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# (2*E*,6*E*)-2,6-Bis(2,4,5-trimethoxybenzylidene)cyclohexanone

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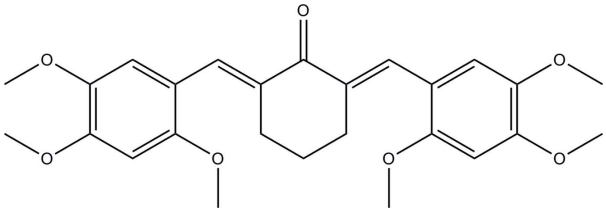
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.003 Å; disorder in main residue; $R$ factor = 0.070; $wR$ factor = 0.155; data-to-parameter ratio = 21.1.

In the title compound, $C_{26}H_{30}O_7$, one atom in the cyclohexanone ring is disordered over two positions with a site-occupancy ratio of 0.871 (6):0.129 (6). The dihedral angles formed between the mean plane through the six C atoms of the major component of the cyclohexanone ring and two benzene rings are 35.09 (10) and 34.21 (10)°; the corresponding angles for the minor component are 20.1 (2) and 19.5 (2)°. Both the major and minor disordered components of the cyclohexanone ring adopt half-boat conformations. In the crystal packing, intermolecular C—H···O hydrogen bonds connect the molecules into a three-dimensional network.

## Related literature

For natural biocides, see: Geiger & Conn (1945); Marian *et al.* (1947). For the biological activity and biological properties of chalcones, see: Srivastava *et al.* (1997); Kuhn & Hensel (1953); Hosni & Saad (1995); Ishida *et al.* (1960); Mehata & Parikh (1978); Mudaliar & Joshi (1995). For ring conformations, see: Cremer & Pople (1975). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For bond-length data, see: Allen *et al.* (1987).



‡ Thomson Reuters ResearcherID: A-3561-2009.

## Experimental

### Crystal data

$C_{26}H_{30}O_7$
$M_r$ = 454.50
Monoclinic, $P2_1/n$
$a$ = 9.0943 (1) Å
$b$ = 13.4947 (1) Å
$c$ = 18.8293 (2) Å
$\beta$ = 100.449 (1)°
$V$ = 2272.50 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 296 K
0.37 × 0.21 × 0.18 mm

### Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.965, $T_{max}$ = 0.983
29097 measured reflections
6691 independent reflections
4027 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.036

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.155
$S$ = 1.06
6691 reflections
317 parameters
4 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.24 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C12$A$—H12$B$···O1$^{i}$ | 0.97 | 2.43 | 3.364 (3) | 160 |
| C25—H25$B$···O1$^{ii}$ | 0.96 | 2.55 | 3.450 (3) | 156 |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; 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* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2721).

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# supplementary materials

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# (2*E*,6*E*)-2,6-Bis(2,4,5-trimethoxybenzylidene)cyclohexanone

**T. Shahani, H.-K. Fun, G. L. Balaji, V. Vijayakumar and S. Sarveswari**

## Comment

The chemistry of chalcones has generated intensive scientific interest due to their biological and industrial applications. Chalcones are natural biocides (Geiger & Conn, 1945; Marian *et al.*, 1947) and are well known intermediates in the synthesis of heterocyclic compounds exhibiting various biological activities (Srivastava *et al.*, 1997; Kuhn & Hensel, 1953). Chalcones and their derivatives possess some interesting biological properties such as antibacterial (Ishida *et al.*, 1960), antifungal (Mehata *et al.*, 1978), insecticidal (Mudaliar & Joshi, 1995), anesthetic (Hosni & Saad, 1995), anti inflammatory, analgesic and ulcerogenic activities.

In the title compound (Fig. 1), the C12 atom is disordered over two positions with a site-occupancy ratio of 0.871 (6):0.129 (6). The dihedral angles formed between major component (C8–C11/C12A/C13) and two benzene rings (C1—C6 and C15—C20) are 35.09 (10) and 34.21 (10)°, and between the minor component (C8–C11/C12B/C13) and two benzene rings are 20.1 (2) and 19.5 (2)°. The major and minor disordered components adopt half-boat conformations (Cremer & Pople, 1975) with puckering of Q = 0.487 (2) Å, Θ = 128.3 (2) ° & φ = 57.8 (3)° and Q = 0.387 (9) Å, Θ = 58.5 (9)° & φ = 242.6 (9)° respectively. In the crystal packing (Fig. 2), intermolecular C12A —H12B···O1 & C25— H25B···O1 hydrogen bonds link the molecules into a 3D network.

