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# 2,6-Bis(4-methoxybenzylidene)cyclohexanone

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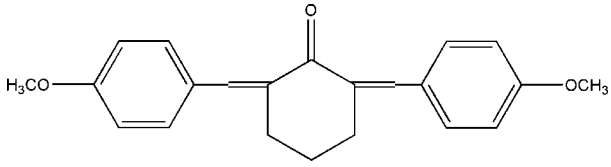
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.061; $wR$ factor = 0.170; data-to-parameter ratio = 13.6.

In the title molecule, $C_{22}H_{22}O_3$, the central cyclohexanone ring adopts an envelope conformation. The two outer aromatic rings form a dihedral angle of 19.3 (2)°. The crystal packing exhibits weak intermolecular C—H···O hydrogen bonds.

## Related literature

For background, see: Tanaka *et al.* (2000). For a related structure, see: Brinda, Mudakavi *et al.* (2007).




## Experimental

### Crystal data

$C_{22}H_{22}O_3$
$M_r$ = 334.40
Monoclinic, $P2_1/c$
$a$ = 9.0129 (8) Å
$b$ = 9.4874 (10) Å
$c$ = 20.9416 (17) Å
$\beta$ = 100.518 (1)°
$V$ = 1760.6 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 298 K
0.45 × 0.17 × 0.15 mm

### Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.964, $T_{max}$ = 0.988
9092 measured reflections
3105 independent reflections
1233 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.065

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.170
$S$ = 0.87
3105 reflections
228 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.12 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C14—H14$B$···O2$^{i}$ | 0.96 | 2.67 | 3.512 (5) | 146 |
| C4—H4$A$···O1$^{ii}$ | 0.97 | 2.61 | 3.510 (5) | 154 |

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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*.

This project was supported by the Foundation of Liaocheng Vocational and Technical College.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2532).

## References

Brinda, Mudakavi, R., Chopra, D., Murthy, M. S. & Row, T. N. G. (2007). *Acta Cryst.* E**63**, o4494.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Tanaka, T. & Toda, F. (2000). *Chem. Rev.* **100**, 1025–1074.

# supplementary materials

*Acta Cryst.* (2009). E**65**, o928 [ **doi:10.1107/S160053680901112X** ]

# 2,6-Bis(4-methoxybenzylidene)cyclohexanone

**D. Liu and G. Chen**

## Comment

The use of organic syntheses without volatile, often flammable, expensive and toxic solvents strongly reduces the waste production and many fundamental processes have proven to be achievable through efficient procedures characterized by high simplicity of set-up and work-up (Tanaka *et al.*, 2000). In this paper, we describe a solvent-free protocol used in the synthesis of the title compound, (I), starting from the fragrant aldehydes and cyclohexanone in the presence of NaOH.

In (I) (Fig. 1), the bond lengths and angles are normal and correspond to those observed in 4-methyl-2,6-bis(2-naphthylmethylene) cyclohexan-1-one (Brinda, Mudakavi *et al.*, 2007). The central cyclohexanone ring adopts an envelope conformation. The mean planes of two rings - C8—C13 and C16—C21 - form a dihedral angle of 19.3 (2)°. The crystal packing exhibits weak intermolecular C—H···O hydrogen bonds (Table 1).

## Experimental

2-Methoxylbenzaldehyde (4 mmol) and cyclohexanone (2.0 mmol), NaOH (4.0 mmol) were mixed in 50 ml flash under sovlent-free condtions After stirring for 15 min at 293 K, tthe resulting mixture was washed with water for several times for removing NaOH, and recrystalized from ethanol, and afforded the title compound as a crystalline solid. Elemental analysis: calculated for $C_{22}H_{22}O_3$: C 79.02, H 6.63%; found: C 69.93, H 6.65%.

## Refinement

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined using a riding model, with $U_{iso}$(H) = 1.2–1.5$U_{eq}$(C).

## Figures

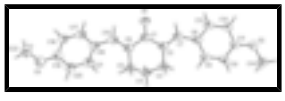


Fig. 1. View of (I) showing the atomic numbering scheme and 40% probability displacement ellipsoids.

