Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

N-Benzyl-*N*-(4-methoxyphenyl)cyclohex-1-enecarbothioamide

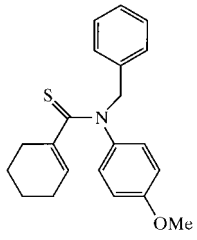
Hiroyuki Hosomi,[a] Shigeru Ohba[a]* and Hiromu Aoyama[b]

[a]Department of Chemistry, Faculty of Science and Technology, Keio University, Hiyoshi 3-14-1, Kohoku-ku, Yokohama 223-8522, Japan, and [b]Department of Material Chemistry, Faculty of Textile Science and Technology, Shinshu University, Tokida 3-15-1, Ueda 386-0081, Japan
Correspondence e-mail: ohba@chem.keio.ac.jp

Received 3 March 2000
Accepted 15 March 2000

Data validation number: IUC0000081

The crystal structure of the title thioamide, $C_{21}H_{23}NOS$, was determined to investigate the relationship between the photostability in the solid state and the structure.



(I)

Experimental

The title compound, (I), was prepared by one of the authors (HA) in a study on the photocyclization of enamides and thioamides in the solid state (Aoyama, 2000). Crystals were grown from a hexane solution.

Crystal data

$C_{21}H_{23}NOS$
M_r = 337.48
Orthorhombic, *Pbca*
a = 10.880 (2) Å
b = 16.458 (2) Å
c = 21.261 (2) Å
V = 3807.1 (8) Å^3
Z = 8
D_x = 1.177 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 28.5–29.6°
μ = 1.544 mm^{-1}
T = 248 (1) K
Sphere, pale yellow
0.25 mm (radius)

Data collection

Rigaku AFC-7*R* diffractometer
θ–2θ scans
Absorption correction: spherical (*International Tables for X-ray Crystallography*)
T_{min} = 0.566, T_{max} = 0.598
4142 measured reflections
3630 independent reflections
2723 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 75.0°
$h = -14 \rightarrow 0$
$k = 0 \rightarrow 21$
$l = -13 \rightarrow 27$
3 standard reflections
every 150 reflections
intensity decay: 1.6%

Refinement

Refinement on F^2
$R(F)$ = 0.062
$wR(F^2)$ = 0.168
S = 1.24
3630 reflections
217 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 2.0P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected geometric parameters (Å).

S1—C4	1.672 (3)	C6—C7	1.479 (11)
N3—C4	1.338 (4)	C7—C8	1.366 (13)
C4—C5	1.487 (4)	C8—C9	1.443 (9)
C5—C6	1.481 (5)	C9—C10	1.475 (6)
C5—C10	1.351 (5)		

X-ray intensity data were measured for $-h,+k,+l$ ($\theta < 75°$) and for $-h,+k,-l$ ($\theta < 30°$). The completeness of symmetry unique reflections ($\theta < 75°$) was 92.7%, which was due to the blind region of the low-temperature apparatus. All H-atom positional parameters were calculated geometrically and fixed with U_{iso}(H) = 1.2U_{eq}(parent atom). For the cyclohexene ring, the C5—C6 and C5═C10 axes were assigned to be single and double bonds, respectively, based on the bond lengths [1.481 (5) and 1.351 (5) Å, respectively]. The short C7—C8 bond length of 1.366 (13) Å may be an artifact due to the conformational disorder of the six-membered ring.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *TEXSAN*.

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Aoyama, H. (2000). To be published.
Molecular Structure Corporation (1993). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Molecular Structure Corporation (1999). *TEXSAN*. Version 1.10. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.