

Crystal structure and Hirshfeld surface analysis of 4-azido-2-(3,5-dimethylphenyl)-5-(4-nitrophenyl)-2H-1,2,3-triazole

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Received 7 July 2023

Accepted 8 September 2023

Edited by A. Briceno, Venezuelan Institute of Scientific Research, Venezuela

Keywords: crystal structure; hydrogen bonds; azido group; 2H-1,2,3-triazole; Hirshfeld surface analysis.

CCDC reference: 2293836

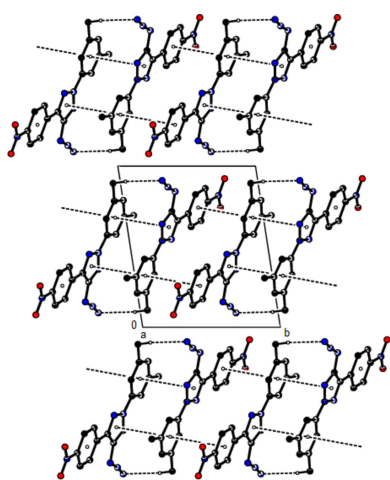
Supporting information: this article has supporting information at journals.iucr.org/e

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In the title compound, C₁₆H₁₃N₇O₂, the 3,5-dimethylphenyl and 4-nitrophenyl rings are inclined to the central 2H-1,2,3-triazole ring by 1.80 (7)° and 1.79 (7)°, respectively, and to one another by 2.16 (7)°. In the crystal, the molecules are linked by C—H···N hydrogen bonds and π – π stacking interactions [centroid-to-centroid distances = 3.7295 (9) and 3.7971 (9) Å], forming ribbons along the *b*-axis direction. These ribbons are connected to each other by weak van der Waals interactions and the stability of the crystal structure is ensured. A Hirshfeld surface analysis of the crystal structure indicates that the most important contributions to the crystal packing are from H···H (31.5%), N···H/H···N (19.2%), O···H/H···O (14.5%), N···C/C···C (10.9%) and C···H/H···C (10.2%) contacts.

1. Chemical context

Triazoles are used as biological agents for their anti-inflammatory, anti-thrombotic and anti-viral activities (Blass *et al.*, 2006; Caliendo *et al.*, 1999; Phillips *et al.*, 2009). At the same time, 2H-1,2,3-triazoles are effective catalysts (Zhao *et al.*, 2008; Yan *et al.*, 2006; Chandrasekhar *et al.*, 2010) and are used as ionic liquids (Yoshida *et al.*, 2012) in organic synthesis. It should be noted that many methods for their preparation are quite complicated, which limits the possibilities of studying the biological activity of these compounds and also their use in other fields of science and technology. In general, the development of new synthesis methods for 2H-1,2,3-triazole derivatives has been a constant in the field of organic synthesis. Since the 1,2,3-triazole ring is an integral part of many medicinal preparations, research on their synthesis (Kamijo *et al.*, 2002; Liu *et al.*, 2008; Ghazlan *et al.*, 2006; Kalisiak *et al.*, 2008; Koszytkowska-Stawińska *et al.*, 2012) and biological activities is constantly increasing (Ferreira *et al.*, 2013; Tan *et al.*, 2002; Prusiner & Sundaralingam, 1973; Toniolo *et al.*, 2017; Caliendo *et al.*, 1999; Blass *et al.*, 2006; von Mutius *et al.*, 2012; Ferreira *et al.*, 2013). In particular, we can mention the synthesis of new 1,2,3-triazole-based drugs against tuberculosis (Sanna *et al.*, 2000). In this regard, the synthesis of 4-azido-2H-1,2,3-triazole derivatives (Tsyrenova *et al.*, 2021)



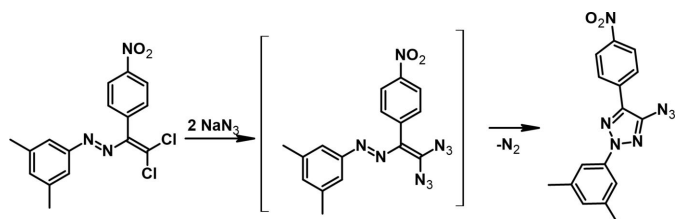
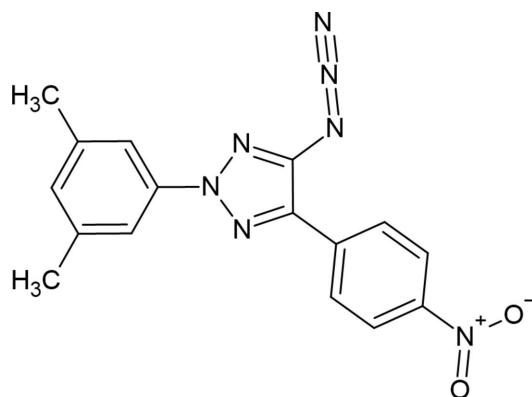


Figure 1
Reaction scheme for the synthesis of the title compound.

from the reaction of dichlorodiazadienes with NaN_3 is a relevant issue. In the following scheme (Fig. 1), on the basis of the synthesis of (*E*)-1-(2,2-dichloro-1-(4-nitrophenyl)vinyl)-2-(3,5-dimethylphenyl) diazene obtained from the reaction of *N*-substituted hydrazone (Maharramov *et al.*, 2018; Nenajdenko *et al.*, 2020; Shikhaliyev *et al.*, 2018, 2019*a,b*, 2021*a,b*) and CCl_4 with NaN_3 , 4-azido-2-(3,5-dimethylphenyl)-5-(4-nitrophenyl)-2*H*-1,2,3-triazole was synthesized and its structure was confirmed by single-crystal X-ray analysis.



