

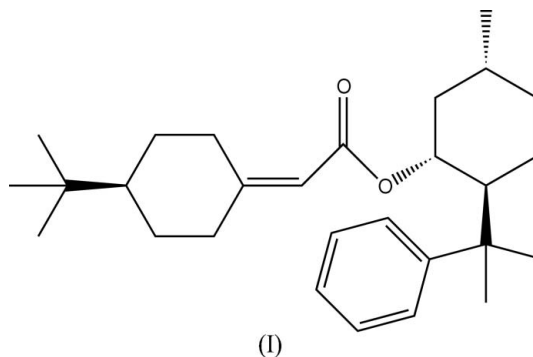
**(a*R*,1*R*,2*S*,5*R*)-5-Methyl-2-(1-methyl-1-phenylethyl)cyclohexyl (4-*tert*-butylcyclohexylidene)-acetate****Jin-Long Wu**Laboratory of Asymmetric Catalysis and  
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Republic of ChinaCorrespondence e-mail: [wyz@zju.edu.cn](mailto:wyz@zju.edu.cn)**Key indicators**Single-crystal X-ray study  
 $T = 298$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.033  
 $wR$  factor = 0.061  
Data-to-parameter ratio = 21.3For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.In the molecule of the title compound,  $\text{C}_{28}\text{H}_{42}\text{O}_2$ , the two cyclohexane rings adopt chair conformations. The olefinic bond and carbonyl group are approximately coplanar.

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**Comment**

The Wittig reaction of ylides with aldehydes or ketones is an olefination method extensively used in organic synthesis (Maryanoff &amp; Reitz, 1989; Kolodiazhnyi, 1999; Hoffmann, 2001; Rein &amp; Pedersen, 2002). The reactions of 4-substituted cyclohexanone with chiral Wittig or Horner–Wadsworth–Emmons (HWE) reagents are typical asymmetric Wittig reactions (Tomoskozi &amp; Janzso, 1962; Rein &amp; Reiser, 1996; Furuta &amp; Iwamura, 1994).

In the molecule of the title compound, (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987).Rings *A* (C1–C6) and *C* (C19–C24), with total puckering amplitudes,  $Q_T$ , of 0.564 (3) and 0.560 (4) Å, respectively, have chair conformations [ $\varphi = 160.12$  (2)° and  $\theta = 4.13$  (3)°, and  $\varphi = -144.71$  (1.7)° and  $\theta = 0.68$  (3)°] (Cremer & Pople, 1975). Ring *B* (C10–C15) is, of course, planar. The O2/C17–C19 unit is planar, with a maximum deviation of 0.0211 (3) Å (C17) from the least-squares plane.As can be seen from the packing diagram (Fig. 2), the molecules are extended along the *c* axis and stacked along the *a* axis. Dipole–dipole and van der Waals interactions are effective in the molecular packing.**Experimental**A solution of *n*-BuLi in hexane (0.39 ml, 0.624 mmol) was added to a suspension of (1*R*,2*S*,5*R*)-[5-methyl-2-(1-methyl-1-phenylethyl)cyclohexyloxycarbonylmethyl]triphenylarsonium bromide (411.0 mg, 0.625 mmol) in tetrahydrofuran (THF, 5 ml) and stirred for 30 min at 195 K under  $\text{N}_2$ ; a solution of 4-*tert*-cyclohexanone (77.1 mg,

0.50 mmol) in THF (1 ml) was then added. The temperature was raised to 258 K, and stirring continued for an additional 91 h. The reaction mixture was quenched with aqueous saturated  $\text{NH}_4\text{Cl}$  solution (5 ml) and extracted with ethyl acetate ( $3 \times 10$  ml); the combined organic layer was washed with brine, dried over anhydrous  $\text{MgSO}_4$  and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel, eluting with EtOAc in hexane (5:95) to afford (I) (yield 69%). The diastereomeric ratio of (I) was determined to be 86:14 by  $^1\text{H}$  NMR on a 600 MHz instrument (Dai *et al.*, 1997). It was recrystallized from hexane, giving colorless crystals suitable for X-ray diffraction analysis.

