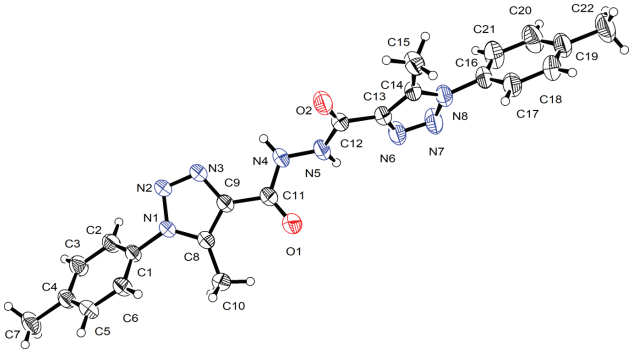
Obaid Fahad Aldosari, Bakr F. Abdel-Wahab, Mohammad Hayal Alotaibi, Amany S. Hegazy, Benson M. Kariuki* and Gamal A. El-Hiti*

# 5-Methyl-*N'*-[5-methyl-1-(4-methylphenyl)-1*H*-1,2,3-triazole-4-carbonyl]-1-(4-methylphenyl)-1*H*-1,2,3-triazole-4-carbohydrazide, $C_{22}H_{22}N_8O_2$




https://doi.org/10.1515/ncrs-2019-0241
Received April 3, 2019; accepted May 13, 2019; available online May 24, 2019

## Abstract

$C_{22}H_{22}N_8O_2$, monoclinic, $P2_1/c$ (no. 14), $a = 15.5175(5)$ Å, $b = 7.9715(3)$ Å, $c = 17.3941(5)$ Å, $\beta = 90.005(3)°$, $V = 2151.61(12)$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.0592$, $wR_{ref}(F^2) = 0.1780$, $T = 293(2)$ K.

**CCDC no.:** 1915609

***Corresponding authors: Benson M. Kariuki,** School of Chemistry, Cardiff University, Main Building, Park Place, Cardiff CF10 3AT, UK, e-mail: kariukib@cardiff.ac.uk; and **Gamal A. El-Hiti,** Department of Optometry, College of Applied Medical Sciences, King Saud University, P.O. Box 10219, Riyadh 11433, Saudi Arabia, e-mail: gelhiti@ksu.edu.sa
**Obaid Fahad Aldosari:** Department of Chemistry, College of Science and Human Studies at Hautat Sudair, Majmaah University, P.O. Box 66, 11952 Majmaah, Saudi Arabia; and Chemistry Department, College of Science and Humanities, Prince Sattam bin Abdulaziz University, P.O. Box 83, 11942 Alkharj, Saudi Arabia
**Bakr F. Abdel-Wahab:** Department of Chemistry, College of Science and Humanities, Shaqra University, Duwadimi, Saudi Arabia; and Applied Organic Chemistry Department, National Research Centre, Dokki, Giza, Egypt
**Mohammad Hayal Alotaibi:** National Center for Petrochemicals Technology, King Abdulaziz City for Science and Technology, P.O. Box 6086, Riyadh 11442, Saudi Arabia
**Amany S. Hegazy:** School of Chemistry, Cardiff University, Main Building, Park Place, Cardiff CF10 3AT, UK

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

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Colourless needle |
| Size: | 0.38 × 0.20 × 0.10 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 0.09 mm$^{-1}$ |
| Diffractometer, scan mode: | SuperNova, Atlas, $\omega$ |
| $\theta_{max}$, completeness: | 29.9°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 19956, 5492, 0.027 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 3531 |
| $N(param)_{refined}$: | 293 |
| Programs: | CrysAlis$^{PRO}$ [1], SHELX [2, 3], WinGX/ORTEP [4] |

## Source of material

The title compound was synthesized as previously reported [5] from reaction of ethyl 2-cyano-3-ethoxyacrylate and 5-methyl-1-(4-methylphenyl)-1*H*-1,2,3-triazole-4-carbohydrazide in boiling ethanol for 2 h. The solid obtained was collected by filtration, washed with ethanol, dried and recrystallized from dimethylformamide to give colourless crystals in 73% yield (Mp. 296–297 °C; lit. Mp. 296–297 °C [5]).

## Experimental details

All H atoms were placed in calculated positions and refined using a riding model. For the methyl groups, C—H bonds were fixed at 0.96 Å and $U_{iso}$(H) set to 1.5$U_{eq}$(C) with free rotation around the C—C bond (HFIX 137 in SHELX [3]). For the rest of the hydrogens, $U_{iso}$(H) was set to 1.2$U_{eq}$(C, N) with aromatic C—H and N—H distances of 0.93 and 0.86 Å respectively.

