Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

N-(4-Methoxybenzoyl)-*N*′-[2-(morpholinium-1-yl)-ethyl]thiourea thiocyanate

M. Sukeri M. Yusof[a] and Bohari M. Yamin[b]*

[a]Department of Chemistry, Kolej Universiti Sains dan Teknologi Malaysia, Mengabang Telipot, 21030 Kuala Terengganu, Malaysia, and [b]School of Chemical Sciences and Food Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

Correspondence e-mail:
bohari@pkrisc.cc.ukm.my

Received 29 June 2005
Accepted 4 July 2005
Online 9 July 2005

The title compound {systematic name: 4-[2-({[(4-methoxybenzoyl)amino]carbonothioyl}amino)ethyl]morpholin-4-ium thiocyanate}, $C_{15}H_{22}N_3O_3S^+\cdot NCS^-$, is a *p*-anisoylthiourea salt with a protonated morpholine group and a thiocyanate counter-anion. The morpholinium group has a chair conformation and the dihedral angle between the *p*-anisoyl group and the central thiourea N_2CS group is 32.51 (8)°. The crystal structure is stablized by intra- and intermolecular hydrogen bonds.

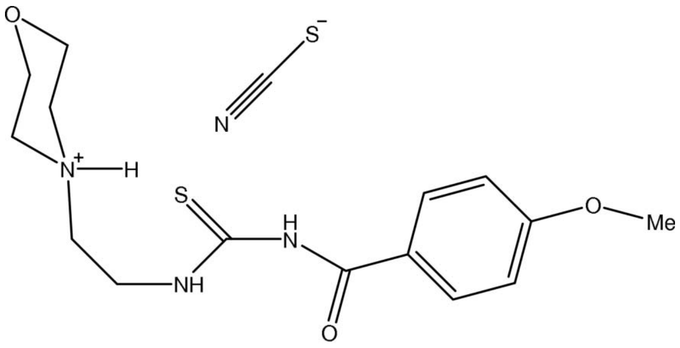
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.054
wR factor = 0.125
Data-to-parameter ratio = 15.2

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

Comment

Most anisoylthiourea derivatives have a *trans-cis* configuration with respect to the thiono group across their C—N bonds such as in *N*-(1-benzylpiperidine-4-yl)-*N*′-(4-methoxybenzoyl)thiourea (Yusof & Yamin, 2005). The title compound, (I), is also an anisoylthiourea derivative that has similar properties, but the morpholinium-1-ylethyl group bound to the terminal atom N3 is protonated. The charged thiourea molecule is balanced by the thiocyanate counter-anion (Fig.1).



(I)

The bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and in agreement with those in *N*-(1-benzylpiperidine-4-yl)-*N*′-(4-methoxybenzoyl)thiourea. The thiocyanate C16—S2 bond length is slightly shorter than the thiono C8═S1 (Table 1). The *p*-anisoyl fragment [C7/C1–C6/O2/C15, maximum deviation 0.019 (2) Å at C6] is inclined to the central thiourea group [N1/C8/S1/N3/C9, maximum deviation 0.022 (2)° at C9] by 32.51 (8)°. There are two intramolecular hydrogen bonds, N2—H2*A*···O1 and C9—H9*A*···S1, resulting in the formation of a six-membered ring (H2*A*···O1—C7—N1—C8—N2) and a five-membered ring (H9*A*···S1—C8—N2—C9), respectively. The N3—H3*A*···N4 hydrogen bond links the cation to the counter-anion. In the crystal structure, there are intermolecular hydrogen bonds of the type C—H···O, N—H···S and C—H···S (Table 2) to form a polymeric chain network (Fig. 2).

Experimental

A solution of 2-(morpholine-4-yl)ethylamine (0.53 g, 4.1 mmol) in acetone (50 ml) was added dropwise to an acetone solution (50 ml) containing an equimolar amount of *p*-methoxybenzoyl isothiocyanate in a 200 ml two-necked round-bottomed flask. The solution was refluxed for 2 h and then poured into a beaker containing some crushed ice. The white precipitate was washed with cold ethanol and distilled water, then dried in a vacumm (yield 80%). Recrystallization from acetone yielded single cystals suitable for X-ray analysis (m.p. 417.1–419.0 K). Analysis calculated: C 50.24, H 5.80, N 14.65, S 16.77, O 12.55%; found: C 49.3, H 5.60, N 14.13, S 15.86, O 11.983%.

