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Short Note

(*Z*)-2-(1-(5-Methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)ethylidene)-*N*-phenylhydrazine-1-carbothioamide

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Abstract: Reaction of equimolar equivalents of 1-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)ethan-1-one (**1**) and *N*-phenylhydrazinecarbothioamide (**2**) in boiling ethanol containing a catalytic amount of concentrated hydrochloric acid for 4 h gave (*Z*)-2-(1-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)ethylidene)-*N*-phenylhydrazine-1-carbothioamide (**3**) with 88% yield. The structure of **3** was established using single-crystal X-ray diffraction and magnetic resonance spectroscopy.

Keywords: synthesis; 1,2,3-triazole; *N*-phenylhydrazinecarbothioamide; X-ray diffraction; heterocycles; hydrazones

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Citation: Kariuki, B.M.; Abdel-Wahab, B.F.; Bekheit, M.S.; El-Hiti, G.A. (*Z*)-2-(1-(5-Methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)ethylidene)-*N*-phenylhydrazine-1-carbothioamide. *Molbank* **2022**, *2022*, M1462. https://doi.org/10.3390/M1462

Academic Editor: Rodrigo Abonia

Received: 15 September 2022
Accepted: 30 September 2022
Published: 9 October 2022

1. Introduction

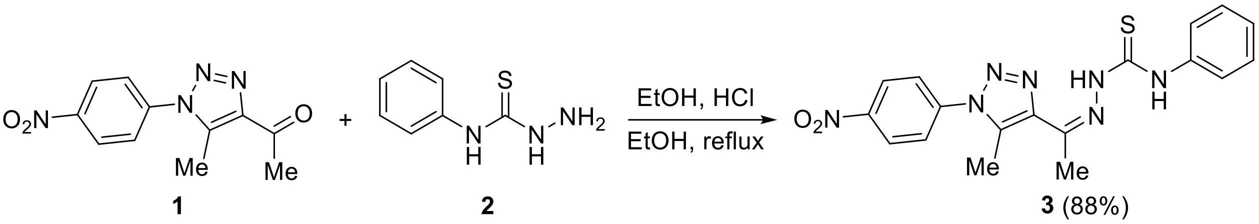
Thiosemicarbazones are compelling from different perspectives, including their bonding modes, biological implications, structural diversity, and ion-sensing capability [1–5]. They are effective chelating ligands for the production of metal complexes due to the presence of flexible donor atoms (sulfur and nitrogen). The biological activities of thiosemicarbazones can be altered through complexation with transition metals such as the copper(II) ion [6,7]. Thiosemicarbazone–metal (Cu, Ni, and Co) complexes act as antibacterial, antifungal, antiviral, and anti-inflammatory agents [8–12]. In addition, the metal complexes of thiosemicarbazones have potential for application in nonlinear optics [13,14], electrochemical sensing [14,15], and generation of Langmuir films [16–18].

1,2,3-Triazoles present a wide range of biological activities [19–23]. The 1,2,3-triazole ring system can be synthesized through click chemistry, which involves simple and effective processes that can lead to the formation of a variety of substituted derivatives in high yields [24]. The 1,3-cycloaddition of substituted nitriles containing an active methylene moiety and aryl azides is an efficient procedure for the production of 1,2,3-triazoles [25,26]. In addition, 1,2,3-triazoles can be synthesized from reactions of diazo compounds and amines or amides [27] and reaction of azides and amines [28] or alkynes [29–31]. The current work covers the synthesis of a new 1,2,3-triazole derivative in continuation of our interest in the synthesis of new heterocycles [32–35].

2. Results and Discussion

2.1. Synthesis of 3

Reaction of equimolar equivalents of 1-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)ethan-1-one (**1**) and *N*-phenylhydrazinecarbothioamide (**2**) in boiling ethanol containing a catalytic amount of concentrated hydrochloric acid (HCl) for 4 h gave (*Z*)-2-(1-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)ethylidene)-*N*-phenylhydrazine-1-carbothioamide (**3**) in 88% yield (Scheme 1). The structure of **3** was determined using single-crystal X-ray diffraction (Section 2.3) and nuclear magnetic resonance (NMR) spectroscopy (Section 2.2).



Scheme 1. Synthesis of **3**.

