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# Bis(2-formylphenyl) benzene-1,2-dicarboxylate

Shaaban K. Mohamed,[a,b] Joel T. Mague,[c] Mehmet Akkurt,[d] Farouq E. Hawaiz,[e]* Mzgin M. Ayoob[e] and Awaz J. Hussein[e]

[a]Chemistry and Environmental Division, Manchester Metropolitan University, Manchester M1 5GD, England, [b]Chemistry Department, Faculty of Science, Minia University, 61519 El-Minia, Egypt, [c]Department of Chemistry, Tulane University, New Orleans, LA 70118, USA, [d]Department of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Turkey, and [e]Chemistry Department, College of Education, Salahaddin University-Hawler, Erbil, Kurdistan Region, Iraq. *Correspondence e-mail: shaabankamel@yahoo.com

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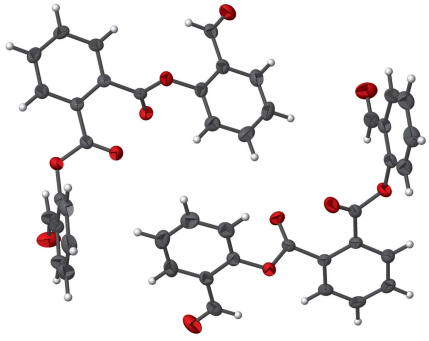
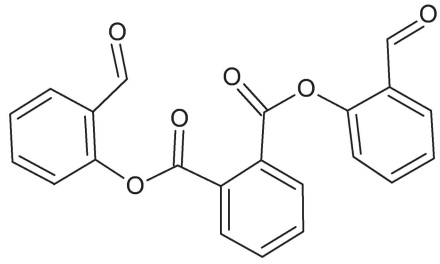
**Structural data**: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title compound, $C_{22}H_{14}O_6$, consists of two independent molecules differing in the orientations of the ester groups. In one molecule, the two terminal benzene rings are inclined to the central benzene ring by 4.99 (13) and 77.46 (13)°, while in the other the corresponding angles are 11.03 (13) and 88.09 (12)°. In the crystal, molecules are connected into a ribbon structure running along [101] *via* C—H···O and C—H···$\pi$ interactions. Adjacent ribbons are further linked by additional C—H···O and C—H···$\pi$ interactions. The crystal studied was a non-merohedral twin [twin law (0.986 − 0.073 − 0.008, 0.323 1.036 0.148, −0.121 − 0.102 0.942)], the ratio of components being 0.937 (4):0.063 (4).

**3D view**



**Chemical scheme**



## Structure description

The synthesis of bis-aldehydes is of great importance as this bi-functional unit is a useful synthon for the preparation of different intermediates such as bis-chalcones and bis-imines, which can be easily converted to many important pharmaceutical and biologically active heterocyclic compounds such as pyrazolines (Hawaiz & Shekh Omer, 2017) and thiazolidinones (Hussein & Azeez, 2013). Based on such findings, we herein report the synthesis and crystal structure of the title compound.

The asymmetric unit of the title compound consists of two independent molecules, which differ in the orientations of the ester groups (Fig. 1). In one molecule, the terminal C8–C13 and C16–C21 benzene rings are inclined to the central C1–C6 ring by 4.99 (13) and 77.46 (13)°, respectively, while in the other the C30–C35 and C38–C43 benzene rings are inclined to the C23–C28 ring by 88.09 (12) and 11.03 (13)°, respectively.

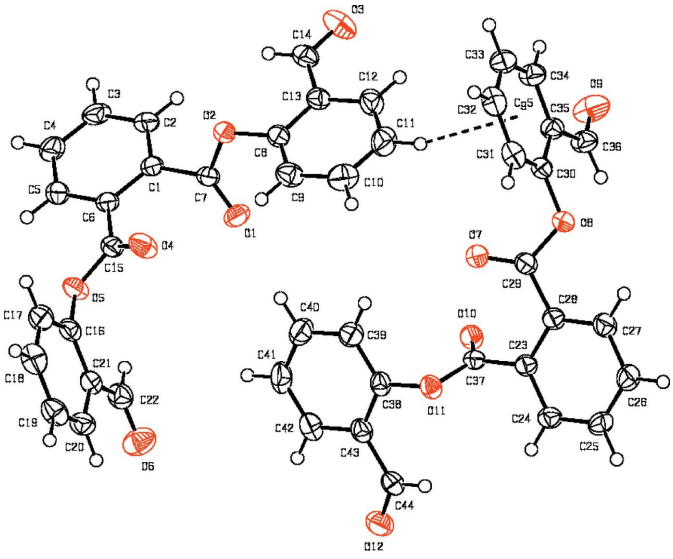



**Figure 1**
The asymmetric unit of the title compound with labeling scheme and 50% probability ellipsoids. The C—H···$\pi$ interaction is shown as a dashed line.

In the crystal (Fig. 2), the molecules are linked into a ribbon structure running along [101] *via* three predominant C—H···O hydrogen bonds and a C—H···$\pi$ interaction (C3—H3···O10$^{i}$, C22—H22···O3$^{i}$, C36—H36···O12$^{iii}$ and C11—H11···*Cg*5; symmetry codes as in Table 1; *Cg*5 is the centroid of the C30–C35 benzene ring). The ribbons are further linked by other weak C—H···O and C—H···$\pi$ interactions (C18—H18···O6$^{ii}$, C32—H32···O9$^{ii}$ and C26—H26···*Cg*3$^{iv}$; Table 1; *Cg*3 is the centroid of the C16–C21 benzene ring).

## Synthesis and crystallization

A mixture of phthaloyl chloride (0.5 g, 0.0025 mol), 2-hydroxybenzaldehyde (0.61 g, 0.005 mol) and triethylamine (0.5 g, 0.005 mol) in dioxane (20 ml) was stirred for a few minutes. The solid amine salt ($Et_3N$·HCl) was separated by filtration and the filtrate was refluxed for 30 min. On cooling, good quality crystals of the title compound were obtained, filtered and dried (0.9 g, 92%).