Crystal data

$\text{C}_{28}\text{H}_{42}\text{O}_2$	$D_x = 1.063 \text{ Mg m}^{-3}$
$M_r = 410.64$	Mo $K\alpha$ radiation
Tetragonal, $P4_1$	$\mu = 0.06 \text{ mm}^{-1}$
$a = 11.909 (4) \text{ \AA}$	$T = 298 (1) \text{ K}$
$c = 18.083 (4) \text{ \AA}$	Block, colorless
$V = 2564.5 (12) \text{ \AA}^3$	$0.42 \times 0.39 \times 0.36 \text{ mm}$
$Z = 4$	

Data collection

Rigaku R-AXIS RAPID-S diffractometer	5828 independent reflections
$\omega$ scans	2994 reflections with $F^2 > 2\sigma(F^2)$
Absorption correction: none	$R_{\text{int}} = 0.040$
24932 measured reflections	$\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on $F^2$	$\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$
$R[F^2 > 2\sigma(F^2)] = 0.033$	$\Delta\rho_{\text{min}} = -0.15 \text{ e \AA}^{-3}$
$wR(F^2) = 0.061$	Extinction correction: Larson (1970)
$S = 1.00$	Extinction coefficient: 768 (34)
5828 reflections	Absolute structure: Flack (1983), with 671 Friedel pairs
273 parameters	Flack parameter: 0.46 (8)
H-atom parameters constrained	
$w = 1/[0.7900\sigma(F_o^2)]/(4F_o^2)$	
$(\Delta/\sigma)_{\text{max}} < 0.001$	

The title compound was synthesized from 4-*tert*-butylcyclohexanone and chiral (1*R*,2*S*,5*R*)-5-methyl-2-(1-methyl-1-phenylethyl)cyclohexyl dimethoxyphosponylacetate but only a 36:64 diastereomer mixture was obtained (Furuta *et al.*, 1994). It has been found (Dai *et al.*, 1997) that the chiral arsonium ylide derived from (1*R*,2*S*,5*R*)-[5-methyl-2-(1-methyl-1-phenylethyl)cyclohexyloxycarbonylmethyl]-triphenylarsonium bromide could apparently improve the asymmetric induction and diastereoselectivity up to 86:14 was achieved. The absolute stereochemistry was assigned as *aR* by chemical correlation with 4-*tert*-butylcyclohexylideneethanol. H atoms were positioned geometrically, with C–H = 0.93, 0.98 0.97 and 0.96 Å for aromatic, methine, methylene and methyl H atoms, respectively, and constrained to ride on their parent atoms, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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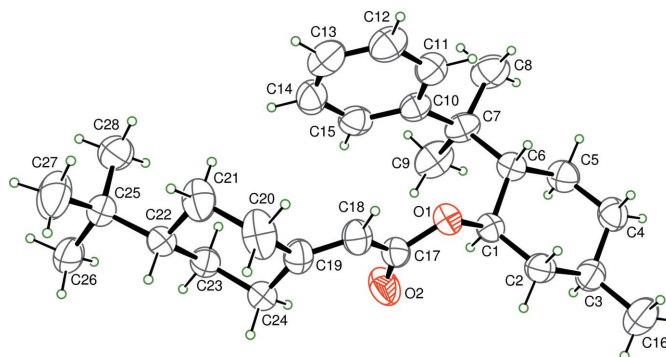


Figure 1 An ORTEP-3 (Farrugia, 1997) drawing of the molecular structure with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

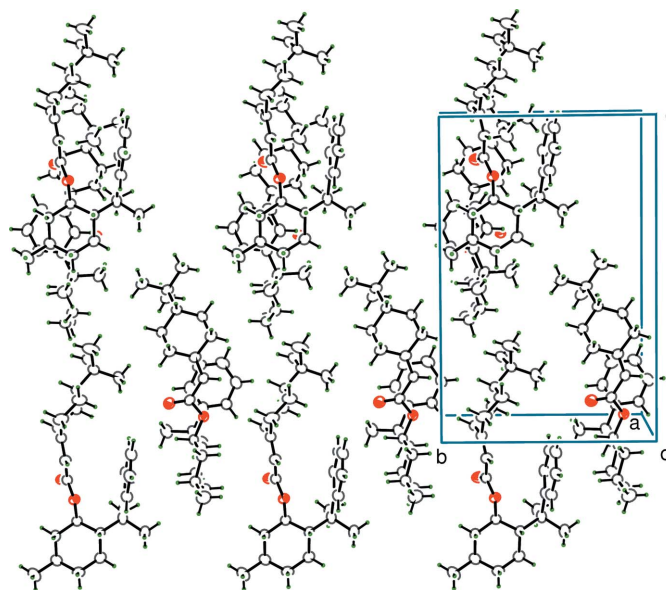


Figure 2 A packing diagram of (I).

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