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1-Acetamido-1-(1-naphthyl)ethylene

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.154
Data-to-parameter ratio = 18.7

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

The crystal structure and synthesis of 1-acetamido-1-(1-naphthyl)ethylene, $C_{14}H_{13}NO$, are reported.

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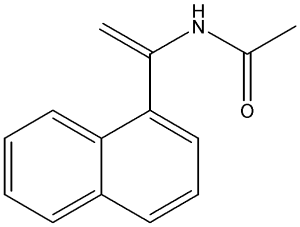
Comment

Enamides have been extensively studied for practical use as prochiral materials for the asymmetric synthesis of chiral amines, which can be used as resolving reagents, chiral auxiliaries and intermediates for the synthesis of many biologically active substances (Burk *et al.*, 1996; Noyori *et al.*, 1986; Kitamura *et al.*, 1994; Tschaen *et al.*, 1995; Meth-Cohn & Westwood, 1984; Mpango *et al.*, 1980; Baldwin & DuPont, 1980). Herein, we report the crystal structure of an enamide, 1-acetamido-1-(1-naphthyl)ethylene, (I). We have been using it in our laboratory as the substrate for the synthesis of the corresponding optically active amide through catalytic hydrogenation.



(I)

Experimental

The title compound was prepared according to the literature method of Burk *et al.* (1998). Acetic anhydride (8.39 g, 82.3 mmol), followed by acetic acid (4.94 g, 82.3 mmol), was added to a solution of 1-acetonaphthone oxime (5.07 g, 27.4 mmol) in toluene (40 ml) under a nitrogen atmosphere. Fe powder (3.08 g, 55 mmol) was then added and the mixture was heated to 343 K for 8 h. The reaction mixture was then cooled to room temperature and filtered through celite to remove solid residues, which were then washed with toluene (2 × 5 ml). The combined filtrates were diluted with dichloromethane (50 ml) and the mixture cooled in an ice-bath and washed with 2 *M* NaOH (2 × 30 ml). The organic phase was separated, dried over Na_2SO_4, and evaporated to a volume of 30 ml, the solution was cooled to room temperature and then the desired enamide was precipitated, After filtration, followed by recrystallization from hexane and ethyl acetate, crystals were obtained.

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Crystal data

$C_{14}H_{13}NO$
$M_r = 211.25$
Orthorhombic, *Pbca*
$a = 11.2035$ (15) Å
$b = 9.2977$ (13) Å
$c = 22.738$ (3) Å
$V = 2368.6$ (6) Å^3
$Z = 8$
$D_x = 1.185$ Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 3813 reflections
$\theta = 1–27.5°$
$\mu = 0.08$ mm^{-1}
$T = 294$ (2) K
Block, colorless
0.30 × 0.16 × 0.14 mm

Data collection

Bruker CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.978$, $T_{\max} = 0.990$
15120 measured reflections

2715 independent reflections
1200 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.047$
$\theta_{\max} = 27.6°$
$h = -14 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -27 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.154$
$S = 1.00$
2715 reflections
145 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.07P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1$^{\rm i}$	0.93	1.87	2.7931 (19)	177

Symmetry code: (i) $\frac{1}{2} - x, y - \frac{1}{2}, z$.

The C-bound H atoms were placed in geometrically calculated positions and included in the final refinement in the riding-model approximation.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995) and *SHELXTL-NT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-NT*; software used to prepare material for publication: *SHELXTL-NT*.

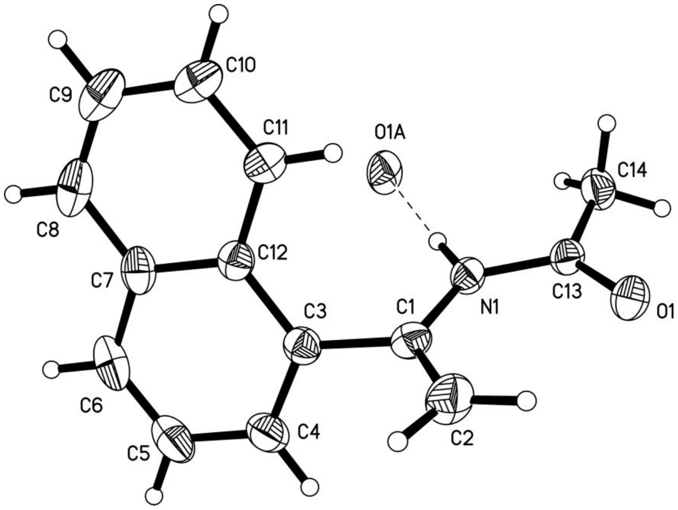

Figure 1
The molecular structure of (I), with ellipsoids at the 30% probability level (Siemens, 1995).

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