

Table 2. Selected geometric parameters (Å, °)

S1—C7	1.728 (4)	N1—C6	1.310 (5)
S1—C6	1.745 (4)	N1—C8	1.386 (5)
S2—C14	1.717 (4)	N2—C13	1.284 (5)
S2—C13	1.759 (4)	N2—C15	1.393 (5)
C7—S1—C6	89.3 (2)	C6—N1—C8	110.6 (3)
C14—S2—C13	88.8 (2)	C13—N2—C15	110.8 (3)

H atoms were added at calculated positions and refined with a common isotropic displacement parameter using a riding model.

Data collection: *DIF4* (Stoe & Cie, 1988a). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Cie, 1988b). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP92* (Vicković, 1994). Geometric calculations: *CSU* (Vicković, 1988). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: SK1029). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-(2-Chlorophenyl)-1-(2-naphthyl)-2-propen-1-one†

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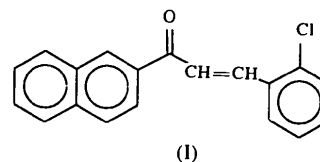
(Received 6 September 1995; accepted 6 December 1995)

Abstract

The carbonyl group in the α,β -unsaturated ketone system of the title compound, C₁₉H₁₃ClO, is in an *s-cis* conformation. The naphthyl ring is twisted by 27.68 (5)° with respect to the phenyl ring. The naphthoyl group is *trans* to the chlorophenyl group.

Comment

Chalcones (α,β -unsaturated ketones) derived from acetophenones are potential germicides, fungicides, bactericides and carcinogenics (Eaton & Davis, 1950; Misra, Kushwaha & Lal, 1970; Misra, Tewari & Nath, 1971; Geiger & Conn, 1945). The effectiveness of these compounds is associated with the tendency of substituents to withdraw electrons from the double bond, since saturation of the double bond by hydrogenation makes the compounds inactive. The substitution in benzene by a halogen enhances the activity (Kamoda, 1954). Even though greater anticancer properties have been found in many unsaturated ketones, the compounds with halogen atoms have higher cytotoxicity to leukemic cells. The structure of 3-(2-chlorophenyl)-1-(2-naphthyl)-2-propen-1-one, (I), is presented here.



The title molecule is found to have an *s-cis* conformation of the α,β -unsaturated ketone system as evidenced by the torsion angle O20—C11—C12—C13 of $-21.4(3)^\circ$ (Fig. 1). The torsion angle C2—C11—C12—C13 of $153.6(2)^\circ$ indicates that the ketone system is not planar.

The C=O bond distance of 1.222 (3) Å is in agreement with the values reported in the literature (Carpy,

† DCB contribution No. 869.

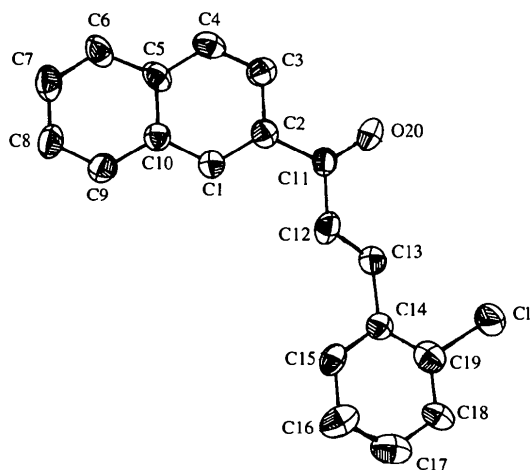


Fig. 1. Perspective view and labelling of the title molecule with displacement ellipsoids drawn at the 50% probability level.

Leger & Nuhlich, 1978; Zabel, Watson, Cassels & Langs, 1980). In this unsaturated ketone moiety, the angle C12—C13—C14 of 126.3 (2)° is larger than expected for an *sp*² hybridized C atom. This may be due to the intermolecular non-bonded interaction of the H12 and H15 atoms [2.21 (4) Å; Tokuno, Matsui, Miyoshi, Asao & Oshashi, 1986].

The Cl atom is close to the phenyl-ring plane [deviation -0.031 (1) Å]. The C—Cl distance of 1.751 (2) Å is larger than the average value of 1.720 (10) Å reported by Allen *et al.* (1987). This elongation may be attributed to the large thermal vibration of the Cl atom. The naphthyl group is twisted by 27.68 (5)° with respect to the phenyl ring.