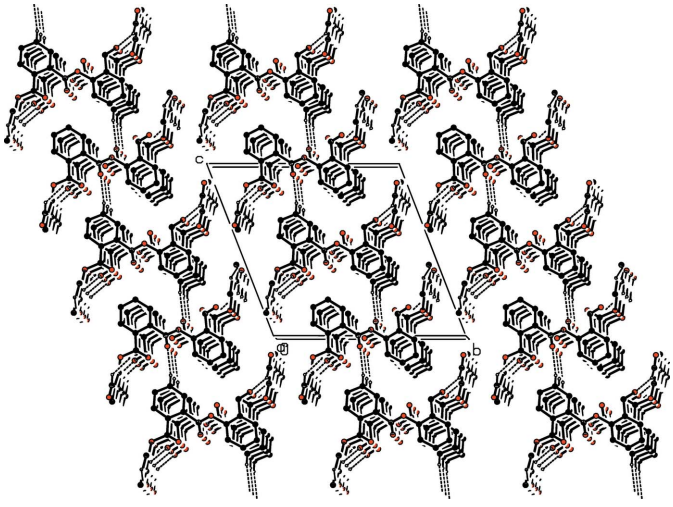



**Figure 2**
A packing diagram viewed along the *a* axis, showing the C—H···O hydrogen bonds (dashed lines).

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

*Cg*3 and *Cg*5 are the centroids of the C16–C21 and C30–C35 benzene rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C3—H3···O10$^{i}$ | 0.95 | 2.35 | 3.221 (3) | 152 |
| C18—H18···O6$^{ii}$ | 0.95 | 2.55 | 3.430 (3) | 155 |
| C22—H22···O3$^{i}$ | 0.95 | 2.29 | 3.147 (3) | 149 |
| C32—H32···O9$^{ii}$ | 0.95 | 2.54 | 3.422 (3) | 155 |
| C36—H36···O12$^{iii}$ | 0.95 | 2.41 | 3.205 (3) | 141 |
| C11—H11···*Cg*5 | 0.95 | 2.75 | 3.664 (3) | 162 |
| C26—H26···*Cg*3$^{iv}$ | 0.95 | 2.68 | 3.546 (3) | 152 |

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

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{22}H_{14}O_6$ |
| $M_r$ | 374.33 |
| Crystal system, space group | Triclinic, $P\overline{1}$ |
| Temperature (K) | 150 |
| *a*, *b*, *c* (Å) | 8.1833 (2), 15.4412 (4), 15.5250 (4) |
| $\alpha, \beta, \gamma$ (°) | 110.657 (1), 104.919 (1), 90.516 (1) |
| *V* (Å$^3$) | 1762.89 (8) |
| *Z* | 4 |
| Radiation type | Cu *K*$\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.87 |
| Crystal size (mm) | 0.17 × 0.12 × 0.08 |
| | |
| Data collection | |
| Diffractometer | Bruker D8 VENTURE PHOTON 100 CMOS |
| Absorption correction | Multi-scan (*TWINABS*; Sheldrick, 2009) |
| $T_{min}$, $T_{max}$ | 0.87, 0.94 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 26732, 26732, 16950 |
| $R_{int}$ | 0.032 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.619 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S* | 0.054, 0.153, 1.03 |
| No. of reflections | 26732 |
| No. of parameters | 507 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.49, −0.37 |

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2015).

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The crystal studied was a non-merohedral twin [twin law (0.986 − 0.073 − 0.008, 0.323 1.036 0.148, −0.121 − 0.102 0.942)], the refined ratio of the two domains being 0.937 (4) and 0.063 (4).

## Funding information

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# full crystallographic data

*IUCrData* (2018). **3**, x180251 [https://doi.org/10.1107/S2414314618002511]

## Bis(2-formylphenyl) benzene-1,2-dicarboxylate

**Shaaban K. Mohamed, Joel T. Mague, Mehmet Akkurt, Farouq E. Hawaiz, Mzgin M. Ayoob and Awaz J. Hussein**

### Bis(2-formylphenyl) benzene-1,2-dicarboxylate

*Crystal data*

$C_{22}H_{14}O_6$
$M_r$ = 374.33
Triclinic, $P\overline{1}$
$a$ = 8.1833 (2) Å
$b$ = 15.4412 (4) Å
$c$ = 15.5250 (4) Å
$\alpha$ = 110.657 (1)°
$\beta$ = 104.919 (1)°
$\gamma$ = 90.516 (1)°
$V$ = 1762.89 (8) Å$^3$
$Z$ = 4
$F(000)$ = 776
$D_x$ = 1.410 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54178 Å
Cell parameters from 9890 reflections
$\theta$ = 3.1–72.5°
$\mu$ = 0.87 mm$^{-1}$
$T$ = 150 K
Block, colourless
0.17 × 0.12 × 0.08 mm

*Data collection*

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC I$\mu$S micro-focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*TWINABS*; Sheldrick, 2009)
$T_{min}$ = 0.87, $T_{max}$ = 0.94
26732 measured reflections
26732 independent reflections
16950 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032
$\theta_{max}$ = 72.6°, $\theta_{min}$ = 3.2°
$h$ = −10→9
$k$ = −18→19
$l$ = −19→19

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.153
$S$ = 1.03
26732 reflections
507 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0601P)^2 + 0.2628P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.49 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.36 e Å$^{-3}$
Extinction correction: *SHELXL2018* (Sheldrick, 2015*b*), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0039 (5)

*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**. Refined as a 2-component twin. Analysis of 1273 reflections having I/$\sigma$(I) > 14 chosen from the full data set with CELL_NOW indicated the crystal to belong to the triclinic system and to contain two components rotated by 10.9° about the real axis [1 -1/4 -3/4]. The refined twin law was (0.986 -0.073 -0.008 0.323 1.036 0.148 -0.121 -0.102 0.942). There were 27338 single reflections, 13636 from component 1 and 13622 from component 2 and 126 full or partial overlaps. The refined twin fractions are 0.937 (4) and 0.063 (4).

