Mohamed Reda Aouad*, Mouslim Messali, Nadjet Rezki, Naveen Shivalingegowda and Ismail Warad*

Crystal structure of 4-phenyl-3-((4-phenyl-1*H*-1,2,3-triazol-1-yl)methyl)-1*H*-1,2,4-triazole-5(4*H*)-thione, C₁₇H₁₄N₆S

<https://doi.org/10.1515/ncrs-2018-0025>

Received January 14, 2018; accepted April 30, 2018; available online May 22, 2018

Abstract

C₁₇H₁₄N₆S, orthorhombic, *P*2₁2₁2₁ (no. 19), *a* = 7.3832(3) Å, *b* = 14.4702(7) Å, *c* = 15.2806(7) Å, *V* = 1632.52(13) Å³, *Z* = 4 and with *R*_{int} = 0.0577, *R*_{gt}(*F*) = 0.0358, *wR*_{ref}(*F*²) = 0.0944, *T* = 296(2) K.

CCDC no.: 1824847

*Corresponding authors: **Mohamed Reda Aouad**, Department of Chemistry, Taibah University, 30002 Al-Madina Al-Mounawara, Saudi Arabia e-mail: aouadmohamedreda@yahoo.fr; and **Ismail Warad**, Department of Chemistry, AN-Najah National University, Nablus, Palestine, e-mail: warad@najah.edu

Mouslim Messali: Department of Chemistry, Taibah University, 30002 Al-Madina Al-Mounawara, Saudi Arabia

Nadjet Rezki: Department of Chemistry, Taibah University, 30002 Al-Madina Al-Mounawara, Saudi Arabia; and Laboratoire de Chimie et Electrochimie des Complexes Metalliques (LCECM) USTO-MB, University of Sciences and Technology Mohamed Boudiaf, BP 1505 Oran, El M nouar, Algeria

Naveen Shivalingegowda: Department of Physics, School of Engineering and Technology, Jain University, Bangalore 562 112, India

Table 1: Data collection and handling.

Crystal:	Rectangle, white
Size:	0.28 × 0.26 × 0.22 mm
Wavelength:	Cu <i>K</i> α radiation (1.54178 Å)
μ:	1.85 mm ^{−1}
Diffractometer, scan mode:	Bruker X8 Proteum, φ and ω-scans
θ _{max} , completeness:	66.57°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	28809, 2869, 0.058
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2848
<i>N</i> (<i>param</i>) _{refined} :	217
Programs:	Bruker programs [1], SHELX [2, 3]

Source of material

A solution of *N*-phenyl-2-(2-(4-phenyl-1*H*-1,2,3-triazol-1-yl)acetyl)hydrazinecarbothioamide (10 mmol, 3.52 g) in aqueous sodium hydroxide 10% (100 ml) was heated under reflux for 6 h. After cooling, the reaction mixture was acidified with diluted hydrochloric acid. The precipitate formed was filtered and recrystallized from ethanol to give the title compound as colorless needles in 89% yield, m.p. 269–270 °C.

Experimental details

Hydrogen atoms were placed at the calculated positions and refined using a riding model with *U*_{iso}(H) = 1.2*U*_{eq} or 1.5*U*_{eq} of the adjacent non-hydrogen atom. H3 Atom position was localized from the difference map and refined with DFIX 0.87 and *U*_{iso}(H) = 1.5*U*_{eq}(O3) [2, 3].

Discussion

Triazole and its derivatives compound have gained a great biological activities like antiproliferative, antibacterial, analgesic, antitubercular, anti-inflammatory, anti-tuberculosis, anti-inflammatory, depressant, anti-HIV and anti-cancer [4–10]. The design and synthesis of novel triazole-thione bearing different aromatic moieties have become a broad area in bio-organic synthesis research [11, 12]. 1,2,4-Triazoles are used as poly-chelate ligands for the synthesis of metal complexes [13]. There is one molecule in the asymmetric unit