## Comment

Compounds containing the triazole moiety show various biological activities [6–9]. Therefore, various efficient

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

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.96790(12) | 0.0586(2) | 0.79433(10) | 0.0437(4) |
| C2 | 0.89047(14) | −0.0239(3) | 0.80137(12) | 0.0547(5) |
| H2 | 0.866434 | −0.079612 | 0.759562 | 0.066* |
| C3 | 0.84903(15) | −0.0227(3) | 0.87167(13) | 0.0602(6) |
| H3 | 0.796371 | −0.077359 | 0.876566 | 0.072* |
| C4 | 0.88377(14) | 0.0576(3) | 0.93492(11) | 0.0518(5) |
| C5 | 0.96172(14) | 0.1376(3) | 0.92605(11) | 0.0529(5) |
| H5 | 0.986290 | 0.191577 | 0.968075 | 0.063* |
| C6 | 1.00460(14) | 0.1398(3) | 0.85619(11) | 0.0505(5) |
| H6 | 1.057095 | 0.194996 | 0.851124 | 0.061* |
| C7 | 0.83736(18) | 0.0535(4) | 1.01108(13) | 0.0710(7) |
| H7A | 0.834672 | −0.059948 | 1.029431 | 0.106* |
| H7B | 0.779980 | 0.096337 | 1.004684 | 0.106* |
| H7C | 0.867871 | 0.121508 | 1.047607 | 0.106* |
| C8 | 1.09266(12) | 0.0225(2) | 0.70254(10) | 0.0442(4) |
| C9 | 1.09452(13) | 0.0445(3) | 0.62392(11) | 0.0469(4) |
| C10 | 1.15761(14) | −0.0406(3) | 0.75754(12) | 0.0553(5) |
| H10A | 1.177426 | 0.050192 | 0.789216 | 0.083* |
| H10B | 1.205401 | −0.087267 | 0.729791 | 0.083* |
| H10C | 1.132189 | −0.125651 | 0.789347 | 0.083* |
| C11 | 1.16846(14) | 0.0198(3) | 0.57203(11) | 0.0514(5) |
| C12 | 1.23475(13) | 0.0625(3) | 0.38549(11) | 0.0517(5) |
| C13 | 1.29432(13) | −0.0268(3) | 0.33374(12) | 0.0527(5) |
| C14 | 1.33624(13) | 0.0293(3) | 0.26919(12) | 0.0520(5) |
| C15 | 1.34021(17) | 0.1947(3) | 0.23121(14) | 0.0688(7) |
| H15A | 1.396466 | 0.242328 | 0.238353 | 0.103* |
| H15B | 1.297701 | 0.267686 | 0.253327 | 0.103* |
| H15C | 1.329043 | 0.181677 | 0.177255 | 0.103* |
| C16 | 1.43024(14) | −0.1322(3) | 0.17649(13) | 0.0599(6) |
| C17 | 1.50621(15) | −0.0466(4) | 0.17030(15) | 0.0695(7) |
| H17 | 1.522426 | 0.029961 | 0.207951 | 0.083* |
| C18 | 1.55879(16) | −0.0753(4) | 0.10730(16) | 0.0755(7) |
| H18 | 1.610103 | −0.015771 | 0.102806 | 0.091* |
| C19 | 1.53726(18) | −0.1886(4) | 0.05159(15) | 0.0756(7) |
| C20 | 1.4600(2) | −0.2720(4) | 0.05912(17) | 0.0926(10) |
| H20 | 1.443611 | −0.348057 | 0.021283 | 0.111* |
| C21 | 1.40587(18) | −0.2460(4) | 0.12147(17) | 0.0831(8) |
| H21 | 1.354226 | −0.304452 | 0.125872 | 0.100* |
| C22 | 1.5959(2) | −0.2185(5) | −0.01578(19) | 0.1049(11) |
| H22A | 1.609256 | −0.113447 | −0.039952 | 0.157* |
| H22B | 1.567777 | −0.290794 | −0.052111 | 0.157* |
| H22C | 1.648162 | −0.270555 | 0.001692 | 0.157* |
| N1 | 1.01030(10) | 0.0598(2) | 0.72083(8) | 0.0457(4) |
| N2 | 0.96332(11) | 0.1015(2) | 0.65742(9) | 0.0553(5) |
| N3 | 1.01520(12) | 0.0900(2) | 0.59885(9) | 0.0554(5) |
| N4 | 1.14820(12) | 0.0166(3) | 0.49665(10) | 0.0616(5) |
| H4 | 1.097628 | 0.045055 | 0.481164 | 0.074* |
| N5 | 1.21055(12) | −0.0333(3) | 0.44502(10) | 0.0617(5) |
| H5A | 1.234788 | −0.129357 | 0.451240 | 0.074* |
| N6 | 1.31080(14) | −0.1927(3) | 0.34385(12) | 0.0720(6) |
| N7 | 1.36070(15) | −0.2444(3) | 0.28864(14) | 0.0797(7) |
| N8 | 1.37675(12) | −0.1092(3) | 0.24288(11) | 0.0604(5) |
| O1 | 1.24163(11) | 0.0029(3) | 0.59494(9) | 0.0775(5) |
| O2 | 1.20913(12) | 0.2028(2) | 0.37390(9) | 0.0722(5) |

synthetic processes have been developed for the production of 1,2,3-triazoles [10–12]. Recently, the crystal structures for related compounds have been published [13, 14].

