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# 2,2′-[1,2-Bis(4-chlorophenyl)ethane-1,2-diyl]bis(1-tetralone)

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**Key indicators**

Single-crystal X-ray study
*T* = 294 K
Mean σ(C–C) = 0.002 Å
*R* factor = 0.037
*wR* factor = 0.088
Data-to-parameter ratio = 13.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.



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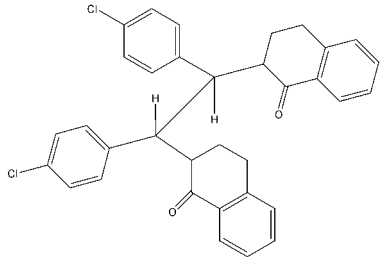
The title compound, $C_{34}H_{28}Cl_2O_2$, was synthesized by the self-coupling reaction of 2-(4-chlorobenzyl)-1-tetralone induced by low-valent titanium reagent ($TiCl_4$/Zn) in tetrahydrofuran. The X-ray analysis reveals that the two cyclohexenone rings adopt different conformations, *viz*. envelope and distorted half-chair.

## Comment

In the early seventies, Tyreik & Wolochowicz (1973), Mukaiyama *et al.* (1973) and McMurry & Fleming (1974) established that low-valent titanium can abstract oxygen from ketones or aldehydes leading to formation of olefins. An increasing interest in coupling reactions induced by low-valent titanium reagents has been observed and a large number of functional groups can be reduced (Shi *et al.*, 1993, 1997, 1998). We report here the crystal structure of the title compound, (I), synthesized by the self-coupling reaction of 2-(4-chlorobenzyl)-1-tetralone induced by low-valent titanium reagent ($TiCl_4$/Zn).




(I)

The molecular structure of (I) is shown in Fig. 1 and selected bond lengths and angles are listed in Table 1. The C7—C24 linkage resulting from dimerization reaction of 2-(4-chlorobenzyl)-1-tetralone has a length of 1.549 (2) Å. The bond angles around C7 and C24 deviate from the ideal value for a $sp^3$ carbon. One of the cyclohexenone rings (C8–C11/C16/C17) adopts an envelope conformation with C9 deviating by 0.652 (2) Å from the plane determined by the other five atoms. The other cyclohexenone ring (C25–C28/C33/C34) adopts a distorted half-chair conformation, with atoms C25 and C26 deviating from the C27/C28/C33/C34 plane by −0.186 (2) and 0.576 (2) Å, respectively. In addition, the X-ray analysis reveals that the configuration of (I) is *threo* and its conformation is *gauche*. Carbonyl atoms O1 and O2 are involved in weak intramolecular C—H···O interactions (Table 2) and the molecular packing in the crystal (Fig. 2) is stabilized by C—H···π interactions involving C30—H30 and phenyl ring (C18–C23) at ($x$, $1 + y$, $z$).

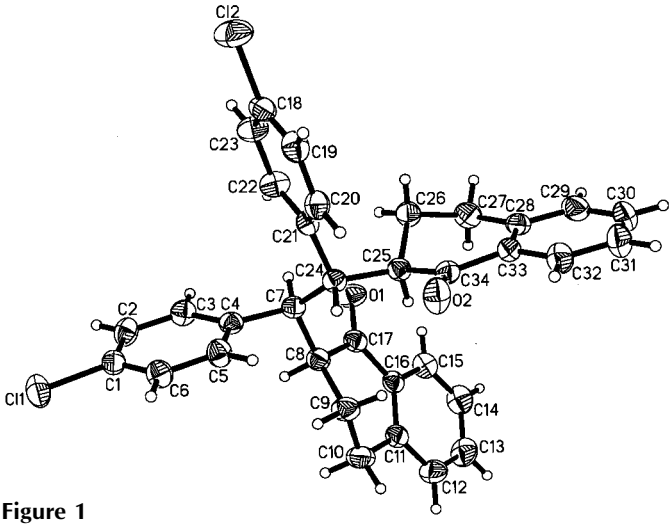



**Figure 1**
The molecular structure of (I), showing 50% probability of displacement ellipsoids and the atom-numbering scheme.

