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# 2,4-Dimethoxy-6-[(*E*)-2-(4-methoxyphenyl)ethenyl]benzaldehyde

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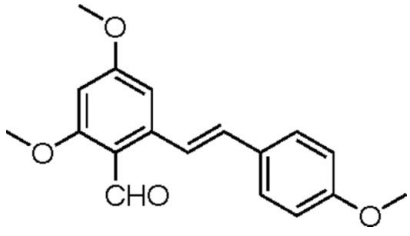
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.048; $wR$ factor = 0.168; data-to-parameter ratio = 13.0.

There are two conformationally similar molecules in the asymmetric unit of he title compound, $C_{18}H_{18}O_4$, in which the dihedral angles between the benzene rings are 23.54 (12) and 31.11 (12)°. In the crystal, C—H···$\pi$ interactions (minimum H···ring centroid distance = 2.66 Å) link the molecules into a layered structure extending down *a*.

## Related literature

The title compound is a derivative of the natural product resveratrol (*trans*-3,4,5-trihydroxystilbene). For background to resveratrol, see: Jang *et al.* (1997); Orsini *et al.* (1997); Pettit *et al.* (2002). For standard bond lengths and angles, see: Boumendjel *et al.* (2008). For the synthesis of the title compound, see: Huang *et al.* (2007).




## Experimental

### Crystal data

$C_{18}H_{18}O_4$
$M_r$ = 298.32
Triclinic, $P\overline{1}$
$a$ = 9.292 (5) Å
$b$ = 9.448 (5) Å
$c$ = 17.547 (5) Å
$\alpha$ = 84.097 (5)°
$\beta$ = 84.040 (5)°
$\gamma$ = 83.315 (5)°
$V$ = 1515.3 (12) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 298 K
0.30 × 0.20 × 0.20 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min}$ = 0.973, $T_{max}$ = 0.982
10535 measured reflections
5272 independent reflections
3060 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.039

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.168
$S$ = 0.79
5272 reflections
404 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$

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

*Cg*2 and *Cg*4 are the centroids of the C12–C17 and C30–C35 rings, respectively.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C14—H14···*Cg*4$^{i}$ | 0.93 | 2.88 | 3.597 (3) | 135 |
| C17—H14···*Cg*4$^{ii}$ | 0.93 | 2.76 | 3.471 (3) | 134 |
| C32—H14···*Cg*2 | 0.93 | 2.82 | 3.530 (3) | 135 |
| C15—H14···*Cg*2$^{iii}$ | 0.93 | 2.66 | 3.371 (3) | 134 |

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: ZS2252).

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supporting information

*Acta Cryst.* (2013). E**69**, o629 [https://doi.org/10.1107/S1600536813007964]

## 2,4-Dimethoxy-6-[(*E*)-2-(4-methoxyphenyl)ethenyl]benzaldehyde

**Xiao-Lin Ge, Qiu-Xiang Guan, Sheng-Song Deng and Ban-Feng Ruan**

### S1. Comment

Resveratrol (*trans*-3,4,,5-trihydroxystilbene), a naturally occurring phytoalexin present in medicinal plants, grape skin, peanuts and red wine, has attracted a great deal of attention because it has been suggested as a possible cancer chemopreventive agent on the basis of its inhibitory effects on tumor initiation, promotion, and progression (Jang *et al.*, 1997; Orsini *et al.*, 1997; Pettit *et al.*, 2002). In order to discover novel antitumor agents with high efficiency, a broad spectrum and safety, several series of derivatives of resveratrol were prepared in our laboratory. As an important intermediate, the title compound 2,4-dimethoxy-6-[(*E*)-2-(4-methoxyphenyl)ethenyl]benzaldehyde, was synthesized by the reaction of resveratrol using the Vilslmeier reaction and herein we report the crystal structure.

The title compound, C18 H18 O4, crystallizes in the triclinic space group *P*-1 with two independent molecules in the asymmetric unit (Fig. 1). All bond lengths are within normal ranges (Boumendjel *et al.*, 2008). The C10—C11 and C28—C29 bond lengths [1.319 (3) and 1.323 (3) Å] are consistent with the expected value for a C—C double bond and similarly, the C7—O1 and C25—O5 bond lengths [1.196 (3) and 1.210 (3) Å] are consistent with the expected value for a carbonyl C—O double bond. The dihedral angle between the two phenyl rings in each are 23.54 (12)° [C1–C6 (ring 1) and C12–C17) (ring 2)] and 31.11 (12)° [C19–C24 (ring 3) and C30–C35 (ring 4)]. As such, the structural differences between the two molecules in the title compound are only marginal.

In the crystal, the two molecules associate through C—H···pi stacking interactions [C14—H14···Cg(4)$^{i}$; C17—H···Cg(4)$^{ii}$; C32—H···Cg(2)$^{iii}$; C35—H···Cg(2)$^{iv}$: H···Cg = 2.88, 2.76, 2.82, 2.66 Å respectively] [for symmetry codes: (i) -*x* + 1, -*y* + 1, -*z* + 1; (ii) -*x* + 1, -*y*, -*z* + 1; (iii) *x*, *y*, *z*; (iv) *x* + 1, *y*, *z*]. Intermolecular methoxy C26—H···O8$^{v}$ interactions [3.041 (4) Å] across an inversion centre generate a sheet structure (Fig. 2) [for symmetry code (v): -*x* + 2, -*y*, -*z* + 1]. Present also in the structure are intramolecular C—H···O interactions between the aldehyde and ethylenic donors and methoxy O-atom acceptors (Table 1).