2. Structural commentary

The molecule of the title compound, (Fig. 2), except for the methyl H atoms, can be described as essentially planar

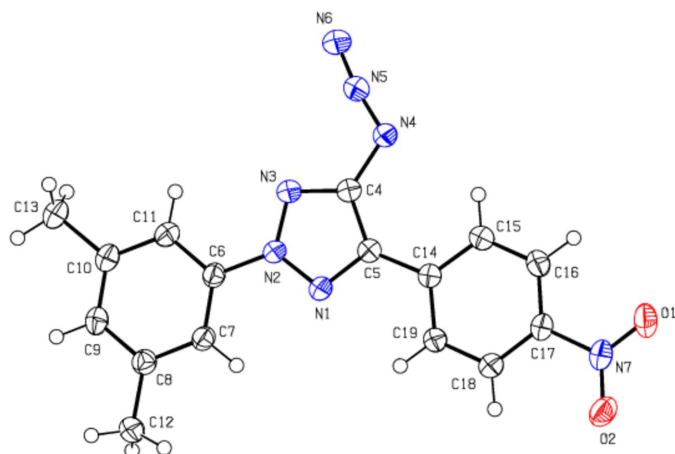


Figure 2
The molecular structure of the title compound, showing the atom labeling and displacement ellipsoids drawn at the 50% probability level.

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C12-H12A\cdots N6^i$	0.98	2.61	3.570 (2)	166

Symmetry code: (i) $-x + 1, -y, -z + 1$.

[maximum deviations = 0.060 (1) \AA for C15 and -0.076 (1) \AA for N6] with the substituents rotated slightly around the triazole centre. The planar 3,5-dimethylphenyl (C6–C11) and 4-nitrophenyl (C14–C19) rings are inclined to the central 2*H*-1,2,3-triazole ring (N1–N3/C4/C5) by 1.80 (7) and 1.79 (7) $^\circ$, respectively, and to one another by 2.16 (7) $^\circ$. The nitro group is co-planar with the benzene ring (C14–C19) to which it is connected [torsion angles $O1-N7-C17-C16 = 0.4$ (2) $^\circ$ and $O2-N7-C17-C16 = -179.18$ (12) $^\circ$]. The azido group ($-N3=N4^+=N5^-$) is almost co-planar with the central 2*H*-1,2,3-triazole ring to which it is connected [$N6-N5-N4 = 171.56$ (14) $^\circ$ and torsion angles $N5-N4-C4-N3 = -1.69$ (19) $^\circ$ and $N5-N4-C4-C5 = 177.54$ (13) $^\circ$].

3. Supramolecular features and Hirshfeld surface analysis

In the crystal, the molecules are linked by $C-H\cdots N$ hydrogen bonds and $\pi-\pi$ stacking interactions [$Cg1\cdots Cg2^i$ =

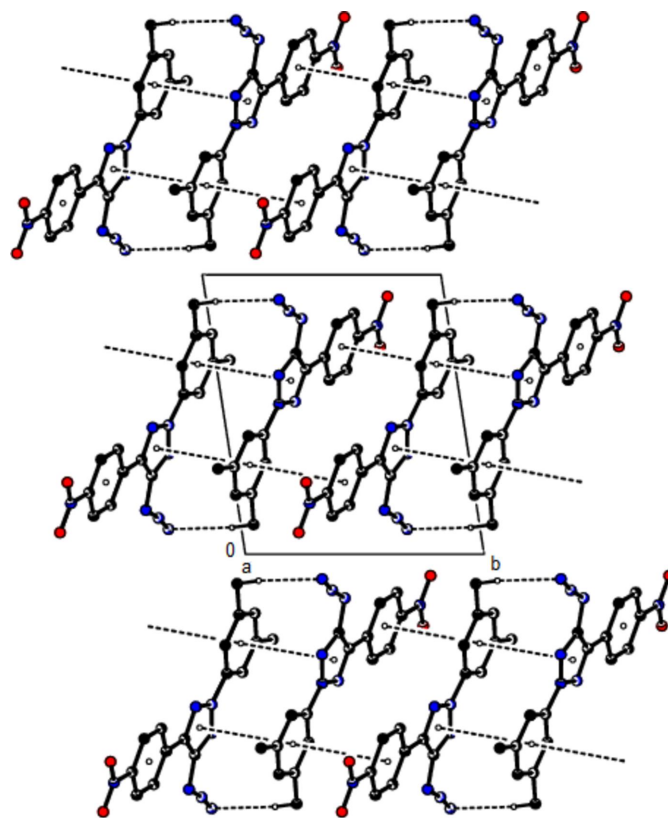


Figure 3
The crystal packing of the title compound viewed along the *a*-axis with intermolecular $C-H\cdots N$ contacts and $\pi-\pi$ stacking interactions shown as dashed lines.

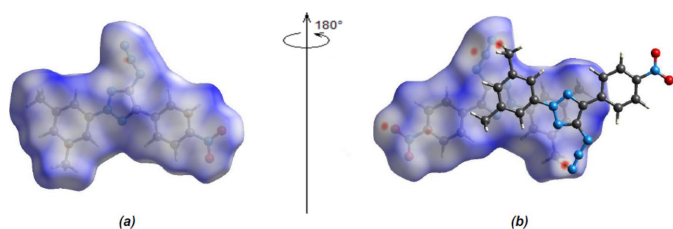


Figure 4
(a) Front and (b) back views of the three-dimensional Hirshfeld surface of the title compound plotted over d_{norm} .

