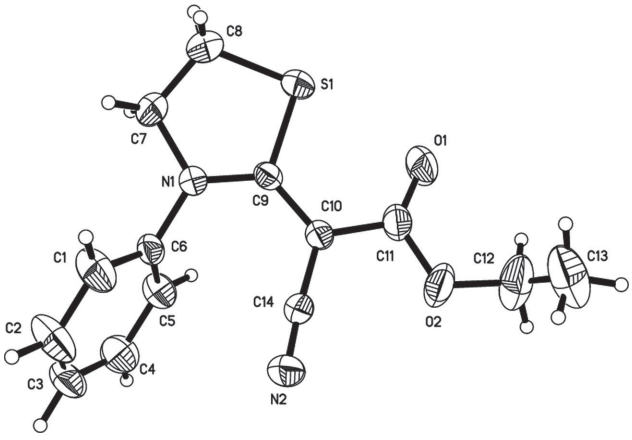
Open Access

Yahia N. Mabkhot*, Hazem A. Ghabbour, Faiz A. Al-aizari and Salim Al-Showiman

Crystal structure of (*Z*)-Ethyl 2-cyano-2-(3-phenylthiazolidin-2-ylidene) acetate, $C_{14}H_{14}N_2O_2S$



DOI 10.1515/ncrs-2016-0121
Received June 19, 2016; accepted September 14, 2016; available online October 10, 2016

Abstract

$C_{14}H_{14}N_2O_2S$, triclinic, $P\bar{1}$, $a = 8.6471(4)$ Å, $b = 9.5576(5)$ Å, $c = 9.7098(5)$ Å, $\alpha = 91.054(2)°$, $\beta = 109.380(2)°$, $\gamma = 113.769(2)°$, $V = 681.95(6)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.061$, $wR_{ref}(F^2) = 0.175$, $T = 296(2)$.

CCDC no.: 1484500

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

Source of material

The synthesis of *(Z)*-ethyl 2-cyano-2-(3-phenylthiazolidin-2-ylidene) acetate proceeded by a reaction of ethylcyanoacetate (1 mmol) in 10 mL DMF/C_2H_5OH and phenylisothiocyanate (1 mmol) at presence of K_2CO_3. The reaction mixture was stirred for 10 h. The residue was recrystallized from DMF/C_2H_5OH. *(Z)*-Ethyl 2-cyano-2-(3-phenylthiazolidin-2-ylidene) acetate ($C_{14}H_{14}N_2O_2S$), yellow crystals; yield (35%); m.p.: 205 °C; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 1.84 (s, CH_3), 3.19 (t, CH_2), 3.27 (t, CH_2), 4.31 (q, CH_2) 7.56–7.50 (m, Ar) ppm; **^{13}C-NMR** (400 MHz, $CDCl_3$): δ 14.28, 17.85, 26.24, 29.57, 60.25, 94.90, 118.47, 123.57, 140.91, 155.42, 158.92, 147.3, 163.91, 198.31 ppm.

Table 1: Data collection and handling.

Crystal:	Colourless block Size 0.32 × 0.26 × 0.24 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	2.4 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	55°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	19934, 3119, 0.049
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2345
$N(param)_{refined}$:	173
Programs:	Bruker programs [12], SHELX [13]

Experimental details

C-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation. The H atoms of the methyl groups were allowed to rotate with a fixed angle around the C—C bond to best fit the experimental electron density.

Discussion

This contribution is part of our continuing interest in the syntheses and crystal structures of small organic compounds [1–4]. Thiazole compounds are an important in heterocyclic [5–8], and also used in coordination chemistry [9] and are known as powerful inhibitors of carbonic anhydrase activity [8–11].

The asymmetric unit cell of the title compound contains one molecule. The bond length of C9—C10 is 1.390(3) Å which is a typical C = C double bond and the configuration around this double bond is *Z*. The molecules are packed in the crystal structure at least by one non-classical

***Corresponding author: Yahia N. Mabkhot,** Department of Chemistry, College of Science, King Saud University, P.O. Box 2455, Riyadh 1451, Saudi Arabia, e-mail: yahia@ksu.edu.sa
Hazem A. Ghabbour: Department of Pharmaceutical Chemistry, College of Pharmacy, King Saud University, P.O. Box 2457, Riyadh 11451, Saudi Arabia; and Department of Medicinal Chemistry, Faculty of Pharmacy, Mansoura University, Mansoura 35516, Egypt
Faiz A. Al-aizari and Salim Al-Showiman: Department of Chemistry, College of Science, King Saud University, P.O. Box 2455, Riyadh 1451, Saudi Arabia

