*Crystal data*

$C_{15}H_{17}NO_3$
$M_r = 259.30$
Orthorhombic
$Pca2_1$
$a = 18.0675\,(9)$ Å
$b = 7.7986\,(4)$ Å
$c = 9.2847\,(4)$ Å
$V = 1308.2\,(1)$ Å$^3$
$Z = 4$
$D_x = 1.317$ Mg m$^{-3}$
$D_m$ not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 6180 reflections
$\theta = 2.25–28.31°$
$\mu = 0.092$ mm$^{-1}$
$T = 293\,(2)$ K
Block
0.48 × 0.40 × 0.40 mm
Colourless

*Data collection*

Siemens SMART CCD area-detector diffractometer
$\omega$ scans
Absorption correction: none
8746 measured reflections
3233 independent reflections
2823 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.028$
$\theta_{\rm max} = 28.31°$
$h = 0 \rightarrow 23$
$k = 0 \rightarrow 10$
$l = -12 \rightarrow 12$
Intensity decay: negligible

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.098$
$S = 1.05$
3233 reflections
173 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.0867P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.15$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.11$ e Å$^{-3}$
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—N1 | 1.202 (2) | N1—C1 | 1.516 (2) |
| O2—N1 | 1.217 (2) | C4—C5 | 1.327 (2) |
| O3—C6 | 1.214 (2) | C9—C10 | 1.321 (3) |
| C8—C14—C11 | 93.8 (1) | | |
| O1—N1—C1—C13 | −22.5 (2) | O2—N1—C1—C2 | −81.2 (2) |

Table 2. *Hydrogen-bonding geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C12—H12···O3$^{\rm i}$ | 0.98 | 2.49 | 3.337 (2) | 144 |

Symmetry code: (i) $1 - x, 2 - y, z - \frac{1}{2}$.

After checking their presence in the difference map, the H atoms were fixed geometrically and allowed to ride on the atoms to which they were attached; rotating-group refinement was used for the methyl groups.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1281). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1999). **C55**, 1130–1132

# (2*RS*,5*RS*)-2,5-Bis[3-(4-methylphenyl)-3-oxopropyl]cyclopentan-1-one

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## Abstract

In the title compound, $C_{25}H_{28}O_3$, the cyclopentane ring adopts a half-chair conformation, with one of the side chains attached in the equatorial position and the other in a bi-axial orientation. The mean plane through the cyclopentane ring forms dihedral angles of 30.9 (2) and 24.3 (2)° with the two phenyl rings. The crystal structure is stabilized by C—H···O hydrogen bonds and C—H···π interactions.

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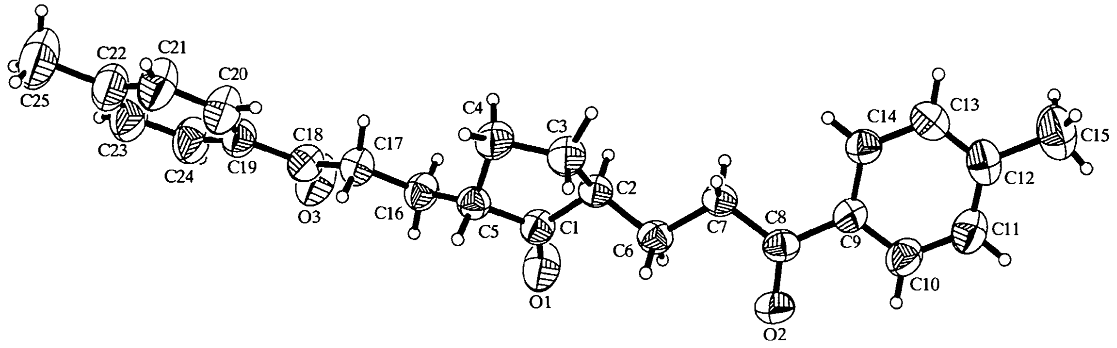
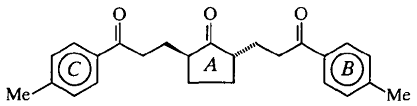


Fig. 1. The structure of (I) showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms are drawn as spheres of arbitrary radii. Only one orientation for each of the disordered methyl groups is shown.

## Comment

While attempting to repeat the procedure of Gill *et al.* (1952) for the synthesis of 1-(4-methylphenyl)-3-(2-oxocyclopentyl)-1-propanone, we found that the reaction furnishes the title *trans*-triketone, (I), along with its *cis* isomer, in yields of 65.5 and 10%, respectively. Such triketones are important as they are expected to show specific metal-binding properties. The present X-ray structure determination was carried out in order to elucidate the molecular conformation of (I).



(I)

The bond lengths and angles observed in the structure of (I) show normal values (Allen *et al.*, 1987). The cyclopentane ring *A* adopts a half-chair conformation, with C3 and C4 twisted from the mean plane by 0.224 (3) and −0.219 (3) Å, respectively; asymmetry parameter $\Delta C_2$(C3—C4) = 1.7 (3)° (Duax *et al.*, 1976). The carbonyl group and one of the side chains (C6–C15) are attached in the equatorial position, whereas the other side chain is in a bi-axial orientation. The carbonyl atoms O2 and O3 are in the synperiplanar conformation with respect to C6 and C16, respectively, while the conformation observed across each of the C6—C7—C8—C9 and C16—C17—C18—C19 linkages is antiperiplanar. The mean plane through the cyclopentane ring forms dihedral angles of 30.9 (2) and 24.3 (2)°, respectively, with phenyl rings *B* and *C*. The two phenyl ring planes form a dihedral angle of 53.8 (2)°. The glide-related molecules are almost stacked along the *b* direction, with C7—H7*A*···O2$^{i}$ [symmetry code: (i) $\frac{3}{2}-x$, $y-\frac{1}{2}$, $z$] hydrogen bonds linking them to form infinite chains (Table 2). The crystal structure is further stabilized by a number of C—H···π interactions involving the two phenyl rings (Brady *et al.*, 1998; Gallagher *et al.*, 1998; Dalton *et al.*, 1999).