2.2. NMR Spectroscopic Analysis

The ^{1}H NMR spectrum of **3** showed two exchangeable singlets, appearing at 9.71 and 10.92 ppm, due to the two NH protons. In addition, the two singlets that appeared at 2.54 and 2.64 ppm were attributed to the protons of the two methyl groups. The ^{13}C NMR spectrum of **3** showed the presence of a singlet at 177.0 ppm due to the thiocarbonyl carbon. Also observed are two singlets at 11.6 and 15.5 ppm due to the carbon atoms of the two methyl groups (see the Supplementary Material for details). It should be noted that the ^{1}H NMR spectrum of crude **3** contains a few signals with very low intensities that are possibly due to the presence of **2**. Following crystallization, the purity of **3** was confirmed by microanalytical analysis (Section 3.2).

2.3. X-ray Crystal Structure Description

The asymmetric unit of the crystal structure comprises three molecules (**M**$_1$, **M**$_2$, and **M**$_3$) and is shown in Figure 1. Each molecule is composed of four groups (**A**–**D**): aryl (**M**$_1$**A**: C1–C6), (**M**$_2$**A**: C19–C24), (**M**$_3$**A**: C37–C42); ethylidenehydrazine-carbothioamide (**M**$_1$**B**:C7–C9, N1–N3, S1), (**M**$_2$**B**:C25-C27, N8-N10, S2), (**M**$_3$**B**:C43-C45, N15-N17, S3); methyltriazole (**M**$_1$**C**: C10–C12, N4–N6), (**M**$_2$**C**: C28–C30, N11–N13), (**M**$_3$**C**: C46–C48, N18–N20); and nitrobenzene (**M**$_1$**D**: C13–C18, N7, O1, O2), (**M**$_2$**D**: C31–C36, N14, O3, O4), (**M**$_3$**D**: C49–C54, N21, O5, O6).

In each of the three unique molecules, group **B** is planar with a maximum deviation from the least squares plane of no more than 0.031(2) Å. Intramolecular N–H . . . N hydrogen bonding contributes to the stabilization of these groups in the planar orientation (Table 1).

Table 1. Intramolecular hydrogen bond geometry of **3**.

Molecule	Distance (Å)		Angle (°)	
M$_1$	N1 . . . N3 N2 . . . N4	2.542(2) 2.707(2)	N1-H1 . . . N3 N2-H2A . . . N4	112.3 132.5
M$_2$	N8 . . . N10 N9 . . . N11	2.546(2) 2.673(2)	N8-H8 . . . N10 N9-H9 . . . N11	113.0 133.2
M$_3$	N15 . . . N17 N16 . . . N18	2.551(2 2.685(2)	N15-H15A . . . N17 N16-H16 . . . N18	114.2 133.4

Groups **B** and **C** are coplanar in each of the three molecules with twist angles of 4.07(8)°, 2.71(8)°, 4.60(9)° for molecules **M**$_1$, **M**$_2$, and **M**$_3$, respectively. Twist angles **A**/**B** are 41.52(5)°, 39.23(5)°, and 24.70(65)° for molecules **M**$_1$, **M**$_2$, and **M**$_3$, respectively, and the corresponding respective **C**/**D** twists are 45.74(42)°, 46.40(4)°, 37.67(5)°. It is notable that in both the cases of **A/B** and **C/D** twist angles, the values for molecule **M**$_3$ deviate significantly from the values for **M**$_1$ and **M**$_2$.