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.5734 (2) | 0.42829 (12) | 0.41266 (11) | 0.0428 (4) |
| O2 | 0.80387 (19) | 0.52408 (10) | 0.51789 (10) | 0.0311 (3) |
| O3 | 0.6591 (3) | 0.77157 (12) | 0.65877 (14) | 0.0543 (5) |
| O4 | 0.8409 (3) | 0.30317 (11) | 0.36575 (11) | 0.0461 (5) |
| O5 | 0.7698 (2) | 0.16274 (10) | 0.36799 (10) | 0.0356 (4) |
| O6 | 0.3825 (2) | 0.04440 (15) | 0.12472 (13) | 0.0543 (5) |
| C1 | 0.7376 (3) | 0.38678 (15) | 0.54099 (14) | 0.0300 (5) |
| C2 | 0.7252 (3) | 0.42400 (16) | 0.63447 (15) | 0.0355 (5) |
| H2 | 0.699632 | 0.486288 | 0.659733 | 0.043* |
| C3 | 0.7502 (3) | 0.37001 (18) | 0.69075 (15) | 0.0405 (6) |
| H3 | 0.741109 | 0.395358 | 0.754464 | 0.049* |
| C4 | 0.7882 (3) | 0.27987 (17) | 0.65466 (16) | 0.0419 (6) |
| H4 | 0.807154 | 0.243657 | 0.693953 | 0.050* |
| C5 | 0.7989 (3) | 0.24170 (16) | 0.56124 (16) | 0.0370 (5) |
| H5 | 0.823710 | 0.179185 | 0.536468 | 0.044* |
| C6 | 0.7734 (3) | 0.29486 (15) | 0.50353 (14) | 0.0304 (5) |
| C7 | 0.6941 (3) | 0.44492 (14) | 0.48077 (14) | 0.0301 (5) |
| C8 | 0.7694 (3) | 0.58694 (15) | 0.46973 (15) | 0.0308 (5) |
| C9 | 0.7879 (3) | 0.56301 (17) | 0.37874 (17) | 0.0398 (5) |
| H9 | 0.823531 | 0.504487 | 0.347907 | 0.048* |
| C10 | 0.7532 (4) | 0.62625 (19) | 0.33305 (18) | 0.0473 (6) |
| H10 | 0.764553 | 0.610711 | 0.270116 | 0.057* |
| C11 | 0.7024 (4) | 0.71172 (19) | 0.3783 (2) | 0.0485 (6) |
| H11 | 0.677560 | 0.754195 | 0.346147 | 0.058* |
| C12 | 0.6877 (3) | 0.73525 (17) | 0.47004 (19) | 0.0415 (6) |
| H12 | 0.654548 | 0.794425 | 0.501197 | 0.050* |
| C13 | 0.7211 (3) | 0.67281 (15) | 0.51767 (16) | 0.0322 (5) |
| C14 | 0.7065 (3) | 0.69903 (16) | 0.61646 (17) | 0.0366 (5) |
| H14 | 0.736100 | 0.656566 | 0.648284 | 0.044* |
| C15 | 0.7984 (3) | 0.25722 (15) | 0.40591 (15) | 0.0321 (5) |
| C16 | 0.8110 (3) | 0.11665 (14) | 0.28143 (15) | 0.0312 (5) |
| C17 | 0.9792 (3) | 0.10939 (17) | 0.28322 (18) | 0.0389 (5) |
| H17 | 1.066574 | 0.136746 | 0.341619 | 0.047* |