## 2,6-Bis(4-methoxybenzylidene)cyclohexanone

*Crystal data*

| | |
|---|---|
| $C_{22}H_{22}O_3$ | $F_{000}$ = 712 |
| $M_r$ = 334.40 | $D_x$ = 1.262 Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| $a$ = 9.0129 (8) Å | Cell parameters from 935 reflections |

| | |
|---|---|
| $b$ = 9.4874 (10) Å | θ = 2.4–25.2º |
| $c$ = 20.9416 (17) Å | μ = 0.08 mm$^{-1}$ |
| β = 100.5180 (10)º | $T$ = 298 K |
| $V$ = 1760.6 (3) Å$^3$ | Prism, yellow |
| $Z$ = 4 | 0.45 × 0.17 × 0.15 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX CCD area-detector diffractometer | 3105 independent reflections |
| Radiation source: fine-focus sealed tube | 1233 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.065 |
| $T$ = 298 K | $\theta_{max}$ = 25.0º |
| φ and ω scans | $\theta_{min}$ = 2.0º |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h$ = −10→10 |
| $T_{min}$ = 0.964, $T_{max}$ = 0.988 | $k$ = −11→11 |
| 9092 measured reflections | $l$ = −23→24 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.061 | H-atom parameters constrained |
| $wR(F^2)$ = 0.170 | $w = 1/[\sigma^2(F_o^2) + (0.076P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 0.87 | $(\Delta/\sigma)_{max} < 0.001$ |
| 3105 reflections | $\Delta\rho_{max}$ = 0.26 e Å$^{-3}$ |
| 228 parameters | $\Delta\rho_{min}$ = −0.12 e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

