Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure and conformational analysis of 2-hydroxy-3-(2-methylprop-1-en-1-yl)naphthalene-1,4-dione

Sannyele Alcantara Emiliano, Sheyla Welma Duarte Silva, Mariano Alves Pereira, Valeria R.dos Santos Malta and Tatiane Luciano Balliano*

Institute of Chemistry and Biotechnology – IQB, Federal University of Alagoas - UFAL, Maceio–Alagoas, Brazil. *Correspondence e-mail: tlb@qui.ufal.br

Received 28 November 2015
Accepted 24 December 2015

Edited by G. Smith, Queensland University of Technology, Australia

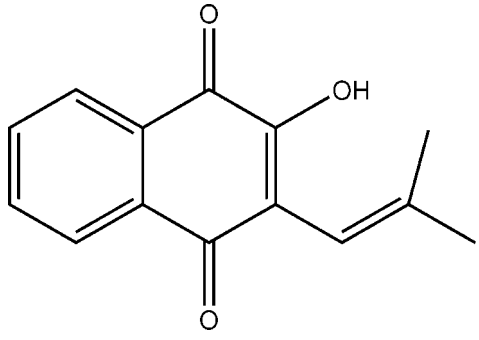
Keywords: crystal structure; naphthoquinone derivative; molecular conformation; hydrogen bonding

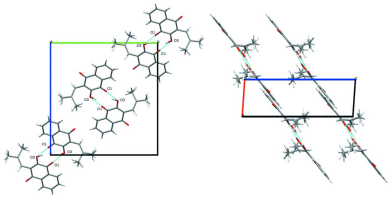
CCDC reference: 1444109
Supporting information: this article has supporting information at journals.iucr.org/e

In the structure of the title compound, $C_{14}H_{12}O_3$, the substituent side chain, in which the H atoms of both methyl groups are disordered over six equivalent sites, lies outside of the plane of the naphthalenedione ring. The ring-to-chain C—C—C—C torsion angles are 50.7 (3), −176.6 (2) and 4.9 (4)°. An intramolecular methyl–hydroxy C—H···O hydrogen bond is present. In the crystal, molecules are primarily connected by intermolecular O—H···O hydrogen bonds, forming a centrosymmetric cyclic dimer motif [graph set $R_2^2(10)$]. Also present is a weak intermolecular C—H···O hydrogen bond linking the dimers and a weak π–π ring interaction [ring centroid separation = 3.7862 (13) Å], giving layers parallel to $(10\overline{3})$.

1. Chemical context

Naphthoquinone compounds exhibit several biological activities, being utilized for the treatment of parasitic diseases (Salas *et al.*, 2008) some types of cancer (Tonholo *et al.*, 1998) and cardiovascular disease (Silva & Torres, 2013). The compound in this study, 2-hydroxy-3-(2-metilprop-1-enol)-naphthalene-1,4-dione, $C_{14}H_{12}O_3$, is a naphthoquinone derivative and the structure is reported herein.





2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. In this structure the side chain is rotated out of the plane of the naphthalenedione ring, with torsion angles C2—C3—C9—C10, C3—C9—C10—C12 and C3—C9—C10—C22 of 50.7 (3), −176.6 (2) and 4.9 (4)°, respectively. Present also in the molecule is an intramolecular methyl C22···O3 [2.959 (3) Å; see Table 1] and a short O3···O1 contact [2.665 (2) Å]. When compared with other analogous struc-

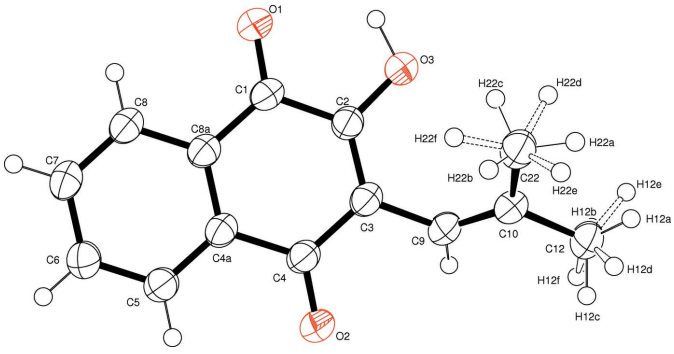


Figure 1
Molecular conformation and atom-numbering scheme, with non-H atoms drawn at the 50% probability level. The H atoms of the rotationally disordered methyl groups are shown as six equivalent half-occupancy sites.

tures in the literature, *e.g.* 2-chloro-3-(4-chlorobenzamido)-1,4-naphthoquinone (Brandy *et al.*, 2009), it is observed that the title compound has similar conformational features with respect to the side chain, which lies out of the naphthoquinone plane.