### S2. Experimental

Title compound was synthesized according to the reported method (Huang *et al.*, 2007). Crystals suitable for X-ray structure analysis were obtained by slow evaporation of a solution of the title compound in a petroleum ether/ dichloromethane mixture (1:1, *v*/*v*) at room temperature.

### S3. Refinement

All the H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93–0.96 Å, and with $U_{iso}$(H) = 1.2$U_{eq}$(carrier) or 1.5$U_{eq}$(methyl groups).

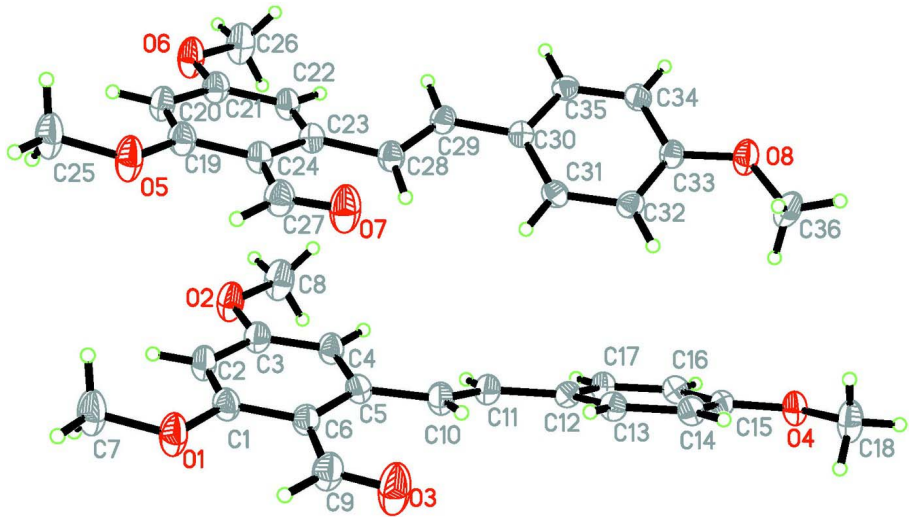


**Figure 1**

The molecular structure and atom numbering scheme for the two independent molecules of the title compound in the asymmetric unit, showing 30% probability displacement ellipsoids.

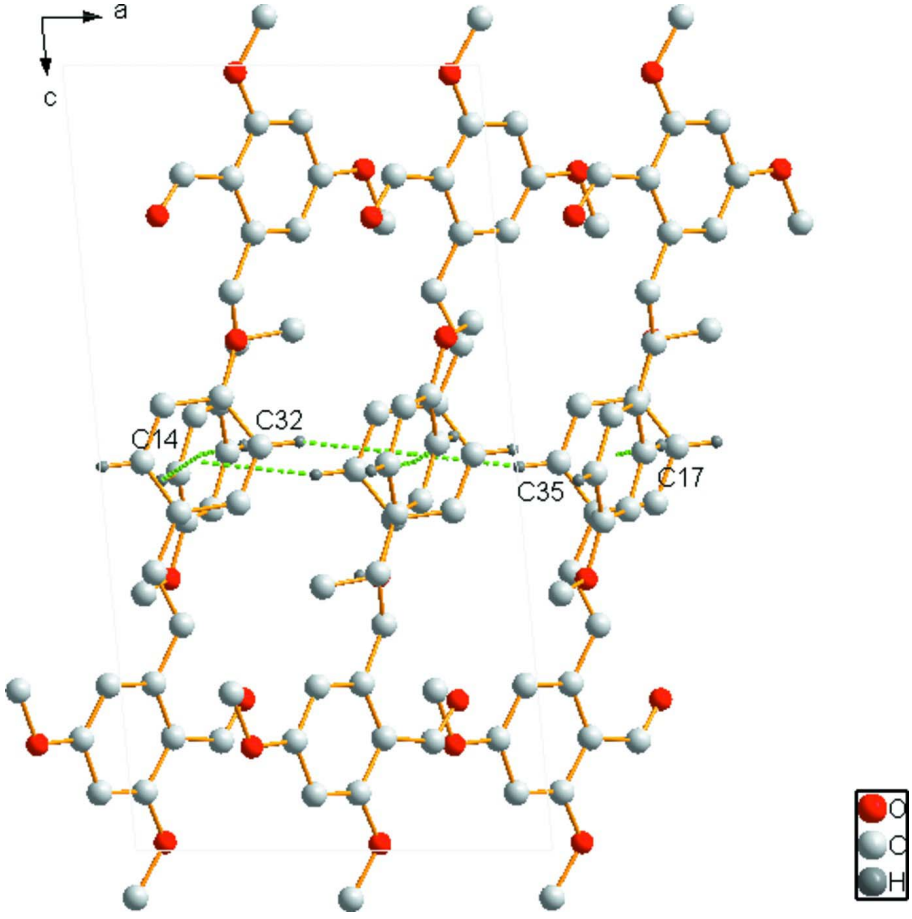


**Figure 2**

The layered structure of title compound formed through C—H···pi stacking interactions. Non-interactive H-atoms are omitted.