*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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| O1 | 0.1376 (3) | −0.1927 (3) | 0.02703 (11) | 0.1012 (9) |
| O2 | 0.4818 (3) | −0.0038 (2) | 0.41111 (13) | 0.0905 (8) |
| O3 | 0.0867 (3) | 0.0536 (3) | −0.35644 (17) | 0.1062 (9) |
| C1 | 0.1981 (4) | −0.0760 (4) | 0.02660 (19) | 0.0782 (10) |
| C2 | 0.2658 (4) | −0.0065 (3) | 0.0884 (2) | 0.0729 (10) |
| C3 | 0.3239 (4) | 0.1430 (3) | 0.08487 (18) | 0.0941 (12) |
| H3A | 0.3018 | 0.1963 | 0.1215 | 0.113* |
| H3B | 0.4327 | 0.1404 | 0.0883 | 0.113* |
| C4 | 0.2551 (5) | 0.2176 (4) | 0.02279 (19) | 0.1055 (14) |
| H4A | 0.1479 | 0.2305 | 0.0217 | 0.127* |
| H4B | 0.3007 | 0.3101 | 0.0220 | 0.127* |
| C5 | 0.2779 (5) | 0.1360 (4) | −0.03590 (18) | 0.0973 (12) |
| H5A | 0.3851 | 0.1263 | −0.0359 | 0.117* |
| H5B | 0.2331 | 0.1869 | −0.0748 | 0.117* |
| C6 | 0.2063 (4) | −0.0095 (3) | −0.0364 (2) | 0.0754 (10) |
| C7 | 0.2725 (4) | −0.0772 (3) | 0.1430 (2) | 0.0744 (10) |
| H7 | 0.2294 | −0.1662 | 0.1361 | 0.089* |
| C8 | 0.3302 (4) | −0.0512 (3) | 0.21109 (18) | 0.0655 (9) |
| C9 | 0.4222 (4) | 0.0636 (4) | 0.2354 (2) | 0.0812 (10) |
| H9 | 0.4495 | 0.1291 | 0.2066 | 0.097* |
| C10 | 0.4728 (4) | 0.0810 (3) | 0.30125 (19) | 0.0760 (10) |
| H10 | 0.5326 | 0.1585 | 0.3158 | 0.091* |
| C11 | 0.4371 (4) | −0.0127 (4) | 0.3454 (2) | 0.0746 (10) |
| C12 | 0.3475 (4) | −0.1281 (3) | 0.3233 (2) | 0.0753 (10) |
| H12 | 0.3210 | −0.1932 | 0.3525 | 0.090* |
| C13 | 0.2984 (4) | −0.1449 (3) | 0.2575 (2) | 0.0789 (10) |
| H13 | 0.2403 | −0.2238 | 0.2434 | 0.095* |
| C14 | 0.5918 (5) | 0.0992 (4) | 0.43567 (18) | 0.1055 (13) |
| H14A | 0.6829 | 0.0810 | 0.4194 | 0.158* |
| H14B | 0.6125 | 0.0951 | 0.4823 | 0.158* |
| H14C | 0.5545 | 0.1911 | 0.4220 | 0.158* |
| C15 | 0.1460 (4) | −0.0755 (3) | −0.0911 (2) | 0.0747 (10) |
| H15 | 0.1021 | −0.1614 | −0.0840 | 0.090* |
| C16 | 0.1343 (4) | −0.0418 (3) | −0.1593 (2) | 0.0709 (10) |
| C17 | 0.0221 (4) | −0.1023 (3) | −0.2053 (2) | 0.0777 (10) |
| H17 | −0.0428 | −0.1659 | −0.1907 | 0.093* |
| C18 | −0.0001 (4) | −0.0757 (4) | −0.2704 (2) | 0.0792 (10) |
| H18 | −0.0786 | −0.1184 | −0.2988 | 0.095* |
| C19 | 0.0973 (5) | 0.0166 (4) | −0.2928 (2) | 0.0862 (12) |
| C20 | 0.2143 (5) | 0.0738 (4) | −0.2494 (2) | 0.0884 (12) |
| H20 | 0.2823 | 0.1329 | −0.2646 | 0.106* |
| C21 | 0.2335 (4) | 0.0466 (4) | −0.1850 (2) | 0.0894 (11) |
| H21 | 0.3142 | 0.0874 | −0.1572 | 0.107* |
| C22 | −0.0240 (5) | −0.0063 (4) | −0.4044 (2) | 0.1223 (16) |
| H22A | −0.0036 | −0.1049 | −0.4082 | 0.184* |
| H22B | −0.0231 | 0.0395 | −0.4452 | 0.184* |
| H22C | −0.1213 | 0.0054 | −0.3925 | 0.184* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.117 (2) | 0.0614 (16) | 0.120 (2) | −0.0281 (15) | 0.0088 (15) | 0.0043 (14) |
| O2 | 0.097 (2) | 0.0870 (18) | 0.087 (2) | −0.0124 (15) | 0.0157 (15) | 0.0058 (14) |
| O3 | 0.128 (3) | 0.090 (2) | 0.104 (2) | 0.0029 (17) | 0.0314 (19) | −0.0034 (17) |
| C1 | 0.072 (3) | 0.059 (2) | 0.105 (3) | 0.0060 (19) | 0.019 (2) | 0.008 (2) |
| C2 | 0.066 (2) | 0.053 (2) | 0.098 (3) | −0.0002 (17) | 0.012 (2) | −0.004 (2) |
| C3 | 0.107 (3) | 0.061 (2) | 0.109 (3) | −0.011 (2) | 0.005 (2) | 0.003 (2) |
| C4 | 0.133 (4) | 0.053 (2) | 0.124 (3) | −0.019 (2) | 0.005 (3) | −0.003 (2) |
| C5 | 0.108 (3) | 0.063 (2) | 0.120 (3) | −0.018 (2) | 0.017 (2) | 0.002 (2) |
| C6 | 0.070 (3) | 0.054 (2) | 0.101 (3) | 0.0013 (17) | 0.012 (2) | −0.010 (2) |
| C7 | 0.065 (2) | 0.053 (2) | 0.107 (3) | 0.0008 (17) | 0.021 (2) | 0.001 (2) |
| C8 | 0.057 (2) | 0.047 (2) | 0.094 (3) | 0.0007 (17) | 0.018 (2) | −0.0063 (19) |
| C9 | 0.078 (3) | 0.069 (2) | 0.101 (3) | −0.010 (2) | 0.027 (2) | 0.010 (2) |
| C10 | 0.073 (3) | 0.064 (2) | 0.090 (3) | −0.0117 (18) | 0.014 (2) | 0.002 (2) |
| C11 | 0.070 (3) | 0.067 (2) | 0.091 (3) | 0.0036 (19) | 0.025 (2) | 0.009 (2) |
| C12 | 0.071 (3) | 0.053 (2) | 0.104 (3) | −0.0076 (18) | 0.022 (2) | 0.0050 (19) |
| C13 | 0.067 (3) | 0.056 (2) | 0.116 (3) | −0.0037 (17) | 0.021 (2) | 0.000 (2) |
| C14 | 0.103 (3) | 0.083 (3) | 0.126 (3) | −0.012 (2) | 0.008 (3) | −0.008 (2) |
| C15 | 0.067 (2) | 0.059 (2) | 0.101 (3) | 0.0070 (18) | 0.022 (2) | 0.001 (2) |
| C16 | 0.060 (2) | 0.050 (2) | 0.104 (3) | 0.0043 (18) | 0.019 (2) | −0.009 (2) |
| C17 | 0.072 (3) | 0.058 (2) | 0.106 (3) | 0.0068 (19) | 0.023 (2) | −0.007 (2) |
| C18 | 0.073 (3) | 0.065 (2) | 0.101 (3) | 0.010 (2) | 0.021 (2) | −0.008 (2) |
| C19 | 0.102 (4) | 0.065 (3) | 0.097 (4) | 0.026 (2) | 0.031 (3) | 0.001 (2) |
| C20 | 0.090 (3) | 0.069 (2) | 0.111 (4) | −0.011 (2) | 0.031 (3) | −0.010 (2) |
| C21 | 0.086 (3) | 0.076 (3) | 0.109 (4) | −0.007 (2) | 0.023 (3) | −0.009 (2) |
| C22 | 0.135 (4) | 0.134 (4) | 0.099 (3) | 0.024 (3) | 0.025 (3) | −0.013 (3) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.236 (4) | C10—C11 | 1.363 (4) |
| O2—C11 | 1.364 (4) | C10—H10 | 0.9300 |
| O2—C14 | 1.420 (4) | C11—C12 | 1.388 (4) |
| O3—C19 | 1.364 (4) | C12—C13 | 1.377 (4) |
| O3—C22 | 1.400 (4) | C12—H12 | 0.9300 |
| C1—C6 | 1.477 (5) | C13—H13 | 0.9300 |
| C1—C2 | 1.481 (5) | C14—H14A | 0.9600 |
| C2—C7 | 1.318 (4) | C14—H14B | 0.9600 |
| C2—C3 | 1.518 (4) | C14—H14C | 0.9600 |
| C3—C4 | 1.510 (4) | C15—C16 | 1.448 (4) |
| C3—H3A | 0.9700 | C15—H15 | 0.9300 |
| C3—H3B | 0.9700 | C16—C17 | 1.388 (4) |
| C4—C5 | 1.498 (4) | C16—C21 | 1.403 (5) |
| C4—H4A | 0.9700 | C17—C18 | 1.364 (4) |
| C4—H4B | 0.9700 | C17—H17 | 0.9300 |
| C5—C6 | 1.523 (4) | C18—C19 | 1.382 (5) |
| C5—H5A | 0.9700 | C18—H18 | 0.9300 |