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} [*] /U _{eq}
S1	−0.02291(6)	−0.68235(3)	−0.89262(4)	0.06769(16)
N1	−0.33020(17)	−0.78853(8)	−0.87530(8)	0.0480(3)
N2	−0.38008(19)	−0.64350(9)	−0.88009(9)	0.0550(3)
H2	−0.3640	−0.5847	−0.8833	0.066
N3	−0.54711(18)	−0.68324(9)	−0.87164(9)	0.0531(3)
N4	−0.68993(17)	−0.86710(9)	−0.76957(8)	0.0498(3)
N5	−0.6828(2)	−0.95080(9)	−0.65555(9)	0.0588(3)
N6	−0.6413(2)	−0.94984(9)	−0.73887(9)	0.0595(3)
C1	−0.2441(2)	−0.70438(10)	−0.88287(9)	0.0513(3)
C2	−0.5139(2)	−0.77161(10)	−0.86984(9)	0.0482(3)
C3	−0.2396(2)	−0.87673(10)	−0.87094(10)	0.0514(3)
C4	−0.1890(3)	−0.92074(13)	−0.94624(13)	0.0674(4)
H4	−0.2152	−0.8949	−1.0005	0.081
C5	−0.0984(4)	−1.00414(16)	−0.94056(18)	0.0884(6)
H5	−0.0640	−1.0347	−0.9914	0.106
C6	−0.0589(3)	−1.04186(14)	−0.86126(19)	0.0870(7)
H6	0.0029	−1.0978	−0.8582	0.104
C7	−0.1099(4)	−0.99780(17)	−0.78625(18)	0.0924(7)
H7	−0.0830	−1.0237	−0.7321	0.111
C8	−0.2012(3)	−0.91491(14)	−0.79070(13)	0.0743(5)
H8	−0.2367	−0.8849	−0.7397	0.089
C9	−0.6575(2)	−0.84319(11)	−0.86167(9)	0.0513(3)
H9A	−0.7688	−0.8203	−0.8875	0.062
H9B	−0.6213	−0.8982	−0.8935	0.062
C10	−0.7623(2)	−0.81429(11)	−0.70616(10)	0.0542(3)
H10	−0.8064	−0.7543	−0.7112	0.065
C11	−0.7570(2)	−0.86804(10)	−0.63250(10)	0.0517(3)
C12	−0.8083(2)	−0.84733(12)	−0.54128(10)	0.0549(4)
C13	−0.7714(3)	−0.76039(14)	−0.50696(13)	0.0714(5)
H13	−0.7218	−0.7148	−0.5425	0.086
C14	−0.8082(4)	−0.74174(19)	−0.42030(14)	0.0871(6)
H14	−0.7838	−0.6835	−0.3975	0.104
C15	−0.8811(3)	−0.80935(19)	−0.36726(12)	0.0835(6)
H15	−0.9018	−0.7974	−0.3083	0.100
C16	−0.9232(3)	−0.89474(16)	−0.40187(13)	0.0787(6)
H16	−0.9772	−0.9395	−0.3668	0.094
C17	−0.8854(3)	−0.91398(13)	−0.48846(12)	0.0674(4)
H17	−0.9119	−0.9720	−0.5113	0.081

as in the figure; the compound has not been detected in dimer form. All the angles and bond lengths are in their expected ranges. The two phenyl groups are almost perpendicular to the plane of the 1,2,4-triazole-5(4*H*)-thione ring. The molecules packing in the crystal structure is stabilized via several interaction types. N—H···N hydrogen bonds with 2.701 Å, 2.674 Å and 2.576 Å, C—H···S hydrogen bonds 2.741 Å, aromatic C—H···π stacking interactions with 2.89 Å and 2.85 Å were also detected. These interactions link the molecules to a three-dimensional arrangement.

