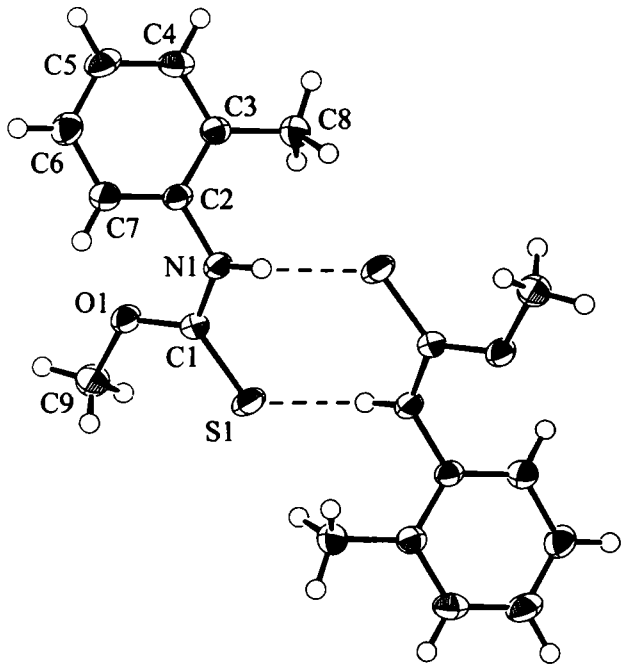
Crystal structure of *o*-methyl *N*-(*o*-tolyl)thiocarbamate, $SC(OCH_3)NH(C_6H_4CH_3)$

F. S. Kuan[I], 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. 1267/1560



Abstract

$C_9H_{11}NOS$, triclinic, $P\bar{1}$ (no. 2), $a = 6.887(2)$ Å, $b = 8.251(2)$ Å, $c = 9.419(2)$ Å, $\alpha = 114.180(5)°$, $\beta = 101.572(5)°$, $\gamma = 98.849(5)°$, $V = 461.4$ Å^3, $Z = 2$, $R_{gt}(F) = 0.045$, $wR_{ref}(F^2) = 0.140$, $T = 223$ K.

Source of material

The title compound was prepared from the reaction between methanol and *o*-tolylthiocyanate using a literature procedure [1]. Crystals were obtained by the slow evaporation of an ether/hexane (1:2) solution of the compound (m.p. 345-347 K).

Discussion

The central COC(=S)NC chromophore in the structure of S=C(OMe)N(H)(C_6H_4Me-2) is essentially planar as evidenced by the S1/C1/N1/C2 and N1/C1/O1/C9 torsion angles of −177.9(1)° and 176.1(1)°, respectively. However, the C1/N1/C2/C3 torsion angle of −127.2(2)° indicates that there is a significant twist between the central chromophore and the aromatic ring, an observation that is consistent with the molecular structure of S=C(OMe)N(H)C_6H_5 [2]. The key geometric parameters d(S1=C1), d(N1—C1) and d(N1—C2) are 1.670(1) Å, 1.335(2) Å and 1.434(2) Å, respectively. Referring to the figure, centrosymmetric molecules associate via N–H···S interactions so that d(H···S^i) = 2.60 Å, d(N1···S1^i) = 3.456(2) Å with an angle of 169° subtended at the H atom; symmetry operation i: $-1-x, 1-y, 1-z$.

Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.05 × 0.39 × 0.49 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	3.01 cm^{-1}
Diffractometer, scan mode:	Bruker AXS SMART CCD, ω
$2\theta_{max}$:	60°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3873, 2616
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2165
$N(param)_{refined}$:	111
Programs:	DIRDIF92 [3], SHELXL-97 [4], ORTEP-II [5], teXsan [6]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	−0.4912	0.4134	0.3250	0.037
H(4)	2*i*	−0.7463	0.1494	−0.2392	0.041
H(5)	2*i*	−0.4981	0.0020	−0.3162	0.046
H(6)	2*i*	−0.2025	0.0432	−0.1211	0.044
H(7)	2*i*	−0.1557	0.2364	0.1530	0.038
H(8A)	2*i*	−0.8718	0.3574	−0.0434	0.054
H(8B)	2*i*	−0.6979	0.5217	0.1056	0.054
H(8C)	2*i*	−0.8120	0.3549	0.1270	0.054
H(9A)	2*i*	0.0750	0.8575	0.4552	0.061
H(9B)	2*i*	0.1994	0.7334	0.3525	0.061
H(9C)	2*i*	0.1683	0.7216	0.5104	0.061

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)	2*i*	−0.22167(6)	0.71629(6)	0.57841(4)	0.0418(2)	0.0423(2)	0.0217(2)	0.0036(2)	0.0117(2)	0.0031(2)
O(1)	2*i*	−0.0817(2)	0.5952(1)	0.3202(1)	0.0301(5)	0.0319(5)	0.0253(5)	−0.0002(4)	0.0100(4)	0.0049(4)
N(1)	2*i*	−0.3919(2)	0.4352(2)	0.2856(1)	0.0270(5)	0.0368(6)	0.0211(5)	0.0017(5)	0.0090(4)	0.0077(5)

* 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)	2*i*	–0.2304(2)	0.5772(2)	0.3879(2)	0.0267(6)	0.0292(6)	0.0217(6)	0.0065(5)	0.0063(5)	0.0099(5)
C(2)	2*i*	–0.4196(2)	0.3148(2)	0.1179(2)	0.0269(6)	0.0263(6)	0.0209(6)	–0.0001(5)	0.0070(5)	0.0075(5)
C(3)	2*i*	–0.6006(2)	0.2898(2)	0.0029(2)	0.0267(6)	0.0280(6)	0.0252(6)	0.0002(5)	0.0068(5)	0.0113(5)
C(4)	2*i*	–0.6260(2)	0.1698(2)	–0.1594(2)	0.0338(7)	0.0325(7)	0.0252(6)	–0.0011(5)	0.0018(5)	0.0098(6)
C(5)	2*i*	–0.4786(3)	0.0800(2)	–0.2058(2)	0.0452(8)	0.0296(7)	0.0250(7)	0.0001(6)	0.0086(6)	0.0019(6)
C(6)	2*i*	–0.3023(2)	0.1048(2)	–0.0897(2)	0.0368(7)	0.0286(7)	0.0347(7)	0.0058(6)	0.0130(6)	0.0055(6)
C(7)	2*i*	–0.2734(2)	0.2212(2)	0.0733(2)	0.0296(6)	0.0304(7)	0.0284(7)	0.0040(5)	0.0065(5)	0.0097(6)
C(8)	2*i*	–0.7597(2)	0.3899(2)	0.0524(2)	0.0296(6)	0.0455(8)	0.0327(7)	0.0084(6)	0.0106(6)	0.0183(7)
C(9)	2*i*	0.1055(2)	0.7386(2)	0.4175(2)	0.0323(7)	0.0355(8)	0.0367(8)	–0.0043(6)	0.0087(6)	0.0053(6)

Acknowledgments. The Queensland Government, for the award of a Smart Returns Fellowship (ERTT), and the National University of Singapore (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. E**59** (2003) o1155-o1156.
3. 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.
4. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
5. Johnson, C. K.: ORTEP-II. A Fortran Thermal-Ellipsoid Plot Program. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA 1976.
6. Molecular Structure Corporation: teXsan. Single Crystal Structure Analysis Software. Version 1.05. MSC, The Woodlands, Texas, USA 1997.