Crystal structure of *N*(ethyl(2-hydroxyethyl)carbamothioyl)-2-iodobenzamide, $C_{12}H_{15}IN_2O_2S$

Nor Wahida Awang, Bohari M. Yamin and Siti Fairus M. Yusoff*

School of Chemical Sciences and Food Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

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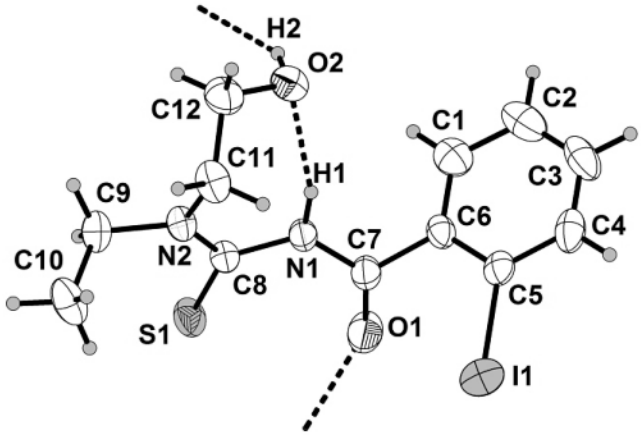

Abstract

$C_{12}H_{15}IN_2O_2S$, orthorhombic, $Pna2_1$ (no. 33), a = 6.9108(4) Å, b = 18.559(1) Å, c = 11.2975(5) Å, V = 1449.0 Å^3, Z = 4, $R_{gt}(F)$ = 0.0314, $wR_{ref}(F^2)$ = 0.0793, T = 296 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.32×0.36×0.50 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	23.50 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX CCD, ω
$2\theta_{max}$:	56.6°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	23668, 3579
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3341
$N(param)_{refined}$:	172
Programs:	SADABS [7], PARST [8], PLATON [9], SHELX [10]

Source of material

An acetone (30 ml) solution of 2-(ethylamino)ethanol (0.18 g, 2 mmol) was added to 2-iodobenzoyl isothiocyanate (0.58 g, 2 mmol). The mixture was refluxed for 3h. After cooling the solution was filtered off and the filtrate was left to evaporate at room temperature. The solid formed was washed with water and cold ethanol. Crystals suitable for X-ray study were obtained by recrystallization from DMSO.

Experimental details

All H atoms attached to C atoms were fixed geometrically and treated as riding with C–H = 0.93-0.97 Å with U_{iso}(H) = 1.2U_{eq}(C methylene and aromatic) and 1.5 for C(methyl). The hydrogen atoms attached to the N and O atoms were located from Fourier maps and refined isotropically.

Discussion

Most carbonoyl thioureas with primary amine possess *trans*-configuration with respect to the position of the carbonoyl group against the thiono group about the C–N bond, whereas the thiourea moiety has *trans*-geometry [1-3]. The configuration is associated with the hydrogen bond between the carbonyl oxygen atom and the amino hydrogen of the other terminal. Similar configurations were also found within secondary amines such as 2-bromo-*N*-(dibenzylcarbamothioyl)-benzamide [4] and 3-({[bis(2-methylpropyl)carbamothioyl]amino}carbonylbenzamide [5]. However, secondary amines provide more flexibility for the formation of the *cis*-configuration and consequently can act as bidentate ligand. Such flexibility in the rational design is demonstrated by the present thiourea compound having an ethylhydroxyethylamine (Fig.). The thiourea moiety S1/N1/N2/C8 and the benzyl fragment (C1-C6)/C7 are planar with maximum deviation of 0.038(4) Å for C1 atom from the least square plane. The thiourea moiety is perpendicular to the benzyl fragment at an angle of 82.45(15)°. The iodobenzoyl group is *cis* to the thiono group about the N1–C8 bond. However, the carbonyl and thione groups are twisted with torsion angle O1–C7–N1–C8 and S1–C8–N1–C7 of 1.6(6) and -21.9(5)°, respectively. The bond lengths and angles are in normal ranges [6]. There is intramolecular hydrogen bond between the hydroxyl oxygen atom, O2 and the hydrogen of the amide (N1–H1A···O2 = 2.795(4) Å, 157°). In the title structure, the molecules are linked by intermolecular hydrogen bonds between the hydroxyl and the keto group with O2–H2···O1 = 2.825(4) Å, 153° forming chains along *c*.

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*a*	0.5828	0.8793	0.4366	0.053
H(2A)	4*a*	0.5956	0.7599	0.4949	0.065
H(3)	4*a*	0.5387	0.6698	0.3607	0.065
H(4)	4*a*	0.4632	0.6970	0.1666	0.054
H(9A)	4*a*	0.2534	1.1785	0.3189	0.050
H(9B)	4*a*	0.0471	1.1577	0.3655	0.050
H(10A)	4*a*	0.1429	1.1709	0.1241	0.082
H(10B)	4*a*	0.0158	1.2261	0.1946	0.082
H(10C)	4*a*	–0.0628	1.1484	0.1694	0.082
H(11A)	4*a*	0.0463	0.9804	0.2792	0.049
H(11B)	4*a*	–0.0892	1.0461	0.3057	0.049
H(12A)	4*a*	0.0397	1.0451	0.5043	0.059
H(12B)	4*a*	–0.0976	0.9804	0.4730	0.059
H(1A)	4*a*	0.329(4)	0.960(2)	0.331(2)	0.029(8)
H(2)	4*a*	0.235(5)	0.966(2)	0.540(2)	0.03(1)

