 (6) | −0.0083 (6) |
| C22 | 0.0565 (10) | 0.0422 (9) | 0.0359 (9) | −0.0074 (7) | 0.0058 (7) | −0.0166 (7) |
| C23 | 0.0557 (9) | 0.0333 (8) | 0.0239 (7) | −0.0079 (7) | −0.0098 (6) | −0.0041 (6) |
| C24 | 0.0480 (9) | 0.0551 (10) | 0.0339 (8) | −0.0188 (8) | −0.0107 (7) | −0.0095 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.3247 (16) | C12—H12A | 0.9500 |
| O1—C17 | 1.4921 (16) | C13—C14 | 1.3924 (19) |
| O2—C1 | 1.2044 (17) | C13—H13A | 0.9500 |
| O3—C3 | 1.3811 (15) | C15—C16 | 1.5168 (19) |
| O3—C2 | 1.4180 (16) | C15—H15A | 0.9900 |
| O4—C14 | 1.3805 (16) | C15—H15B | 0.9900 |
| O4—C15 | 1.4183 (16) | C17—C20 | 1.507 (2) |
| O5—C16 | 1.2038 (16) | C17—C18 | 1.511 (2) |
| O6—C16 | 1.3407 (17) | C17—C19 | 1.513 (2) |
| O6—C21 | 1.4839 (16) | C18—H18A | 0.9800 |
| C1—C2 | 1.5196 (18) | C18—H18B | 0.9800 |
| C2—H2A | 0.9900 | C18—H18C | 0.9800 |

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| C2—H2B | 0.9900 | C19—H19A | 0.9800 |
| C3—C4 | 1.3939 (19) | C19—H19B | 0.9800 |
| C3—C8 | 1.4006 (18) | C19—H19C | 0.9800 |
| C4—C5 | 1.3910 (19) | C20—H20A | 0.9800 |
| C4—H4A | 0.9500 | C20—H20B | 0.9800 |
| C5—C6 | 1.380 (2) | C20—H20C | 0.9800 |
| C5—H5A | 0.9500 | C21—C23 | 1.517 (2) |
| C6—C7 | 1.391 (2) | C21—C22 | 1.518 (2) |
| C6—H6A | 0.9500 | C21—C24 | 1.520 (2) |
| C7—C8 | 1.3938 (17) | C22—H22A | 0.9800 |
| C7—H7A | 0.9500 | C22—H22B | 0.9800 |
| C8—C9 | 1.4950 (18) | C22—H22C | 0.9800 |
| C9—C10 | 1.3966 (19) | C23—H23A | 0.9800 |
| C9—C14 | 1.3967 (19) | C23—H23B | 0.9800 |
| C10—C11 | 1.389 (2) | C23—H23C | 0.9800 |
| C10—H10A | 0.9500 | C24—H24A | 0.9800 |
| C11—C12 | 1.377 (2) | C24—H24B | 0.9800 |
| C11—H11A | 0.9500 | C24—H24C | 0.9800 |
| C12—C13 | 1.386 (2) | | |

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| C1—O1—C17 | 122.25 (11) | C16—C15—H15B | 109.3 |
| C3—O3—C2 | 117.83 (10) | H15A—C15—H15B | 108.0 |
| C14—O4—C15 | 116.99 (11) | O5—C16—O6 | 125.86 (13) |
| C16—O6—C21 | 121.03 (10) | O5—C16—C15 | 123.97 (12) |
| O2—C1—O1 | 126.99 (13) | O6—C16—C15 | 110.14 (11) |
| O2—C1—C2 | 120.93 (12) | O1—C17—C20 | 109.60 (11) |
| O1—C1—C2 | 112.04 (11) | O1—C17—C18 | 102.02 (12) |
| O3—C2—C1 | 115.22 (10) | C20—C17—C18 | 111.65 (15) |
| O3—C2—H2A | 108.5 | O1—C17—C19 | 109.59 (12) |
| C1—C2—H2A | 108.5 | C20—C17—C19 | 112.56 (14) |
| O3—C2—H2B | 108.5 | C18—C17—C19 | 110.90 (16) |
| C1—C2—H2B | 108.5 | C17—C18—H18A | 109.5 |
| H2A—C2—H2B | 107.5 | C17—C18—H18B | 109.5 |
| O3—C3—C4 | 122.53 (11) | H18A—C18—H18B | 109.5 |
| O3—C3—C8 | 116.03 (11) | C17—C18—H18C | 109.5 |
| C4—C3—C8 | 121.43 (11) | H18A—C18—H18C | 109.5 |
| C5—C4—C3 | 119.31 (12) | H18B—C18—H18C | 109.5 |
| C5—C4—H4A | 120.3 | C17—C19—H19A | 109.5 |
| C3—C4—H4A | 120.3 | C17—C19—H19B | 109.5 |
| C6—C5—C4 | 120.34 (12) | H19A—C19—H19B | 109.5 |
| C6—C5—H5A | 119.8 | C17—C19—H19C | 109.5 |
| C4—C5—H5A | 119.8 | H19A—C19—H19C | 109.5 |
| C5—C6—C7 | 119.79 (12) | H19B—C19—H19C | 109.5 |
| C5—C6—H6A | 120.1 | C17—C20—H20A | 109.5 |
| C7—C6—H6A | 120.1 | C17—C20—H20B | 109.5 |
| C6—C7—C8 | 121.50 (12) | H20A—C20—H20B | 109.5 |
| C6—C7—H7A | 119.2 | C17—C20—H20C | 109.5 |
| C8—C7—H7A | 119.2 | H20A—C20—H20C | 109.5 |