## 2,4-Dimethoxy-6-[(*E*)-2-(4-methoxyphenyl)ethenyl]benzaldehyde

*Crystal data*

$C_{18}H_{18}O_4$
$M_r = 298.32$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.292$ (5) Å
$b = 9.448$ (5) Å
$c = 17.547$ (5) Å
$\alpha = 84.097$ (5)°
$\beta = 84.040$ (5)°
$\gamma = 83.315$ (5)°
$V = 1515.3$ (12) Å$^3$
$Z = 4$
$F(000) = 632$
$D_x = 1.308$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
Cell parameters from 1909 reflections
$\theta = 2.2$–$26.7°$
$\mu = 0.09$ mm$^{-1}$
$T = 298$ K
Block, colourless
0.30 × 0.20 × 0.20 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.973$, $T_{max} = 0.982$
10535 measured reflections
5272 independent reflections
3060 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$h = -11 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -20 \rightarrow 19$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.168$
$S = 0.79$
5272 reflections
404 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
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.699P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å$^{-3}$
$\Delta\rho_{min} = -0.18$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.017 (2)

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O4 | 0.14160 (19) | 0.29091 (17) | 0.65390 (9) | 0.0490 (5) |
| O8 | 0.6445 (2) | 0.3074 (2) | 0.65049 (10) | 0.0584 (5) |

| | | | | |
|---|---|---|---|---|
| O2 | 0.6991 (2) | −0.0498 (2) | 0.13171 (10) | 0.0641 (6) |
| O6 | 1.2124 (2) | −0.0612 (2) | 0.13477 (10) | 0.0663 (6) |
| C12 | 0.3000 (2) | 0.2119 (2) | 0.42976 (13) | 0.0377 (6) |
| C15 | 0.1936 (2) | 0.2736 (2) | 0.57966 (13) | 0.0368 (6) |
| O5 | 0.9275 (2) | 0.3222 (2) | 0.00997 (10) | 0.0698 (6) |
| O1 | 0.4129 (2) | 0.3330 (2) | 0.00836 (10) | 0.0674 (6) |
| C33 | 0.6939 (3) | 0.2778 (2) | 0.57720 (13) | 0.0393 (6) |
| C29 | 0.8934 (3) | 0.1922 (2) | 0.35458 (13) | 0.0413 (6) |
| H29 | 0.9886 | 0.1486 | 0.3536 | 0.050* |
| C13 | 0.2388 (2) | 0.3479 (2) | 0.44573 (13) | 0.0400 (6) |
| H13 | 0.2332 | 0.4200 | 0.4055 | 0.048* |
| C11 | 0.3571 (3) | 0.1739 (3) | 0.35295 (14) | 0.0444 (6) |
| H11 | 0.4007 | 0.0807 | 0.3497 | 0.053* |
| C30 | 0.8207 (3) | 0.2192 (2) | 0.43039 (13) | 0.0367 (6) |
| C35 | 0.8980 (3) | 0.1892 (2) | 0.49504 (13) | 0.0404 (6) |
| H35 | 0.9942 | 0.1490 | 0.4892 | 0.048* |
| C31 | 0.6761 (3) | 0.2748 (2) | 0.44293 (13) | 0.0394 (6) |
| H31 | 0.6201 | 0.2922 | 0.4013 | 0.047* |
| C16 | 0.2576 (3) | 0.1382 (2) | 0.56550 (13) | 0.0401 (6) |
| H16 | 0.2659 | 0.0670 | 0.6060 | 0.048* |
| C4 | 0.5278 (3) | 0.1016 (3) | 0.20863 (14) | 0.0463 (6) |
| H4 | 0.5554 | 0.0505 | 0.2539 | 0.056* |
| C19 | 0.9651 (3) | 0.2384 (3) | 0.07433 (14) | 0.0491 (7) |
| C24 | 0.8850 (3) | 0.2751 (3) | 0.14382 (14) | 0.0436 (6) |
| C3 | 0.5943 (3) | 0.0623 (3) | 0.13922 (15) | 0.0471 (6) |
| O7 | 0.7066 (2) | 0.4560 (2) | 0.19247 (11) | 0.0734 (6) |
| C32 | 0.6124 (3) | 0.3051 (2) | 0.51476 (13) | 0.0410 (6) |
| H32 | 0.5156 | 0.3435 | 0.5211 | 0.049* |
| C34 | 0.8365 (3) | 0.2172 (3) | 0.56693 (14) | 0.0443 (6) |
| H34 | 0.8908 | 0.1954 | 0.6090 | 0.053* |
| C23 | 0.9194 (3) | 0.1940 (2) | 0.21264 (13) | 0.0421 (6) |
| C20 | 1.0740 (3) | 0.1273 (3) | 0.07344 (15) | 0.0522 (7) |
| H20 | 1.1262 | 0.1047 | 0.0273 | 0.063* |
| C1 | 0.4531 (3) | 0.2517 (3) | 0.07286 (14) | 0.0473 (6) |
| C5 | 0.4200 (3) | 0.2160 (3) | 0.21234 (13) | 0.0430 (6) |
| C2 | 0.5590 (3) | 0.1378 (3) | 0.07091 (14) | 0.0516 (7) |
| H2 | 0.6062 | 0.1118 | 0.0243 | 0.062* |
| C28 | 0.8397 (3) | 0.2227 (2) | 0.28713 (14) | 0.0444 (6) |
| H28 | 0.7443 | 0.2654 | 0.2869 | 0.053* |
| C21 | 1.1053 (3) | 0.0493 (3) | 0.14175 (15) | 0.0481 (6) |
| C14 | 0.1858 (3) | 0.3803 (2) | 0.51919 (13) | 0.0395 (6) |
| H14 | 0.1453 | 0.4726 | 0.5280 | 0.047* |
| C6 | 0.3793 (3) | 0.2931 (3) | 0.14320 (14) | 0.0441 (6) |
| O3 | 0.1938 (3) | 0.4673 (3) | 0.19099 (12) | 0.0913 (8) |
| C17 | 0.3091 (3) | 0.1078 (2) | 0.49209 (13) | 0.0396 (6) |
| H17 | 0.3509 | 0.0157 | 0.4837 | 0.047* |
| C22 | 1.0291 (3) | 0.0813 (3) | 0.21013 (14) | 0.0475 (6) |
| H22 | 1.0513 | 0.0267 | 0.2554 | 0.057* |