References

1. Bruker. SAINT, APEX2, Bruker Inc., Madison, Wisconsin, USA (2016).
2. Sheldrick, G. M.: SHELXL2014: Program for the Solution of Crystal Structures, University of Göttingen, Göttingen, Germany (2014).
3. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr. A* **64** (2008) 112–122.
4. Balzarini, J.; Orzeszko, B.; Mauri, J. K.; Orzeszko, A.: Synthesis and anti-HIV studies of 2-adamantyl-substituted thiazolidin-4-ones. *Eur. J. Med. Chem.* **42** (2007) 993–1003.
5. El-Emam, A. A.; Al-Tamimi, A.-M. S.; Al-Omar, M. A.; Al-Rashood, K. A.; Habib, E. E.: Synthesis and antimicrobial activity of novel 5-(1-adamantyl)-2-aminomethyl-4-substituted-1,2,4-triazoline-3-thiones. *Eur. J. Med. Chem.* **68** (2013) 96–102.
6. Wang, X.; Dong, Y.; Wittlin, S.; Charman, S. A.; Chiu, F. C. K.; Chollet, J.; Katneni, K.; Mannila, J.; Morizzi, J.; Ryan, E.; Scheurer, C.; Steuten, J.; Tomas, J. S.; Snyder, C.; Vennerstrom, J. L.: Comparative antimalarial activities and ADME profiles of ozonides (1,2,4-trioxolanes) OZ277, OZ439, and their 1,2-dioxolane, 1,2,4-trioxane, and 1,2,4,5-tetraoxane isosteres. *J. Med. Chem.* **56** (2013) 2547–2555.
7. Kouatly, O.; Geronikaki, A.; Kamoutsis, C.; Hadjipavlou-Litina, D.; Eleftheriou, P.: Adamantane derivatives of thiazolyl-N-substituted amide, as possible non-steroidal anti-inflammatory agents. *Eur. J. Med. Chem.* **44** (2009) 1198–1204.
8. Tozkoparan, B.; Küpeli, E.; Ye silada, E.; Ertan, M.: Preparation of 5-aryl-3-alkylthio-1, 2,4-triazoles and corresponding sulfones with anti-inflammatory-analgesic activity. *Bioorg. Med. Chem.* **15** (2007) 1808–1814.
9. Almajan, G. L.; Barbuceanu, S. -F.; Almajan, E.; Draghici, C.; Saramet, G.: Synthesis, characterization and antibacterial activity of some triazole Mannich bases carrying diphenylsulfone moieties. *Eur. J. Med. Chem.* **44** (2009) 3083–3089.
10. Al-Deeb, O. A.; Al-Omar, M. A.; El-Brollosy, N. R.; Habib, E. E.; Ibrahim, T. M.; El-Emam, A. A.: Synthesis, antimicrobial, and anti-inflammatory activities of novel 2-[3-(1-adamantyl)-4-substituted-5-thioxo-1,2,4-triazolin-1-yl]acetic acids, 2-[3-(1-adamantyl)-4-substituted-5-thioxo-1,2,4-triazolin-1-yl]propionic acids and related derivatives. *Arzneim.-Forsch.* **56** (2006) 40–47.
11. Ishibashi, Y.; Umesato, T.; Fujiwara, M.; Une, K.; Yoneda, Y.; Sotome, H.; Katayama, T.; Kobatake, S.; Asahi, T.; Irie, M.; Miyasaka, H.: Solvent polarity dependence of photochromic reactions of a diarylethene derivative as revealed by steady-state and transient spectroscopies. *J. Phys. Chem.* **120** (2016) 1170–1177.
12. Wang, J.; Liu, G.; Fan, C.; Pu, Sh.: Structure and photochromism of 1,2-bis[2-methyl-5-(2-chlorophenyl)-3-thienyl]-3,3,4,4,5,5-hexafluorocyclopent-1-ene, C₂₇H₁₆Cl₂F₆S₂. *Z. Kristallogr. NCS* **232** (2017) 359–361.
13. Castineiras, A.; Garcia-Santos, I.: Desulfuration and cyclization of (Z)-2-[amino(pyridine-2-yl)methylene]hydrazonocarbothioamide in the presence of manganese(II). *Z. Anorg. Allg. Chem.* **634** (2008) 2907–2916.