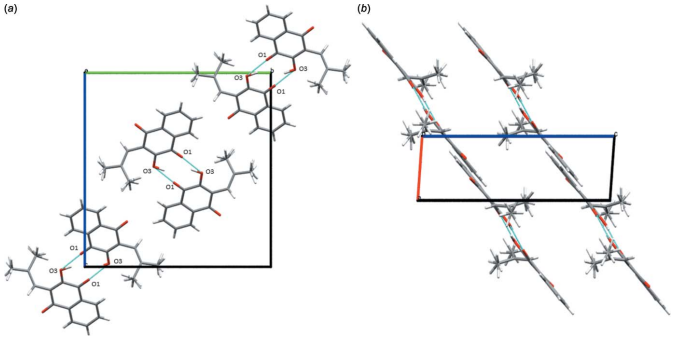


Figure 2
The centrosymmetric dimers formed from the O3—H···O1^{i} hydrogen bonds, viewed (*a*) along *a* and (*b*) along *b*. For symmetry code (i), see Table 1.

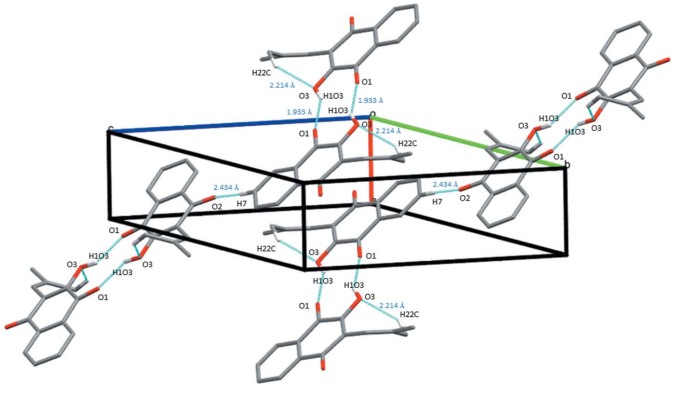


Figure 3
The crystal packing in the unit cell, showing intra- and intermolecular interactions as dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1*O*3···O1^{i}	0.97 (3)	1.93 (3)	2.770 (2)	143 (3)
C7—H7···O2^{ii}	0.93	2.43	3.339 (3)	164
C22—H22*C*···O3	0.96	2.21	2.959 (3)	134

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

3. Supramolecular features

In the crystal, the molecules are connected by classic intermolecular O3—H···O1^{i} hydrogen bonds (Table 1), forming a centrosymmetric cyclic dimer [graph set $R_2^2(10)$] (Bernstein *et al.*, 1995) (Fig. 2*a*). Also present in the structure is a weak intermolecular C7—H···O2^{ii} hydrogen bond [3.339 (3) Å], linking the dimers and a weak π–π ring interaction between the benzene and quinone ring moieties of the parent ring system [ring centroid separation $Cg\cdots Cg^{iii}$ = 3.7862 (13) Å; symmetry code: (iii) $x+1, y, z$], giving layers parallel to $(10\overline{3})$ (Figs. 2*b* and 3).

4. Database survey

A search of the Cambridge Structural Database (Groom & Allen, 2014) revealed the presence of 40 structures containing the 2-hydroxynaphthalene-1,4-dione core moiety. There were 787 structures which possess the naphthalene-1,4-dione

Table 2
Experimental details.