| | | | | |
|---|---|---|---|---|
| C10 | 0.3540 (3) | 0.2567 (3) | 0.28771 (14) | 0.0492 (7) |
| H10 | 0.3060 | 0.3484 | 0.2896 | 0.059* |
| C27 | 0.7704 (3) | 0.3951 (3) | 0.13975 (16) | 0.0545 (7) |
| H27 | 0.7437 | 0.4282 | 0.0909 | 0.065* |
| C18 | 0.0775 (3) | 0.4283 (3) | 0.67268 (16) | 0.0655 (9) |
| H18A | 0.1483 | 0.4957 | 0.6614 | 0.098* |
| H18B | 0.0444 | 0.4241 | 0.7265 | 0.098* |
| H18C | −0.0035 | 0.4581 | 0.6429 | 0.098* |
| C9 | 0.2627 (3) | 0.4111 (3) | 0.13893 (17) | 0.0593 (8) |
| H9 | 0.2396 | 0.4467 | 0.0898 | 0.071* |
| C25 | 1.0115 (4) | 0.2970 (3) | −0.06044 (15) | 0.0743 (9) |
| H25A | 1.1114 | 0.3084 | −0.0557 | 0.111* |
| H25B | 0.9758 | 0.3642 | −0.1008 | 0.111* |
| H25C | 1.0042 | 0.2014 | −0.0725 | 0.111* |
| C8 | 0.7351 (4) | −0.1367 (3) | 0.19987 (16) | 0.0722 (9) |
| H8A | 0.7697 | −0.0791 | 0.2346 | 0.108* |
| H8B | 0.8097 | −0.2118 | 0.1868 | 0.108* |
| H8C | 0.6502 | −0.1779 | 0.2241 | 0.108* |
| C36 | 0.5076 (3) | 0.3858 (3) | 0.66358 (16) | 0.0632 (8) |
| H36A | 0.5030 | 0.4724 | 0.6297 | 0.095* |
| H36B | 0.4933 | 0.4091 | 0.7160 | 0.095* |
| H36C | 0.4329 | 0.3295 | 0.6540 | 0.095* |
| C26 | 1.2461 (4) | −0.1482 (3) | 0.20355 (16) | 0.0727 (9) |
| H26A | 1.2762 | −0.0899 | 0.2394 | 0.109* |
| H26B | 1.3232 | −0.2215 | 0.1914 | 0.109* |
| H26C | 1.1613 | −0.1917 | 0.2260 | 0.109* |
| C7 | 0.4947 (4) | 0.3057 (3) | −0.06241 (15) | 0.0728 (9) |
| H7A | 0.5953 | 0.3159 | −0.0586 | 0.109* |
| H7B | 0.4589 | 0.3728 | −0.1028 | 0.109* |
| H7C | 0.4855 | 0.2101 | −0.0738 | 0.109* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O4 | 0.0641 (12) | 0.0468 (10) | 0.0339 (10) | 0.0028 (9) | −0.0014 (9) | −0.0061 (8) |
| O8 | 0.0587 (12) | 0.0752 (13) | 0.0359 (11) | 0.0159 (10) | −0.0024 (9) | −0.0079 (9) |
| O2 | 0.0753 (14) | 0.0640 (12) | 0.0428 (11) | 0.0229 (11) | 0.0027 (10) | 0.0008 (9) |
| O6 | 0.0766 (14) | 0.0687 (13) | 0.0430 (11) | 0.0281 (11) | 0.0005 (10) | −0.0008 (9) |
| C12 | 0.0364 (13) | 0.0377 (13) | 0.0383 (14) | −0.0038 (10) | −0.0014 (11) | −0.0023 (10) |
| C15 | 0.0364 (13) | 0.0404 (13) | 0.0336 (14) | −0.0026 (11) | −0.0040 (11) | −0.0048 (10) |
| O5 | 0.0817 (14) | 0.0805 (14) | 0.0353 (11) | 0.0236 (11) | 0.0019 (10) | 0.0076 (10) |
| O1 | 0.0782 (14) | 0.0788 (14) | 0.0348 (11) | 0.0208 (11) | 0.0007 (10) | 0.0044 (9) |
| C33 | 0.0438 (15) | 0.0380 (13) | 0.0347 (14) | −0.0009 (11) | −0.0027 (11) | −0.0016 (10) |
| C29 | 0.0428 (14) | 0.0406 (13) | 0.0391 (15) | −0.0001 (11) | −0.0019 (12) | −0.0034 (11) |
| C13 | 0.0452 (15) | 0.0379 (13) | 0.0353 (14) | −0.0010 (11) | −0.0065 (11) | 0.0039 (11) |
| C11 | 0.0488 (15) | 0.0438 (14) | 0.0392 (15) | −0.0006 (12) | −0.0005 (12) | −0.0062 (12) |
| C30 | 0.0395 (14) | 0.0331 (12) | 0.0371 (14) | −0.0032 (10) | −0.0033 (11) | −0.0022 (10) |