## Experimental

A mixture of cyclohexanone (0.5 mmol), 2,4,5-trimethoxybenzaldehyde (4 g) and dry ammonium acetate (0.78 g) was taken in 1:4:2 molar ratio in methanol and heated on water bath till the colour changes to reddish orange, then diethyl ether (50 mmol) was added. The mixture was allowed to stand for 24 h resulting in the formation of needle-shaped crystals. Yield: 60%. *Mp*: 170 °C.

## Refinement

The H atoms bound to C11 and C13 were located in a difference map and then constrained to ride with the parent atoms with $U_{iso}$(H) = 1.2$U_{iso}$(C). The other H atoms were positioned geometrically. [C—H = 0.9300 to 0.9700 Å]. The C11–C12A, C11–C12B, C13–C12A & C13–C12B distances were restrained with to be equal. The distance between H1A and H13D, H20A and H11C were restrainted to be 2.01 Å.

## Figures

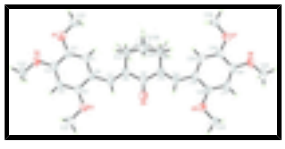

Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids for the non-hydrogen atoms. Open bonds indicate the minor disordered component.

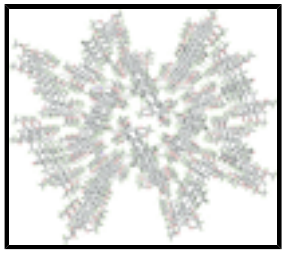

Fig. 2. The crystal packing of the title compound, viewed along the *a* axis, showing the three-dimensional network. Only the major disordered component is shown.

**(2*E*,6*E*)-2,6-Bis(2,4,5-trimethoxybenzylidene)cyclohexanone**

*Crystal data*

| | |
|---|---|
| $C_{26}H_{30}O_7$ | $F(000) = 968$ |
| $M_r = 454.50$ | $D_x = 1.328$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2yn | Cell parameters from 7467 reflections |
| $a = 9.0943$ (1) Å | $\theta = 2.7$–$30.2°$ |
| $b = 13.4947$ (1) Å | $\mu = 0.10$ mm$^{-1}$ |
| $c = 18.8293$ (2) Å | $T = 296$ K |
| $\beta = 100.449$ (1)° | Block, yellow |
| $V = 2272.50$ (4) Å$^3$ | 0.37 × 0.21 × 0.18 mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEXII CCD area-detector diffractometer | 6691 independent reflections |
| Radiation source: fine-focus sealed tube | 4027 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.036$ |
| φ and ω scans | $\theta_{max} = 30.2°$, $\theta_{min} = 1.9°$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2009) | $h = -12 \rightarrow 12$ |
| $T_{min} = 0.965$, $T_{max} = 0.983$ | $k = -16 \rightarrow 19$ |
| 29097 measured reflections | $l = -26 \rightarrow 26$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.070$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.155$ | H-atom parameters constrained |
| $S = 1.06$ | $w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 1.1706P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 6691 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 317 parameters | $\Delta\rho_{max} = 0.24$ e Å$^{-3}$ |