* Correspondence author (e-mail: ctfairus@ukm.my)

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}
I(1)	4*a*	0.41562(3)	0.82817(1)	0.00714(4)	0.0548(1)	0.0621(2)	0.0414(1)	–0.00054(9)	0.0017(1)	–0.0054(1)
S(1)	4*a*	0.5334(1)	1.10906(4)	0.20741(9)	0.0429(4)	0.0291(3)	0.0561(5)	–0.0031(3)	0.0106(4)	0.0048(3)
O(1)	4*a*	0.6534(5)	0.9556(1)	0.1521(3)	0.077(2)	0.036(1)	0.076(2)	–0.004(1)	0.047(2)	0.001(1)
O(2)	4*a*	0.1785(4)	0.9532(1)	0.4803(2)	0.043(1)	0.042(1)	0.039(2)	–0.004(1)	–0.0034(9)	0.0046(8)
N(1)	4*a*	0.4023(4)	0.9794(1)	0.2788(3)	0.038(1)	0.026(1)	0.039(2)	0.0033(9)	0.012(1)	0.003(1)
N(2)	4*a*	0.1921(4)	1.0736(1)	0.2976(2)	0.031(1)	0.028(1)	0.040(1)	0.0022(9)	–0.002(1)	0.0018(9)
C(1)	4*a*	0.5621(5)	0.8427(2)	0.3816(4)	0.035(2)	0.047(2)	0.050(2)	–0.002(1)	0.004(1)	0.006(2)
C(2)	4*a*	0.5679(5)	0.7714(2)	0.4166(4)	0.043(2)	0.060(2)	0.059(3)	–0.000(2)	0.002(2)	0.025(2)
C(3)	4*a*	0.5331(5)	0.7177(2)	0.3365(4)	0.041(2)	0.040(2)	0.081(3)	0.006(1)	0.019(2)	0.026(2)
C(4)	4*a*	0.4896(5)	0.7338(2)	0.2200(4)	0.037(2)	0.028(1)	0.070(3)	–0.002(1)	0.019(2)	–0.005(2)
C(5)	4*a*	0.4853(4)	0.8053(2)	0.1826(3)	0.025(1)	0.033(1)	0.041(2)	0.004(1)	0.009(1)	–0.001(1)
C(6)	4*a*	0.5252(4)	0.8597(2)	0.2633(3)	0.028(1)	0.026(1)	0.041(2)	0.001(1)	0.007(1)	0.004(1)
C(7)	4*a*	0.5362(5)	0.9366(2)	0.2239(3)	0.038(1)	0.027(1)	0.039(2)	0.001(1)	0.006(1)	0.000(1)
C(8)	4*a*	0.3680(5)	1.0530(2)	0.2629(3)	0.036(1)	0.027(1)	0.032(2)	0.003(1)	–0.000(1)	0.001(1)
C(9)	4*a*	0.1388(5)	1.1501(2)	0.3017(3)	0.041(2)	0.031(1)	0.053(2)	0.008(1)	–0.002(2)	–0.002(1)
C(10)	4*a*	0.0506(7)	1.1762(2)	0.1870(5)	0.057(2)	0.039(2)	0.068(3)	0.004(1)	–0.017(2)	0.012(2)
C(11)	4*a*	0.0327(5)	1.0236(2)	0.3268(4)	0.030(1)	0.038(2)	0.056(2)	–0.001(1)	–0.007(1)	0.003(2)
C(12)	4*a*	0.0261(6)	1.0025(2)	0.4552(4)	0.046(2)	0.047(2)	0.055(2)	0.001(2)	0.016(2)	0.010(2)

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References

1. Yusoff, M. S. M.; Azmi, N. R.; Yamin, B. M.: *N*-(4-Chlorobutanoyl)-*N*′-(2,5-dimethoxyphenyl)thiourea. Acta Crystallogr. **E67** (2011) o2609.
2. Yamin, B. M.: *N*-(4-Bromobutanoyl)-*N*′-phenylthiourea. Acta Crystallogr. **E67** (2011) o1629.
3. Hassan, I. N.; Wan Daud, W. R.; Yamin, B. M.; Kassim, M. B.: (*E*)-2-(3-Cinnamoylthioureido)acetic acid dimethyl sulfoxide disolvate. Acta Crystallogr. **E67** (2011) o2686.
4. Nasir, M. F. M.; Hassan, I. N.; Wan Daud, W. R.; Yamin, B. M.; Kassim, M. B.: 2-Bromo-*N*-(dibenzylcarbamothioyl)benzamide. Acta Crystallogr. **E67** (2011) o1218.
5. Selvakumaran, N.; Karvembu, T.; Ng, S. W.; Tienink, E. R. T.: 3-({[Bis(2-methylpropyl)carbamothioyl]amino}carbonyl)benzamide. Acta Crystallogr. **E69** (2013) o1183.
6. Allen, F. H.; Kennard, O.; Watson, D.G.; Brammer, L.; Orpen, A. G.; Taylor, R.: (1987).
7. Bruker (2000): SMART, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
8. Nardelli, M.: PARST95 - an update to PARST: a system of Fortran routines for calculating molecular structure parameters from the results of crystal structure analyses. J. Appl. Crystallogr. **28** (1995) 659.
9. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.
10. Spek, A. L.: Structure validation in chemical crystallography. Acta Crystallogr. **D65** (2009) 148-155.