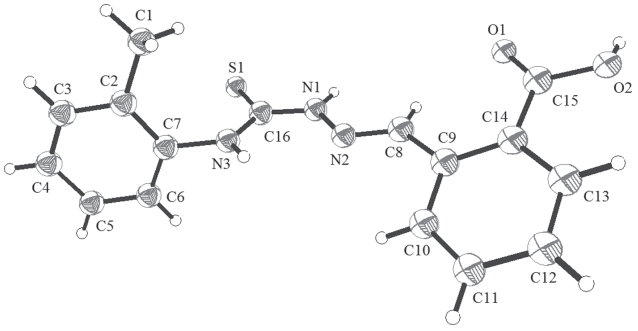
Tai Xi-Shi*, Li Peng-Fei and Liu Qing-Yun

The crystal structure of (*E*)-2-((2-(*o*-tolylcarbamothioyl)hydrazono)methyl)benzoic acid, $C_{16}H_{15}N_3O_2S$



https://doi.org/10.1515/ncrs-2017-0221
Received September 2, 2017; accepted November 24, 2017; available online December 14, 2017

Abstract

$C_{16}H_{15}N_3O_2S$, triclinic, $P\bar{1}$ (no. 2), $a = 7.6394(15)$ Å, $b = 9.2445(18)$ Å, $c = 11.428(2)$ Å, $\alpha = 98.75(3)°$, $\beta = 106.34(3)°$, $\gamma = 91.18(3)°$, $V = 763.7(3)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0548$, $wR_{ref}(F^2) = 0.1411$, $T = 293(2)$ K.

CCDC no.: 1587438

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

0.9013 g 4-(2-methylphenyl)-3-thiosemicarbazide (0.5 mmol) and 0.7506 g 2-carboxybenzaldehyde (0.5 mmol) were dissolved in 15 mL 95% C_2H_5OH. Then the mixture was heated at 60 °C for 4 h and cooled to room temperature. The solution was evaporated to obtain block crystals within 20 days.

***Corresponding author: Tai Xi-Shi,** College of Chemistry and Chemical Engineering, Weifang University, Weifang, Shandong 261061, P.R. China, e-mail: taixs@wfu.edu.cn
Li Peng-Fei: College of Chemical Engineering, Qingdao University of Science and Technology, Qingdao,Shandong 266061, P.R. China
Liu Qing-Yun: College of Chemistry and Environmental Engineering, Shandong University of Science and Technology, Qingdao, Shandong 266590, P.R. China

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.19 × 0.18 × 0.170 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.22 mm^{-1}
Diffractometer, scan mode:	Bruker, APEX2, ω and ϕ scans
$2\theta_{max}$, completeness:	27.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7428, 3461, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3139
$N(param)_{refined}$:	200
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3], DIAMOND [4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.48227(5)	0.73308(4)	0.15236(4)	0.04350(16)
O1	1.18235(17)	0.35086(12)	0.03524(13)	0.0519(3)
O2	1.37644(17)	0.17585(15)	0.05773(13)	0.0582(4)
H2	1.4095	0.2088	0.0041	0.087*
N1	0.69310(17)	0.51243(14)	0.14418(11)	0.0372(3)
H1	0.7143	0.5431	0.0818	0.045*
N2	0.77189(16)	0.38893(13)	0.18398(11)	0.0340(3)
N3	0.55869(18)	0.53450(14)	0.30140(11)	0.0384(3)
H3	0.6132	0.4581	0.3229	0.046*
C1	0.7095(3)	0.7827(3)	0.4965(2)	0.0654(5)
H1A	0.7920	0.7159	0.5358	0.098*
H1B	0.7248	0.8749	0.5510	0.098*
H1C	0.7353	0.7975	0.4215	0.098*
C2	0.5154(2)	0.71973(18)	0.46684(14)	0.0425(3)
C3	0.3989(3)	0.7791(2)	0.53331(16)	0.0532(4)
H3A	0.4424	0.8578	0.5972	0.064*
C4	0.2198(3)	0.7232(2)	0.50625(16)	0.0557(5)
H4	0.1445	0.7647	0.5517	0.067*
C5	0.1535(2)	0.6077(2)	0.41326(17)	0.0550(4)
H5	0.0333	0.5701	0.3953	0.066*
C6	0.2664(2)	0.54647(19)	0.34554(15)	0.0463(4)
H6	0.2218	0.4677	0.2819	0.056*
C7	0.4450(2)	0.60240(15)	0.37252(13)	0.0357(3)
C8	0.87311(18)	0.32873(15)	0.12093(13)	0.0332(3)
H8	0.8941	0.3706	0.0567	0.040*
C9	0.95591(18)	0.19169(14)	0.15207(12)	0.0307(3)
C10	0.8632(2)	0.09791(16)	0.20312(14)	0.0395(3)
H10	0.7520	0.1234	0.2157	0.047*