4 restraints $\Delta\rho_{min} = -0.24$ e Å$^{-3}$

*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**. 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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| O1 | 0.26290 (19) | 0.81506 (13) | −0.25518 (8) | 0.0701 (5) | |
| O2 | 0.46504 (19) | 0.94889 (13) | −0.21805 (8) | 0.0677 (5) | |
| O3 | 0.57350 (17) | 0.81575 (11) | 0.02537 (8) | 0.0560 (4) | |
| O4 | 0.2953 (2) | 0.57310 (12) | 0.10823 (8) | 0.0769 (6) | |
| O5 | 0.29543 (19) | 0.29369 (11) | 0.23732 (8) | 0.0618 (4) | |
| O6 | −0.0235 (2) | 0.00816 (12) | 0.17156 (9) | 0.0732 (5) | |
| O7 | −0.11209 (18) | 0.08047 (12) | 0.04346 (8) | 0.0627 (4) | |
| C1 | 0.3099 (2) | 0.74316 (15) | −0.13474 (11) | 0.0458 (5) | |
| H1A | 0.2355 | 0.6960 | −0.1483 | 0.055* | |
| C2 | 0.3373 (2) | 0.81086 (16) | −0.18504 (11) | 0.0481 (5) | |
| C3 | 0.4469 (2) | 0.88311 (16) | −0.16507 (11) | 0.0483 (5) | |
| C4 | 0.5267 (2) | 0.88492 (15) | −0.09551 (11) | 0.0456 (5) | |
| H4A | 0.6001 | 0.9328 | −0.0823 | 0.055* | |
| C5 | 0.4985 (2) | 0.81575 (14) | −0.04479 (10) | 0.0418 (4) | |
| C6 | 0.3896 (2) | 0.74210 (14) | −0.06348 (10) | 0.0405 (4) | |
| C7 | 0.3571 (2) | 0.67221 (14) | −0.00909 (10) | 0.0414 (4) | |
| H7A | 0.3853 | 0.6931 | 0.0385 | 0.050* | |
| C8 | 0.2925 (2) | 0.58240 (14) | −0.01745 (10) | 0.0413 (4) | |
| C9 | 0.2669 (2) | 0.53078 (14) | 0.04976 (10) | 0.0441 (5) | |
| C10 | 0.2049 (2) | 0.42752 (14) | 0.04458 (10) | 0.0390 (4) | |
| C11 | 0.1613 (3) | 0.38201 (15) | −0.02935 (10) | 0.0502 (5) | |
| H11A | 0.1699 | 0.3105 | −0.0253 | 0.060* | 0.871 (6) |
| H11B | 0.0577 | 0.3979 | −0.0485 | 0.060* | 0.871 (6) |
| H11C | 0.0364 | 0.3580 | −0.0455 | 0.060* | 0.129 (6) |
| H11D | 0.2383 | 0.3151 | −0.0082 | 0.060* | 0.129 (6) |
| C12A | 0.2580 (3) | 0.41870 (14) | −0.08081 (12) | 0.0498 (8) | 0.871 (6) |
| H12A | 0.3610 | 0.3997 | −0.0632 | 0.060* | 0.871 (6) |
| H12B | 0.2261 | 0.3881 | −0.1277 | 0.060* | 0.871 (6) |
| C12B | 0.1315 (14) | 0.4496 (10) | −0.0939 (6) | 0.082 (8) | 0.129 (6) |
| H12C | 0.1311 | 0.4113 | −0.1375 | 0.098* | 0.129 (6) |
| H12D | 0.0337 | 0.4797 | −0.0970 | 0.098* | 0.129 (6) |

