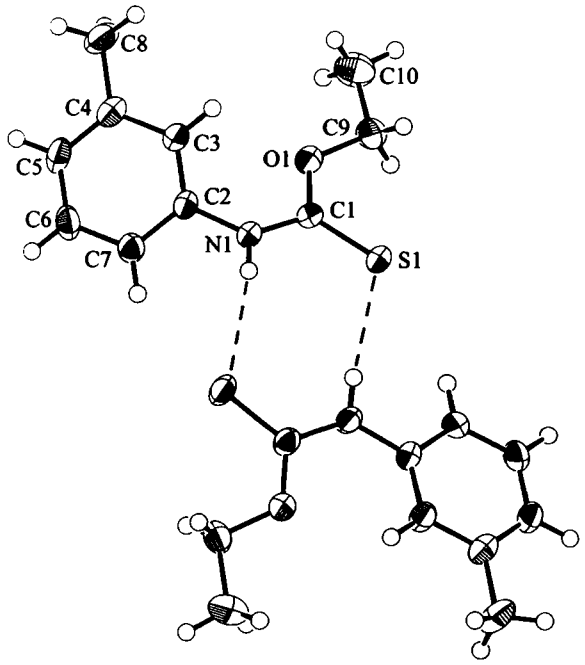
Crystal structure of *o*-ethyl *N*-(*m*-tolyl)thiocarbamate, $SC(OC_2H_5)NH(C_6H_4CH_3)$

P. Tadbuppa[I] and E. R. T. Tiekink*[,II]

[I] National University of Singapore, Department of Chemistry, Singapore 117543
[II] Griffith University, School of Science, Nathan, Queensland 4111, Australia

Received May 24, 2005, accepted and available on-line July 17, 2005; CCDC no. 1267/1561



Abstract

$C_{10}H_{13}NOS$, monoclinic, *C*12/*c*1 (no. 15), $a = 17.975(2)$ Å, $b = 7.2265(9)$ Å, $c = 17.343(2)$ Å, $\beta = 110.687(3)°$, $V = 2107.6$ Å^3, $Z = 8$, $R_{gt}(F) = 0.054$, $wR_{ref}(F^2) = 0.151$, $T = 223$ K.

Source of material

The title compound was prepared from the reaction between ethanol and *m*-tolylthiocyanate using a literature procedure [1]. Crystals were obtained by the slow evaporation of a chloroform/hexane (1:1) solution of the compound (m.p. 337-339 K).

Discussion

Consistent with related structures [1-3], the central COC(=S)NC chromophore in S=C(OEt)N(H)(C_6H_4Me-3) is essentially planar as seen in the S1/C1/N1/C2 and N1/C1/O1/C9 torsion angles of −178.5(1)° and −179.6(2)°, respectively, and the aromatic ring is twisted with respect to this plane as the C1/N1/C2/C3 torsion angle is −32.6(3)°. The key geometric parameters *d*(S1=C1), *d*(N1—C1) and *d*(N1—C2) are 1.672(2) Å, 1.337(2) Å and 1.426(2) Å, respectively. Again consistent with related structures [1-3], molecules associate via N–H···S interactions with $d(H\cdots S^i) = 2.58$ Å, $d(N1\cdots S1^i) = 3.414(2)$ Å and an angle of 160° subtended at the H atom for symmetry operation i: $-x,y,½-z$.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.10 × 0.34 × 0.52 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	2.68 cm^{-1}
Diffractometer, scan mode:	Bruker AXS SMART CCD, ω
$2\theta_{max}$:	60°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8456, 3065
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2305
$N(param)_{refined}$:	119
Programs:	DIRDIF92 [4], SHELXL-97 [5], ORTEP-II [6], teXsan [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*f*	0.0890	0.1577	0.2709	0.043
H(3)	8*f*	0.2466	0.1489	0.2022	0.043
H(5)	8*f*	0.4090	0.3113	0.4188	0.051
H(6)	8*f*	0.3087	0.3588	0.4711	0.051
H(7)	8*f*	0.1764	0.3077	0.3890	0.044
H(8A)	8*f*	0.3881	0.0943	0.2304	0.079
H(8B)	8*f*	0.4521	0.1603	0.3150	0.079
H(8C)	8*f*	0.4122	0.3059	0.2442	0.079
H(9A)	8*f*	0.0448	0.3780	0.0156	0.059
H(9B)	8*f*	0.0577	0.1636	0.0037	0.059
H(10A)	8*f*	0.1273	0.3383	−0.0616	0.095
H(10B)	8*f*	0.1883	0.2256	0.0121	0.095
H(10C)	8*f*	0.1744	0.4391	0.0226	0.095