3.7295 (9) Å; slippage = 1.489 Å and $Cg2 \cdots Cg3^{ii}$ = 3.7971 (9) Å; slippage = 1.783 Å; symmetry codes: (i) $-x + 1, -y, -z + 1$, (ii) $-x + 1, -y + 1, -z + 1$; $Cg1$, $Cg2$ and $Cg3$ are the centroids of the 2*H*-1,2,3-triazole (N1–N3/C4/C5), 3,5-di-

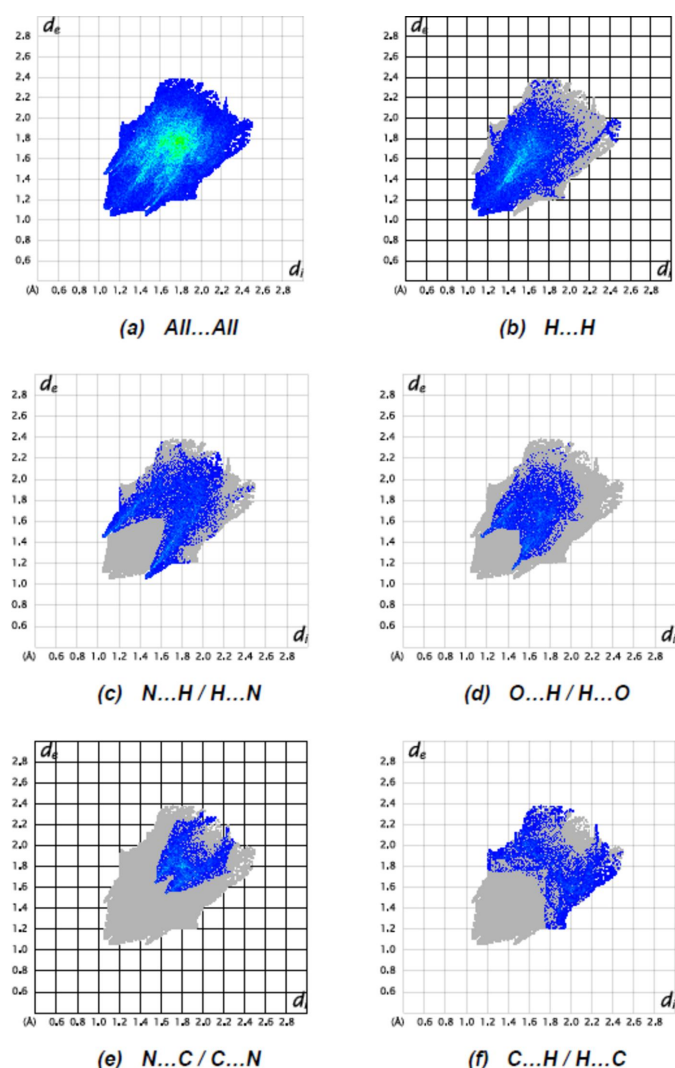


Figure 5
The full two-dimensional fingerprint plots for the title compound, showing (a) all interactions, and delineated into (b) H...H, (c) N...H/H...N, (d) O...H/H...O, (e) N...C/C...N and (f) C...H/H...C interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

Table 2
Summary of short interatomic contacts (Å) in the title compound.

O1...C12	3.36	$x, 1 + y, 1 + z$
N5...O1	2.98	$1 - x, 1 - y, 2 - z$
H13A...O2	2.90	$1 - x, 1 - y, 1 - z$
H7...H13A	2.59	$-1 + x, y, z$
H19...H18	2.37	$-x, 1 - y, 1 - z$
H12A...N6	2.61	$1 - x, -y, 1 - z$
H13C...H11	2.51	$2 - x, -y, 1 - z$
H15...N6	2.75	$2 - x, 1 - y, 2 - z$
H12A...C12	2.93	$-x, -y, -z$

methylphenyl (C6–C11) and 4-nitrophenyl (C14–C19) rings, respectively], forming ribbons along the *b*-axis direction (Tables 1 and 2; Fig. 3). These ribbons are connected to each other by weak van der Waals interactions and the stability of the crystal structure is ensured.

In order to investigate the intermolecular interactions in a visual manner, a Hirshfeld surface analysis was performed using *CrystalExplorer 17.5* (Spackman *et al.*, 2021). The bright-red spots on the Hirshfeld surface mapped over d_{norm} (Fig. 4) indicate the presence of C–H...N interactions. The fingerprint plots (Fig. 5) are given for all contacts, and those delineated into H...H (31.5%), N...H/H...N (19.2%), O...H/H...O (14.5%), N...C/C...N (10.9%), C...H/H...C (10.2%), C...C (5.2%), O...N/N...O (4.0%), O...C/C...O (2.4%) and N...N (2.1%). The most important contributions to the crystal packing are H...H and N...H/H...N contacts.

4. Database survey

The ten most similar compounds found in a search of the Cambridge Structural Database (CSD, Version 5.42, update of September 2021; Groom *et al.*, 2016) for the 2*H*-1,2,3-triazole group are JADSEP (Canseco-González *et al.*, 2015), JELTEC (Zukerman-Schpector *et al.*, 2017), HUYTEC (Haslinger *et al.*, 2015), FEVLIE, FEVLOK, FEVLUQ, FEVMAX, FEVMEB and FEVMOL (Farrán *et al.*, 2018) and SECQUO (Altimari *et al.*, 2012).