| C35 | 0.0334 (13) | 0.0438 (14) | 0.0440 (15) | −0.0008 (11) | −0.0076 (11) | −0.0035 (11) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C31 | 0.0423 (15) | 0.0405 (13) | 0.0350 (14) | −0.0011 (11) | −0.0105 (11) | 0.0018 (10) |
| C16 | 0.0483 (15) | 0.0328 (12) | 0.0385 (14) | −0.0036 (11) | −0.0077 (12) | 0.0028 (10) |
| C4 | 0.0518 (16) | 0.0506 (15) | 0.0336 (14) | 0.0021 (13) | −0.0040 (12) | 0.0007 (11) |
| C19 | 0.0574 (17) | 0.0488 (15) | 0.0383 (15) | 0.0006 (13) | −0.0054 (13) | 0.0030 (12) |
| C24 | 0.0491 (15) | 0.0428 (14) | 0.0368 (15) | 0.0013 (12) | −0.0022 (12) | −0.0033 (11) |
| C3 | 0.0511 (16) | 0.0482 (15) | 0.0399 (15) | 0.0024 (13) | −0.0018 (12) | −0.0050 (12) |
| O7 | 0.0889 (16) | 0.0706 (13) | 0.0507 (13) | 0.0321 (12) | −0.0004 (11) | −0.0093 (10) |
| C32 | 0.0351 (14) | 0.0435 (14) | 0.0421 (15) | 0.0026 (11) | −0.0037 (11) | −0.0012 (11) |
| C34 | 0.0406 (15) | 0.0536 (15) | 0.0386 (15) | 0.0018 (12) | −0.0113 (12) | −0.0037 (12) |
| C23 | 0.0475 (15) | 0.0406 (13) | 0.0377 (14) | −0.0028 (12) | −0.0022 (12) | −0.0052 (11) |
| C20 | 0.0574 (17) | 0.0561 (16) | 0.0388 (16) | 0.0063 (14) | 0.0032 (13) | −0.0062 (13) |
| C1 | 0.0549 (16) | 0.0515 (15) | 0.0335 (14) | −0.0017 (13) | −0.0058 (12) | 0.0026 (12) |
| C5 | 0.0472 (15) | 0.0472 (14) | 0.0351 (14) | −0.0050 (12) | −0.0052 (12) | −0.0047 (11) |
| C2 | 0.0611 (18) | 0.0555 (16) | 0.0344 (15) | 0.0036 (14) | 0.0016 (13) | −0.0035 (12) |
| C28 | 0.0486 (15) | 0.0427 (14) | 0.0403 (15) | 0.0015 (12) | −0.0029 (12) | −0.0050 (11) |
| C21 | 0.0515 (16) | 0.0460 (15) | 0.0441 (16) | 0.0047 (12) | −0.0025 (13) | −0.0051 (12) |
| C14 | 0.0439 (14) | 0.0323 (12) | 0.0412 (15) | 0.0031 (11) | −0.0073 (11) | −0.0031 (11) |
| C6 | 0.0463 (15) | 0.0476 (15) | 0.0369 (15) | −0.0012 (12) | −0.0023 (12) | −0.0034 (11) |
| O3 | 0.1072 (18) | 0.0979 (17) | 0.0522 (14) | 0.0503 (15) | 0.0014 (13) | −0.0063 (12) |
| C17 | 0.0452 (14) | 0.0311 (12) | 0.0414 (15) | −0.0005 (10) | −0.0030 (11) | −0.0036 (10) |
| C22 | 0.0546 (16) | 0.0511 (15) | 0.0346 (15) | 0.0019 (13) | −0.0033 (12) | −0.0035 (12) |
| C10 | 0.0545 (16) | 0.0524 (15) | 0.0390 (15) | 0.0053 (13) | −0.0042 (12) | −0.0078 (12) |
| C27 | 0.0600 (18) | 0.0560 (17) | 0.0437 (16) | 0.0056 (14) | −0.0046 (14) | −0.0004 (13) |
| C18 | 0.088 (2) | 0.0557 (17) | 0.0473 (18) | 0.0156 (16) | 0.0027 (16) | −0.0153 (14) |
| C9 | 0.0634 (19) | 0.0636 (18) | 0.0450 (17) | 0.0100 (15) | 0.0002 (15) | −0.0005 (14) |
| C25 | 0.091 (2) | 0.086 (2) | 0.0361 (17) | 0.0159 (19) | 0.0019 (16) | 0.0065 (15) |
| C8 | 0.087 (2) | 0.0664 (19) | 0.0513 (19) | 0.0222 (17) | −0.0011 (16) | 0.0105 (15) |
| C36 | 0.0549 (18) | 0.076 (2) | 0.0524 (18) | 0.0074 (15) | 0.0112 (14) | −0.0083 (15) |
| C26 | 0.084 (2) | 0.070 (2) | 0.0526 (19) | 0.0292 (17) | −0.0030 (16) | 0.0004 (15) |
| C7 | 0.091 (2) | 0.080 (2) | 0.0374 (17) | 0.0167 (18) | 0.0009 (16) | 0.0060 (15) |