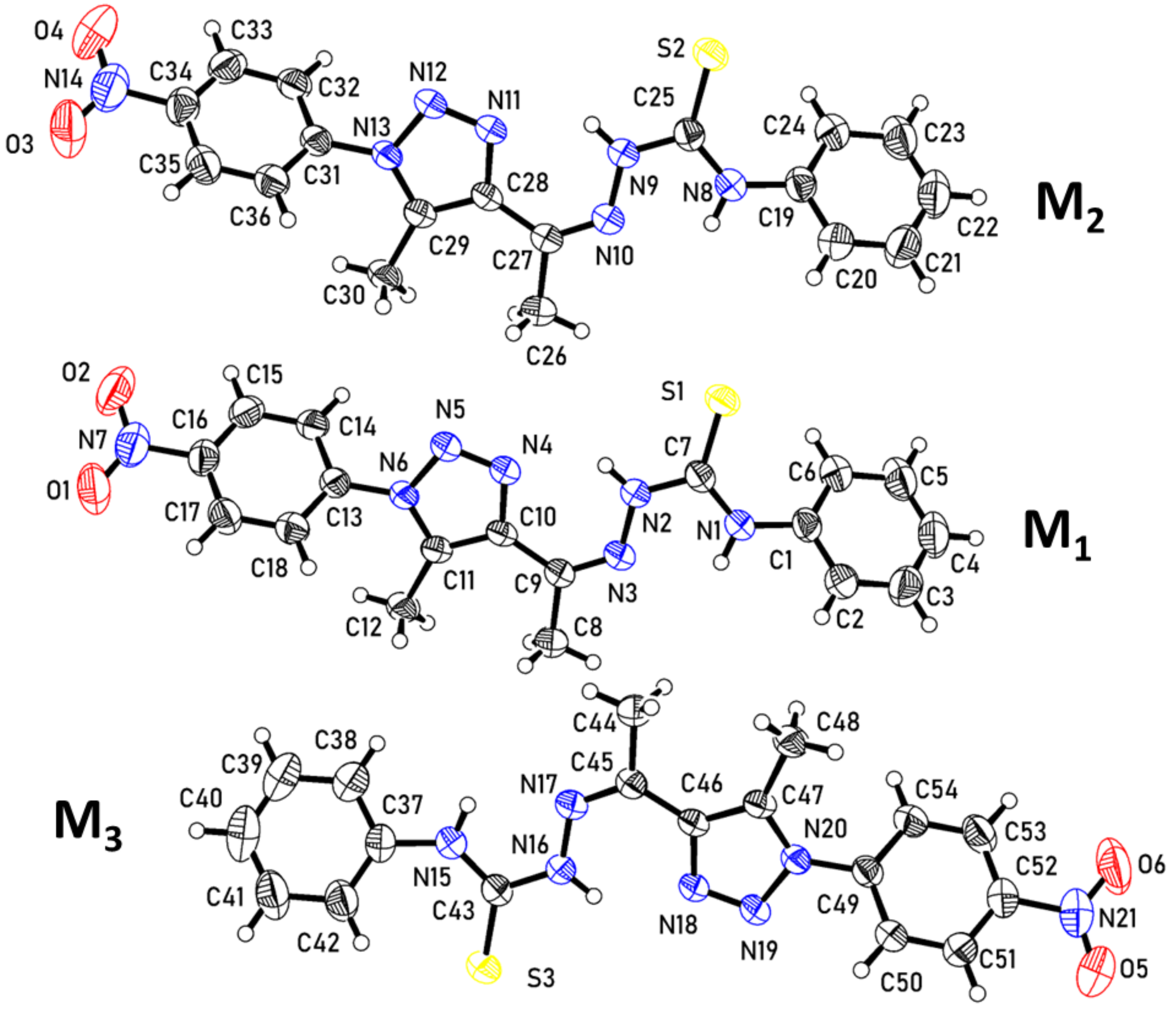


Figure 1. An ortep representation of the three unique molecules in the crystal structure of **3** showing 50% probability atomic displacement ellipsoids.

In the crystal structure, the molecules are stacked to form columns parallel to the *a* axis, with the long axis of the molecules aligned roughly in line with the *b* axis (Figure 2). Two types of columns are observed. In one type of column, molecules $\mathbf{M}_1$ and $\mathbf{M}_2$ alternate, whereas the other type of column consists of only molecules of type $\mathbf{M}_3$.