In the crystal of JADSEP, molecules are linked *via* C–H...I hydrogen bonds, forming slabs parallel to the *ab* plane. Within the slabs there are weak π – π interactions present involving the mesityl and phenyl rings. In the crystal of JELTEC, the three-dimensional packing is stabilized by a combination of methylene-C–H...O, methylene-C–H... π , C–H... π and nitro-O... π (nitrobenzene) interactions, along with weak π (triazolyl)– π (nitrobenzene) contacts. In the crystal of HUYTEC, the water molecules are connected into [010] chains by O–H...O hydrogen bonds, while O–H...N hydrogen bonds connect the water molecules to the organic molecules, generating corrugated (100) sheets. In the crystals of FEVLIE, FEVLOK, FEVLUQ, FEVMAX, FEVMEB and FEVMOL, there are no $C_{\text{ar}}\text{--H}\cdots\text{F}\cdots\text{C}$ intramolecular contacts. If the aryl groups were coplanar with the triazole ring, the C–F and the C–H atoms would be too close. Thus, the steric effect is more efficient than the weak hydrogen bond. Only compound FEVMOL clearly shows a hydrogen bond (O–H...N). In the crystal of SECQUO, the molecules

Table 3

Experimental details.

Crystal data	
Chemical formula	C ₁₆ H ₁₃ N ₇ O ₂
<i>M_r</i>	335.33
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	7.4400 (8), 9.920 (1), 11.5200 (13)
α , β , γ (°)	93.071 (9), 105.349 (10), 108.879 (11)
<i>V</i> (Å ³)	766.71 (16)
<i>Z</i>	2
Radiation type	Synchrotron, $\lambda = 0.79313$ Å
μ (mm ⁻¹)	0.13
Crystal size (mm)	0.09 × 0.05 × 0.03
Data collection	
Diffractometer	Rayonix SX165 CCD
Absorption correction	Multi-scan (<i>SCALA</i> ; Evans, 2006)
<i>T_{min}</i> , <i>T_{max}</i>	0.969, 0.990
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	13293, 3466, 2976
<i>R_{int}</i>	0.025
(sin θ/λ) _{max} (Å ⁻¹)	0.650
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.046, 0.133, 1.08
No. of reflections	3466
No. of parameters	229
H-atom treatment	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.27, -0.22

Computer programs: *Marccd* (Doyle, 2011), *iMosflm* (Battye *et al.*, 2011), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

pack in a head-to-tail arrangement along the *a*-axis direction with closest inter-centroid distances between the triazole rings of 3.7372 (12) Å.

5. Synthesis and crystallization

The title compound was synthesized according to a literature protocol (Tsyrenova *et al.*, 2021). A 20 ml screw-neck vial was charged with DMSO (20 ml), (*E*)-1-[2,2-dichloro-1-(4-nitrophenyl)vinyl]-2-(3,5-dimethylphenyl)diazene (350 mg, 1 mmol) and sodium azide (NaN₃; 390 mg; 3 mmol). After 1–3 h (until TLC analysis showed complete consumption of the corresponding triazole), the reaction mixture was poured into a 0.01 *M* solution of HCl (100 ml, pH = 2–3), and extracted with dichloromethane (3 × 20 ml). The combined organic phase was washed with water (3 × 50 ml), brine (30 ml), dried over anhydrous Na₂SO₄ and concentrated *in vacuo* using a rotary evaporator. The residue was purified by column chromatography on silica gel using appropriate mixtures of hexane and dichloromethane (*v/v*: 3/1–1/1). Red solid (yield 75%); m.p. 375 K. Analysis calculated for C₁₆H₁₃N₇O₂ (*M* = 335.33): ¹H NMR (300 MHz, chloroform-*d*) δ 8.84 (*s*, 1H), 8.40–8.27 (*m*, 1H), 8.20 (*d*, *J* = 8.2 Hz, 1H), 7.68 (*s*, 2H), 7.62 (*t*, *J* = 8.0 Hz, 1H), 7.02 (*s*, 1H), 2.44 (*s*, 6H). ¹³C NMR (75 MHz, CDCl₃) δ 143.9, 134.7, 134.4, 130.1, 127.3, 126.1, 125.0, 118.3, 116.5, 111.4, 16.8. The compound was dissolved in dichloromethane and then left at room temperature for slow evaporation; red crystal of the title compound suitable for X-rays started to form after *ca* 2 d.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were positioned geometrically and allowed to ride on their parent atoms (C–H = 0.95–0.98 Å) with *U*_{iso}(H) = 1.2 or 1.5*U*_{eq}(C). Owing to poor agreement between observed and calculated intensities, sixteen outliers (6 $\bar{1}0$ 1, $\bar{5}$ 12 0, $\bar{7}$ 11 0, $\bar{6}$ 12 1, 6 $\bar{1}2$ 1, 7 $\bar{1}1$ 1, 5 $\bar{1}0$ 1, $\bar{2}$ 2 6, 2 $\bar{1}$ 1, 1 1 4, $\bar{2}$ 4 1, $\bar{1}$ 1 1, 0 2 0, $\bar{2}$ 1 2, $\bar{1}$ 4 4, 2 0 5) were omitted during the final refinement cycle.

Acknowledgements

The authors' contributions are as follows. Conceptualization, ZA, MA and AB; synthesis, AA, AN and GTA; X-ray analysis, VK, SG and MA; writing (review and editing of the manuscript) ZA, MA and AB; funding acquisition, NQS, and AM; supervision, NQS, MA and AB.

Funding information

This work was funded by the Science Development Foundation under the President of the Republic of Azerbaijan, grant No. EIF-BGM-4-RFTF-1/2017–21/13/4.