The packing of the molecules, viewed down the *c* axis, is shown in Fig. 2. The naphthyl rings stack closer whereas the chlorophenyl rings point away from each other in the symmetry-related molecules. This type of packing creates a hollow channel down the *c* direction. The structure is stabilized by van der Waals interactions. The intermolecular short contact C12...O20 ($x, -y + \frac{3}{2}, z - \frac{1}{2}$) is 3.415 (3) Å.

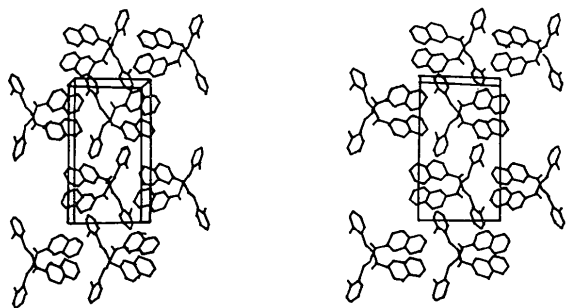


Fig. 2. Stereoview of the packing of the molecules viewed down the *c* axis; the *b* axis points upwards and the *a* axis to the right.

Experimental

The title compound was prepared according to Bonsignore, Cabiddu, Maccioni & Marogiu (1976) and crystallized by slow evaporation from a chloroform–methanol mixture. The density D_m was measured by flotation in benzene/bromoform solution.

Crystal data

C₁₉H₁₃ClO
 $M_r = 292.77$
 Monoclinic
*P*2₁/*c*
 $a = 10.710$ (1) Å
 $b = 16.485$ (2) Å
 $c = 9.124$ (1) Å
 $\beta = 113.59$ (1)°
 $V = 1476.3$ (3) Å³
 $Z = 4$
 $D_x = 1.32$ Mg m⁻³
 $D_m = 1.31$ Mg m⁻³

Cu $K\alpha$ radiation
 $\lambda = 1.5418$ Å
 Cell parameters from 21 reflections
 $\theta = 10$ –22°
 $\mu = 2.3$ mm⁻¹
 $T = 293$ K
 Transparent plate
 0.22 × 0.20 × 0.15 mm
 Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
 $T_{\min} = 0.845$, $T_{\max} = 0.998$
 2777 measured reflections
 2705 independent reflections
 2507 observed reflections
 $[F \geq 6\sigma(F)]$

$R_{\text{int}} = 0.017$
 $\theta_{\text{max}} = 70^\circ$
 $h = -13 \rightarrow 13$
 $k = 0 \rightarrow 20$
 $l = 0 \rightarrow 11$
 3 standard reflections monitored every 100 reflections
 intensity decay: <1%

Refinement

Refinement on F
 $R = 0.055$
 $wR = 0.076$
 $S = 1.30$
 2507 reflections
 242 parameters
 All H-atom parameters refined
 $w = 1/[\sigma^2(F_o) + 0.0063F_o^2]$

$(\Delta/\sigma)_{\text{max}} = 0.01$
 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.38$ e Å⁻³
 Extinction correction: none
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C1	0.7177 (2)	0.7766 (1)	-0.04320 (2)	0.0400 (6)
C2	0.6470 (2)	0.8234 (1)	0.02310 (2)	0.0420 (6)
C3	0.7042 (2)	0.8986 (1)	0.09600 (2)	0.0633 (7)
C4	0.8295 (2)	0.9222 (1)	0.10750 (2)	0.0484 (6)
C5	0.9062 (2)	0.8731 (1)	0.04880 (2)	0.0422 (6)
C6	1.0382 (2)	0.8944 (1)	0.05890 (3)	0.0544 (7)
C7	1.1075 (2)	0.8465 (1)	-0.00530 (3)	0.0602 (8)
C8	1.0475 (2)	0.7761 (1)	-0.08800 (3)	0.0588 (8)
C9	0.9209 (2)	0.7520 (1)	-0.10180 (2)	0.0492 (7)
C10	0.8471 (2)	0.8002 (1)	-0.03440 (2)	0.0401 (6)
C11	0.5179 (2)	0.7959 (1)	0.03280 (2)	0.0442 (6)