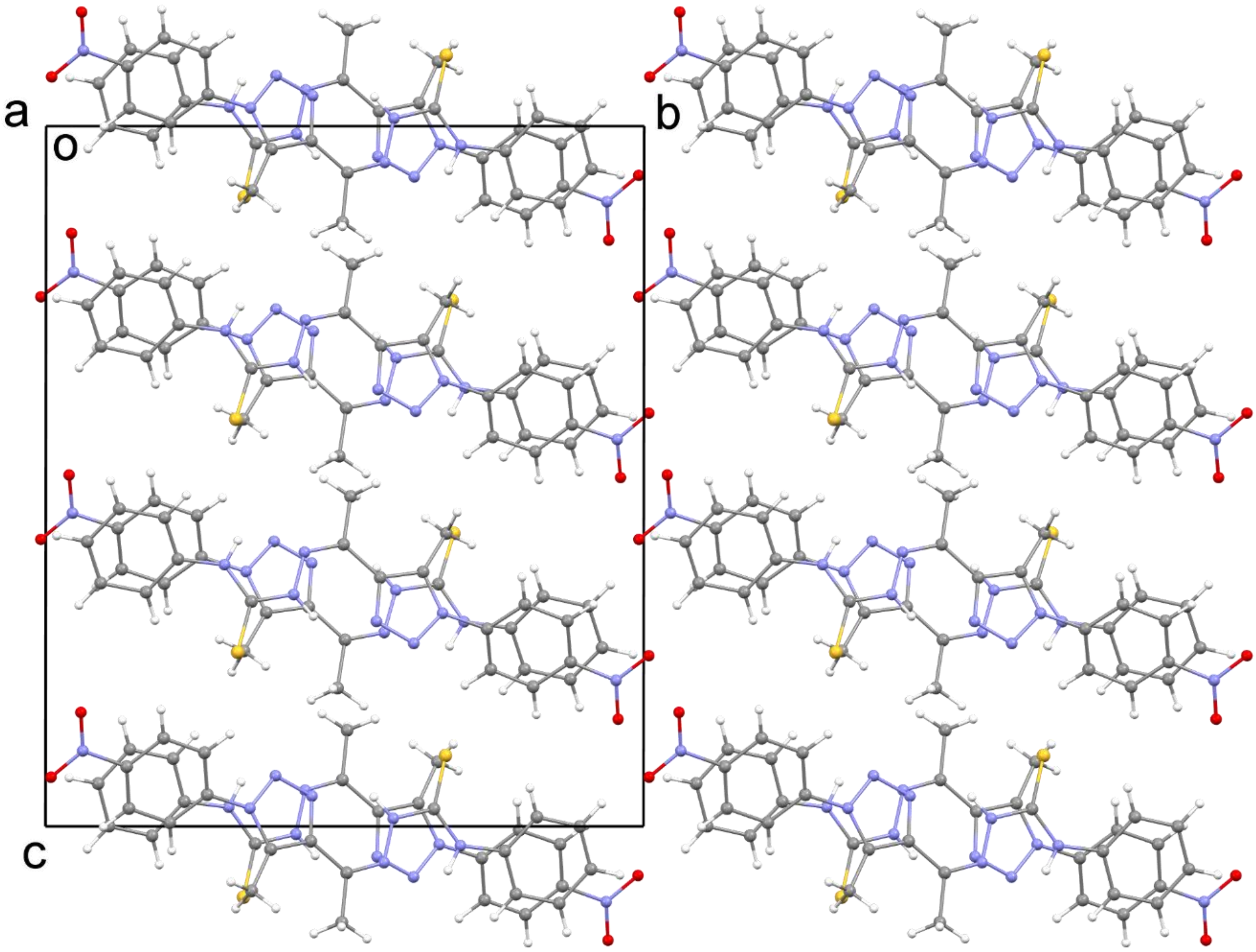


Figure 2. A view of the crystal structure of **3** down the *a* axis with the unit cell outline shown (**a**–**c**).

3. Materials and Methods

3.1. General

A Shimadzu IR Affinity-1 Spectrometer was used to record the IR spectrum of **3**. A JEOLNMR spectrometer was used to record the ^{1}H (500 MHz) and ^{13}C NMR (125 MHz) spectra in deuterated dimethyl sulfoxide (DMSO-d_6). The chemical shift (δ) was recorded in ppm and the coupling constant (*J*) was measured in Hz. Compound **2** was prepared based on a literature procedure [36].