References

- Altimari, J. M., Healy, P. C. & Henderson, L. C. (2012). *Acta Cryst.* **E68**, o3159.
- Battye, T. G. G., Kontogiannis, L., Johnson, O., Powell, H. R. & Leslie, A. G. W. (2011). *Acta Cryst.* **D67**, 271–281.
- Blass, B. E., Coburn, K., Lee, W., Fairweather, N., Fluxe, A., Wu, S., Janusz, J. M., Murawsky, M., Fadayel, G. M., Fang, B., Hare, M., Ridgeway, J., White, R., Jackson, C., Djandjighian, L., Hedges, R., Wireko, F. C. & Ritter, A. L. (2006). *Bioorg. Med. Chem. Lett.* **16**, 4629–4632.
- Caliendo, G., Fiorino, F., Grieco, P., Perissutti, E., Santagada, V., Meli, R., Raso, G. M., Zanesco, A. & De Nucci, G. (1999). *Eur. J. Med. Chem.* **34**, 1043–1051.
- Canseco-González, D., García, J. J. & Flores-Alamo, M. (2015). *Acta Cryst.* **E71**, o1041–o1042.
- Chandrasekhar, S., Kumar, T. P., Haribabu, K. & Reddy, C. R. (2010). *Tetrahedron Asymmetry*, **21**, 2372–2375.
- Doyle, R. A. (2011). *Marccd software manual*. Rayonix LLC, Evanston, IL 60201, USA.
- Evans, P. (2006). *Acta Cryst.* **D62**, 72–82.
- Farrán, M. Á., Bonet, M. Á., Claramunt, R. M., Torralba, M. C., Alkorta, I. & Elguero, J. (2018). *Acta Cryst.* **C74**, 513–522.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Ferreira, V. F., da Rocha, D. R., da Silva, F. C., Ferreira, P. G., Boechat, N. A. & Magalhães, J. L. (2013). *Expert Opin. Ther. Pat.* **23**, 319–331.
- Ghozlan, S. A., Abdelhamid, I. A., Ibrahim, H. M. & Elnagdi, M. H. (2006). *Arkivoc*, **2006**, 53–60.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Haslinger, S., Laus, G., Wurst, K. & Schottenberger, H. (2015). *Acta Cryst.* **E71**, o945–o946.
- Kalisiak, J., Sharpless, K. B. & Fokin, V. V. (2008). *Org. Lett.* **10**, 3171–3174.
- Kamijo, S., Jin, T., Huo, Z. & Yamamoto, Y. (2002). *Tetrahedron Lett.* **43**, 9707–9710.

- Koszytkowska-Stawińska, M., Mironiuk-Puchalska, E. & Rowicki, T. (2012). *Tetrahedron*, **68**, 214–225.
- Liu, Y., Yan, W., Chen, Y., Petersen, J. L. & Shi, X. (2008). *Org. Lett.* **10**, 5389–5392.
- Maharramov, A. M., Shikhaliyev, N. Q., Suleymanova, G. T., Gurbanov, A. V., Babayeva, G. V., Mammadova, G. Z., Zubkov, F. I., Nenajdenko, V. G., Mahmudov, K. T. & Pombeiro, A. J. (2018). *Dyes Pigments*, **159**, 135–141.
- Mutius, E. von & Drazen, J. M. (2012). *N. Engl. J. Med.* **366**, 827–834.
- Nenajdenko, V. G., Shikhaliyev, N. G., Maharramov, A. M., Bagirova, K. N., Suleymanova, G. T., Novikov, A. S., Khrustalev, V. N. & Tskhovrebov, A. G. (2020). *Molecules*, **25**, 5013.
- Phillips, O. A., Udo, E. E., Abdel-Hamid, M. E. & Varghese, R. (2009). *Eur. J. Med. Chem.* **44**, 3217–3227.
- Prusiner, P. T. & Sundaralingam, M. (1973). *Nature New Biol.* **244**, 116–118.
- Sanna, P., Carta, A. & Nikookar, M. E. (2000). *Eur. J. Med. Chem.* **35**, 535–543.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Shikhaliyev, N. G., Maharramov, A. M., Bagirova, K. N., Suleymanova, G. T., Tsyrenova, B. D., Nenajdenko, V. G., Novikov, A. S., Khrustalev, V. N. & Tskhovrebov, A. G. (2021a). *Mendeleev Commun.* **31**, 191–193.
- Shikhaliyev, N. G., Maharramov, A. M., Suleymanova, G. T., Babayeva, G. V., Mammadova, G. Z., Shikhaliyeva, I. M., Babazade, A. A. & Nenajdenko, V. G. (2021b). *Organic Chemistry, (part iii)*, 67–75.
- Shikhaliyev, N. G., Suleymanova, G. T., İsrayılova, A. A., Ganbarov, K. G., Babayeva, G. V., Garazadeh, K. A., Mammadova, G. Z. & Nenajdenko, V. G. (2019b). *Organic Chemistry, (part vi)*, 64–73.
- Shikhaliyev, N. Q., Ahmadova, N. E., Gurbanov, A. V., Maharramov, A. M., Mammadova, G. Z., Nenajdenko, V. G., Zubkov, F. I., Mahmudov, K. T. & Pombeiro, A. J. (2018). *Dyes Pigments*, **150**, 377–381.
- Shikhaliyev, N. Q., Kuznetsov, M. L., Maharramov, A. M., Gurbanov, A. V., Ahmadova, N. E., Nenajdenko, V. G., Mahmudov, K. T. & Pombeiro, A. J. (2019a). *CrystEngComm*, **21**, 5032–5038.
- Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). *J. Appl. Cryst.* **54**, 1006–1011.
- Spek, A. L. (2020). *Acta Cryst.* **E76**, 1–11.
- Tan, S. L., Pause, A., Shi, Y. & Sonenberg, N. (2002). *Nat. Rev. Drug Discov.* **1**, 867–881.
- Toniolo, N., Taveri, G., Hurle, K., Roether, J., Ercole, P., Dlouhy, I. & Boccaccini, A. R. (2017). *Journal of Ceramic Science and Technology*, **8**, 411–420.
- Tsyrenova, B. D., Khrustalev, V. N. & Nenajdenko, V. G. (2021). *Org. Biomol. Chem.* **19**, 8140–8152.
- Yan, Z. Y., Niu, Y. N., Wei, H. L., Wu, L. Y., Zhao, Y. B. & Liang, Y. M. (2006). *Tetrahedron Asymmetry*, **17**, 3288–3293.
- Yoshida, Y., Takizawa, S. & Sasai, H. (2012). *Tetrahedron Asymmetry*, **23**, 843–851.
- Zhao, Y. B., Zhang, L. W., Wu, L. Y., Zhong, X., Li, R. & Ma, J. T. (2008). *Tetrahedron Asymmetry*, **19**, 1352–1355.
- Zukerman-Schpector, J., Dallasta Pedroso, S., Sousa Madureira, L., Weber Paixão, M., Ali, A. & Tiekink, E. R. T. (2017). *Acta Cryst.* **E73**, 1716–1720.