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.57896(10)	0.36123(8)	0.14783(8)	0.0524(3)
O1	0.2177(3)	0.2648(3)	0.0047(3)	0.0708(7)
O2	0.0428(3)	0.3411(3)	0.0835(3)	0.0779(8)
N1	0.6838(3)	0.6103(2)	0.3276(2)	0.0391(5)
N2	0.2674(4)	0.6137(3)	0.3722(3)	0.0662(8)
C1	0.7642(5)	0.7946(4)	0.5420(3)	0.0672(9)
H1A	0.8039	0.7305	0.6041	0.081*
C2	0.7753(7)	0.9313(5)	0.6024(5)	0.0902(13)
H2A	0.8224	0.9611	0.7071	0.108*
C3	0.7203(5)	1.0241(4)	0.5149(5)	0.0750(11)
H3A	0.7259	1.1169	0.5582	0.090*
C4	0.6564(5)	0.9837(4)	0.3639(5)	0.0703(9)
H4A	0.6196	1.0496	0.3026	0.084*
C5	0.6449(4)	0.8474(3)	0.3000(3)	0.0541(7)
H5A	0.6032	0.8204	0.1952	0.065*
C6	0.6945(3)	0.7523(3)	0.3900(3)	0.0387(5)
C7	0.8553(4)	0.6050(4)	0.3404(4)	0.0622(8)
H7A	0.9201	0.6852	0.2909	0.075*
H7B	0.9346	0.6257	0.4462	0.075*
C8	0.8136(5)	0.4511(5)	0.2705(5)	0.0839(12)
H8A	0.8357	0.3872	0.3472	0.101*
H8B	0.8928	0.4600	0.2141	0.101*
C9	0.5325(3)	0.4940(3)	0.2338(3)	0.0344(5)
C10	0.3550(3)	0.4713(3)	0.2064(3)	0.0381(5)
C11	0.2045(4)	0.3489(3)	0.0895(3)	0.0503(7)
C12	−0.1216(5)	0.2288(6)	−0.0327(4)	0.0952(14)
H12A	−0.2047	0.2772	−0.0753	0.114*
H12B	−0.0904	0.1956	−0.1129	0.114*
C13	−0.2075(8)	0.1007(5)	0.0242(6)	0.1189(19)
H13A	−0.3290	0.0353	−0.0493	0.178*
H13B	−0.2184	0.1354	0.1147	0.178*
H13C	−0.1359	0.0408	0.0475	0.178*
C14	0.3118(3)	0.5551(3)	0.2986(3)	0.0441(6)

intermolecular hydrogen bond C3—H3A$\cdots$N2^{i}. The H$\cdots$A distance is 2.62 Å and the angle is 169°. Symmetry codes: (i) $-x+1, -y+2, -z+1$.

Acknowledgements: The authors extend their sincere appreciation to the Deanship of Scientific Research at king Saud University for its funding this Prolific Research group (PRG-1437-29).

References

1. Mabkhot, Y, N.; Al-Majid, A. M.; Barakat, A.; Choudhary, M. I.; Yousuf, S.: Crystal structure of 1-(5-acetyl-3,4-dimethyl-thieno[2,3-b]thiophen-2-yl)-ethanone, $C_{12}H_{12}O_2S_2$. Z. Kristallogr. NCS **229** (2014) 39–40.
2. Mabkhot, Y. N.; Barakat, A.; Ghabbour, H. A.; Fun, H.-K.: Crystal structure of 2-amino-N-(4-methoxyphenyl)benzamide, $C_{14}H_{14}N_2O_2$. Z. Kristallogr. NCS **231** (2016) 203–204.
3. Mabkhot, Y. N.; Al-Showiman, S. S.; Barakat, A.; Yousuf, S.; Choudhary, M. I.: Crystal structure of 1-acetylimidazolidine-2,4-dione monohydrate, $C_5H_8N_2O_4$. Z. Kristallogr. NCS **230** (2015) 255–256.
4. Mabkhot, Y. N.; Al-Showiman, S. S.; Barakat, A.; Yousuf, S.; Choudhary, M. I.: Regioselective synthesis and crystal structure of ethyl-4-acetyl-5-((2-ethoxy-2-oxoethyl)thio)-3-hydroxythiophene-2-carboxylate, $C_{13}H_{16}O_6S_2$. Z. Kristallogr. NCS **230** (2015) 241–242.
5. Rouf, A.; Tanyeli, C.: Bioactive thiazole and benzothiazole derivatives. Eur. J. Med. Chem. **97** (2015) 911–927.
6. Cristiani, F.; Devillanova, F. A.; Diaz, A.; Verani, G.: Thio-amido and seleno-amido groups as reducing agents towards OsO4. A kinetic study with some imidazolidine-2-selones. Phosphorus, Sulfur Relat. Elem. **12** (1982) 305–315.
7. Cox, P. J.; Aslanidis, P.; Karagiannidis, P.; Hadjikakou, S.: Dinuclear complexes of silver(I) bromide: the crystal structure of bis[bromo-(pyrimidine-2-thione)(triphenylphosphine)silver(I)], Inorg. Chim. Acta **310** (2000) 268–272.
8. Toplak, R. B.; Rocherulle, P.; Lorcy. D.: Unexpected reaction of dimethoxycarbonyl dithiole-2-thione or tetramethoxy-carbonyl TTF as dipolarophiles. Tetrahedron Lett. **43** (2002) 3879–3882.
9. Raper, E. S.: Complexes of heterocyclic thione donors. Coord. Chem. Rev. **61** (1985) 115–185.
10. Supuran, C.; Briganti, T.; Scozzafava, F. A.: Sulfenamidosulfonamides as inhibitors of carbonic anhydrase isozymes I, II and IV. J. Enzyme Inhib. **12** (1997) 175–190.
11. Brezeanu, M. R.; Olar, G.; Manole, C. T.; Supuran, C. T.: 1,3,4-Thiadiazole derivatives. Zn(II) and Hg(II) coordination compounds of 2-amino-5-mercapto-1,3,4-thiadiazole as enzyme-inhibitors. Rev. Roum. Chim. **37** (1992) 425–431.
12. Brucker. APEX2, SAINT and SADABS. Brucker AXS Inc., Madison, Wisconsin, USA, 2009.
13. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.