| | | | | |
|---|---|---|---|---|
| C18 | 1.0193 (3) | 0.06153 (18) | 0.19846 (19) | 0.0430 (6) |
| H18 | 1.135038 | 0.057669 | 0.198343 | 0.052* |
| C19 | 0.8911 (3) | 0.01932 (17) | 0.11396 (17) | 0.0411 (6) |
| H19 | 0.919116 | −0.013859 | 0.056228 | 0.049* |
| C20 | 0.7233 (3) | 0.02544 (16) | 0.11364 (16) | 0.0361 (5) |
| H20 | 0.635927 | −0.004304 | 0.055714 | 0.043* |
| C21 | 0.6803 (3) | 0.07516 (15) | 0.19799 (15) | 0.0314 (5) |
| C22 | 0.4995 (3) | 0.07987 (17) | 0.19666 (17) | 0.0392 (5) |
| H22 | 0.473487 | 0.112361 | 0.255175 | 0.047* |
| O7 | 0.4612 (2) | 0.68794 (11) | 0.13827 (11) | 0.0421 (4) |
| O8 | 0.4103 (2) | 0.83121 (10) | 0.13783 (10) | 0.0343 (4) |
| O9 | 0.2576 (2) | 0.96930 (16) | 0.37361 (13) | 0.0564 (5) |
| O10 | 0.1470 (2) | 0.56231 (10) | 0.07902 (10) | 0.0344 (4) |
| O11 | 0.2885 (2) | 0.47254 (10) | −0.02015 (10) | 0.0307 (3) |
| O12 | −0.0216 (2) | 0.22964 (12) | −0.14418 (12) | 0.0445 (4) |
| C23 | 0.1995 (3) | 0.60988 (15) | −0.04291 (14) | 0.0287 (4) |
| C24 | 0.1012 (3) | 0.57557 (16) | −0.13781 (14) | 0.0330 (5) |
| H24 | 0.050852 | 0.512756 | −0.166770 | 0.040* |
| C25 | 0.0767 (3) | 0.63330 (17) | −0.19027 (15) | 0.0373 (5) |
| H25 | 0.010341 | 0.609567 | −0.255340 | 0.045* |
| C26 | 0.1480 (3) | 0.72480 (17) | −0.14857 (16) | 0.0391 (5) |
| H26 | 0.130032 | 0.763998 | −0.184761 | 0.047* |
| C27 | 0.2459 (3) | 0.75960 (16) | −0.05380 (16) | 0.0361 (5) |
| H27 | 0.294766 | 0.822699 | −0.025161 | 0.043* |
| C28 | 0.2730 (3) | 0.70253 (15) | −0.00049 (14) | 0.0301 (5) |
| C29 | 0.3900 (3) | 0.73626 (15) | 0.09826 (15) | 0.0306 (5) |
| C30 | 0.5356 (3) | 0.87237 (14) | 0.22533 (15) | 0.0298 (5) |
| C31 | 0.7047 (3) | 0.86742 (16) | 0.22777 (17) | 0.0373 (5) |
| H31 | 0.736192 | 0.834619 | 0.171062 | 0.045* |
| C32 | 0.8280 (3) | 0.91077 (17) | 0.3137 (2) | 0.0421 (6) |
| H32 | 0.944924 | 0.907067 | 0.316452 | 0.051* |
| C33 | 0.7813 (3) | 0.95957 (17) | 0.39587 (18) | 0.0420 (6) |
| H33 | 0.866240 | 0.988609 | 0.455021 | 0.050* |
| C34 | 0.6121 (3) | 0.96609 (17) | 0.39196 (16) | 0.0377 (5) |
| H34 | 0.581216 | 1.000895 | 0.448302 | 0.045* |
| C35 | 0.4856 (3) | 0.92221 (15) | 0.30632 (15) | 0.0303 (5) |
| C36 | 0.3052 (3) | 0.93132 (17) | 0.30308 (16) | 0.0374 (5) |
| H36 | 0.222211 | 0.906132 | 0.242657 | 0.045* |
| C37 | 0.2092 (3) | 0.54900 (14) | 0.01415 (14) | 0.0274 (4) |
| C38 | 0.3023 (3) | 0.41139 (15) | 0.03022 (15) | 0.0303 (5) |
| C39 | 0.4192 (3) | 0.43683 (17) | 0.11928 (16) | 0.0377 (5) |
| H39 | 0.488630 | 0.494788 | 0.146572 | 0.045* |
| C40 | 0.4332 (3) | 0.37622 (19) | 0.16793 (17) | 0.0444 (6) |
| H40 | 0.512796 | 0.392745 | 0.229272 | 0.053* |
| C41 | 0.3319 (4) | 0.29152 (18) | 0.12787 (19) | 0.0462 (6) |
| H41 | 0.341554 | 0.250581 | 0.162010 | 0.055* |
| C42 | 0.2169 (3) | 0.26673 (17) | 0.03832 (18) | 0.0402 (5) |
| H42 | 0.148523 | 0.208449 | 0.010831 | 0.048* |