| | | | | | |
|---|---|---|---|---|---|
| C13 | 0.2482 (3) | 0.52947 (15) | −0.08856 (11) | 0.0575 (6) | |
| H13A | 0.1466 | 0.5477 | −0.1098 | 0.069* | 0.871 (6) |
| H13B | 0.3130 | 0.5510 | −0.1212 | 0.069* | 0.871 (6) |
| H13C | 0.3357 | 0.4916 | −0.0669 | 0.069* | 0.129 (6) |
| H13D | 0.2519 | 0.5469 | −0.1344 | 0.069* | 0.129 (6) |
| C14 | 0.1980 (2) | 0.38183 (14) | 0.10713 (11) | 0.0437 (5) | |
| H14A | 0.2338 | 0.4193 | 0.1481 | 0.052* | |
| C15 | 0.1438 (2) | 0.28305 (14) | 0.12215 (10) | 0.0395 (4) | |
| C16 | 0.1928 (2) | 0.24048 (14) | 0.19020 (10) | 0.0423 (4) | |
| C17 | 0.1381 (2) | 0.14907 (15) | 0.20811 (11) | 0.0469 (5) | |
| H17A | 0.1707 | 0.1224 | 0.2538 | 0.056* | |
| C18 | 0.0359 (2) | 0.09808 (14) | 0.15832 (11) | 0.0464 (5) | |
| C19 | −0.0115 (2) | 0.13695 (15) | 0.08948 (11) | 0.0439 (5) | |
| C20 | 0.0398 (2) | 0.22886 (14) | 0.07311 (11) | 0.0433 (5) | |
| H20A | 0.0041 | 0.2559 | 0.0278 | 0.052* | |
| C21 | 0.1569 (3) | 0.74047 (18) | −0.27866 (12) | 0.0603 (6) | |
| H21A | 0.1141 | 0.7511 | −0.3285 | 0.090* | |
| H21B | 0.0794 | 0.7426 | −0.2502 | 0.090* | |
| H21C | 0.2049 | 0.6769 | −0.2732 | 0.090* | |
| C22 | 0.5693 (3) | 1.02662 (17) | −0.20014 (14) | 0.0640 (7) | |
| H22A | 0.5703 | 1.0672 | −0.2419 | 0.096* | |
| H22B | 0.6671 | 0.9993 | −0.1840 | 0.096* | |
| H22C | 0.5414 | 1.0662 | −0.1623 | 0.096* | |
| C23 | 0.6873 (3) | 0.88797 (17) | 0.04552 (12) | 0.0570 (6) | |
| H23A | 0.7343 | 0.8776 | 0.0949 | 0.086* | |
| H23B | 0.6439 | 0.9530 | 0.0404 | 0.086* | |
| H23C | 0.7606 | 0.8820 | 0.0149 | 0.086* | |
| C24 | 0.3591 (3) | 0.2507 (2) | 0.30446 (12) | 0.0772 (8) | |
| H24A | 0.4324 | 0.2950 | 0.3304 | 0.116* | |
| H24B | 0.4060 | 0.1891 | 0.2963 | 0.116* | |
| H24C | 0.2821 | 0.2389 | 0.3322 | 0.116* | |
| C25 | −0.0341 (3) | −0.01589 (18) | 0.24289 (13) | 0.0663 (7) | |
| H25A | −0.0779 | −0.0805 | 0.2441 | 0.099* | |
| H25B | −0.0955 | 0.0322 | 0.2612 | 0.099* | |
| H25C | 0.0640 | −0.0159 | 0.2722 | 0.099* | |
| C26 | −0.1422 (3) | 0.10874 (19) | −0.02995 (12) | 0.0634 (7) | |
| H26A | −0.2049 | 0.0598 | −0.0575 | 0.095* | |
| H26B | −0.0500 | 0.1139 | −0.0477 | 0.095* | |
| H26C | −0.1923 | 0.1717 | −0.0346 | 0.095* | |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0786 (11) | 0.0790 (12) | 0.0460 (9) | −0.0247 (9) | −0.0067 (8) | 0.0191 (8) |
| O2 | 0.0777 (11) | 0.0669 (11) | 0.0557 (10) | −0.0219 (9) | 0.0045 (8) | 0.0270 (8) |
| O3 | 0.0667 (10) | 0.0512 (9) | 0.0453 (8) | −0.0205 (7) | −0.0026 (7) | 0.0099 (7) |
| O4 | 0.1479 (17) | 0.0475 (9) | 0.0382 (9) | −0.0341 (10) | 0.0246 (9) | −0.0076 (7) |
| O5 | 0.0905 (12) | 0.0490 (9) | 0.0402 (8) | −0.0184 (8) | −0.0030 (8) | 0.0028 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O6 | 0.1228 (15) | 0.0437 (9) | 0.0548 (10) | −0.0316 (9) | 0.0207 (10) | 0.0047 (7) |
| O7 | 0.0733 (10) | 0.0562 (10) | 0.0541 (9) | −0.0266 (8) | −0.0009 (8) | 0.0056 (7) |
| C1 | 0.0488 (11) | 0.0429 (11) | 0.0450 (11) | −0.0095 (9) | 0.0067 (9) | 0.0055 (9) |
| C2 | 0.0509 (12) | 0.0528 (13) | 0.0392 (11) | −0.0045 (10) | 0.0044 (9) | 0.0088 (9) |