## Experimental

Following the procedure of Gill *et al.* (1952), a mixture of cyclopentanone (2.207 g, 26.28 mmol) and 3-dimethylamino-1-(4-methylphenyl)-1-propanone (1.673 g, 8.76 mmol) was heated in an oil bath maintained at 433 K for 30 min. The resulting reaction mixture was applied to a silica-gel column (100–200 mesh, 2 × 40 cm) and eluted with an increasing percentage of ethyl acetate in hexanes (5.95 to 15.85%). The column purification furnished the di- and triketones in the ratio of 6:1. The isomeric triketones, which eluted from the column essentially as mixtures, were subjected to fractional crystallization (hexane, ethyl acetate). The *trans* isomer, (I), was obtained through fractional crystallization (m.p. 374 K).

*Crystal data*

$C_{25}H_{28}O_3$
$M_r$ = 376.47
Orthorhombic
*Pbca*
*a* = 11.7954 (5) Å
*b* = 7.6537 (3) Å
*c* = 46.732 (2) Å
*V* = 4218.9 (3) Å$^3$
*Z* = 8
$D_x$ = 1.185 Mg m$^{-3}$
$D_m$ not measured

Mo *Kα* radiation
λ = 0.71073 Å
Cell parameters from 6910 reflections
θ = 1.74–25.00°
μ = 0.076 mm$^{-1}$
*T* = 293 (2) K
Plate
0.38 × 0.32 × 0.14 mm
Colourless

*Data collection*

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
20 844 measured reflections
3705 independent reflections
2485 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.059
$\theta_{\rm max}$ = 25°
*h* = −14 → 13
*k* = −9 → 9
*l* = −55 → 41
Intensity decay: negligible

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.148
*S* = 1.14

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.12 e Å$^{-3}$
$\Delta\rho_{\rm min}$ = −0.14 e Å$^{-3}$
Extinction correction: none

3705 reflections
253 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0345P)^2 + 2.4171P]$
where $P = (F_o^2 + 2F_c^2)/3$

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.210 (3) | O3—C18 | 1.217 (3) |
| O2—C8 | 1.219 (3) | | |
| C5—C1—C2—C3 | 12.6 (3) | C6—C7—C8—C9 | 170.7 (2) |
| C1—C2—C3—C4 | −30.9 (3) | C7—C8—C9—C10 | −169.4 (2) |
| C2—C3—C4—C5 | 38.3 (3) | C4—C5—C16—C17 | −58.7 (3) |
| C2—C1—C5—C4 | 10.5 (3) | C5—C16—C17—C18 | −173.3 (2) |
| C3—C4—C5—C1 | −29.6 (3) | C16—C17—C18—O3 | 0.8 (4) |
| C3—C2—C6—C7 | −67.5 (3) | C16—C17—C18—C19 | −179.4 (3) |
| C2—C6—C7—C8 | −171.2 (2) | C17—C18—C19—C20 | 7.2 (5) |
| C6—C7—C8—O2 | −7.2 (4) | | |

Table 2. *Hydrogen-bonding geometry (Å, °)*

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C7—H7$A$···O2$^{i}$ | 0.97 | 2.41 | 3.113 (3) | 129 |
| C10—H10$A$···π(ring $B$)$^{ii}$ | 0.93 | 3.05 | 3.863 (3) | 146 |
| C15—H15$F$···π(ring $C$)$^{iii}$ | 0.96 | 2.72 | 3.637 (3) | 160 |
| C17—H17$B$···π(ring $B$)$^{iv}$ | 0.97 | 3.16 | 3.986 (3) | 143 |
| C25—H25$E$···π(ring $C$)$^{v}$ | 0.96 | 3.10 | 3.970 (3) | 152 |

Symmetry codes: (i) $\frac{3}{2} - x, y - \frac{1}{2}, z$; (ii) $\frac{3}{2} - x, \frac{1}{2} + y, z$; (iii) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (iv) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$; (v) $1 - x, 2 - y, 1 - z$.

All H atoms were located from a difference electron-density map. Both methyl groups were found to be disordered, with two positions rotated from each other by 60°, and they were treated as idealized disordered methyl groups. The remaining H atoms were also placed at geometrically calculated positions and a riding model was used for their refinement. The $U_{iso}$ values for the methyl H atoms were set at $1.5U_{eq}$ of the attached atom and those for the remaining H atoms were set at $1.2U_{eq}$.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1083). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1999). C**55**, 1132–1134

# 1-(4-Benzylpiperidin-1-yl)glyoxal dioxime

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## Abstract

The title compound, $C_{14}H_{19}N_3O_2$, contains two independent molecules in the asymmetric unit. The piperidine rings assume chair conformations. The dihedral angle between the piperidine and benzyl ring planes is 86.6 (1)° in molecule *A* and 77.2 (1)° in molecule *B*. Both O—H···O and O—H···N intermolecular hydrogen bonds are present.

## Comment

Oximes and dioximes are of much analytical and medicinal interest, and for this reason their chemistry has been investigated extensively (Hamilton, 1961; Calleri *et al.*, 1966; Chakravorty, 1974). The interesting property they possess of forming complexes with transition metals has also been studied (Bekaroğlu *et al.*, 1978; Endres, 1978). Structural information on oximes and dioximes and their derivatives is therefore useful, and so the structure of the title compound, (I), has been determined.