

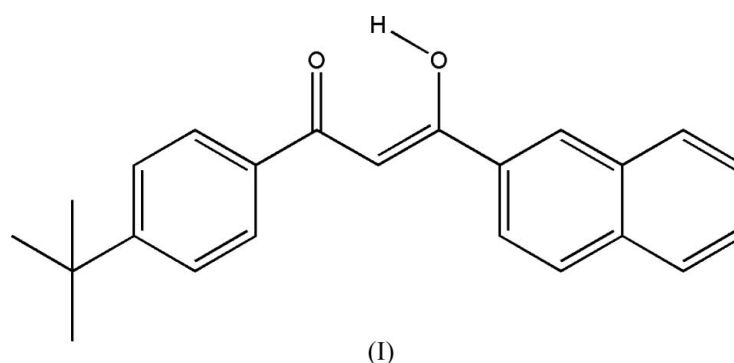
1-(4-*tert*-Butylphenyl)-3-hydroxy-3-(2-naphthyl)-prop-2-en-1-oneDun-Jia Wang,^{a*} Chun-Yang Zheng^b and Ling Fan^b^aHubei Key Laboratory of Bioanalytical Technique, Department of Chemistry and Environmental Engineering, Hubei Normal University, Huangshi 435002, People's Republic of China, and ^bCollege of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China

Correspondence e-mail: dunjiawang@163.com

Key indicators

Single-crystal X-ray study
T = 292 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.061
wR factor = 0.164
Data-to-parameter ratio = 17.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The crystal structure of the title compound, $\text{C}_{23}\text{H}_{22}\text{O}_2$, which exists in the enol form, is stabilized by an intramolecular hydrogen bond. The dihedral angle between the naphthyl ring system and the aromatic ring of the *t*- BuC_6H_4 group is $23.50 (10)^\circ$.Received 4 July 2006
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Comment

1,3-Diketones have long been widely employed as effective chelating reagents for a large number of metallic systems (Melby *et al.*, 1964; Liang *et al.*, 2003). Molecular crystals of 1,3-diketones are found to comprise enol tautomers stabilized by a strong intramolecular hydrogen bond (Vila *et al.*, 1991). The structure of the title 1,3-diketone derivative, (I), is presented here (Fig. 1).The geometric data (Table 1) are in agreement with reported literature values (Bertolasi *et al.*, 1991; Gilli *et al.*, 2004). The dihedral angle between the naphthyl ring system and the aromatic ring of the *t*- BuC_6H_4 group is $23.50 (10)^\circ$. The enol form found in (I) is stabilized by an intramolecular hydrogen bond; details are presented in Table 2.

Experimental

2-Acetylnaphthalene (6.80 g, 0.04 mol), methyl 4-*tert*-butylbenzoate (7.68 g, 0.04 mol), NaNH_2 (1.95 g, 0.05 mol) and toluene (50 ml) were placed in a three-necked, round-bottom flask. The mixture was heated with stirring to 353 K and maintained there for 6 h under a nitrogen atmosphere. The reaction mixture was then cooled to room temperature and acidified with dilute hydrochloric acid; stirring was continued until all solids had dissolved. The toluene layer was separated and washed with a saturated NaHCO_3 solution, dried over anhydrous Na_2SO_4 and the solvent removed by evaporation. The residual oil solidified on standing and was recrystallized from an ethanol solution to give the title compound (I) (yield 8.58 g, 65.0%; m.p. 395 K). Crystals suitable for X-ray diffraction were grown by slow evaporation of a CH_2Cl_2 -EtOH (2:1) solution at room

temperature. Calculated for $C_{23}H_{22}O_2$: C 83.64, H 6.67%; found: C 83.70, H 6.69%. IR (KBr, ν cm^{-1}): 1598 (C=O), 1525 (C=C), 2961 (C–H, alkyl).

Crystal data

$C_{23}H_{22}O_2$
 $M_r = 330.41$
 Monoclinic, $C2/c$
 $a = 32.490$ (4) Å
 $b = 6.2231$ (8) Å
 $c = 22.533$ (3) Å
 $\beta = 128.605$ (2)°
 $V = 3560.2$ (8) Å³

$Z = 8$
 $D_x = 1.233$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 292$ (2) K
 Block, colorless
 0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.977$, $T_{max} = 0.982$

15619 measured reflections
 4055 independent reflections
 2810 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.101$
 $\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.164$
 $S = 1.02$
 4055 reflections
 234 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0858P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.22$ e Å⁻³
 $\Delta\rho_{min} = -0.25$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

| | | | |
|----------------|-------------|----------------|-------------|
| O1–C11 | 1.2881 (19) | C11–C12 | 1.397 (2) |
| O2–C13 | 1.2908 (18) | C12–C13 | 1.393 (2) |
| O1–C11–C12 | 119.92 (14) | O2–C13–C12 | 120.04 (15) |
| C11–C12–C13 | 120.70 (14) | | |
| O1–C11–C12–C13 | 4.6 (2) | C11–C12–C13–O2 | −5.6 (2) |

Table 2

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------|----------|-------------|-------------|---------------|
| O2–H1 \cdots O1 | 1.14 (3) | 1.36 (3) | 2.4441 (17) | 156 (2) |

The C-bound H atoms were included in the riding-model approximation, with C–H = 0.93–0.97 Å and $U_{iso}(H) = 1.2$ (1.5 for

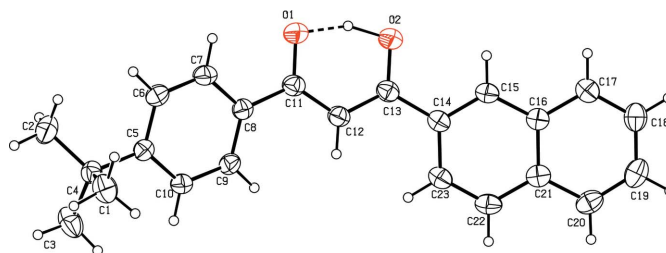


Figure 1

View of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. The dashed line indicates a hydrogen bond.

methyl) times $U_{eq}(C)$. The O-bound H atom was refined freely; see Table 2 for geometrical data.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2001); software used to prepare material for publication: SHELXTL.

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References

- Bertolasi, V., Cilli, P., Ferretti, V. & Gilli, G. (1991). *J. Am. Chem. Soc.* **113**, 4917–4925.
 Bruker (1997). SMART. Version 5.054. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (1999). SAINT. Version 6.01. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (2001). SHELXTL. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
 Gilli, P., Bertolasi, V., Pretto, L., Ferretti, V. & Gilli, G. (2004). *J. Am. Chem. Soc.* **126**, 3845–3855.
 Liang, F. S., Zhou, Q. G., Cheng, Y. X., Wang, L. X., Ma, D. G., Jing, X. B. & Wang, F. S. (2003). *Chem. Mater.* **15**, 1935–1937.
 Melby, L. R., Rose, N. J., Abramson, E. & Caris, J. C. (1964). *J. Am. Chem. Soc.* **86**, 5117–5125.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Vila, A. J., Lagier, C. M. & Olivieri, A. C. (1991). *J. Phys. Chem.* **95**, 5069–5073.