*Geometric parameters (Å, º)*

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|---|---|---|---|
| O4—C15 | 1.361 (3) | C3—C2 | 1.380 (3) |
| O4—C18 | 1.420 (3) | O7—C27 | 1.206 (3) |
| O8—C33 | 1.365 (3) | C32—H32 | 0.9300 |
| O8—C36 | 1.405 (3) | C34—H34 | 0.9300 |
| O2—C3 | 1.360 (3) | C23—C22 | 1.386 (3) |
| O2—C8 | 1.426 (3) | C23—C28 | 1.468 (3) |
| O6—C21 | 1.362 (3) | C20—C21 | 1.380 (3) |
| O6—C26 | 1.430 (3) | C20—H20 | 0.9300 |
| C12—C13 | 1.387 (3) | C1—C2 | 1.372 (3) |
| C12—C17 | 1.396 (3) | C1—C6 | 1.417 (3) |
| C12—C11 | 1.461 (3) | C5—C6 | 1.412 (3) |
| C15—C16 | 1.382 (3) | C5—C10 | 1.468 (3) |
| C15—C14 | 1.388 (3) | C2—H2 | 0.9300 |
| O5—C19 | 1.363 (3) | C28—H28 | 0.9300 |
| O5—C25 | 1.418 (3) | C21—C22 | 1.372 (3) |

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|---|---|---|---|
| O1—C1 | 1.361 (3) | C14—H14 | 0.9300 |
| O1—C7 | 1.418 (3) | C6—C9 | 1.463 (4) |
| C33—C32 | 1.382 (3) | O3—C9 | 1.194 (3) |
| C33—C34 | 1.383 (3) | C17—H17 | 0.9300 |
| C29—C28 | 1.324 (3) | C22—H22 | 0.9300 |
| C29—C30 | 1.461 (3) | C10—H10 | 0.9300 |
| C29—H29 | 0.9300 | C27—H27 | 0.9300 |
| C13—C14 | 1.383 (3) | C18—H18A | 0.9600 |
| C13—H13 | 0.9300 | C18—H18B | 0.9600 |
| C11—C10 | 1.319 (3) | C18—H18C | 0.9600 |
| C11—H11 | 0.9300 | C9—H9 | 0.9300 |
| C30—C31 | 1.389 (3) | C25—H25A | 0.9600 |
| C30—C35 | 1.392 (3) | C25—H25B | 0.9600 |
| C35—C34 | 1.368 (3) | C25—H25C | 0.9600 |
| C35—H35 | 0.9300 | C8—H8A | 0.9600 |
| C31—C32 | 1.379 (3) | C8—H8B | 0.9600 |
| C31—H31 | 0.9300 | C8—H8C | 0.9600 |
| C16—C17 | 1.374 (3) | C36—H36A | 0.9600 |
| C16—H16 | 0.9300 | C36—H36B | 0.9600 |
| C4—C3 | 1.373 (3) | C36—H36C | 0.9600 |
| C4—C5 | 1.387 (3) | C26—H26A | 0.9600 |
| C4—H4 | 0.9300 | C26—H26B | 0.9600 |
| C19—C20 | 1.371 (3) | C26—H26C | 0.9600 |
| C19—C24 | 1.414 (3) | C7—H7A | 0.9600 |
| C24—C23 | 1.407 (3) | C7—H7B | 0.9600 |
| C24—C27 | 1.464 (3) | C7—H7C | 0.9600 |

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|---|---|---|---|
| C15—O4—C18 | 118.68 (19) | C1—C2—C3 | 119.0 (2) |
| C33—O8—C36 | 119.0 (2) | C1—C2—H2 | 120.5 |
| C3—O2—C8 | 117.7 (2) | C3—C2—H2 | 120.5 |
| C21—O6—C26 | 117.2 (2) | C29—C28—C23 | 124.8 (2) |
| C13—C12—C17 | 116.8 (2) | C29—C28—H28 | 117.6 |
| C13—C12—C11 | 123.9 (2) | C23—C28—H28 | 117.6 |
| C17—C12—C11 | 119.2 (2) | O6—C21—C22 | 124.1 (2) |
| O4—C15—C16 | 115.8 (2) | O6—C21—C20 | 114.9 (2) |
| O4—C15—C14 | 124.9 (2) | C22—C21—C20 | 121.0 (2) |
| C16—C15—C14 | 119.3 (2) | C13—C14—C15 | 119.3 (2) |
| C19—O5—C25 | 117.6 (2) | C13—C14—H14 | 120.4 |
| C1—O1—C7 | 117.6 (2) | C15—C14—H14 | 120.4 |
| O8—C33—C32 | 124.7 (2) | C5—C6—C1 | 118.2 (2) |
| O8—C33—C34 | 115.8 (2) | C5—C6—C9 | 124.4 (2) |
| C32—C33—C34 | 119.5 (2) | C1—C6—C9 | 117.4 (2) |
| C28—C29—C30 | 127.5 (2) | C16—C17—C12 | 121.5 (2) |
| C28—C29—H29 | 116.2 | C16—C17—H17 | 119.2 |
| C30—C29—H29 | 116.2 | C12—C17—H17 | 119.2 |
| C14—C13—C12 | 122.5 (2) | C21—C22—C23 | 120.9 (2) |
| C14—C13—H13 | 118.8 | C21—C22—H22 | 119.6 |
| C12—C13—H13 | 118.8 | C23—C22—H22 | 119.6 |

