Zhe Wang, Wei Hong* and Hao Wang*

Crystal structure of methyl 2-((4-((2-nitrophenoxy)methyl)-1H,1,2,3-triazol-1-yl)methyl) benzoate, C₁₈H₁₆N₄O₅

https://doi.org/10.1515/ncrs-2018-0041
Received April 19, 2018; accepted July 19, 2018; available online July 31, 2018

Abstract

C₁₈H₁₆N₄O₅, triclinic, Pī (no. 2), a = 8.4076(6) Å, b = 8.6630(5) Å, c = 12.0763(8) Å, α = 102.046(3)°, β = 99.280(3)°, γ = 99.820(3)°, V = 836.70(9) Å³, Z = 2, R₁(F) = 0.0396, wR₂(F²) = 0.0972, T = 150(2) K.

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

Source of material

1-Nitro-2-(prop-2-yn-1-yl)benzene (200 mg, 1.13 mmol) and methy 2-(azidomethyl)benzoate (220 mg, 1.13 mmol) were added to tert-butanol:water = 8 mL:8 mL in a 50 mL flask. After that CuSO₄·5H₂O (50 mg, 0.21 mmol) and VcNa (100 mg, 0.52 mmol) were added. The mixture was stirred at room temperature for 15 h. The solution was diluted with water and extracted with ethyl acetate. The organic layer was treated as riding: C—H…π···π interaction play a major role in the molecular encapsulation

organic residue was purified by silica gel column chromatography, eluted with ethyl acetate and petroleum ether (1:1, v/v) to give a yellow solid (yield 63%, 250 mg); m.p. 104.8–105.1 °C; ¹H NMR (400 MHz, CDCl₃) 8.05 (d, J = 8.0, 1H, Ar-H), 7.83 (m, J = 8.0, 1H, Ar-H), 7.52 (m, J = 24.0, 2H, Ar-H), 7.42 (td, J = 16.0, 1H, Ar-H), 7.32 (dd, J = 16.0, 1H, Ar-H), 7.15 (dt, J = 8.0, 1H, Ar-H), 7.05 (dd, J = 16.0, 1H, Ar-H), 5.98 (s, 2H, CH₂), 5.37 (s, 2H, CH₂), 3.93 (s, 3H, CH₃). Crystals of the title compound suitable for X-ray analysis were obtained from dichloromethane: methanol (1:1).

Experimental details

All H-atoms were placed in calculated positions and treated as riding: C—H…π···π interaction and the C—H…π interaction play a major role in the molecular encapsulation

1,2,3-Triazole is an important five-membered nitrogen-containing heterocyclic compound [4], as substituted 1,2,3-triazoles have a large biological spectrum, and hence are used as antimicrobial [5], antihistaminic [6], anticancer [7], and antitubercular agents [8]. Modification of existing drugs containing heterocyclic compound [4], as substituted 1,2,3-triazoles have a large biological spectrum, and hence are used as antimicrobial [5], antihistaminic [6], anticancer [7], and antitubercular agents [8]. Modification of existing drugs with 1,2,3-triazole ring to improve the physical and chemical properties of drugs and the biological activity has become one of the hot spots in drug research and development in recent years.

Table 1: Data collection and handling.

| Crystal: | Block, colorless |
| Size: | 0.32 × 0.23 × 0.11 mm |
| Wavelength: | Mo Kα radiation (0.71073 Å) |
| μ: | 0.11 mm⁻¹ |
| Diffractometer, scan mode: | Bruker SMART, φ and ω-scans |
| θmax, completeness: | 25°, >99% |
| N(hkl)measured, N(hkl)gt: | 13503, 2945, 0.043 |
| Criterion for Iobs: | Iobs > 2 σ(Iobs), 2345 |
| N(param)refined: | 245 |
| Programs: | Bruker programs [1], SHELX [2, 3] |

*Corresponding authors: Wei Hong, School of Chemistry and Chemical Engineering, North Minzu University, Yinchuan, Ningxia, China, e-mail: hongwei336@hotmail.com; and Hao Wang, School of Pharmacy, Ningxia Medical University, Yinchuan, Ningxia, China, e-mail: paxhw@yahoo.co.uk

Zhe Wang: School of Pharmacy, Ningxia Medical University, Yinchuan, Ningxia, China

© 2018 Zhe Wang et al., published by De Gruyter. This work is licensed under the Creative Commons Attribution-NonCommercial-NoDerivatives 4.0 License.
in the title crystal structure. All bond lengths and angles in the crystal structure are within the normal range. The dihedral angle between the triazole ring and the 2-nitro phenyl moiety is 5.6°. The dihedral angle between the triazole ring and the benzoate moiety is 79.7°. The N—N lengths are in the range of 1.3160(12)–1.3408(18) Å, and the N(3)—N(2)—N(1) angle is 107.39(13)°. There are typical C—H···O non-classical hydrogen bonds [9] and C—H···N non-classical hydrogen bonds in the crystal structure, and the presence of these hydrogen bonds stabilizes the crystal structure. In the crystal structure, the title structure shows at least three hydrogen bonds, C9—H9B···O1 (angle = 136.6°, C···O distance = 2.51 Å), C1—H1C···O4 (angle = 174.4°, C···O distance = 2.45 Å) and C17—H17···N2 (angle = 153.5°, C···N distance = 2.35 Å).

Acknowledgements: This work was supported by the National Natural Science Foundation of China (No. 81660588, 81773582). The authors would also like to thank Dr. Qingfeng Yang for the help with the analysis of the crystal structure data.

References