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C11	0.9334(2)	−0.03187(17)	0.23523(16)	0.0468(4)
H11	0.8701	−0.0920	0.2700	0.056*
C12	1.0975(3)	−0.07247(17)	0.21568(16)	0.0470(4)
H12	1.1450	−0.1596	0.2374	0.056*
C13	1.1900(2)	0.01715(16)	0.16386(14)	0.0401(3)
H13	1.3004	−0.0104	0.1510	0.048*
C14	1.12161(18)	0.14855(14)	0.13020(12)	0.0312(3)
C15	1.22646(19)	0.23649(15)	0.06986(13)	0.0342(3)
C16	0.58289(18)	0.58518(15)	0.20315(13)	0.0327(3)

Elemental analysis calc. for $C_{16}H_{15}N_3O_2S$: C, 61.27, H, 4.79, N, 13.40(%); Found: C, 61.39, H, 4.56, N, 13.20(%).

Experimental details

Coordinates of hydrogen atoms were refined without any constraints or restraints. Their U_{iso} values were set to $1.2U_{eq}$ of the parent atoms.

Comment

The bond lengths of C16—S1 and C8—N2 are 1.6825(14) Å and 1.2791(18) Å, respectively. The N—N bond length is shorter than in structures with hydrazinyl moieties [5, 6], and the C—S distance is also shorter than a typical C—S single bond. The geometric parameters excellently fit with a directly related structure [7]. In the crystal structure, the dihedral angles between benzene ring 1 (C2—C3—C4—C5—C6—C7) and ring 2 (C9—C10—C11—C12—C13—C14) is 68.3°. There are weak intermolecular hydrogen bonds and π–π stacking interactions.

Acknowledgements: This project was supported by the National Natural Science Foundation of China (No. 21171132), the Natural Science Foundation of Shandong (ZR2014BL003), the project of Shandong Province Higher Educational Science and Technology Program (J14LC01) and Science Foundation of Weifang.

References

1. Bruker: APEX2, SAINT and SADABS. Brucker AXS Inc., Madison, WI, USA (2009).
2. Sheldrick, G. M.: Crystal structure refinement with *SHELXTL*. Acta Crystallogr. **C71** (2015) 3–8.
3. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. J. Appl. Crystallogr. **42** (2009) 339–341.
4. Brandenburg, K.: *DIAMOND*. Visual Crystal Structure Information System. Ver. 3.2. Crystal Impact, Bonn, Germany (2012).
5. Tai, X.-S.; Wang, X.: Synthesis, structural characterization and antitumor activity of a Ca(II) coordination polymer based on 4-formyl-1,3-benzenedisulfonate-2-furoic acid hydrazide ligands. Crystallogr. Rep. **62** (2017) 242–245.
6. Amr, A.-E.; Khalifa, N.-M.; Al-Omar, M.-A.; Ghabbour, H.-A.: Crystal structure of N^2,N^6-bis(1-hydrazinyl-2-methyl-1-oxopropan-2-yl)pyridine-2,6-dicarboxamide, $C_{15}H_{23}N_7O_4$. Z. Kristallogr. NCS **232** (2017) 537–539.
7. Madanhire, T.; Abrahams, A.; Hosten, E. C.; Betz, R.: Crystal structure of 4-(4-bromophenyl)-1-(propan-2-ylidene)thiosemicarbazide, $C_{10}H_{12}BrN_3S$. Z. Kristallogr. NCS **230** (2015) 67–68.