| | | | | |
|---|---|---|---|---|
| C43 | 0.2006 (3) | 0.32701 (15) | −0.01224 (15) | 0.0317 (5) |
| C44 | 0.0783 (3) | 0.29935 (15) | −0.10841 (16) | 0.0335 (5) |
| H44 | 0.078098 | 0.338378 | −0.143977 | 0.040* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0479 (10) | 0.0383 (9) | 0.0337 (8) | −0.0107 (8) | −0.0082 (7) | 0.0175 (7) |
| O2 | 0.0334 (8) | 0.0251 (8) | 0.0309 (7) | −0.0009 (6) | 0.0035 (6) | 0.0097 (6) |
| O3 | 0.0635 (12) | 0.0340 (10) | 0.0613 (11) | 0.0048 (9) | 0.0306 (10) | 0.0033 (8) |
| O4 | 0.0803 (13) | 0.0286 (8) | 0.0332 (8) | −0.0009 (8) | 0.0221 (8) | 0.0116 (7) |
| O5 | 0.0536 (10) | 0.0238 (8) | 0.0294 (8) | −0.0004 (7) | 0.0162 (7) | 0.0066 (6) |
| O6 | 0.0378 (10) | 0.0713 (13) | 0.0448 (10) | 0.0075 (9) | 0.0083 (8) | 0.0129 (9) |
| C1 | 0.0334 (11) | 0.0281 (11) | 0.0238 (9) | −0.0020 (9) | 0.0017 (8) | 0.0087 (8) |
| C2 | 0.0424 (13) | 0.0322 (12) | 0.0245 (10) | 0.0006 (10) | 0.0041 (9) | 0.0053 (9) |
| C3 | 0.0504 (14) | 0.0449 (14) | 0.0210 (10) | −0.0025 (11) | 0.0054 (9) | 0.0093 (9) |
| C4 | 0.0564 (15) | 0.0416 (14) | 0.0291 (11) | −0.0011 (12) | 0.0051 (10) | 0.0195 (10) |
| C5 | 0.0499 (14) | 0.0293 (11) | 0.0302 (11) | 0.0006 (10) | 0.0063 (10) | 0.0124 (9) |
| C6 | 0.0370 (12) | 0.0267 (11) | 0.0235 (10) | −0.0026 (9) | 0.0030 (8) | 0.0084 (8) |
| C7 | 0.0356 (12) | 0.0248 (11) | 0.0252 (10) | −0.0010 (9) | 0.0053 (9) | 0.0060 (8) |
| C8 | 0.0302 (11) | 0.0267 (11) | 0.0333 (11) | −0.0022 (9) | 0.0041 (9) | 0.0119 (9) |
| C9 | 0.0460 (14) | 0.0350 (13) | 0.0379 (12) | −0.0016 (11) | 0.0142 (10) | 0.0110 (10) |
| C10 | 0.0552 (16) | 0.0504 (16) | 0.0379 (13) | −0.0102 (13) | 0.0115 (11) | 0.0194 (11) |
| C11 | 0.0520 (16) | 0.0434 (15) | 0.0540 (15) | −0.0054 (12) | 0.0044 (12) | 0.0300 (13) |
| C12 | 0.0390 (13) | 0.0312 (12) | 0.0524 (14) | 0.0003 (10) | 0.0060 (11) | 0.0180 (11) |
| C13 | 0.0295 (11) | 0.0271 (11) | 0.0356 (11) | −0.0016 (9) | 0.0052 (9) | 0.0092 (9) |
| C14 | 0.0351 (12) | 0.0287 (12) | 0.0400 (12) | −0.0026 (10) | 0.0091 (10) | 0.0064 (9) |
| C15 | 0.0432 (13) | 0.0240 (10) | 0.0269 (10) | 0.0006 (9) | 0.0066 (9) | 0.0089 (8) |
| C16 | 0.0429 (13) | 0.0228 (10) | 0.0303 (10) | 0.0032 (9) | 0.0137 (9) | 0.0102 (8) |
| C17 | 0.0391 (13) | 0.0350 (13) | 0.0426 (13) | −0.0006 (10) | 0.0084 (10) | 0.0164 (10) |
| C18 | 0.0365 (13) | 0.0416 (14) | 0.0591 (15) | 0.0083 (11) | 0.0221 (12) | 0.0218 (12) |
| C19 | 0.0515 (15) | 0.0351 (13) | 0.0428 (13) | 0.0088 (11) | 0.0264 (11) | 0.0120 (10) |
| C20 | 0.0430 (13) | 0.0326 (12) | 0.0309 (11) | 0.0038 (10) | 0.0124 (9) | 0.0080 (9) |
| C21 | 0.0381 (12) | 0.0260 (11) | 0.0327 (11) | 0.0061 (9) | 0.0136 (9) | 0.0112 (9) |
| C22 | 0.0431 (14) | 0.0375 (13) | 0.0387 (12) | 0.0112 (11) | 0.0169 (11) | 0.0119 (10) |
| O7 | 0.0549 (11) | 0.0288 (8) | 0.0346 (8) | 0.0018 (8) | −0.0017 (7) | 0.0123 (7) |
| O8 | 0.0441 (9) | 0.0246 (8) | 0.0281 (7) | 0.0035 (7) | 0.0041 (6) | 0.0066 (6) |
| O9 | 0.0343 (9) | 0.0830 (15) | 0.0398 (9) | 0.0076 (9) | 0.0121 (7) | 0.0070 (9) |
| O10 | 0.0497 (9) | 0.0327 (8) | 0.0259 (7) | 0.0115 (7) | 0.0168 (7) | 0.0122 (6) |
| O11 | 0.0392 (8) | 0.0275 (8) | 0.0298 (7) | 0.0077 (7) | 0.0149 (6) | 0.0118 (6) |
| O12 | 0.0445 (10) | 0.0334 (9) | 0.0472 (10) | 0.0009 (8) | 0.0099 (8) | 0.0069 (7) |
| C23 | 0.0352 (11) | 0.0284 (11) | 0.0241 (9) | 0.0058 (9) | 0.0112 (8) | 0.0092 (8) |
| C24 | 0.0403 (12) | 0.0314 (11) | 0.0244 (10) | 0.0031 (10) | 0.0089 (9) | 0.0070 (9) |
| C25 | 0.0455 (13) | 0.0430 (13) | 0.0232 (10) | 0.0084 (11) | 0.0085 (9) | 0.0126 (9) |
| C26 | 0.0532 (15) | 0.0401 (13) | 0.0305 (11) | 0.0096 (11) | 0.0135 (10) | 0.0195 (10) |
| C27 | 0.0486 (14) | 0.0306 (12) | 0.0316 (11) | 0.0044 (10) | 0.0133 (10) | 0.0129 (9) |
| C28 | 0.0371 (12) | 0.0290 (11) | 0.0254 (10) | 0.0051 (9) | 0.0108 (9) | 0.0099 (8) |
| C29 | 0.0397 (12) | 0.0252 (10) | 0.0271 (10) | 0.0029 (9) | 0.0106 (9) | 0.0089 (8) |

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|---|---|---|---|---|---|---|
| C30 | 0.0340 (11) | 0.0232 (10) | 0.0301 (10) | 0.0004 (9) | 0.0053 (9) | 0.0098 (8) |
| C31 | 0.0397 (13) | 0.0308 (12) | 0.0468 (13) | 0.0070 (10) | 0.0191 (10) | 0.0158 (10) |
| C32 | 0.0286 (12) | 0.0361 (13) | 0.0645 (16) | 0.0029 (10) | 0.0134 (11) | 0.0217 (12) |
| C33 | 0.0299 (12) | 0.0375 (13) | 0.0464 (13) | −0.0044 (10) | −0.0022 (10) | 0.0104 (11) |
| C34 | 0.0337 (12) | 0.0385 (13) | 0.0333 (11) | −0.0008 (10) | 0.0046 (9) | 0.0075 (10) |
| C35 | 0.0281 (11) | 0.0291 (11) | 0.0312 (10) | −0.0011 (9) | 0.0061 (8) | 0.0099 (9) |
| C36 | 0.0297 (12) | 0.0404 (13) | 0.0341 (11) | 0.0000 (10) | 0.0034 (9) | 0.0081 (10) |
| C37 | 0.0318 (11) | 0.0249 (10) | 0.0219 (9) | 0.0016 (9) | 0.0054 (8) | 0.0057 (8) |
| C38 | 0.0350 (11) | 0.0297 (11) | 0.0314 (10) | 0.0109 (10) | 0.0143 (9) | 0.0136 (9) |
| C39 | 0.0389 (13) | 0.0386 (13) | 0.0343 (11) | 0.0081 (11) | 0.0097 (9) | 0.0121 (10) |
| C40 | 0.0494 (15) | 0.0519 (15) | 0.0328 (12) | 0.0187 (13) | 0.0093 (10) | 0.0177 (11) |
| C41 | 0.0610 (17) | 0.0452 (15) | 0.0471 (14) | 0.0219 (13) | 0.0214 (12) | 0.0296 (12) |
| C42 | 0.0494 (14) | 0.0324 (12) | 0.0453 (13) | 0.0103 (11) | 0.0196 (11) | 0.0172 (10) |
| C43 | 0.0359 (12) | 0.0287 (11) | 0.0332 (11) | 0.0098 (10) | 0.0147 (9) | 0.0108 (9) |
| C44 | 0.0367 (12) | 0.0284 (11) | 0.0353 (11) | 0.0084 (10) | 0.0142 (9) | 0.0085 (9) |