Crystal data

$C_{15}H_{22}N_3O_3S^+\cdot NCS^-$	D_x = 1.346 Mg m^{-3}
M_r = 382.50	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 4016 reflections
a = 9.0195 (18) Å	θ = 2.0–25.5°
b = 10.402 (2) Å	μ = 0.31 mm^{-1}
c = 20.289 (4) Å	T = 293 (2) K
β = 97.477 (3)°	Block, colourless
V = 1887.3 (7) Å^3	0.42 × 0.33 × 0.25 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3505 independent reflections
ω scans	3107 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{\rm int}$ = 0.018
$T_{\rm min}$ = 0.882, $T_{\rm max}$ = 0.927	$\theta_{\rm max}$ = 25.5°
9351 measured reflections	$h = -10 \rightarrow 10$
	$k = -5 \rightarrow 12$
	$l = -23 \rightarrow 24$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 0.9468P]$
$R[F^2 > 2\sigma(F^2)]$ = 0.054	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.125	$(\Delta/\sigma)_{\rm max} < 0.001$
S = 1.17	$\Delta\rho_{\rm max}$ = 0.33 e Å^{-3}
3505 reflections	$\Delta\rho_{\rm min}$ = −0.27 e Å^{-3}
231 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected bond lengths (Å).

S1—C8	1.674 (2)	N2—C8	1.322 (3)
S2—C16	1.617 (3)	N2—C9	1.451 (3)
O1—C7	1.220 (3)	N3—C10	1.492 (3)
N1—C8	1.380 (3)	N3—C14	1.494 (3)
N1—C7	1.381 (3)	N3—C11	1.500 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*A*···O1	0.86	1.94	2.629 (3)	136
C9—H9*A*···S1	0.97	2.63	3.123 (3)	112
N3—H3*A*···N4	0.91 (2)	1.92 (2)	2.800 (4)	163 (2)
N1—H1*A*···S1$^{\rm i}$	0.86	2.77	3.625 (2)	171
C2—H2···O2$^{\rm ii}$	0.93	2.60	3.357 (3)	139
C9—H9*A*···O3$^{\rm iii}$	0.97	2.40	3.206 (3)	141
C10—H10*A*···S2$^{\rm iv}$	0.97	2.82	3.786 (3)	172
C15—H15*C*···S2$^{\rm v}$	0.96	2.85	3.805 (4)	171

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+2, -y, -z$; (iii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iv) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (v) $x+1, y, z$.

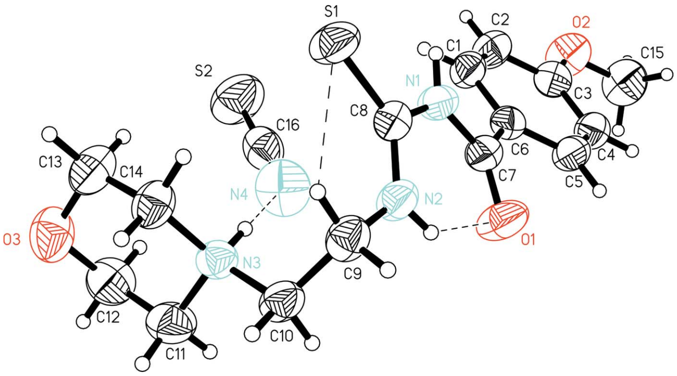


Figure 1
A view of the molecule of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

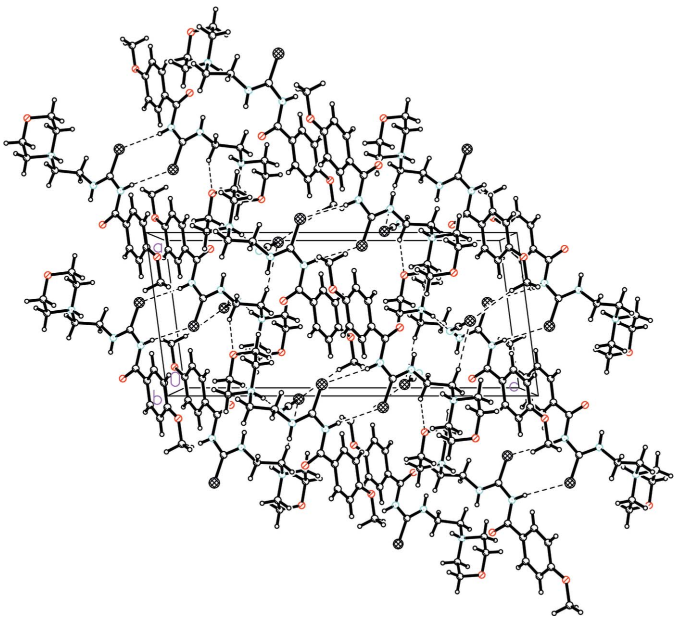


Figure 2
The crystal packing of the cation and anion of (I), viewed along the *b* axis. Dashed lines indicate hydrogen bonds.

After their location in a difference map, all H atoms except H3*A* were positioned geometrically at ideal positions and allowed to ride on the parent atom, with C—H = 0.93–0.96 Å and N—H = 0.86 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(CH$_2$ and NH) or 1.5$U_{\rm eq}$(CH$_3$). The protonated atom H3*A*, bonded to N3, was located in a difference map and refined freely.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

The authors thank the Malaysian Government for research grant IRPA No.09–02-02–0163.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L. Orpen, A. G., & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Yusof, M. S. & Yamin, B. M. (2005). *Acta Cryst.* E**61**, o2265–o2266.