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1-Chloro-2,2-dimethyl-1,3-diphenylpent-4-ene

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.055
wR factor = 0.113
Data-to-parameter ratio = 14.9

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

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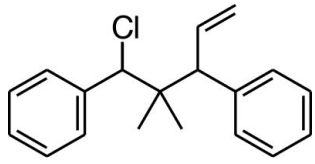
The title compound, $C_{19}H_{21}Cl$, exhibits a symmetrically arranged 1,3-diphenyl-2,2-dimethylpropyl entity, with a 1-chloro and a 3-vinyl substituent displaced on opposite sides of the plane defined by the three C atoms that bridge the two aromatic subunits.

Comment

1-Chloro-2,2-dimethyl-1,3-diphenyl-4-pentene, (I), was prepared by treatment of racemic 2,2-dimethyl-1,3-diphenyl-4-penten-1-ol with triphosgene and pyridine (Špehar, 2004). The identity of (I) was verified by X-ray diffraction analysis (Fig. 1).



(I)

The vinyl group and Cl atom are disordered over two positions in a 55.0 (3):45.0 (3) ratio. The two phenyl groups are pseudo-centrosymmetrically positioned relative to the 2,2-dimethylpropyl entity [C9—C8—C5—C4 = 98.7 (2)° and C4—C3—C14—C19 = 101.5 (2)°]. The chloro and vinyl substituents are displaced from the plane defined by atoms C3, C4 and C5 [Cl1—C5—C4—C3 = 51.3 (2)°, Cl1′—C3—C4—C5 = 57.5 (2)°, C2—C3—C4—C5 = 65.3 (7)° and C2′—C5—C4—C3 = 56.7 (6)]. The C—Cl bond lengths [Cl1—C5 = 1.747 (3) Å and Cl1′—C3 = 1.760 (3) Å] are shorter than the unweighted mean for the Csp^3—Cl bond [1.803 (3) Å for the substructure $C_2CH—Cl$] (Allen *et al.*, 1987). The olefinic double bonds [C1—C2 = 1.331 (13) Å and C1′—C2′ = 1.376 (12) A] are in reasonable agreement with the unweighted sample mean for the Csp^2═Csp^2 entity [1.30 (3) Å for the substructure C*CH═CH_2] (Allen *et al.*, 1987).

Experimental

Compound (I) was prepared by treatment of 1,3-diphenyl-2,2-dimethylpent-4-en-1-ol (38.5 mg, 0.15 mmol) with a mixture of triphosgene (22.0 mg, 0.72 mmol) and pyridine (58 mg, 0.72 mmol) for 1 h at 273 K (Ollivier & Renauld, 2000). Target compound (I) was isolated from the reaction mixture as follows. The solvent was distilled off. The residue was treated with EtOAc to afford a solid that was removed by filtration. The filtrate was concentrated under reduced pressure to furnish an oil, which was purified by column chromatography [SiO_2, *n*-hexane/Et_2O (1:1, *v*/*v*), $R_f = \frac{1}{2}$]. Yield 18 mg

(42%). Crystals suitable for X-ray analysis were obtained by slowly concentrating a saturated solution of (I) in Et_2O (m.p. 412–414 K). Analysis calculated for $C_{19}H_{21}Cl$: C 80.12, H 7.43%; found: C 80.32, H 7.56%; ^{1}H NMR (250 MHz, $CDCl_3$, p.p.m.): 0.72 (*s*, 3H), 0.85 (*s*, 3H), 3.85 (*d*, *J* = 10.4 Hz, 1H), 5.03 (*s*, 1H), 5.27 (*dd*, *J* = 10.1 and 2.2 Hz, 1H), 5.39 (*dd*, *J* = 16.8 and 1.8 Hz, 1H), 6.35 (*dt*, *J* = 16.8 and 10.1 Hz, 1H), 7.20–7.40 (m_C, 10H); ^{13}C NMR (63 MHz, $CDCl_3$, p.p.m.): 20.0, 21.0, 43.1, 57.0, 71.4, 118.7, 126.8, 128.2, 128.3, 129.7, 130.2, 137.1, 139.7, 141.8. MS [EI, 70 eV, *m*/*z* (%)]: 284 [M^+] (30), 249 (1.1), 132 (100), 117 (97), 91 (28).

Crystal data

$C_{19}H_{21}Cl$	D_x = 1.224 Mg m^{-3}
M_r = 284.81	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 25 reflections
a = 13.8829 (9) Å	θ = 10.0–25.0°
b = 7.2027 (5) Å	μ = 0.24 mm^{-1}
c = 15.4632 (10) Å	T = 100 (2) K
β = 92.142 (2)°	Prism, colourless
V = 1545.15 (18) Å^3	0.30 × 0.20 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	θ_{max} = 26.4°
$\omega/2\theta$ scans	$h = -17 \rightarrow 17$
Absorption correction: none	$k = 0 \rightarrow 9$
3134 measured reflections	$l = 0 \rightarrow 19$
3134 independent reflections	3 standard reflections
2604 reflections with $I > 2\sigma(I)$	every 100 reflections
	intensity decay: 0.3%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 1.1424P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.055	
$wR(F^2)$ = 0.113	
S = 1.13	$(\Delta/\sigma)_{max}$ = 0.001
3134 reflections	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
211 parameters	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
H-atom parameters constrained	

All H atoms were placed in geometrically idealized positions (C—H = 0.95–0.98 Å) and treated as riding, with U_{iso}(H) = 1.2U_{eq}(C). The occupancy factors of the vinyl group (atoms C1, C2, C1′ and C2′) and the Cl atom (Cl1 and Cl1′) refined to 0.550 (3) and 0.450 (3). The disorder of atoms, which mimics a pseudo-twofold axis, was restrained to provide the same bond lengths for corresponding pairs of atoms.

Data collection: *CAD-4 Diffractometer Control Software* (Enraf–Nonius, 1993); cell refinement: *CAD-4 Diffractometer Control Software*; data reduction: *CAD-4 Diffractometer Control Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON2002* (Spek, 2002) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

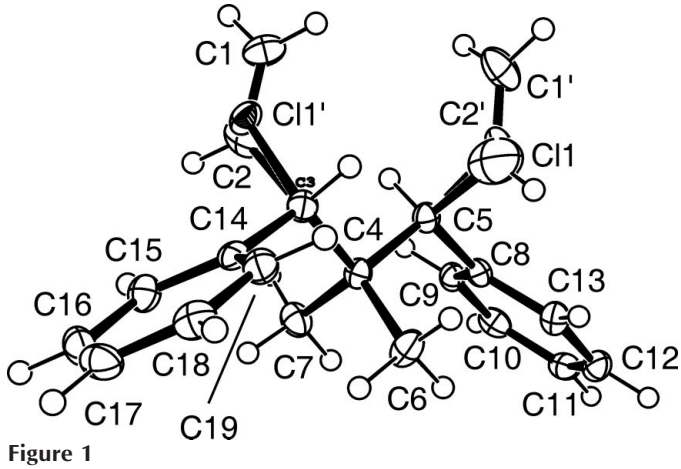


Figure 1
Molecular structure of (I), showing the disordered Cl atom and vinyl group. Atoms labelled with a prime (′) correspond to the minor disorder component. Displacement ellipsoids are plotted at the 50% probability level.

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