3.2. Synthesis of **3**

A mixture of **1** (0.49 g, 2 mmol) and **2** (0.33 g, 2 mmol) in dry EtOH (20 mL) and concentrated HCl (0.2 mL) was refluxed for 4 h. The yellow solid obtained was filtered, washed with EtOH (2 × 15 mL), and recrystallized from DMF to give yellow crystals of **3** (88%), mp. 236–237 °C. IR: 3271, 3204, 1612, 1600, 1472, 1440 cm^{-1}. ^{1}H NMR: 2.54 (s, 3H, Me), 2.64 (s, 3H, Me), 7.14 (t, *J* = 7.6 Hz, 1H, H4 of Ph), 7.32 (t, *J* = 7.6 Hz, 2H, H3/H5 of Ph), 7.68 (d, *J* = 7.6 Hz, 2H, H2/H6 of Ph), 7.91 (d, *J* = 8.6 Hz, 2H, Ar), 8.44 (d, *J* = 8.6 Hz, 2H, Ar), 9.71 (s, exch., 1H, NH), 10.92 (s, exch., 1H, NH). ^{13}C NMR: 11.6 (Me), 15.5 (Me), 124.3 (C2/C6 of Ph), 125.5 (C3/C5 of Ar), 125.7 (C4 of Ph), 126.7 (C2/C6 of Ar), 128.8 (C3/C5 of Ph), 134.0 (C4 of triazolyl), 139.4 (C1 of Ph), 140.8 (C5 of triazolyl), 143.3 (C1 of Ar), 145.4 (C=N), 148.3 (C4 of Ar), 177.0 (C=S). Anal. Calcd. for $C_{18}H_{17}N_7O_2S$ (395.44): C, 54.67; H, 4.33; N, 24.79%. Found: C, 54.77; H, 4.28; N, 24.88%.

3.3. Data Collection and Structure Refinement Details

An Agilent SuperNova Dual Atlas diffractometer using mirror monochromated MoKα radiation was used to collect single-crystal diffraction data. The structure of **3** was solved by direct methods using SHELXT [37] and refined by full-matrix least-squares methods on F^2 with SHELXL-2014 [38]. $C_{18}H_{17}N_7O_2S$: FW = 395.45; T = 296(2) K; λ = 0.71073 Å; triclinic, $P\bar{1}$; a = 7.7173(3) Å; b = 17.6318(6) Å; c =20.8501(9) Å; α = 89.689(3)°; β = 80.918(4)°; γ = 87.656(3)°; V = 2799.16(19) Å^3; Z = 6; calculated density = 1.408 Mg/m^3; absorption coefficient = 0.204 mm^{-1}; F(000) = 1236; crystal size = 0.390 × 0.330 × 0.070 mm^3; reflections collected = 26,301; independent reflections = 13,464; R(int) = 0.0280; parameters = 764; goodness-of-fit on F^2 = 1.023; R1 = 0.0504 and wR2 = 0.1142 for (I > 2sigma(I)); R1 = 0.0935 and wR2 = 0.1415 for all data; largest difference peak and hole = 0.218 and −0.220 e.Å^{-3}. The X-ray crystallographic data for **3** have been deposited in the Cambridge Crystallographic Data Center with CCDC reference number 2207521.

4. Conclusions

(*Z*)-2-(1-(5-Methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)ethylidene)-*N*-phenylhydrazine-1-carbothioamide was synthesized in high yield and its structure was established using single-crystal X-ray diffraction and nuclear magnetic resonance.

Supplementary Materials: IR, ^{1}H, and ^{13}C NMR spectra, CIF, and checkcif reports for the title compound.

Author Contributions: Conceptualization, B.M.K. and G.A.E.-H.; methodology: B.M.K., B.F.A.-W. and G.A.E.-H.; software, B.M.K.; validation, B.M.K., B.F.A.-W., M.S.B. and G.A.E.-H.; formal analysis: B.M.K., B.F.A.-W., M.S.B. and G.A.E.-H.; investigation: B.M.K., B.F.A.-W., M.S.B. and G.A.E.-H.; resources: B.M.K. and G.A.E.-H.; data curation: B.M.K., B.F.A.-W., M.S.B. and G.A.E.-H.; writing—original draft preparation, B.M.K., B.F.A.-W., M.S.B. and G.A.E.-H.; writing—review and editing, B.M.K., B.F.A.-W., M.S.B. and G.A.E.-H.; visualization, B.M.K.; supervision: B.F.A.-W.; project administration, B.F.A.-W. and M.S.B.; funding acquisition: B.M.K. and G.A.E.-H. All authors have read and agreed to the published version of the manuscript.

Funding: G.A.E.-H. is grateful to the Deanship of Scientific Research, King Saud University for funding through Vice Deanship of Scientific Research Chairs.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data are contained within the article and the supplementary material.

Acknowledgments: We thank Cardiff University and National Research Centre for technical support.

Conflicts of Interest: The authors declare no conflict of interest.

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