*Geometric parameters (Å, º)*

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| O1—C7 | 1.194 (3) | O7—C29 | 1.196 (3) |
| O2—C7 | 1.360 (3) | O8—C29 | 1.365 (3) |
| O2—C8 | 1.410 (3) | O8—C30 | 1.401 (3) |
| O3—C14 | 1.207 (3) | O9—C36 | 1.211 (3) |
| O4—C15 | 1.197 (3) | O10—C37 | 1.198 (2) |
| O5—C15 | 1.359 (3) | O11—C37 | 1.359 (3) |
| O5—C16 | 1.410 (2) | O11—C38 | 1.412 (2) |
| O6—C22 | 1.213 (3) | O12—C44 | 1.210 (3) |
| C1—C2 | 1.391 (3) | C23—C24 | 1.389 (3) |
| C1—C6 | 1.397 (3) | C23—C28 | 1.399 (3) |
| C1—C7 | 1.494 (3) | C23—C37 | 1.493 (3) |
| C2—C3 | 1.388 (3) | C24—C25 | 1.389 (3) |
| C2—H2 | 0.9500 | C24—H24 | 0.9500 |
| C3—C4 | 1.377 (4) | C25—C26 | 1.379 (3) |
| C3—H3 | 0.9500 | C25—H25 | 0.9500 |
| C4—C5 | 1.386 (3) | C26—C27 | 1.387 (3) |
| C4—H4 | 0.9500 | C26—H26 | 0.9500 |
| C5—C6 | 1.395 (3) | C27—C28 | 1.391 (3) |
| C5—H5 | 0.9500 | C27—H27 | 0.9500 |
| C6—C15 | 1.489 (3) | C28—C29 | 1.487 (3) |
| C8—C9 | 1.377 (3) | C30—C31 | 1.378 (3) |
| C8—C13 | 1.390 (3) | C30—C35 | 1.389 (3) |
| C9—C10 | 1.388 (4) | C31—C32 | 1.382 (4) |
| C9—H9 | 0.9500 | C31—H31 | 0.9500 |
| C10—C11 | 1.384 (4) | C32—C33 | 1.385 (4) |
| C10—H10 | 0.9500 | C32—H32 | 0.9500 |
| C11—C12 | 1.377 (4) | C33—C34 | 1.377 (3) |
| C11—H11 | 0.9500 | C33—H33 | 0.9500 |
| C12—C13 | 1.398 (3) | C34—C35 | 1.393 (3) |
| C12—H12 | 0.9500 | C34—H34 | 0.9500 |

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| C13—C14 | 1.479 (3) | C35—C36 | 1.474 (3) |
| C14—H14 | 0.9500 | C36—H36 | 0.9500 |
| C16—C17 | 1.376 (3) | C38—C39 | 1.382 (3) |
| C16—C21 | 1.386 (3) | C38—C43 | 1.384 (3) |
| C17—C18 | 1.388 (4) | C39—C40 | 1.384 (3) |
| C17—H17 | 0.9500 | C39—H39 | 0.9500 |
| C18—C19 | 1.386 (4) | C40—C41 | 1.388 (4) |
| C18—H18 | 0.9500 | C40—H40 | 0.9500 |
| C19—C20 | 1.376 (4) | C41—C42 | 1.382 (4) |
| C19—H19 | 0.9500 | C41—H41 | 0.9500 |
| C20—C21 | 1.400 (3) | C42—C43 | 1.401 (3) |
| C20—H20 | 0.9500 | C42—H42 | 0.9500 |
| C21—C22 | 1.477 (3) | C43—C44 | 1.479 (3) |
| C22—H22 | 0.9500 | C44—H44 | 0.9500 |

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| C7—O2—C8 | 115.88 (16) | C29—O8—C30 | 116.81 (16) |
| C15—O5—C16 | 117.47 (16) | C37—O11—C38 | 115.03 (15) |
| C2—C1—C6 | 120.02 (19) | C24—C23—C28 | 119.85 (19) |
| C2—C1—C7 | 117.62 (19) | C24—C23—C37 | 118.41 (19) |
| C6—C1—C7 | 122.07 (18) | C28—C23—C37 | 121.44 (18) |
| C3—C2—C1 | 119.9 (2) | C25—C24—C23 | 119.9 (2) |
| C3—C2—H2 | 120.0 | C25—C24—H24 | 120.1 |
| C1—C2—H2 | 120.0 | C23—C24—H24 | 120.1 |
| C4—C3—C2 | 120.3 (2) | C26—C25—C24 | 120.5 (2) |
| C4—C3—H3 | 119.9 | C26—C25—H25 | 119.8 |
| C2—C3—H3 | 119.9 | C24—C25—H25 | 119.8 |
| C3—C4—C5 | 120.3 (2) | C25—C26—C27 | 120.0 (2) |
| C3—C4—H4 | 119.8 | C25—C26—H26 | 120.0 |
| C5—C4—H4 | 119.8 | C27—C26—H26 | 120.0 |
| C4—C5—C6 | 120.2 (2) | C26—C27—C28 | 120.3 (2) |
| C4—C5—H5 | 119.9 | C26—C27—H27 | 119.9 |
| C6—C5—H5 | 119.9 | C28—C27—H27 | 119.9 |
| C5—C6—C1 | 119.31 (19) | C27—C28—C23 | 119.5 (2) |
| C5—C6—C15 | 120.6 (2) | C27—C28—C29 | 120.9 (2) |
| C1—C6—C15 | 119.91 (18) | C23—C28—C29 | 119.39 (18) |
| O1—C7—O2 | 123.6 (2) | O7—C29—O8 | 123.5 (2) |
| O1—C7—C1 | 125.3 (2) | O7—C29—C28 | 125.5 (2) |
| O2—C7—C1 | 110.90 (18) | O8—C29—C28 | 111.05 (18) |
| C9—C8—C13 | 122.2 (2) | C31—C30—C35 | 121.8 (2) |
| C9—C8—O2 | 119.8 (2) | C31—C30—O8 | 119.43 (19) |
| C13—C8—O2 | 118.03 (19) | C35—C30—O8 | 118.69 (19) |
| C8—C9—C10 | 118.6 (2) | C30—C31—C32 | 119.2 (2) |
| C8—C9—H9 | 120.7 | C30—C31—H31 | 120.4 |
| C10—C9—H9 | 120.7 | C32—C31—H31 | 120.4 |
| C11—C10—C9 | 120.6 (2) | C31—C32—C33 | 120.1 (2) |
| C11—C10—H10 | 119.7 | C31—C32—H32 | 119.9 |
| C9—C10—H10 | 119.7 | C33—C32—H32 | 119.9 |
| C12—C11—C10 | 120.1 (2) | C34—C33—C32 | 120.1 (2) |