| C3 | 0.0524 (12) | 0.0465 (12) | 0.0470 (12) | −0.0039 (10) | 0.0117 (9) | 0.0142 (9) |
| C4 | 0.0463 (11) | 0.0394 (11) | 0.0506 (12) | −0.0083 (9) | 0.0077 (9) | 0.0085 (9) |
| C5 | 0.0467 (11) | 0.0365 (10) | 0.0415 (11) | −0.0021 (8) | 0.0064 (8) | 0.0047 (8) |
| C6 | 0.0470 (10) | 0.0355 (10) | 0.0398 (10) | −0.0018 (8) | 0.0098 (8) | 0.0056 (8) |
| C7 | 0.0506 (11) | 0.0367 (10) | 0.0367 (10) | −0.0028 (9) | 0.0071 (8) | 0.0029 (8) |
| C8 | 0.0546 (11) | 0.0352 (10) | 0.0358 (10) | −0.0046 (9) | 0.0129 (8) | 0.0015 (8) |
| C9 | 0.0636 (13) | 0.0346 (10) | 0.0358 (10) | −0.0067 (9) | 0.0133 (9) | 0.0000 (8) |
| C10 | 0.0484 (11) | 0.0318 (10) | 0.0381 (10) | −0.0015 (8) | 0.0111 (8) | 0.0002 (8) |
| C11 | 0.0741 (14) | 0.0354 (11) | 0.0415 (11) | −0.0105 (10) | 0.0110 (10) | −0.0025 (9) |
| C12A | 0.0751 (19) | 0.0390 (14) | 0.0384 (13) | −0.0028 (12) | 0.0182 (12) | −0.0078 (10) |
| C12B | 0.11 (2) | 0.081 (16) | 0.062 (13) | 0.035 (14) | 0.026 (12) | 0.000 (11) |
| C13 | 0.0890 (17) | 0.0492 (13) | 0.0369 (11) | −0.0140 (12) | 0.0186 (11) | −0.0012 (9) |
| C14 | 0.0568 (12) | 0.0356 (10) | 0.0392 (10) | −0.0073 (9) | 0.0098 (9) | −0.0004 (8) |
| C15 | 0.0485 (11) | 0.0328 (10) | 0.0391 (10) | −0.0014 (8) | 0.0133 (8) | 0.0027 (8) |
| C16 | 0.0551 (12) | 0.0354 (10) | 0.0380 (10) | −0.0029 (9) | 0.0129 (8) | −0.0020 (8) |
| C17 | 0.0694 (14) | 0.0356 (11) | 0.0367 (10) | 0.0006 (10) | 0.0126 (9) | 0.0051 (8) |
| C18 | 0.0645 (13) | 0.0316 (10) | 0.0461 (11) | −0.0049 (9) | 0.0179 (10) | 0.0032 (8) |
| C19 | 0.0488 (11) | 0.0376 (11) | 0.0456 (11) | −0.0069 (9) | 0.0093 (9) | 0.0002 (9) |
| C20 | 0.0484 (11) | 0.0404 (11) | 0.0410 (11) | −0.0026 (9) | 0.0074 (8) | 0.0075 (8) |
| C21 | 0.0647 (14) | 0.0613 (15) | 0.0513 (13) | −0.0012 (12) | 0.0007 (11) | −0.0027 (11) |
| C22 | 0.0683 (15) | 0.0491 (14) | 0.0767 (17) | −0.0051 (12) | 0.0186 (13) | 0.0241 (12) |
| C23 | 0.0634 (14) | 0.0538 (13) | 0.0512 (13) | −0.0166 (11) | 0.0030 (10) | −0.0013 (10) |
| C24 | 0.113 (2) | 0.0700 (17) | 0.0398 (13) | −0.0172 (16) | −0.0096 (13) | 0.0031 (12) |
| C25 | 0.0842 (17) | 0.0513 (14) | 0.0646 (15) | −0.0113 (13) | 0.0168 (13) | 0.0208 (12) |
| C26 | 0.0717 (16) | 0.0591 (15) | 0.0535 (14) | −0.0157 (12) | −0.0048 (11) | 0.0007 (11) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C2 | 1.372 (2) | C12A—H12B | 0.9700 |
| O1—C21 | 1.408 (3) | C12A—H13C | 1.2119 |
| O2—C3 | 1.367 (2) | C12B—C13 | 1.504 (2) |
| O2—C22 | 1.413 (3) | C12B—H12C | 0.9700 |
| O3—C5 | 1.372 (2) | C12B—H12D | 0.9700 |
| O3—C23 | 1.422 (2) | C13—H13A | 0.9700 |
| O4—C9 | 1.225 (2) | C13—H13B | 0.9700 |
| O5—C16 | 1.369 (2) | C13—H13C | 0.9715 |
| O5—C24 | 1.416 (3) | C13—H13D | 0.9016 |
| O6—C18 | 1.369 (2) | C14—C15 | 1.467 (3) |
| O6—C25 | 1.402 (3) | C14—H14A | 0.9300 |
| O7—C19 | 1.372 (2) | C15—C16 | 1.401 (3) |
| O7—C26 | 1.412 (3) | C15—C20 | 1.402 (3) |
| C1—C2 | 1.371 (3) | C16—C17 | 1.394 (3) |
| C1—C6 | 1.405 (3) | C17—C18 | 1.379 (3) |
| C1—H1A | 0.9300 | C17—H17A | 0.9300 |
| C2—C3 | 1.396 (3) | C18—C19 | 1.392 (3) |