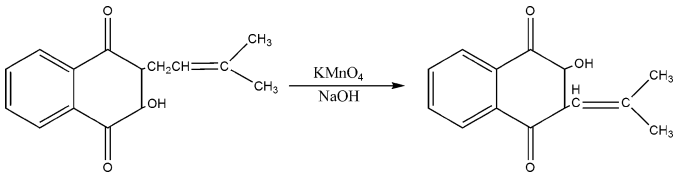
Crystal data	
Chemical formula	$C_{14}H_{12}O_3$
M_r	228.24
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, *b*, *c* (Å)	4.3564 (2), 16.4069 (8), 15.8598 (7)
β (°)	94.793 (2)
V (Å^3)	1129.62 (9)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.14 × 0.11 × 0.10
Data collection	
Diffractometer	Nonius KappaCCD
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4661, 2585, 1802
R_{int}	0.041
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.061, 0.191, 1.03
No. of reflections	2585
No. of parameters	158
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.31, −0.30

Computer programs: *COLLECT* (Enraf–Nonius, 2001), *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008), *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

moiety. There are structures similar to the title compound, whichvary depending on the oxidant used in the synthesis.

5. Synthesis and crystallization

The compound was obtained through to the lapachol oxidation product as can be seen in the scheme below (Hooker, 1936). The sample was subjected to an ethyl acetate solution at 301 K for crystallization.



6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The O3-bound H atom was located in a difference Fourier map and was freely refined. The remaining H atoms were positioned geometrically with aromatic C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C). Rotational disorder was identified in the hydrogen atoms of the methyl carbon atoms C12 and C22 and these were included in the refinement over six equivalent 60° sites with 50% occupation, with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C).

Acknowledgements

UFAL, IQB, LabCriMM, CNPq and FAPEAL are acknowledged for support. We thank Professor Dr Antonio Ventura Pinto (in memorium) for his collaboration in the works of this research group, specifically for the synthesis of the title compound.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Brandy, Y., Butcher, R. J., Adesiyun, T. A., Berhe, S. & Bakare, O. (2009). *Acta Cryst.* E**65**, o64.
Enraf–Nonius (2001). *Kappa CCD Operation Manual*. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed.* **53**, 662–671.
Hooker, S. C. (1936). *J. Am. Chem. Soc.* **58**, 1168–1173.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. **276**, *Macromolecular Crystallography*, Part A, Editors C. W. Carter & R. M. Sweet, pp. 307–326. New York: Academic Press.
Salas, C., Tapia, R. A., Ciudad, K., Armstrong, V., Orellana, M., Kemmerling, U., Ferreira, J., Maya, J. D. & Morello, A. (2008). *Bioorg. Med. Chem.* **16**, 668–674.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Silva, A. K. Soares e, de Oliveira Cipriano Torres, D., Santos Rocha, S. W., dos Santos Gomes, F. O., dos Santos Silva, B., Donato, M. A. M., Raposo, C., Santos, A. C. O., de Lima, M. do C. A., Galdino, S. L., da Rocha Pitta, I., de Souza, J. R. B. & Peixoto, C. A. (2013). *Cardiovascular Pathol.* **22**, 81–90.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Tonholo, J. L. R., Freitas, L. R., de Abreu, F. C., Azevedo, D. C., Zani, C. L., de Oliveira, A. B. & Goulart, M. O. F. (1998). *J. Braz. Chem. Soc.* **9**, 163–169.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2016). E**72**, 188-190 [doi:10.1107/S2056989015024755]

Crystal structure and conformational analysis of 2-hydroxy-3-(2-methylprop-1-en-1-yl)naphthalene-1,4-dione

Sannyele Alcantara Emiliano, Sheyla Welma Duarte Silva, Mariano Alves Pereira, Valeria R.dos Santos Malta and Tatiane Luciano Balliano

Computing details

Data collection: *COLLECT* (Enraf–Nonius, 2001); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 2012), *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