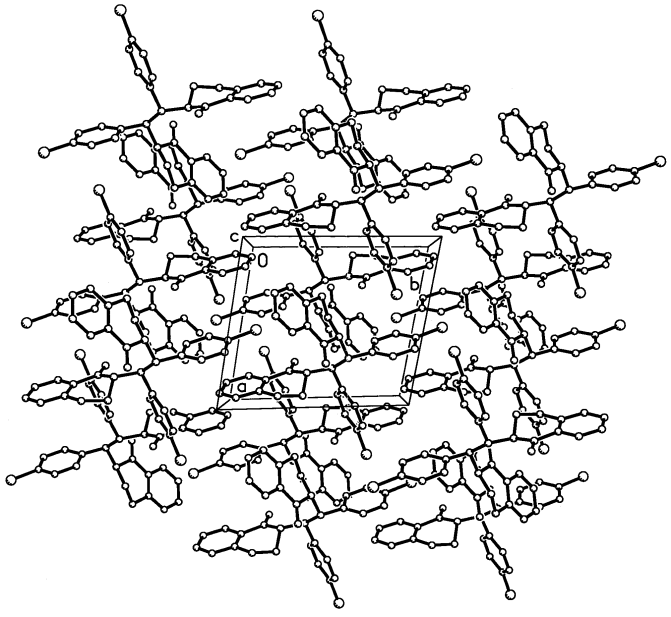



**Figure 2**
The molecular packing of (I), viewed down the *c* axis.

## Experimental

The title compound, (I), was prepared by the self-coupling reaction of 2-(4-chlorobenzyl)-1-tetralone induced by low-valent titanium reagent ($TiCl_4$/Zn); m.p. 491–493 K. Single crystals were obtained by slow evaporation of an ethanol solution.

*Crystal data*

$C_{34}H_{28}Cl_2O_2$
$M_r$ = 539.46
Triclinic, $P\overline{1}$
$a$ = 10.411 (2) Å
$b$ = 10.975 (1) Å
$c$ = 13.296 (2) Å
$\alpha$ = 103.54 (1)°
$\beta$ = 112.97 (1)°
$\gamma$ = 93.89 (1)°
$V$ = 1338.2 (4) Å$^3$
$Z$ = 2
$D_x$ = 1.339 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 31 reflections
$\theta$ = 4.4–15.0°
$\mu$ = 0.27 mm$^{-1}$
$T$ = 294 (2) K
Block, colorless
0.52 × 0.48 × 0.48 mm

*Data collection*

Siemens $P4$ diffractometer
$\omega$ scans
Absorption correction: $\psi$ scan (*XSCANS*; Siemens, 1994)
$T_{min}$ = 0.865, $T_{max}$ = 0.877
5121 measured reflections
4671 independent reflections
3392 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.015
$\theta_{max}$ = 25.0°
$h = 0 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -15 \rightarrow 14$
3 standard reflections
every 97 reflections
intensity decay: 4.3%

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.088
$S$ = 1.05
4671 reflections
344 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0415P)^2 + 0.0134P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0082 (11)

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

| | | | |
|---|---|---|---|
| Cl1—C1 | 1.7420 (18) | C7—C24 | 1.549 (2) |
| Cl2—C18 | 1.7409 (19) | C7—C8 | 1.555 (2) |
| O1—C17 | 1.214 (2) | C21—C24 | 1.526 (2) |
| O2—C34 | 1.217 (2) | C24—C25 | 1.546 (2) |
| C4—C7 | 1.520 (2) | | |
| | | | |
| C4—C7—C24 | 111.85 (14) | C21—C24—C25 | 110.19 (14) |
| C4—C7—C8 | 108.79 (14) | C21—C24—C7 | 111.58 (14) |
| C24—C7—C8 | 115.78 (14) | C25—C24—C7 | 114.44 (14) |
| O1—C17—C16 | 120.68 (17) | O2—C34—C33 | 121.16 (16) |
| O1—C17—C8 | 121.60 (17) | O2—C34—C25 | 121.86 (16) |
| C16—C17—C8 | 117.68 (15) | C33—C34—C25 | 116.91 (15) |
| | | | |
| C4—C7—C24—C21 | −59.28 (18) | C4—C7—C24—C25 | 174.74 (14) |
| C8—C7—C24—C21 | 175.33 (13) | C8—C7—C24—C25 | 49.4 (2) |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C7—H7···O1 | 0.98 | 2.37 | 2.820 (2) | 107 |
| C20—H20···O2 | 0.93 | 2.55 | 3.183 (3) | 125 |
| C24—H24···O2 | 0.98 | 2.37 | 2.797 (2) | 106 |
| C30—H30···$CgP^i$ | 0.93 | 2.72 | 3.626 (3) | 166 |

Symmetry code: (i) $x, 1 + y, z$. *CgP* is the centroid of the phenyl ring C18–C23.

H atoms were fixed geometrically and allowed to ride on their parent C atoms, with C—H distances fixed in the range 0.93–0.98 Å and $U_{iso}$(H) values set equal to $1.2U_{eq}$(C).

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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