| | | | |
|---|---|---|---|
| C3—C4 | 1.378 (3) | C19—C20 | 1.380 (3) |
| C4—C5 | 1.392 (3) | C20—H20A | 0.9300 |
| C4—H4A | 0.9300 | C21—H21A | 0.9600 |
| C5—C6 | 1.402 (3) | C21—H21B | 0.9600 |
| C6—C7 | 1.462 (3) | C21—H21C | 0.9600 |
| C7—C8 | 1.344 (3) | C22—H22A | 0.9600 |
| C7—H7A | 0.9300 | C22—H22B | 0.9600 |
| C8—C9 | 1.499 (3) | C22—H22C | 0.9600 |
| C8—C13 | 1.506 (3) | C23—H23A | 0.9600 |
| C9—C10 | 1.500 (3) | C23—H23B | 0.9600 |
| C10—C14 | 1.341 (3) | C23—H23C | 0.9600 |
| C10—C11 | 1.507 (3) | C24—H24A | 0.9600 |
| C11—C12B | 1.504 (2) | C24—H24B | 0.9600 |
| C11—C12A | 1.505 (2) | C24—H24C | 0.9600 |
| C11—H11A | 0.9700 | C25—H25A | 0.9600 |
| C11—H11B | 0.9700 | C25—H25B | 0.9600 |
| C11—H11C | 1.1673 | C25—H25C | 0.9600 |
| C11—H11D | 1.1672 | C26—H26A | 0.9600 |
| C12A—C13 | 1.503 (2) | C26—H26B | 0.9600 |
| C12A—H12A | 0.9700 | C26—H26C | 0.9600 |
| C2—O1—C21 | 117.58 (17) | C12B—C13—H13A | 62.9 |
| C3—O2—C22 | 118.17 (17) | C8—C13—H13A | 109.1 |
| C5—O3—C23 | 117.86 (16) | C12A—C13—H13B | 109.1 |
| C16—O5—C24 | 118.96 (17) | C12B—C13—H13B | 132.5 |
| C18—O6—C25 | 118.51 (18) | C8—C13—H13B | 109.1 |
| C19—O7—C26 | 117.20 (16) | H13A—C13—H13B | 107.8 |
| C2—C1—C6 | 122.64 (19) | C12A—C13—H13C | 53.6 |
| C2—C1—H1A | 118.7 | C12B—C13—H13C | 99.5 |
| C6—C1—H1A | 118.7 | C8—C13—H13C | 79.0 |
| C1—C2—O1 | 124.94 (19) | H13A—C13—H13C | 162.5 |
| C1—C2—C3 | 119.33 (18) | H13B—C13—H13C | 83.0 |
| O1—C2—C3 | 115.72 (18) | C12A—C13—H13D | 109.9 |
| O2—C3—C4 | 124.73 (19) | C12B—C13—H13D | 105.8 |
| O2—C3—C2 | 115.56 (18) | C8—C13—H13D | 132.7 |
| C4—C3—C2 | 119.71 (18) | H13A—C13—H13D | 74.8 |
| C3—C4—C5 | 120.64 (19) | H13C—C13—H13D | 111.7 |
| C3—C4—H4A | 119.7 | C10—C14—C15 | 131.17 (18) |
| C5—C4—H4A | 119.7 | C10—C14—H14A | 114.4 |
| O3—C5—C4 | 122.63 (17) | C15—C14—H14A | 114.4 |
| O3—C5—C6 | 116.53 (16) | C16—C15—C20 | 116.95 (17) |
| C4—C5—C6 | 120.84 (18) | C16—C15—C14 | 119.13 (17) |
| C5—C6—C1 | 116.83 (17) | C20—C15—C14 | 123.87 (17) |
| C5—C6—C7 | 120.34 (17) | O5—C16—C17 | 122.59 (18) |
| C1—C6—C7 | 122.73 (17) | O5—C16—C15 | 116.35 (17) |
| C8—C7—C6 | 129.79 (18) | C17—C16—C15 | 121.05 (18) |
| C8—C7—H7A | 115.1 | C18—C17—C16 | 120.22 (18) |
| C6—C7—H7A | 115.1 | C18—C17—H17A | 119.9 |
| C7—C8—C9 | 116.86 (17) | C16—C17—H17A | 119.9 |
| C7—C8—C13 | 124.92 (17) | O6—C18—C17 | 123.93 (19) |