2-Hydroxy-3-(2-methylprop-1-en-1-yl)naphthalene-1,4-dione

Crystal data

$C_{14}H_{12}O_3$
M_r = 228.24
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 4.3564 (2) Å
b = 16.4069 (8) Å
c = 15.8598 (7) Å
β = 94.793 (2)°
V = 1129.62 (9) Å^3
Z = 4
$F(000)$ = 480
D_x = 1.342 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2659 reflections
θ = 1.0–27.5°
μ = 0.09 mm^{-1}
T = 293 K
Block, red
0.14 × 0.11 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: Enraf-Nonius FR590
Graphite monochromator
Detector resolution: 9 pixels mm^{-1}
CCD rotation images, thick slices scans
4661 measured reflections
2585 independent reflections
1802 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 27.5°, θ_{min} = 2.6°
h = −5→5
k = −19→21
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.191
S = 1.03
2585 reflections
158 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 atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0946P)^2 + 0.4119P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}	Occ. (<1)
O3	0.3690 (4)	0.37038 (10)	0.48362 (10)	0.0407 (4)	
O1	0.2205 (4)	0.52143 (9)	0.43377 (9)	0.0404 (4)	
O2	−0.3406 (4)	0.27382 (9)	0.26721 (10)	0.0481 (5)	
C10	0.0940 (5)	0.19603 (13)	0.47932 (13)	0.0395 (5)	
C9	0.0749 (5)	0.23006 (12)	0.40272 (13)	0.0386 (5)	
H9	0.1126	0.1961	0.3578	0.046*	
H1O3	0.448 (7)	0.424 (2)	0.5005 (19)	0.073 (9)*	
C4A	−0.3114 (5)	0.41650 (12)	0.28476 (13)	0.0349 (5)	
C8A	−0.1689 (5)	0.48205 (13)	0.32860 (13)	0.0348 (5)	
C2	0.1441 (5)	0.38047 (12)	0.42115 (13)	0.0351 (5)	
C1	0.0733 (5)	0.46622 (12)	0.39675 (13)	0.0350 (5)	
C5	−0.5333 (5)	0.43122 (14)	0.21845 (13)	0.0400 (5)	
H5	−0.6268	0.3879	0.1885	0.048*	
C3	0.0007 (5)	0.31557 (12)	0.38155 (12)	0.0358 (5)	
C4	−0.2235 (5)	0.33077 (13)	0.30859 (13)	0.0369 (5)	
C6	−0.6151 (5)	0.51093 (14)	0.19709 (14)	0.0426 (5)	
H6	−0.7628	0.5207	0.1524	0.051*	
C8	−0.2532 (5)	0.56203 (13)	0.30691 (14)	0.0386 (5)	
H8	−0.1584	0.6056	0.3362	0.046*	
C7	−0.4789 (5)	0.57607 (13)	0.24159 (14)	0.0413 (5)	
H7	−0.5389	0.6291	0.2277	0.05*	
C12	0.1899 (6)	0.10858 (13)	0.49043 (15)	0.0475 (6)	
H12A	0.1919	0.0939	0.5491	0.071*	0.5
H12B	0.3923	0.1015	0.4718	0.071*	0.5
H12C	0.0468	0.0744	0.4575	0.071*	0.5
H12D	0.2288	0.086	0.4365	0.071*	0.5
H12E	0.0283	0.0784	0.5138	0.071*	0.5
H12F	0.3738	0.1055	0.5281	0.071*	0.5
C22	0.0189 (6)	0.23815 (14)	0.55869 (14)	0.0452 (6)	
H22A	0.0507	0.2013	0.6056	0.068*	0.5
H22B	−0.1923	0.2554	0.553	0.068*	0.5
H22C	0.1503	0.2848	0.5684	0.068*	0.5