supporting information

Acta Cryst. (2023). E79, 905-909 [https://doi.org/10.1107/S2056989023007855]

Crystal structure and Hirshfeld surface analysis of 4-azido-2-(3,5-dimethylphenyl)-5-(4-nitrophenyl)-2H-1,2,3-triazole

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Computing details

Data collection: *Marccd* (Doyle, 2011); cell refinement: *iMosflm* (Battye *et al.*, 2011); data reduction: *iMosflm* (Battye *et al.*, 2011); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2020).

4-Azido-2-(3,5-dimethylphenyl)-5-(4-nitrophenyl)-2H-1,2,3-triazole

Crystal data

$C_{16}H_{13}N_7O_2$	$Z = 2$
$M_r = 335.33$	$F(000) = 348$
Triclinic, $P\bar{1}$	$D_x = 1.452 \text{ Mg m}^{-3}$
$a = 7.4400$ (8) Å	Synchrotron radiation, $\lambda = 0.79313$ Å
$b = 9.920$ (1) Å	Cell parameters from 1000 reflections
$c = 11.5200$ (13) Å	$\theta = 2.1\text{--}28.0^\circ$
$\alpha = 93.071$ (9) $^\circ$	$\mu = 0.13 \text{ mm}^{-1}$
$\beta = 105.349$ (10) $^\circ$	$T = 100 \text{ K}$
$\gamma = 108.879$ (11) $^\circ$	Prism, red
$V = 766.71$ (16) Å ³	$0.09 \times 0.05 \times 0.03 \text{ mm}$

Data collection

Rayonix SX165 CCD diffractometer	3466 independent reflections
/f scan	2976 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (<i>SCALA</i> ; Evans, 2006)	$R_{\text{int}} = 0.025$
$T_{\text{min}} = 0.969$, $T_{\text{max}} = 0.990$	$\theta_{\text{max}} = 31.0^\circ$, $\theta_{\text{min}} = 2.1^\circ$
13293 measured reflections	$h = -9 \rightarrow 9$
	$k = -12 \rightarrow 12$
	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	229 parameters
Least-squares matrix: full	0 restraints
$R[F^2 > 2\sigma(F^2)] = 0.046$	Primary atom site location: difference Fourier map
$wR(F^2) = 0.133$	Secondary atom site location: difference Fourier map
$S = 1.08$	
3466 reflections	

Hydrogen site location: inferred from
neighbouring sites
H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0764P)^2 + 0.1976P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$
Extinction correction: SHELXL,
 $F_c^* = kFc[1 + 0.001x\text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.074 (7)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.18207 (18)	0.76197 (12)	0.92289 (10)	0.0446 (3)
O2	-0.03369 (17)	0.69879 (12)	0.74319 (11)	0.0423 (3)
N1	0.47322 (16)	0.30620 (12)	0.54670 (10)	0.0295 (3)
N2	0.59776 (16)	0.23870 (12)	0.53829 (10)	0.0280 (3)
N3	0.74669 (16)	0.25675 (12)	0.64053 (10)	0.0295 (3)
N4	0.83239 (17)	0.38805 (13)	0.84246 (10)	0.0326 (3)
N5	0.96905 (17)	0.34031 (13)	0.87091 (10)	0.0332 (3)
N6	1.10029 (19)	0.30585 (14)	0.91074 (12)	0.0387 (3)
N7	0.11839 (19)	0.69804 (13)	0.81792 (11)	0.0343 (3)
C4	0.71164 (19)	0.34137 (14)	0.71900 (12)	0.0291 (3)
C5	0.54163 (19)	0.37371 (14)	0.66201 (12)	0.0282 (3)
C6	0.57495 (19)	0.15443 (14)	0.42750 (12)	0.0279 (3)
C7	0.41649 (19)	0.14311 (14)	0.32646 (12)	0.0284 (3)
H7	0.3240	0.1885	0.3323	0.034*
C8	0.3946 (2)	0.06480 (14)	0.21667 (12)	0.0305 (3)
C9	0.5333 (2)	-0.00175 (14)	0.21177 (13)	0.0325 (3)
H9	0.5185	-0.0562	0.1373	0.039*
C10	0.6915 (2)	0.00999 (15)	0.31301 (13)	0.0331 (3)
C11	0.7126 (2)	0.08921 (15)	0.42256 (13)	0.0323 (3)
H11	0.8196	0.0984	0.4929	0.039*
C12	0.2269 (2)	0.05374 (16)	0.10597 (13)	0.0361 (3)
H12A	0.1186	-0.0385	0.0952	0.054*
H12B	0.2744	0.0595	0.0341	0.054*
H12C	0.1783	0.1330	0.1163	0.054*
C13	0.8416 (2)	-0.05959 (18)	0.30537 (15)	0.0420 (4)
H13A	0.9732	0.0151	0.3219	0.063*
H13B	0.8026	-0.1137	0.2236	0.063*
H13C	0.8465	-0.1254	0.3657	0.063*
C14	0.43732 (19)	0.45871 (14)	0.70518 (12)	0.0280 (3)
C15	0.5043 (2)	0.53230 (15)	0.82427 (12)	0.0319 (3)
H15	0.6221	0.5286	0.8796	0.038*
C16	0.4004 (2)	0.61052 (15)	0.86228 (12)	0.0330 (3)
H16	0.4453	0.6600	0.9433	0.040*