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| C10—C11—C12 | 127.4 (2) | C11—C10—C5 | 125.5 (2) |
| C10—C11—H11 | 116.3 | C11—C10—H10 | 117.3 |
| C12—C11—H11 | 116.3 | C5—C10—H10 | 117.3 |
| C31—C30—C35 | 116.5 (2) | O7—C27—C24 | 127.4 (3) |
| C31—C30—C29 | 123.9 (2) | O7—C27—H27 | 116.3 |
| C35—C30—C29 | 119.6 (2) | C24—C27—H27 | 116.3 |
| C34—C35—C30 | 121.9 (2) | O4—C18—H18A | 109.5 |
| C34—C35—H35 | 119.0 | O4—C18—H18B | 109.5 |
| C30—C35—H35 | 119.0 | H18A—C18—H18B | 109.5 |
| C32—C31—C30 | 122.4 (2) | O4—C18—H18C | 109.5 |
| C32—C31—H31 | 118.8 | H18A—C18—H18C | 109.5 |
| C30—C31—H31 | 118.8 | H18B—C18—H18C | 109.5 |
| C17—C16—C15 | 120.6 (2) | O3—C9—C6 | 127.8 (3) |
| C17—C16—H16 | 119.7 | O3—C9—H9 | 116.1 |
| C15—C16—H16 | 119.7 | C6—C9—H9 | 116.1 |
| C3—C4—C5 | 121.1 (2) | O5—C25—H25A | 109.5 |
| C3—C4—H4 | 119.4 | O5—C25—H25B | 109.5 |
| C5—C4—H4 | 119.4 | H25A—C25—H25B | 109.5 |
| O5—C19—C20 | 123.4 (2) | O5—C25—H25C | 109.5 |
| O5—C19—C24 | 115.3 (2) | H25A—C25—H25C | 109.5 |
| C20—C19—C24 | 121.3 (2) | H25B—C25—H25C | 109.5 |
| C23—C24—C19 | 118.3 (2) | O2—C8—H8A | 109.5 |
| C23—C24—C27 | 123.8 (2) | O2—C8—H8B | 109.5 |
| C19—C24—C27 | 117.9 (2) | H8A—C8—H8B | 109.5 |
| O2—C3—C4 | 123.9 (2) | O2—C8—H8C | 109.5 |
| O2—C3—C2 | 114.9 (2) | H8A—C8—H8C | 109.5 |
| C4—C3—C2 | 121.1 (2) | H8B—C8—H8C | 109.5 |
| C31—C32—C33 | 119.3 (2) | O8—C36—H36A | 109.5 |
| C31—C32—H32 | 120.3 | O8—C36—H36B | 109.5 |
| C33—C32—H32 | 120.3 | H36A—C36—H36B | 109.5 |
| C35—C34—C33 | 120.2 (2) | O8—C36—H36C | 109.5 |
| C35—C34—H34 | 119.9 | H36A—C36—H36C | 109.5 |
| C33—C34—H34 | 119.9 | H36B—C36—H36C | 109.5 |
| C22—C23—C24 | 119.3 (2) | O6—C26—H26A | 109.5 |
| C22—C23—C28 | 118.8 (2) | O6—C26—H26B | 109.5 |
| C24—C23—C28 | 121.9 (2) | H26A—C26—H26B | 109.5 |
| C19—C20—C21 | 119.2 (2) | O6—C26—H26C | 109.5 |
| C19—C20—H20 | 120.4 | H26A—C26—H26C | 109.5 |
| C21—C20—H20 | 120.4 | H26B—C26—H26C | 109.5 |
| O1—C1—C2 | 122.8 (2) | O1—C7—H7A | 109.5 |
| O1—C1—C6 | 115.6 (2) | O1—C7—H7B | 109.5 |
| C2—C1—C6 | 121.5 (2) | H7A—C7—H7B | 109.5 |
| C4—C5—C6 | 119.0 (2) | O1—C7—H7C | 109.5 |
| C4—C5—C10 | 119.5 (2) | H7A—C7—H7C | 109.5 |
| C6—C5—C10 | 121.6 (2) | H7B—C7—H7C | 109.5 |
| | | | |
| C18—O4—C15—C16 | 178.6 (2) | C7—O1—C1—C6 | 173.1 (2) |
| C18—O4—C15—C14 | −1.7 (3) | C3—C4—C5—C6 | −0.2 (4) |