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| C12—C11—H11 | 120.0 | C34—C33—H33 | 119.9 |
| C10—C11—H11 | 120.0 | C32—C33—H33 | 119.9 |
| C11—C12—C13 | 120.6 (2) | C33—C34—C35 | 120.8 (2) |
| C11—C12—H12 | 119.7 | C33—C34—H34 | 119.6 |
| C13—C12—H12 | 119.7 | C35—C34—H34 | 119.6 |
| C8—C13—C12 | 118.0 (2) | C30—C35—C34 | 118.0 (2) |
| C8—C13—C14 | 121.8 (2) | C30—C35—C36 | 121.96 (19) |
| C12—C13—C14 | 120.2 (2) | C34—C35—C36 | 120.01 (19) |
| O3—C14—C13 | 123.4 (2) | O9—C36—C35 | 123.3 (2) |
| O3—C14—H14 | 118.3 | O9—C36—H36 | 118.4 |
| C13—C14—H14 | 118.3 | C35—C36—H36 | 118.4 |
| O4—C15—O5 | 123.68 (19) | O10—C37—O11 | 123.38 (19) |
| O4—C15—C6 | 125.1 (2) | O10—C37—C23 | 124.77 (19) |
| O5—C15—C6 | 111.23 (17) | O11—C37—C23 | 111.73 (16) |
| C17—C16—C21 | 121.9 (2) | C39—C38—C43 | 122.2 (2) |
| C17—C16—O5 | 119.1 (2) | C39—C38—O11 | 119.0 (2) |
| C21—C16—O5 | 118.9 (2) | C43—C38—O11 | 118.81 (19) |
| C16—C17—C18 | 119.0 (2) | C38—C39—C40 | 118.7 (2) |
| C16—C17—H17 | 120.5 | C38—C39—H39 | 120.6 |
| C18—C17—H17 | 120.5 | C40—C39—H39 | 120.6 |
| C19—C18—C17 | 120.2 (2) | C39—C40—C41 | 120.6 (2) |
| C19—C18—H18 | 119.9 | C39—C40—H40 | 119.7 |
| C17—C18—H18 | 119.9 | C41—C40—H40 | 119.7 |
| C20—C19—C18 | 120.1 (2) | C42—C41—C40 | 120.0 (2) |
| C20—C19—H19 | 120.0 | C42—C41—H41 | 120.0 |
| C18—C19—H19 | 120.0 | C40—C41—H41 | 120.0 |
| C19—C20—C21 | 120.5 (2) | C41—C42—C43 | 120.4 (2) |
| C19—C20—H20 | 119.7 | C41—C42—H42 | 119.8 |
| C21—C20—H20 | 119.7 | C43—C42—H42 | 119.8 |
| C16—C21—C20 | 118.2 (2) | C38—C43—C42 | 118.1 (2) |
| C16—C21—C22 | 121.97 (19) | C38—C43—C44 | 121.9 (2) |
| C20—C21—C22 | 119.8 (2) | C42—C43—C44 | 119.9 (2) |
| O6—C22—C21 | 123.4 (2) | O12—C44—C43 | 123.5 (2) |
| O6—C22—H22 | 118.3 | O12—C44—H44 | 118.3 |
| C21—C22—H22 | 118.3 | C43—C44—H44 | 118.3 |

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|---|---|---|---|
| C6—C1—C2—C3 | 0.7 (3) | C28—C23—C24—C25 | 0.2 (3) |
| C7—C1—C2—C3 | 174.6 (2) | C37—C23—C24—C25 | 174.05 (19) |
| C1—C2—C3—C4 | 0.4 (4) | C23—C24—C25—C26 | −0.6 (3) |
| C2—C3—C4—C5 | −1.1 (4) | C24—C25—C26—C27 | 0.4 (4) |
| C3—C4—C5—C6 | 0.8 (4) | C25—C26—C27—C28 | 0.1 (4) |
| C4—C5—C6—C1 | 0.2 (4) | C26—C27—C28—C23 | −0.5 (3) |
| C4—C5—C6—C15 | 175.0 (2) | C26—C27—C28—C29 | 174.5 (2) |
| C2—C1—C6—C5 | −0.9 (3) | C24—C23—C28—C27 | 0.3 (3) |
| C7—C1—C6—C5 | −174.6 (2) | C37—C23—C28—C27 | −173.3 (2) |
| C2—C1—C6—C15 | −175.8 (2) | C24—C23—C28—C29 | −174.7 (2) |
| C7—C1—C6—C15 | 10.5 (3) | C37—C23—C28—C29 | 11.6 (3) |
| C8—O2—C7—O1 | −1.1 (3) | C30—O8—C29—O7 | 7.1 (3) |