C17	0.2301 (2)	0.61509 (14)	0.77992 (12)	0.0303 (3)
C18	0.1596 (2)	0.54308 (15)	0.66123 (13)	0.0326 (3)
H18	0.0421	0.5477	0.6063	0.039*
C19	0.2631 (2)	0.46465 (15)	0.62443 (12)	0.0317 (3)
H19	0.2160	0.4142	0.5437	0.038*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0571 (7)	0.0466 (6)	0.0388 (6)	0.0239 (5)	0.0224 (5)	0.0007 (5)
O2	0.0407 (6)	0.0442 (6)	0.0509 (6)	0.0247 (5)	0.0162 (5)	0.0061 (5)
N1	0.0288 (5)	0.0328 (6)	0.0295 (6)	0.0152 (5)	0.0077 (4)	0.0019 (4)
N2	0.0263 (5)	0.0323 (6)	0.0273 (5)	0.0144 (4)	0.0065 (4)	0.0030 (4)
N3	0.0273 (5)	0.0334 (6)	0.0283 (6)	0.0133 (4)	0.0059 (4)	0.0041 (4)
N4	0.0292 (6)	0.0376 (6)	0.0305 (6)	0.0154 (5)	0.0050 (5)	−0.0014 (5)
N5	0.0331 (6)	0.0367 (6)	0.0288 (6)	0.0134 (5)	0.0065 (5)	0.0032 (5)
N6	0.0351 (6)	0.0455 (7)	0.0364 (6)	0.0196 (5)	0.0057 (5)	0.0058 (5)
N7	0.0402 (6)	0.0317 (6)	0.0383 (6)	0.0160 (5)	0.0193 (5)	0.0052 (5)
C4	0.0276 (6)	0.0317 (6)	0.0286 (6)	0.0123 (5)	0.0069 (5)	0.0046 (5)
C5	0.0278 (6)	0.0304 (6)	0.0272 (6)	0.0116 (5)	0.0077 (5)	0.0035 (5)
C6	0.0290 (6)	0.0288 (6)	0.0290 (6)	0.0127 (5)	0.0106 (5)	0.0035 (5)
C7	0.0268 (6)	0.0289 (6)	0.0311 (7)	0.0117 (5)	0.0091 (5)	0.0027 (5)
C8	0.0298 (6)	0.0294 (6)	0.0320 (7)	0.0104 (5)	0.0093 (5)	0.0026 (5)
C9	0.0360 (7)	0.0306 (6)	0.0344 (7)	0.0139 (5)	0.0143 (6)	0.0014 (5)
C10	0.0342 (7)	0.0332 (7)	0.0379 (7)	0.0167 (6)	0.0146 (6)	0.0052 (5)
C11	0.0306 (6)	0.0369 (7)	0.0334 (7)	0.0174 (6)	0.0093 (5)	0.0058 (5)
C12	0.0359 (7)	0.0384 (7)	0.0314 (7)	0.0152 (6)	0.0049 (6)	−0.0029 (5)
C13	0.0411 (8)	0.0484 (8)	0.0449 (8)	0.0268 (7)	0.0136 (6)	0.0019 (7)
C14	0.0279 (6)	0.0292 (6)	0.0273 (6)	0.0110 (5)	0.0080 (5)	0.0031 (5)
C15	0.0312 (6)	0.0357 (7)	0.0279 (6)	0.0133 (5)	0.0056 (5)	0.0021 (5)
C16	0.0366 (7)	0.0344 (7)	0.0278 (6)	0.0131 (6)	0.0091 (5)	0.0004 (5)
C17	0.0329 (7)	0.0301 (6)	0.0324 (7)	0.0136 (5)	0.0139 (5)	0.0038 (5)
C18	0.0310 (6)	0.0366 (7)	0.0320 (7)	0.0160 (5)	0.0075 (5)	0.0028 (5)
C19	0.0319 (7)	0.0361 (7)	0.0275 (6)	0.0159 (6)	0.0055 (5)	−0.0002 (5)

Geometric parameters (Å, °)

O1—N7	1.2284 (16)	C9—H9	0.9500
O2—N7	1.2296 (17)	C10—C11	1.3944 (19)
N1—N2	1.3261 (15)	C10—C13	1.5085 (19)
N1—C5	1.3410 (16)	C11—H11	0.9500
N2—N3	1.3435 (15)	C12—H12A	0.9800
N2—C6	1.4268 (17)	C12—H12B	0.9800
N3—C4	1.3323 (17)	C12—H12C	0.9800
N4—N5	1.2317 (16)	C13—H13A	0.9800
N4—C4	1.4242 (17)	C13—H13B	0.9800
N5—N6	1.1299 (17)	C13—H13C	0.9800
N7—C17	1.4681 (17)	C14—C15	1.3996 (18)