| | | | |
|---|---|---|---|
| C5—H5B | 0.9700 | C19—C20 | 1.372 (5) |
| C6—C15 | 1.330 (4) | C20—C21 | 1.352 (5) |
| C7—C8 | 1.447 (4) | C20—H20 | 0.9300 |
| C7—H7 | 0.9300 | C21—H21 | 0.9300 |
| C8—C13 | 1.385 (4) | C22—H22A | 0.9600 |
| C8—C9 | 1.407 (4) | C22—H22B | 0.9600 |
| C9—C10 | 1.380 (4) | C22—H22C | 0.9600 |
| C9—H9 | 0.9300 | | |
| C11—O2—C14 | 117.8 (3) | C10—C11—C12 | 118.9 (4) |
| C19—O3—C22 | 120.6 (4) | O2—C11—C12 | 115.6 (3) |
| O1—C1—C6 | 118.9 (3) | C13—C12—C11 | 119.0 (3) |
| O1—C1—C2 | 120.3 (3) | C13—C12—H12 | 120.5 |
| C6—C1—C2 | 120.7 (4) | C11—C12—H12 | 120.5 |
| C7—C2—C1 | 118.6 (3) | C12—C13—C8 | 123.9 (3) |
| C7—C2—C3 | 123.8 (3) | C12—C13—H13 | 118.0 |
| C1—C2—C3 | 117.6 (3) | C8—C13—H13 | 118.0 |
| C4—C3—C2 | 112.9 (3) | O2—C14—H14A | 109.5 |
| C4—C3—H3A | 109.0 | O2—C14—H14B | 109.5 |
| C2—C3—H3A | 109.0 | H14A—C14—H14B | 109.5 |
| C4—C3—H3B | 109.0 | O2—C14—H14C | 109.5 |
| C2—C3—H3B | 109.0 | H14A—C14—H14C | 109.5 |
| H3A—C3—H3B | 107.8 | H14B—C14—H14C | 109.5 |
| C5—C4—C3 | 111.6 (3) | C6—C15—C16 | 133.6 (3) |
| C5—C4—H4A | 109.3 | C6—C15—H15 | 113.2 |
| C3—C4—H4A | 109.3 | C16—C15—H15 | 113.2 |
| C5—C4—H4B | 109.3 | C17—C16—C21 | 114.5 (4) |
| C3—C4—H4B | 109.3 | C17—C16—C15 | 120.4 (4) |
| H4A—C4—H4B | 108.0 | C21—C16—C15 | 125.0 (4) |
| C4—C5—C6 | 110.7 (3) | C18—C17—C16 | 124.9 (4) |
| C4—C5—H5A | 109.5 | C18—C17—H17 | 117.5 |
| C6—C5—H5A | 109.5 | C16—C17—H17 | 117.5 |
| C4—C5—H5B | 109.5 | C17—C18—C19 | 118.1 (4) |
| C6—C5—H5B | 109.5 | C17—C18—H18 | 121.0 |
| H5A—C5—H5B | 108.1 | C19—C18—H18 | 121.0 |
| C15—C6—C1 | 119.3 (3) | O3—C19—C20 | 117.0 (4) |
| C15—C6—C5 | 122.6 (3) | O3—C19—C18 | 124.0 (4) |
| C1—C6—C5 | 118.1 (3) | C20—C19—C18 | 118.9 (4) |
| C2—C7—C8 | 136.0 (3) | C21—C20—C19 | 121.9 (4) |
| C2—C7—H7 | 112.0 | C21—C20—H20 | 119.0 |
| C8—C7—H7 | 112.0 | C19—C20—H20 | 119.0 |
| C13—C8—C9 | 115.2 (3) | C20—C21—C16 | 121.5 (4) |
| C13—C8—C7 | 119.9 (3) | C20—C21—H21 | 119.3 |
| C9—C8—C7 | 124.8 (3) | C16—C21—H21 | 119.3 |
| C10—C9—C8 | 121.3 (3) | O3—C22—H22A | 109.5 |
| C10—C9—H9 | 119.3 | O3—C22—H22B | 109.5 |
| C8—C9—H9 | 119.3 | H22A—C22—H22B | 109.5 |
| C11—C10—C9 | 121.6 (3) | O3—C22—H22C | 109.5 |
| C11—C10—H10 | 119.2 | H22A—C22—H22C | 109.5 |
| C9—C10—H10 | 119.2 | H22B—C22—H22C | 109.5 |

