electronic papers

N-Isopropyl-N-[(E)-2-phenylpropenyl]-thiobenzamide and N-isopropyl-3-methoxy-N-[(E)-2-phenylpropenyl]-thiobenzamide

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The crystal structures of the two title thiobenzamides, $C_{19}H_{21}NS$, (I), and $C_{20}H_{23}NOS$, (II), were determined to investigate the relationship between the photoreactivity in solid state and the structure. Their geometry was confirmed to be the E isomer in each case.

(I) R = H
(II) R = OMe
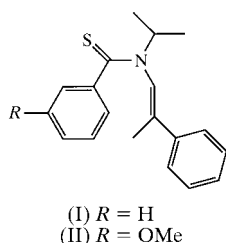
Experimental

The title compounds, (I) and (II), were prepared by one of the authors (HA) in a study on photocyclization of enamides and thio-amides in the solid state (Aoyama, 2000). Crystals were grown from hexane solutions.

Compound (I)

Crystal data

$C_{19}H_{21}NS$	$D_x = 1.173$ Mg m^{-3}
$M_r = 295.44$	Cu $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 25
$a = 17.479 (3)$ Å	reflections
$b = 9.100 (2)$ Å	$\theta = 29.4$–30.0°
$c = 21.494 (3)$ Å	$\mu = 1.641$ mm^{-1}
$\beta = 101.94 (1)°$	$T = 248 (1)$ K
$V = 3344.7 (11)$ Å^3	Plate-like, yellow
$Z = 8$	$0.6 \times 0.4 \times 0.3$ mm

Data collection

Rigaku AFC-7R diffractometer	$R_{int} = 0.055$
θ–2θ scans	$\theta_{max} = 75°$
Absorption correction: by integra-tion (Coppens *et al.*, 1965)	$h = -22 \rightarrow 22$
	$k = -5 \rightarrow 11$
$T_{min} = 0.459$, $T_{max} = 0.651$	$l = -27 \rightarrow 0$
3812 measured reflections	3 standard reflections
3210 independent reflections	every 150 reflections
2923 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0773P)^2$
$R(F) = 0.046$	$+ 2.6174P]$
$wR(F^2) = 0.134$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.001$
3210 reflections	$\Delta\rho_{max} = 0.30$ e Å^{-3}
274 parameters	$\Delta\rho_{min} = -0.42$ e Å^{-3}
All H-atom parameters refined	

Table 1
Selected geometric parameters (Å) for (I).

S1—C3	1.666 (2)	N2—C13	1.436 (2)
N2—C3	1.348 (2)	C13—C14	1.329 (2)

Compound (II)

Crystal data

$C_{20}H_{23}NOS$	$D_x = 1.169$ Mg m^{-3}
$M_r = 325.47$	Cu $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 25
$a = 19.483 (1)$ Å	reflections
$b = 9.111 (1)$ Å	$\theta = 27.3$–29.4°
$c = 21.611 (2)$ Å	$\mu = 1.570$ mm^{-1}
$\beta = 105.39 (1)°$	$T = 248 (1)$ K
$V = 3698.7 (6)$ Å^3	Prism, yellow
$Z = 8$	$0.4 \times 0.3 \times 0.3$ mm

Data collection

Rigaku AFC-7R diffractometer	$R_{int} = 0.028$
θ–2θ scans	$\theta_{max} = 75°$
Absorption correction: by integra-tion (Coppens *et al.*, 1965)	$h = -24 \rightarrow 0$
	$k = -5 \rightarrow 11$
$T_{min} = 0.546$, $T_{max} = 0.689$	$l = -27 \rightarrow 27$
4076 measured reflections	3 standard reflections
3539 independent reflections	every 150 reflections
3121 reflections with $I > 2\sigma(I)$	intensity decay: 1.0%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2$
$R(F) = 0.035$	$+ 2.4391P]$
$wR(F^2) = 0.101$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
3539 reflections	$\Delta\rho_{max} = 0.24$ e Å^{-3}
301 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
All H-atom parameters refined	Extinction correction: *SHELXL*97 (Sheldrick, 1997)
	Extinction coefficient: 0.00076 (7)

Table 2
Selected geometric parameters (Å) for (II).

S1—C4	1.671 (1)	N3—C15	1.435 (2)
N3—C4	1.345 (2)	C15—C16	1.329 (2)

X-ray intensity data were measured for $\pm h, +k, -l$ ($\theta < 75°$) and $\pm h, -k, -l$ ($\theta < 30°$) of (I), and for $-h, +k, \pm l$ ($\theta < 75°$) and $-h, -k, \pm l$ ($\theta < 30°$) of (II). The completeness of symmetry unique reflections ($\theta < 75°$) was 92.7% for both (I) and (II), which was due to the blind region of the low-temperature apparatus. All H atoms were located from difference syntheses and refined isotropically. The C—H bond distances are 0.92 (3)–1.13 (3) Å in (I) and 0.89 (2)–1.01 (3) Å in (II).

For both compounds, 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.
Coppens, P., Leiserowitz, L. & Rabinovich, D. (1965). *Acta Cryst.* **18**, 1035–1038.
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.