The asymmetric unit consists of one molecule of the title compound. The molecule consists of two tolyl rings [**A** (C1—C7) and **D** (C16–C22)] and two triazolyl rings [**B** (N1—N3, C8—C10) and **C** (N6–N8, C13–C15)]. The twist angles between pairs of rings are 50.6(1)°, 59.2(1)° and 61.0(1)° for **A**/**B**, **B**/**C** and **C**/**D** respectively. The torsion angle (C11—N4—N5—C12) for the formyl dihydrazide group is −124.5(2)°.

The molecules are aligned roughly parallel to [−101] in the crystal. Interactions of π-π type occur between identical tolyl groups (**D**) of neighbouring molecules with a centroid-to-centroid distance of 4.8 Å. Tolyl (**A**) and triazolyl (**B**) rings also interact with a centroid-to-centroid distance of 4.0 Å. Weak C—H···X interactions occur with O and N atoms acting as acceptors and C···O and C···N distances in the range 3.14 Å to 3.46 Å.

**Acknowledgements:** The authors thank Majmaah and Cardiff Universities for their continuous support.

## References

1. Rigaku Oxford Diffraction. CrysAlis PRO. Rigaku Oxford Diffraction, Yarnton, England (2015).
2. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. Acta Crystallogr. **C71** (2015) 3–8.
4. Farrugia, L. J.: WinGX and ORTEP for Windows: an update. J. Appl. Crystallogr. **45** (2012) 849–854.
5. Abdel-Wahab, B. F.; Alotaibi, M. H.; El-Hiti, G. A.: Synthesis of new symmetrical *N*,*N*′-diacylhydrazines and 2-(1,2,3-triazol-4-yl)-1,3,4-oxadiazoles. Lett. Org. Chem. **14** (2017) 591–596.
6. Bonandi, E.; Christodoulou, M. S.; Fumagalli, G.; Perdicchia, D.; Rastelli, G.; Passarella, D.: The 1,2,3-triazole ring as a bioisostere in medicinal chemistry. Drug Discovery Today **22** (2017) 1572–1581.
7. Ferreira, S. B.; Sodero, A. C.; Cardoso, M. F.; Lima, E. S.; Kaiser, C. R.; Silva, F. P.; Ferreira, V. F.: Synthesis, biological activity, and molecular modeling studies of 1*H*-1,2,3-triazole derivatives of carbohydrates as alpha-glucosidases inhibitors. J. Med. Chem. **53** (2010) 2364–2375.
8. Slámová, K.; Marhol, P.; Bezouska, K.; Lindkvist, L.; Hansen, S. G.; Kren, V.; Jensen, H. H.: Synthesis and biological activity of glycosyl-1*H*-1,2,3-triazoles. Bioorg. Med. Chem. Lett. **20** (2010) 4263–4265.
9. Xu, J.; Cao, Y.; Zhang, J.; Yu, S.; Zou, Y.; Chai, X.; Wu, Q.; Zhang, D.; Jiang, Y.; Sun, Q.: Design, synthesis and antifungal activities of novel 1,2,4-triazole derivatives. Eur. J. Med. Chem. **46** (2011) 3142–3148.
10. Fletcher, J. T.; Christensen, J. A.; Villa, E. M.: Tandem synthesis of 1-formyl-1,2,3-triazoles. Tetrahedron Lett. **58** (2017) 4450–4454.
11. Smith, C. D.; Greaney, M. F.: Zinc mediated azide–alkyne ligation to 1,5-and 1,4,5-substituted 1,2,3-triazoles. Org. Lett. **15** (2013) 4826–4829.

12. Totobenazara, J.; Burke, A. J.: New click-chemistry methods for 1,2,3-triazoles synthesis: recent advances and applications. Tetrahedron Lett. **56** (2015) 2853–2859.
13. Abu El-Enin, M. A. B.; Abdel-Wahab, B. F.; Baashen, M.; Ghabbour, H. A.; El-Hiti, G. A.: (*E*)-1-[5-Methyl-1-(*p*-tolyl)-1*H*-1,2,3-triazol-4-yl]-3-{3-[5-methyl-1-(*p*-tolyl)-1*H*-1,2,3-triazol-4-yl]-1-phenyl-1*H*-pyrazol-4-yl}prop-2-en-1-one. IUCrData **2** (2017) x171729.
14. El-Hiti, G. A.; Abdel-Wahab, B. F.; Mostafa, M. S.; Khidre, R. E.; Hegazy, A. S.; Kariuki, B. M.: 5-Methyl-*N*′-(5-methyl-1-phenyl-1*H*-1,2,3-triazole-4-carbonyl)-1-phenyl-1*H*-1,2,3-triazole-4-carbohydrazide. IUCrData **3** (2018) x180424.