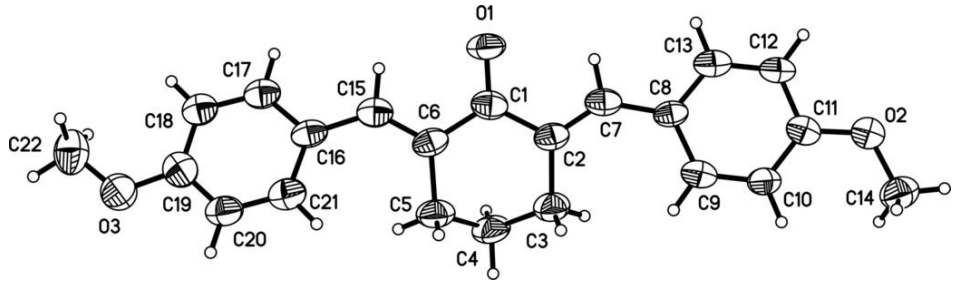
| | |
|---|---|
| C10—C11—O2 | 125.5 (3) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C14—H14B···O2$^{i}$ | 0.96 | 2.67 | 3.512 (5) | 146 |
| C4—H4A···O1$^{ii}$ | 0.97 | 2.61 | 3.510 (5) | 154 |

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

**Fig. 1**


O1
C13
C12
C17
C15
C1
C8
C11
O2
C18
C6
C7
C22
C16
C2
O3
C19
C21
C5
C9
C10
C14
C20
C3
C4