H22D	−0.0449	0.2931	0.5457	0.068*	0.5
H22E	0.1981	0.2389	0.5983	0.068*	0.5
H22F	−0.1445	0.2095	0.583	0.068*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0435 (9)	0.0323 (8)	0.0450 (9)	−0.0013 (6)	−0.0051 (6)	−0.0002 (7)
O1	0.0456 (9)	0.0318 (8)	0.0431 (8)	−0.0041 (6)	−0.0005 (6)	−0.0022 (6)
O2	0.0651 (11)	0.0308 (8)	0.0460 (9)	−0.0033 (7)	−0.0096 (7)	−0.0026 (7)
C10	0.0438 (12)	0.0302 (10)	0.0439 (12)	−0.0025 (8)	−0.0013 (9)	−0.0004 (9)
C9	0.0462 (12)	0.0282 (10)	0.0409 (11)	0.0009 (9)	0.0011 (9)	−0.0027 (9)
C4A	0.0422 (11)	0.0292 (10)	0.0340 (10)	−0.0011 (8)	0.0061 (8)	0.0012 (8)
C8A	0.0398 (11)	0.0305 (11)	0.0346 (10)	−0.0016 (8)	0.0057 (8)	−0.0001 (8)
C2	0.0384 (11)	0.0314 (11)	0.0355 (10)	0.0003 (8)	0.0035 (8)	0.0009 (8)
C1	0.0398 (11)	0.0288 (10)	0.0367 (10)	−0.0019 (8)	0.0055 (8)	−0.0034 (8)
C5	0.0486 (13)	0.0343 (11)	0.0368 (11)	−0.0025 (9)	0.0015 (9)	−0.0004 (9)
C3	0.0431 (11)	0.0292 (10)	0.0355 (10)	−0.0006 (8)	0.0064 (8)	−0.0006 (8)
C4	0.0453 (12)	0.0299 (10)	0.0357 (11)	−0.0025 (9)	0.0040 (9)	−0.0011 (8)
C6	0.0500 (13)	0.0383 (12)	0.0391 (11)	0.0013 (9)	0.0003 (9)	0.0039 (9)
C8	0.0460 (12)	0.0293 (10)	0.0410 (11)	−0.0005 (8)	0.0062 (9)	−0.0002 (8)
C7	0.0493 (12)	0.0312 (11)	0.0436 (11)	0.0027 (9)	0.0060 (9)	0.0055 (9)
C12	0.0651 (15)	0.0315 (11)	0.0447 (12)	0.0020 (10)	−0.0024 (10)	0.0011 (9)
C22	0.0587 (14)	0.0346 (11)	0.0420 (12)	0.0006 (10)	0.0033 (10)	0.0011 (9)

Geometric parameters (Å, º)

O3—C2	1.344 (3)	C3—C4	1.472 (3)
O3—H1O3	0.97 (4)	C6—C7	1.387 (3)
O1—C1	1.230 (2)	C6—H6	0.93
O2—C4	1.228 (2)	C8—C7	1.387 (3)
C10—C9	1.333 (3)	C8—H8	0.93
C10—C22	1.496 (3)	C7—H7	0.93
C10—C12	1.501 (3)	C12—H12A	0.96
C9—C3	1.472 (3)	C12—H12B	0.96
C9—H9	0.93	C12—H12C	0.96
C4A—C5	1.389 (3)	C12—H12D	0.96
C4A—C8A	1.398 (3)	C12—H12E	0.96
C4A—C4	1.498 (3)	C12—H12F	0.96
C8A—C8	1.398 (3)	C22—H22A	0.96
C8A—C1	1.469 (3)	C22—H22B	0.96
C2—C3	1.361 (3)	C22—H22C	0.96
C2—C1	1.485 (3)	C22—H22D	0.96
C5—C6	1.390 (3)	C22—H22E	0.96
C5—H5	0.93	C22—H22F	0.96
C2—O3—H1O3	108.3 (18)	C10—C12—H12C	109.5
C9—C10—C22	124.9 (2)	H12A—C12—H12C	109.5