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|---|---|---|---|
| C8—O2—C7—C1 | −176.66 (17) | C30—O8—C29—C28 | −171.30 (18) |
| C2—C1—C7—O1 | −111.7 (3) | C27—C28—C29—O7 | −156.0 (2) |
| C6—C1—C7—O1 | 62.1 (3) | C23—C28—C29—O7 | 19.0 (3) |
| C2—C1—C7—O2 | 63.7 (3) | C27—C28—C29—O8 | 22.4 (3) |
| C6—C1—C7—O2 | −122.5 (2) | C23—C28—C29—O8 | −162.62 (18) |
| C7—O2—C8—C9 | −68.1 (3) | C29—O8—C30—C31 | 69.3 (3) |
| C7—O2—C8—C13 | 113.4 (2) | C29—O8—C30—C35 | −114.7 (2) |
| C13—C8—C9—C10 | −1.5 (4) | C35—C30—C31—C32 | 2.1 (3) |
| O2—C8—C9—C10 | −179.9 (2) | O8—C30—C31—C32 | 177.9 (2) |
| C8—C9—C10—C11 | 0.4 (4) | C30—C31—C32—C33 | −0.9 (4) |
| C9—C10—C11—C12 | 0.8 (4) | C31—C32—C33—C34 | −0.8 (4) |
| C10—C11—C12—C13 | −1.0 (4) | C32—C33—C34—C35 | 1.5 (4) |
| C9—C8—C13—C12 | 1.2 (3) | C31—C30—C35—C34 | −1.4 (3) |
| O2—C8—C13—C12 | 179.7 (2) | O8—C30—C35—C34 | −177.30 (19) |
| C9—C8—C13—C14 | −178.1 (2) | C31—C30—C35—C36 | 177.1 (2) |
| O2—C8—C13—C14 | 0.4 (3) | O8—C30—C35—C36 | 1.2 (3) |
| C11—C12—C13—C8 | 0.1 (4) | C33—C34—C35—C30 | −0.3 (3) |
| C11—C12—C13—C14 | 179.4 (2) | C33—C34—C35—C36 | −178.9 (2) |
| C8—C13—C14—O3 | −177.2 (2) | C30—C35—C36—O9 | 175.2 (2) |
| C12—C13—C14—O3 | 3.4 (4) | C34—C35—C36—O9 | −6.3 (4) |
| C16—O5—C15—O4 | 7.3 (3) | C38—O11—C37—O10 | −3.7 (3) |
| C16—O5—C15—C6 | −171.59 (19) | C38—O11—C37—C23 | −179.93 (17) |
| C5—C6—C15—O4 | −151.2 (3) | C24—C23—C37—O10 | −110.6 (2) |
| C1—C6—C15—O4 | 23.6 (4) | C28—C23—C37—O10 | 63.2 (3) |
| C5—C6—C15—O5 | 27.7 (3) | C24—C23—C37—O11 | 65.6 (3) |
| C1—C6—C15—O5 | −157.6 (2) | C28—C23—C37—O11 | −120.7 (2) |
| C15—O5—C16—C17 | 75.0 (3) | C37—O11—C38—C39 | −74.2 (2) |
| C15—O5—C16—C21 | −108.8 (2) | C37—O11—C38—C43 | 107.2 (2) |
| C21—C16—C17—C18 | 2.0 (3) | C43—C38—C39—C40 | −0.9 (3) |
| O5—C16—C17—C18 | 178.0 (2) | O11—C38—C39—C40 | −179.45 (19) |
| C16—C17—C18—C19 | −2.0 (4) | C38—C39—C40—C41 | 0.1 (3) |
| C17—C18—C19—C20 | 0.6 (4) | C39—C40—C41—C42 | 0.6 (4) |
| C18—C19—C20—C21 | 0.8 (4) | C40—C41—C42—C43 | −0.6 (4) |
| C17—C16—C21—C20 | −0.6 (3) | C39—C38—C43—C42 | 0.8 (3) |
| O5—C16—C21—C20 | −176.64 (18) | O11—C38—C43—C42 | 179.45 (18) |
| C17—C16—C21—C22 | 177.6 (2) | C39—C38—C43—C44 | −178.7 (2) |
| O5—C16—C21—C22 | 1.6 (3) | O11—C38—C43—C44 | −0.1 (3) |
| C19—C20—C21—C16 | −0.8 (3) | C41—C42—C43—C38 | −0.1 (3) |
| C19—C20—C21—C22 | −179.1 (2) | C41—C42—C43—C44 | 179.4 (2) |
| C16—C21—C22—O6 | 179.9 (2) | C38—C43—C44—O12 | −173.9 (2) |
| C20—C21—C22—O6 | −1.9 (4) | C42—C43—C44—O12 | 6.5 (3) |

*Hydrogen-bond geometry (Å, º)*

*Cg*3 and *Cg*5 are the centroids of the C16–C21 and C30–C35 benzene rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C3—H3···O10$^{i}$ | 0.95 | 2.35 | 3.221 (3) | 152 |
| C18—H18···O6$^{ii}$ | 0.95 | 2.55 | 3.430 (3) | 155 |

| | | | | |
|---|---|---|---|---|
| C22—H22···O3$^{i}$ | 0.95 | 2.29 | 3.147 (3) | 149 |
| C32—H32···O9$^{ii}$ | 0.95 | 2.54 | 3.422 (3) | 155 |
| C36—H36···O12$^{iii}$ | 0.95 | 2.41 | 3.205 (3) | 141 |
| C11—H11···*Cg*5 | 0.95 | 2.75 | 3.664 (3) | 162 |
| C26—H26···*Cg*3$^{iv}$ | 0.95 | 2.68 | 3.546 (3) | 152 |

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