| | | | |
|---|---|---|---|
| C9—C8—C13 | 118.21 (16) | O6—C18—C19 | 115.96 (18) |
| O4—C9—C8 | 120.15 (17) | C17—C18—C19 | 120.11 (18) |
| O4—C9—C10 | 120.45 (17) | O7—C19—C20 | 124.91 (18) |
| C8—C9—C10 | 119.39 (16) | O7—C19—C18 | 115.92 (17) |
| C14—C10—C9 | 116.54 (17) | C20—C19—C18 | 119.12 (18) |
| C14—C10—C11 | 125.28 (17) | C19—C20—C15 | 122.47 (18) |
| C9—C10—C11 | 118.13 (16) | C19—C20—H20A | 118.8 |
| C12B—C11—C12A | 47.3 (6) | C15—C20—H20A | 118.8 |
| C12B—C11—C10 | 118.5 (6) | O1—C21—H21A | 109.5 |
| C12A—C11—C10 | 111.96 (17) | O1—C21—H21B | 109.5 |
| C12B—C11—H11A | 131.8 | H21A—C21—H21B | 109.5 |
| C12A—C11—H11A | 109.2 | O1—C21—H21C | 109.5 |
| C10—C11—H11A | 109.2 | H21A—C21—H21C | 109.5 |
| C12B—C11—H11B | 62.9 | H21B—C21—H21C | 109.5 |
| C12A—C11—H11B | 109.2 | O2—C22—H22A | 109.5 |
| C10—C11—H11B | 109.2 | O2—C22—H22B | 109.5 |
| H11A—C11—H11B | 107.9 | H22A—C22—H22B | 109.5 |
| C12B—C11—H11C | 86.2 | O2—C22—H22C | 109.5 |
| C12A—C11—H11C | 125.3 | H22A—C22—H22C | 109.5 |
| C10—C11—H11C | 115.7 | H22B—C22—H22C | 109.5 |
| H11A—C11—H11C | 78.7 | O3—C23—H23A | 109.5 |
| C12B—C11—H11D | 138.9 | O3—C23—H23B | 109.5 |
| C12A—C11—H11D | 95.1 | H23A—C23—H23B | 109.5 |
| C10—C11—H11D | 87.6 | O3—C23—H23C | 109.5 |
| H11B—C11—H11D | 141.2 | H23A—C23—H23C | 109.5 |
| H11C—C11—H11D | 111.6 | H23B—C23—H23C | 109.5 |
| C13—C12A—C11 | 111.0 (2) | O5—C24—H24A | 109.5 |
| C13—C12A—H12A | 109.4 | O5—C24—H24B | 109.5 |
| C11—C12A—H12A | 109.4 | H24A—C24—H24B | 109.5 |
| C13—C12A—H12B | 109.4 | O5—C24—H24C | 109.5 |
| C11—C12A—H12B | 109.4 | H24A—C24—H24C | 109.5 |
| H12A—C12A—H12B | 108.0 | H24B—C24—H24C | 109.5 |
| C11—C12A—H13C | 120.9 | O6—C25—H25A | 109.5 |
| H12A—C12A—H13C | 69.6 | O6—C25—H25B | 109.5 |
| H12B—C12A—H13C | 127.5 | H25A—C25—H25B | 109.5 |
| C13—C12B—C11 | 111.1 (2) | O6—C25—H25C | 109.5 |
| C13—C12B—H12C | 109.4 | H25A—C25—H25C | 109.5 |
| C11—C12B—H12C | 109.4 | H25B—C25—H25C | 109.5 |
| C13—C12B—H12D | 109.4 | O7—C26—H26A | 109.5 |
| C11—C12B—H12D | 109.4 | O7—C26—H26B | 109.5 |
| H12C—C12B—H12D | 108.0 | H26A—C26—H26B | 109.5 |
| C12A—C13—C12B | 47.3 (6) | O7—C26—H26C | 109.5 |
| C12A—C13—C8 | 112.57 (17) | H26A—C26—H26C | 109.5 |
| C12B—C13—C8 | 118.1 (6) | H26B—C26—H26C | 109.5 |
| C12A—C13—H13A | 109.1 | | |