C12	0.4610 (2)	0.7161 (1)	-0.03500 (2)	0.0504 (7)
C13	0.3834 (2)	0.6750 (1)	0.01930 (2)	0.0446 (6)
C14	0.3286 (2)	0.5931 (1)	-0.03040 (2)	0.0436 (6)
C15	0.3618 (2)	0.5473 (1)	-0.13910 (2)	0.0501 (7)
C16	0.3114 (2)	0.4704 (1)	-0.18420 (3)	0.0582 (8)
C17	0.2238 (3)	0.4362 (1)	-0.12390 (3)	0.0630 (8)
C18	0.1893 (2)	0.4797 (1)	-0.01510 (3)	0.0610 (8)
C19	0.2426 (2)	0.5558 (1)	0.03080 (2)	0.0518 (7)
O20	0.4658 (2)	0.8365 (1)	0.10530 (2)	0.0638 (7)
Cl	0.1966 (1)	0.6067 (1)	0.17020 (1)	0.0808 (3)

Table 2. Geometric parameters (\AA , $^\circ$)

C1—C2	1.379 (3)	C11—C12	1.477 (2)
C1—C10	1.410 (3)	C11—O20	1.222 (3)
C2—C3	1.424 (2)	C12—C13	1.313 (3)
C2—C11	1.491 (3)	C13—C14	1.470 (2)
C3—C4	1.360 (3)	C14—C15	1.401 (3)
C4—C5	1.403 (3)	C14—C19	1.396 (3)
C5—C6	1.423 (3)	C15—C16	1.375 (2)
C5—C10	1.427 (2)	C16—C17	1.383 (4)
C6—C7	1.366 (4)	C17—C18	1.389 (4)
C7—C8	1.393 (2)	C18—C19	1.372 (2)
C8—C9	1.369 (3)	C19—Cl	1.751 (2)
C9—C10	1.422 (3)		
C2—C1—C10	121.3 (2)	C2—C11—O20	120.1 (2)
C1—C2—C11	122.7 (2)	C2—C11—C12	118.9 (2)
C1—C2—C3	118.9 (2)	C12—C11—O20	120.9 (2)
C3—C2—C11	118.2 (2)	C11—C12—C13	121.2 (2)
C2—C3—C4	120.8 (2)	C12—C13—C14	126.3 (2)
C3—C4—C5	121.0 (2)	C13—C14—C19	121.8 (2)
C4—C5—C10	119.1 (2)	C13—C14—C15	122.0 (2)
C4—C5—C6	123.4 (2)	C15—C14—C19	116.1 (2)
C6—C5—C10	117.4 (2)	C14—C15—C16	121.9 (2)
C5—C6—C7	121.7 (2)	C15—C16—C17	120.3 (2)
C6—C7—C8	120.1 (2)	C16—C17—C18	119.4 (2)
C7—C8—C9	121.1 (2)	C17—C18—C19	119.5 (2)
C8—C9—C10	119.9 (2)	C14—C19—C18	122.7 (2)
C5—C10—C9	119.7 (2)	C18—C19—Cl	117.1 (2)
C1—C10—C9	121.6 (2)	C14—C19—Cl	120.1 (1)
C1—C10—C5	118.6 (2)		
O20—C11—C12—C13	-21.4 (3)	C11—C12—C13—C14	-175.6 (2)
C2—C11—C12—C13	153.6 (2)		

H atoms were located in ΔF maps.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *SDP* (Frenz, 1978). Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL76* (Sheldrick, 1976). Molecular graphics: *ORTEPII* (Johnson, 1976), *PLUTO* (Motherwell & Clegg, 1978). Geometrical calculations: *PARST* (Nardelli, 1983).

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Lists of structure factors, least-squares-planes data, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: AB1316). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$(\mu_2$ -Pinacolato-*O,O'*)-bis(pinacolato-*O,O'*)-diboron

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Abstract

The title compound, 2,2'-(2,3-dimethyl-2,3-butanediyl-dioxy)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane, $\text{C}_{18}\text{H}_{36}\text{B}_2\text{O}_6$, B_2pin_3), has two trigonal planar B atoms, each chelated by one pinacolato group, linked by a third pinacolato. The molecule is centrosymmetric, with an exactly *anti* conformation for the bridging pinacolato, and the two Bpin five-membered rings have a twist conformation with torsion angle $\text{O—C—C—O} =$