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Crystal structure of methyl 2-((4-((2-nitrophenoxy)methyl)-1*H*-1,2,3-triazol-1-yl)methyl)benzoate, $C_{18}H_{16}N_4O_5$

Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.32 × 0.23 × 0.11 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.11 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
$\theta_{\max}$, completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	13503, 2945, 0.043
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2345
$N(\text{param})_{\text{refined}}$:	245
Programs:	Bruker programs [1], SHELX [2, 3]

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Abstract

$C_{18}H_{16}N_4O_5$, triclinic, $P\bar{1}$ (no. 2), $a = 8.4076(6)$ Å, $b = 8.6630(5)$ Å, $c = 12.0763(8)$ Å, $\alpha = 102.046(3)^\circ$, $\beta = 97.453(3)^\circ$, $\gamma = 99.280(3)^\circ$, $V = 836.70(9)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0396$, $wR_{\text{ref}}(F^2) = 0.0972$, $T = 150(2)$ K.

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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 methyl 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 $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (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 dried over Na_2SO_4 . After being filtered and concentrated, the

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, CDCl_3) 8.05 (d, $J = 8.0$, 1H, Ar-*H*), 7.83 (m, $J = 8.0$, 2H, Ar-*H*), 7.52 (m, $J = 24.0$, 2H, Ar-*H*), 7.42 (td, $J = 16.0$, 1H, Ar-*H*), 7.32 (dd, $J = 8.0$, 1H, Ar-*H*), 7.15 (dt, $J = 8.0$, 1H, Ar-*H*), 7.05 (ddd, $J = 16.0$, 1H, Ar-*H*), 5.98 (s, 2H, CH_2), 5.37 (s, 2H, CH_2), 3.93 (s, 3H, CH_3). 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 = 0.95–0.99 Å, with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}(\text{parent C-atom})$.

Discussion

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 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.

There is one molecule in the asymmetric unit (*cf.* the figure). The intermolecular π - π interaction and the C–H $\cdots \pi$ interaction play a major role in the molecular encapsulation

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

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O1	−0.01300(17)	0.90989(17)	1.13160(12)	0.0415(4)
O2	−0.21560(14)	0.79185(14)	0.98537(10)	0.0283(3)
O3	0.20752(13)	0.97190(13)	0.50510(9)	0.0219(3)
O4	0.30660(17)	1.19395(16)	0.69004(11)	0.0371(3)
O5	0.54414(18)	1.33980(18)	0.70737(12)	0.0491(4)
N1	−0.07676(16)	0.69827(16)	0.66396(11)	0.0194(3)
N2	−0.16297(16)	0.58914(16)	0.57133(12)	0.0211(3)
N3	−0.10834(16)	0.62464(16)	0.48047(12)	0.0210(3)
N4	0.42263(18)	1.24631(17)	0.64845(13)	0.0261(4)
C1	−0.3210(2)	0.8863(2)	1.04268(17)	0.0349(5)
H1A	−0.431627	0.856403	0.997580	0.052*
H1B	−0.278612	1.000848	1.050113	0.052*
H1C	−0.324308	0.865645	1.119186	0.052*
C2	−0.0615(2)	0.8167(2)	1.03980(15)	0.0240(4)
C3	0.0425(2)	0.71685(19)	0.97706(14)	0.0219(4)
C4	0.1719(2)	0.6819(2)	1.04606(15)	0.0264(4)
H4	0.187706	0.721566	1.126997	0.032*
C5	0.2774(2)	0.5908(2)	0.99898(16)	0.0292(4)
H5	0.364446	0.567103	1.047063	0.035*
C6	0.2552(2)	0.5342(2)	0.88113(16)	0.0296(4)
H6	0.327278	0.471368	0.847905	0.036*
C7	0.1277(2)	0.5691(2)	0.81122(15)	0.0258(4)
H7	0.113860	0.529653	0.730344	0.031*
C8	0.0199(2)	0.66052(19)	0.85714(14)	0.0202(4)
C9	−0.1165(2)	0.6972(2)	0.77758(14)	0.0231(4)
H9A	−0.216805	0.615541	0.769888	0.028*
H9B	−0.139697	0.803522	0.812238	0.028*
C10	0.0344(2)	0.8044(2)	0.63306(14)	0.0205(4)
H10	0.110293	0.893324	0.681834	0.025*
C11	0.01394(19)	0.75617(18)	0.51664(14)	0.0175(4)
C12	0.1035(2)	0.82741(19)	0.43488(14)	0.0207(4)
H12A	0.168895	0.752944	0.397466	0.025*
H12B	0.026447	0.852292	0.374834	0.025*
C13	0.31234(19)	1.06010(19)	0.45574(14)	0.0185(4)
C14	0.3213(2)	1.0217(2)	0.33959(15)	0.0242(4)
H14	0.249872	0.929780	0.290471	0.029*
C15	0.4333(2)	1.1157(2)	0.29442(16)	0.0304(4)
H15	0.438301	1.086836	0.214794	0.036*
C16	0.5378(2)	1.2509(2)	0.36352(17)	0.0313(5)
H16	0.613219	1.315591	0.331656	0.038*
C17	0.5313(2)	1.2906(2)	0.47893(16)	0.0278(4)
H17	0.603260	1.382802	0.527272	0.033*
C18	0.42052(19)	1.19703(19)	0.52509(14)	0.0201(4)

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 1.3176(19)–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).

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