| | | | |
|---|---|---|---|
| C6—C1—C2—O1 | 180.0 (2) | C12B—C11—C12A—C13 | 50.4 (7) |
| C6—C1—C2—C3 | −1.1 (3) | C10—C11—C12A—C13 | −58.8 (3) |
| C21—O1—C2—C1 | −4.3 (3) | C12A—C11—C12B—C13 | −50.4 (7) |
| C21—O1—C2—C3 | 176.8 (2) | C10—C11—C12B—C13 | 44.1 (15) |

| | | | |
|---|---|---|---|
| C22—O2—C3—C4 | −2.2 (3) | C11—C12A—C13—C12B | −50.4 (7) |
| C22—O2—C3—C2 | 177.0 (2) | C11—C12A—C13—C8 | 57.7 (3) |
| C1—C2—C3—O2 | −178.7 (2) | C11—C12B—C13—C12A | 50.4 (7) |
| O1—C2—C3—O2 | 0.3 (3) | C11—C12B—C13—C8 | −45.3 (15) |
| C1—C2—C3—C4 | 0.6 (3) | C7—C8—C13—C12A | 149.0 (2) |
| O1—C2—C3—C4 | 179.6 (2) | C9—C8—C13—C12A | −29.3 (3) |
| O2—C3—C4—C5 | 178.8 (2) | C7—C8—C13—C12B | −158.6 (7) |
| C2—C3—C4—C5 | −0.4 (3) | C9—C8—C13—C12B | 23.1 (7) |
| C23—O3—C5—C4 | −1.9 (3) | C9—C10—C14—C15 | 179.5 (2) |
| C23—O3—C5—C6 | 178.41 (19) | C11—C10—C14—C15 | −3.1 (4) |
| C3—C4—C5—O3 | −178.9 (2) | C10—C14—C15—C16 | 158.9 (2) |
| C3—C4—C5—C6 | 0.7 (3) | C10—C14—C15—C20 | −23.7 (3) |
| O3—C5—C6—C1 | 178.49 (18) | C24—O5—C16—C17 | 5.9 (3) |
| C4—C5—C6—C1 | −1.2 (3) | C24—O5—C16—C15 | −174.4 (2) |
| O3—C5—C6—C7 | 2.0 (3) | C20—C15—C16—O5 | 179.37 (18) |
| C4—C5—C6—C7 | −177.69 (19) | C14—C15—C16—O5 | −3.1 (3) |
| C2—C1—C6—C5 | 1.4 (3) | C20—C15—C16—C17 | −0.9 (3) |
| C2—C1—C6—C7 | 177.8 (2) | C14—C15—C16—C17 | 176.60 (19) |
| C5—C6—C7—C8 | −159.1 (2) | O5—C16—C17—C18 | −179.4 (2) |
| C1—C6—C7—C8 | 24.6 (3) | C15—C16—C17—C18 | 1.0 (3) |
| C6—C7—C8—C9 | −176.7 (2) | C25—O6—C18—C17 | 27.4 (3) |
| C6—C7—C8—C13 | 5.0 (4) | C25—O6—C18—C19 | −153.5 (2) |
| C7—C8—C9—O4 | 4.8 (3) | C16—C17—C18—O6 | −179.9 (2) |
| C13—C8—C9—O4 | −176.8 (2) | C16—C17—C18—C19 | 1.0 (3) |
| C7—C8—C9—C10 | −175.86 (18) | C26—O7—C19—C20 | 13.3 (3) |
| C13—C8—C9—C10 | 2.6 (3) | C26—O7—C19—C18 | −169.2 (2) |
| O4—C9—C10—C14 | −6.9 (3) | O6—C18—C19—O7 | 0.2 (3) |
| C8—C9—C10—C14 | 173.78 (19) | C17—C18—C19—O7 | 179.32 (19) |
| O4—C9—C10—C11 | 175.5 (2) | O6—C18—C19—C20 | 177.82 (19) |
| C8—C9—C10—C11 | −3.9 (3) | C17—C18—C19—C20 | −3.0 (3) |
| C14—C10—C11—C12B | 162.0 (7) | O7—C19—C20—C15 | −179.5 (2) |
| C9—C10—C11—C12B | −20.6 (7) | C18—C19—C20—C15 | 3.1 (3) |
| C14—C10—C11—C12A | −145.8 (2) | C16—C15—C20—C19 | −1.1 (3) |
| C9—C10—C11—C12A | 31.6 (3) | C14—C15—C20—C19 | −178.53 (19) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C12A—H12B···O1$^{i}$ | 0.97 | 2.43 | 3.364 (3) | 160 |
| C25—H25B···O1$^{ii}$ | 0.96 | 2.55 | 3.450 (3) | 156 |

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

**Fig. 1**

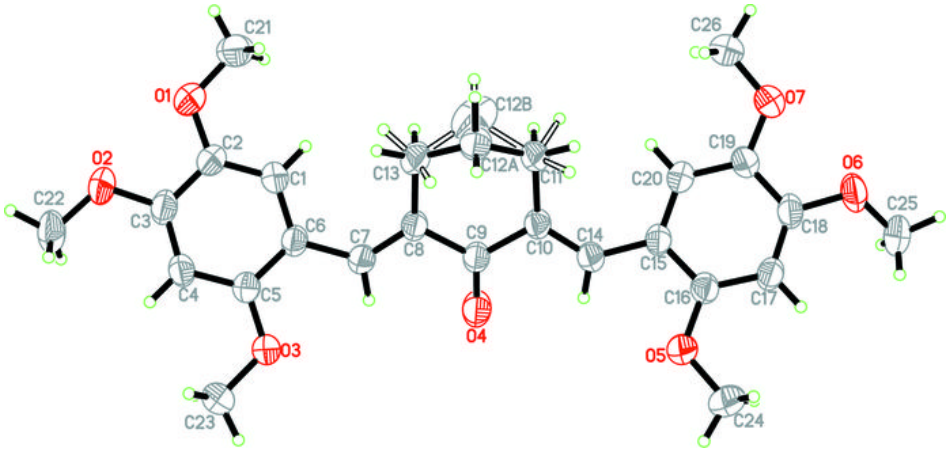
C21
O1
C2
O2
C22
C3
C1
C6
C7
C8
C13
C12B
C12A
C11
C9
C10
C14
C15
C26
O7
C19
C20
O6
C25
C18
C16
C17
O4
O5
O3
C23
C24
C4
C5

**Fig. 2**