C4—C5	1.4069 (18)	C14—C19	1.4020 (18)
C5—C14	1.4640 (18)	C15—C16	1.3866 (19)
C6—C11	1.3876 (18)	C15—H15	0.9500
C6—C7	1.3897 (18)	C16—C17	1.383 (2)
C7—C8	1.3905 (18)	C16—H16	0.9500
C7—H7	0.9500	C17—C18	1.3886 (19)
C8—C9	1.4043 (19)	C18—C19	1.3798 (19)
C8—C12	1.4995 (19)	C18—H18	0.9500
C9—C10	1.389 (2)	C19—H19	0.9500
N2—N1—C5	104.92 (11)	C6—C11—H11	120.4
N1—N2—N3	115.31 (11)	C10—C11—H11	120.4
N1—N2—C6	121.86 (11)	C8—C12—H12A	109.5
N3—N2—C6	122.82 (11)	C8—C12—H12B	109.5
C4—N3—N2	102.74 (11)	H12A—C12—H12B	109.5
N5—N4—C4	113.98 (11)	C8—C12—H12C	109.5
N6—N5—N4	171.56 (14)	H12A—C12—H12C	109.5
O1—N7—O2	123.90 (12)	H12B—C12—H12C	109.5
O1—N7—C17	117.93 (12)	C10—C13—H13A	109.5
O2—N7—C17	118.17 (12)	C10—C13—H13B	109.5
N3—C4—C5	110.21 (11)	H13A—C13—H13B	109.5
N3—C4—N4	122.95 (12)	C10—C13—H13C	109.5
C5—C4—N4	126.83 (12)	H13A—C13—H13C	109.5
N1—C5—C4	106.82 (11)	H13B—C13—H13C	109.5
N1—C5—C14	120.20 (12)	C15—C14—C19	119.20 (12)
C4—C5—C14	132.97 (12)	C15—C14—C5	122.24 (12)
C11—C6—C7	121.96 (12)	C19—C14—C5	118.56 (12)
C11—C6—N2	119.59 (12)	C16—C15—C14	120.60 (13)
C7—C6—N2	118.43 (11)	C16—C15—H15	119.7
C6—C7—C8	119.39 (12)	C14—C15—H15	119.7
C6—C7—H7	120.3	C17—C16—C15	118.67 (12)
C8—C7—H7	120.3	C17—C16—H16	120.7
C7—C8—C9	118.67 (13)	C15—C16—H16	120.7
C7—C8—C12	120.20 (12)	C16—C17—C18	122.11 (13)
C9—C8—C12	121.13 (12)	C16—C17—N7	119.61 (12)
C10—C9—C8	121.71 (13)	C18—C17—N7	118.28 (12)
C10—C9—H9	119.1	C19—C18—C17	118.84 (13)
C8—C9—H9	119.1	C19—C18—H18	120.6
C9—C10—C11	119.17 (13)	C17—C18—H18	120.6
C9—C10—C13	120.99 (13)	C18—C19—C14	120.58 (12)
C11—C10—C13	119.84 (13)	C18—C19—H19	119.7
C6—C11—C10	119.10 (13)	C14—C19—H19	119.7
C5—N1—N2—N3	-0.26 (15)	C8—C9—C10—C11	0.4 (2)
C5—N1—N2—C6	-179.58 (11)	C8—C9—C10—C13	-178.68 (13)
N1—N2—N3—C4	0.38 (15)	C7—C6—C11—C10	0.0 (2)
C6—N2—N3—C4	179.70 (12)	N2—C6—C11—C10	-178.46 (12)
N2—N3—C4—C5	-0.34 (14)	C9—C10—C11—C6	-0.1 (2)

N2—N3—C4—N4	179.00 (12)	C13—C10—C11—C6	178.98 (13)
N5—N4—C4—N3	-1.69 (19)	N1—C5—C14—C15	-179.28 (12)
N5—N4—C4—C5	177.54 (13)	C4—C5—C14—C15	1.9 (2)
N2—N1—C5—C4	0.03 (14)	N1—C5—C14—C19	1.52 (19)
N2—N1—C5—C14	-179.08 (11)	C4—C5—C14—C19	-177.31 (14)
N3—C4—C5—N1	0.21 (15)	C19—C14—C15—C16	-0.1 (2)
N4—C4—C5—N1	-179.10 (12)	C5—C14—C15—C16	-179.32 (12)
N3—C4—C5—C14	179.16 (13)	C14—C15—C16—C17	-0.4 (2)
N4—C4—C5—C14	-0.1 (2)	C15—C16—C17—C18	0.5 (2)
N1—N2—C6—C11	178.22 (12)	C15—C16—C17—N7	-179.59 (12)
N3—N2—C6—C11	-1.1 (2)	O1—N7—C17—C16	0.4 (2)
N1—N2—C6—C7	-0.33 (19)	O2—N7—C17—C16	-179.18 (12)
N3—N2—C6—C7	-179.60 (11)	O1—N7—C17—C18	-179.69 (12)
C11—C6—C7—C8	-0.3 (2)	O2—N7—C17—C18	0.70 (19)
N2—C6—C7—C8	178.25 (12)	C16—C17—C18—C19	-0.1 (2)
C6—C7—C8—C9	0.5 (2)	N7—C17—C18—C19	-179.95 (12)
C6—C7—C8—C12	-178.79 (12)	C17—C18—C19—C14	-0.5 (2)
C7—C8—C9—C10	-0.6 (2)	C15—C14—C19—C18	0.6 (2)
C12—C8—C9—C10	178.72 (13)	C5—C14—C19—C18	179.82 (12)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C7—H7...N1	0.95	2.47	2.7900 (18)	100
C12—H12 <i>A</i> ...N6 ⁱ	0.98	2.61	3.570 (2)	166
C15—H15...N4	0.95	2.51	3.174 (2)	127
C19—H19...N1	0.95	2.47	2.810 (2)	101

Symmetry code: (i) $-x+1, -y, -z+1$.