C9—C10—C12	120.2 (2)	H12B—C12—H12C	109.5
C22—C10—C12	114.91 (19)	C10—C12—H12D	109.5
C10—C9—C3	127.1 (2)	H12A—C12—H12D	141.1
C10—C9—H9	116.5	H12B—C12—H12D	56.3
C3—C9—H9	116.5	H12C—C12—H12D	56.3
C5—C4A—C8A	119.67 (19)	C10—C12—H12E	109.5
C5—C4A—C4	120.09 (19)	H12A—C12—H12E	56.3
C8A—C4A—C4	120.23 (18)	H12B—C12—H12E	141.1
C4A—C8A—C8	120.21 (19)	H12C—C12—H12E	56.3
C4A—C8A—C1	119.46 (19)	H12D—C12—H12E	109.5
C8—C8A—C1	120.32 (19)	C10—C12—H12F	109.5
O3—C2—C3	121.45 (19)	H12A—C12—H12F	56.3
O3—C2—C1	115.56 (18)	H12B—C12—H12F	56.3
C3—C2—C1	122.95 (19)	H12C—C12—H12F	141.1
O1—C1—C8A	122.31 (19)	H12D—C12—H12F	109.5
O1—C1—C2	119.00 (18)	H12E—C12—H12F	109.5
C8A—C1—C2	118.68 (18)	C10—C22—H22A	109.5
C4A—C5—C6	119.8 (2)	C10—C22—H22B	109.5
C4A—C5—H5	120.1	H22A—C22—H22B	109.5
C6—C5—H5	120.1	C10—C22—H22C	109.5
C2—C3—C4	118.61 (19)	H22A—C22—H22C	109.5
C2—C3—C9	123.83 (19)	H22B—C22—H22C	109.5
C4—C3—C9	117.35 (18)	C10—C22—H22D	109.5
O2—C4—C3	120.66 (19)	H22A—C22—H22D	141.1
O2—C4—C4A	119.58 (18)	H22B—C22—H22D	56.3
C3—C4—C4A	119.76 (18)	H22C—C22—H22D	56.3
C7—C6—C5	120.7 (2)	C10—C22—H22E	109.5
C7—C6—H6	119.6	H22A—C22—H22E	56.3
C5—C6—H6	119.6	H22B—C22—H22E	141.1
C7—C8—C8A	119.7 (2)	H22C—C22—H22E	56.3
C7—C8—H8	120.2	H22D—C22—H22E	109.5
C8A—C8—H8	120.2	C10—C22—H22F	109.5
C6—C7—C8	119.9 (2)	H22A—C22—H22F	56.3
C6—C7—H7	120	H22B—C22—H22F	56.3
C8—C7—H7	120	H22C—C22—H22F	141.1
C10—C12—H12A	109.5	H22D—C22—H22F	109.5
C10—C12—H12B	109.5	H22E—C22—H22F	109.5
H12A—C12—H12B	109.5		

O1—C1—C2—O3	0.2 (3)	O2—C4—C4A—C5	2.6 (3)
O1—C1—C2—C3	−177.5 (2)	O2—C4—C4A—C8A	−176.8 (2)
C8A—C1—C2—O3	179.69 (19)	C3—C4—C4A—C5	−177.0 (2)
C8A—C1—C2—C3	2.0 (3)	C3—C4—C4A—C8A	3.5 (3)
O1—C1—C8A—C4A	175.2 (2)	C4—C4A—C5—C6	179.6 (2)
O1—C1—C8A—C8	−3.9 (3)	C8A—C4A—C5—C6	−1.0 (3)
C2—C1—C8A—C4A	−4.3 (3)	C4—C4A—C8A—C1	1.6 (3)
C2—C1—C8A—C8	176.6 (2)	C4—C4A—C8A—C8	−179.4 (2)
O3—C2—C3—C4	−174.50 (19)	C5—C4A—C8A—C1	−177.9 (2)

O3—C2—C3—C9	0.2 (3)	C5—C4A—C8A—C8	1.2 (3)
C1—C2—C3—C4	3.1 (3)	C4A—C5—C6—C7	−0.4 (3)
C1—C2—C3—C9	177.8 (2)	C5—C6—C7—C8	1.5 (3)
C2—C3—C4—O2	174.5 (2)	C6—C7—C8—C8A	−1.3 (3)
C2—C3—C4—C4A	−5.9 (3)	C7—C8—C8A—C1	179.0 (2)
C9—C3—C4—O2	−0.5 (3)	C7—C8—C8A—C4A	−0.1 (3)
C9—C3—C4—C4A	179.15 (19)	C3—C9—C10—C12	−176.6 (2)
C2—C3—C9—C10	50.7 (3)	C3—C9—C10—C22	4.9 (4)
C4—C3—C9—C10	−134.6 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1*O*3···O1^{i}	0.97 (3)	1.93 (3)	2.770 (2)	143 (3)
C7—H7···O2^{ii}	0.93	2.43	3.339 (3)	164
C22—H22*C*···O3	0.96	2.21	2.959 (3)	134

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