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| C7—C8—C3 | 117.62 (12) | H20B—C20—H20C | 109.5 |
| C7—C8—C9 | 120.83 (11) | O6—C21—C23 | 102.98 (11) |
| C3—C8—C9 | 121.45 (11) | O6—C21—C22 | 109.69 (12) |
| C10—C9—C14 | 117.97 (12) | C23—C21—C22 | 110.79 (13) |
| C10—C9—C8 | 119.72 (11) | O6—C21—C24 | 109.62 (12) |
| C14—C9—C8 | 122.25 (11) | C23—C21—C24 | 110.38 (13) |
| C11—C10—C9 | 121.56 (13) | C22—C21—C24 | 112.92 (13) |
| C11—C10—H10A | 119.2 | C21—C22—H22A | 109.5 |
| C9—C10—H10A | 119.2 | C21—C22—H22B | 109.5 |
| C12—C11—C10 | 119.28 (14) | H22A—C22—H22B | 109.5 |
| C12—C11—H11A | 120.4 | C21—C22—H22C | 109.5 |
| C10—C11—H11A | 120.4 | H22A—C22—H22C | 109.5 |
| C11—C12—C13 | 120.73 (13) | H22B—C22—H22C | 109.5 |
| C11—C12—H12A | 119.6 | C21—C23—H23A | 109.5 |
| C13—C12—H12A | 119.6 | C21—C23—H23B | 109.5 |
| C12—C13—C14 | 119.66 (13) | H23A—C23—H23B | 109.5 |
| C12—C13—H13A | 120.2 | C21—C23—H23C | 109.5 |
| C14—C13—H13A | 120.2 | H23A—C23—H23C | 109.5 |
| O4—C14—C13 | 123.23 (12) | H23B—C23—H23C | 109.5 |
| O4—C14—C9 | 115.98 (11) | C21—C24—H24A | 109.5 |
| C13—C14—C9 | 120.78 (12) | C21—C24—H24B | 109.5 |
| O4—C15—C16 | 111.40 (11) | H24A—C24—H24B | 109.5 |
| O4—C15—H15A | 109.3 | C21—C24—H24C | 109.5 |
| C16—C15—H15A | 109.3 | H24A—C24—H24C | 109.5 |
| O4—C15—H15B | 109.3 | H24B—C24—H24C | 109.5 |

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| C17—O1—C1—O2 | 5.8 (2) | C8—C9—C10—C11 | 177.90 (12) |
| C17—O1—C1—C2 | −176.69 (10) | C9—C10—C11—C12 | 0.5 (2) |
| C3—O3—C2—C1 | 63.42 (14) | C10—C11—C12—C13 | −1.0 (2) |
| O2—C1—C2—O3 | −168.21 (12) | C11—C12—C13—C14 | 0.4 (2) |
| O1—C1—C2—O3 | 14.12 (15) | C15—O4—C14—C13 | 9.65 (18) |
| C2—O3—C3—C4 | 20.01 (17) | C15—O4—C14—C9 | −171.68 (11) |
| C2—O3—C3—C8 | −160.90 (11) | C12—C13—C14—O4 | 179.23 (13) |
| O3—C3—C4—C5 | 179.27 (12) | C12—C13—C14—C9 | 0.6 (2) |
| C8—C3—C4—C5 | 0.22 (19) | C10—C9—C14—O4 | −179.80 (11) |
| C3—C4—C5—C6 | −1.2 (2) | C8—C9—C14—O4 | 2.91 (17) |
| C4—C5—C6—C7 | 0.8 (2) | C10—C9—C14—C13 | −1.10 (19) |
| C5—C6—C7—C8 | 0.5 (2) | C8—C9—C14—C13 | −178.39 (12) |
| C6—C7—C8—C3 | −1.45 (19) | C14—O4—C15—C16 | 67.83 (14) |
| C6—C7—C8—C9 | 174.84 (12) | C21—O6—C16—O5 | 1.3 (2) |
| O3—C3—C8—C7 | −178.03 (11) | C21—O6—C16—C15 | −176.87 (11) |
| C4—C3—C8—C7 | 1.08 (18) | O4—C15—C16—O5 | 21.25 (18) |
| O3—C3—C8—C9 | 5.71 (17) | O4—C15—C16—O6 | −160.51 (11) |
| C4—C3—C8—C9 | −175.19 (11) | C1—O1—C17—C20 | −66.57 (16) |
| C7—C8—C9—C10 | −125.19 (13) | C1—O1—C17—C18 | 174.98 (14) |
| C3—C8—C9—C10 | 50.96 (17) | C1—O1—C17—C19 | 57.41 (17) |
| C7—C8—C9—C14 | 52.05 (17) | C16—O6—C21—C23 | −179.93 (12) |
| C3—C8—C9—C14 | −131.80 (13) | C16—O6—C21—C22 | −61.94 (16) |

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| C14—C9—C10—C11 | 0.54 (19) | C16—O6—C21—C24 | 62.58 (17) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C2—H2*A*···O2$^{i}$ | 0.99 | 2.51 | 3.482 (2) | 166 |
| C20—H20*C*···O5$^{ii}$ | 0.98 | 2.47 | 3.414 (2) | 162 |

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