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	8*f*	−0.02143(2)	0.16541(9)	0.12017(3)	0.0227(2)	0.0941(4)	0.0336(2)	−0.0003(2)	0.0072(2)	−0.0104(2)
O(1)	8*f*	0.12089(7)	0.2566(2)	0.11752(7)	0.0298(6)	0.0546(8)	0.0321(6)	0.0007(5)	0.0104(5)	0.0015(5)
N(1)	8*f*	0.11701(8)	0.1925(2)	0.24151(8)	0.0233(6)	0.0512(8)	0.0314(7)	−0.0025(5)	0.0086(5)	−0.0055(6)

* Correspondence author (e-mail: etiekink@deakin.edu.au)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	8*f*	0.07629(9)	0.2066(2)	0.1607(1)	0.0260(7)	0.0448(9)	0.0325(8)	0.0031(6)	0.0100(6)	–0.0062(6)
C(2)	8*f*	0.19906(9)	0.2254(2)	0.2871(1)	0.0242(7)	0.0375(8)	0.0314(7)	–0.0011(6)	0.0052(6)	–0.0006(6)
C(3)	8*f*	0.25923(9)	0.1932(2)	0.2563(1)	0.0259(7)	0.0430(9)	0.0356(8)	–0.0002(6)	0.0079(6)	–0.0063(6)
C(4)	8*f*	0.3383(1)	0.2260(2)	0.3048(1)	0.0267(8)	0.0392(9)	0.047(1)	–0.0007(6)	0.0107(7)	0.0002(7)
C(5)	8*f*	0.3559(1)	0.2887(3)	0.3854(1)	0.0270(8)	0.052(1)	0.0387(9)	–0.0081(7)	0.0012(7)	0.0048(7)
C(6)	8*f*	0.2960(1)	0.3178(3)	0.4165(1)	0.0390(9)	0.057(1)	0.0256(7)	–0.0098(8)	0.0036(7)	0.0017(7)
C(7)	8*f*	0.2171(1)	0.2871(2)	0.3678(1)	0.0323(8)	0.049(1)	0.0286(8)	–0.0046(7)	0.0094(6)	0.0030(7)
C(8)	8*f*	0.4035(1)	0.1938(3)	0.2705(2)	0.0293(9)	0.062(1)	0.069(1)	–0.0016(8)	0.0190(9)	–0.011(1)
C(9)	8*f*	0.0843(1)	0.2784(3)	0.0289(1)	0.043(1)	0.073(1)	0.0312(9)	0.0113(9)	0.0115(7)	0.0071(8)
C(10)	8*f*	0.1492(2)	0.3243(4)	–0.0022(2)	0.067(2)	0.079(2)	0.054(1)	–0.005(1)	0.034(1)	0.007(1)

Acknowledgments. The Queensland Government, for the award of a Smart Returns Fellowship (ERTT), and the National University of Singapore (grant no. R-143-000-213-112) are thanked for support.

References

1. Taylor, R. L.; Tiekink, E. R. T.: Crystal and molecular structure of *o*-ethyl-*N*-phenylthiocarbamate. Z. Kristallogr. **209** (1994) 64-67.
2. Ho, S. Y.; Lai, C. S.; Tiekink, E. R. T.: *o*-Methyl *N*-phenylthiocarbamate. Acta Crystallogr. **E59** (2003) o1155-o1156.
3. Kuan, F. S.; Tadbuppa P.; Tiekink, E. R. T.: Crystal structure of *o*-methyl *N*-(*o*-tolyl)thiocarbamate, $SC(OCH_3)NH(C_6H_4CH_3)$. Z. Kristallogr. NCS **220** (2005) 393-394.
4. Beurskens, P. T.; Admiraal, G.; Beurskens, G.; Bosman, W. P.; García-Granda, S.; Smits, J. M. M.; Smykalla, C.: The DIRDIF program system. Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands 1992.
5. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
6. Johnson, C. K.: ORTEP-II. A Fortran Thermal-Ellipsoid Plot Program. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA 1976.
7. Molecular Structure Corporation: teXsan. Single Crystal Structure Analysis Software. Version 1.05. MSC, The Woodlands, Texas, USA 1997.