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|---|---|---|---|
| C36—O8—C33—C32 | −7.7 (4) | C3—C4—C5—C10 | −178.5 (2) |
| C36—O8—C33—C34 | 171.8 (2) | O1—C1—C2—C3 | 179.1 (2) |
| C17—C12—C13—C14 | −1.4 (3) | C6—C1—C2—C3 | −0.2 (4) |
| C11—C12—C13—C14 | −179.9 (2) | O2—C3—C2—C1 | 179.2 (2) |
| C13—C12—C11—C10 | −3.6 (4) | C4—C3—C2—C1 | −1.5 (4) |
| C17—C12—C11—C10 | 177.9 (2) | C30—C29—C28—C23 | 179.3 (2) |
| C28—C29—C30—C31 | 4.4 (4) | C22—C23—C28—C29 | 27.9 (4) |
| C28—C29—C30—C35 | −175.4 (2) | C24—C23—C28—C29 | −153.8 (2) |
| C31—C30—C35—C34 | −2.1 (3) | C26—O6—C21—C22 | 0.1 (4) |
| C29—C30—C35—C34 | 177.7 (2) | C26—O6—C21—C20 | −178.1 (3) |
| C35—C30—C31—C32 | 2.7 (3) | C19—C20—C21—O6 | 178.3 (2) |
| C29—C30—C31—C32 | −177.0 (2) | C19—C20—C21—C22 | 0.1 (4) |
| O4—C15—C16—C17 | 177.7 (2) | C12—C13—C14—C15 | 0.0 (4) |
| C14—C15—C16—C17 | −2.0 (3) | O4—C15—C14—C13 | −178.0 (2) |
| C25—O5—C19—C20 | −3.6 (4) | C16—C15—C14—C13 | 1.7 (3) |
| C25—O5—C19—C24 | 175.3 (2) | C4—C5—C6—C1 | −1.4 (4) |
| O5—C19—C24—C23 | −179.3 (2) | C10—C5—C6—C1 | 176.9 (2) |
| C20—C19—C24—C23 | −0.3 (4) | C4—C5—C6—C9 | 176.9 (2) |
| O5—C19—C24—C27 | 1.1 (4) | C10—C5—C6—C9 | −4.8 (4) |
| C20—C19—C24—C27 | −179.9 (2) | O1—C1—C6—C5 | −177.7 (2) |
| C8—O2—C3—C4 | 4.0 (4) | C2—C1—C6—C5 | 1.6 (4) |
| C8—O2—C3—C2 | −176.8 (3) | O1—C1—C6—C9 | 3.8 (4) |
| C5—C4—C3—O2 | −179.1 (2) | C2—C1—C6—C9 | −176.8 (2) |
| C5—C4—C3—C2 | 1.7 (4) | C15—C16—C17—C12 | 0.6 (4) |
| C30—C31—C32—C33 | −0.9 (3) | C13—C12—C17—C16 | 1.0 (3) |
| O8—C33—C32—C31 | 177.8 (2) | C11—C12—C17—C16 | 179.7 (2) |
| C34—C33—C32—C31 | −1.7 (3) | O6—C21—C22—C23 | −178.8 (2) |
| C30—C35—C34—C33 | −0.4 (4) | C20—C21—C22—C23 | −0.7 (4) |
| O8—C33—C34—C35 | −177.2 (2) | C24—C23—C22—C21 | 0.8 (4) |
| C32—C33—C34—C35 | 2.3 (4) | C28—C23—C22—C21 | 179.1 (2) |
| C19—C24—C23—C22 | −0.3 (4) | C12—C11—C10—C5 | 176.5 (2) |
| C27—C24—C23—C22 | 179.3 (2) | C4—C5—C10—C11 | −19.5 (4) |
| C19—C24—C23—C28 | −178.6 (2) | C6—C5—C10—C11 | 162.3 (3) |
| C27—C24—C23—C28 | 1.0 (4) | C23—C24—C27—O7 | 12.8 (5) |
| O5—C19—C20—C21 | 179.3 (3) | C19—C24—C27—O7 | −167.6 (3) |
| C24—C19—C20—C21 | 0.4 (4) | C5—C6—C9—O3 | 6.5 (5) |
| C7—O1—C1—C2 | −6.2 (4) | C1—C6—C9—O3 | −175.2 (3) |

*Hydrogen-bond geometry (Å, º)*

Cg2 and Cg4 are the centroids of the C12–C17 and C30–C35 rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C9—H9···O1 | 0.93 | 2.29 | 2.681 (4) | 105 |
| C10—H10···O3 | 0.93 | 2.25 | 2.863 (4) | 123 |
| C27—H27···O5 | 0.93 | 2.31 | 2.681 (4) | 103 |
| C28—H28···O7 | 0.93 | 2.34 | 2.861 (3) | 115 |
| C14—H14···*Cg*4$^{i}$ | 0.93 | 2.88 | 3.597 (3) | 135 |
| C17—H14···*Cg*4$^{ii}$ | 0.93 | 2.76 | 3.471 (3) | 134 |

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|---|---|---|---|---|
| C32—H14···*Cg*2 | 0.93 | 2.82 | 3.530 (3) | 135 |
| C15—H14···*Cg*2$^{iii}$ | 0.93 | 2.66 | 3.371 (3) | 134 |

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