organic compounds

5-(4-Ethoxybenzyl)-1*H*-tetrazole

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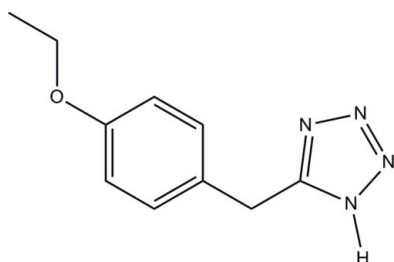Correspondence e-mail: yugp2005@yahoo.com.cn

In the title molecule, $C_{10}H_{12}N_4O$, the tetrazole and benzene rings form a dihedral angle of 67.52 (2)°. In the crystal, intermolecular N—H⋯N hydrogen bonds link the molecules into chains along the *a* axis. The relatively short distance of 3.760 (3) Å between the centroids of the tetrazole rings suggests the existence of π–π interactions.

Related literature

For details of the biological activities of sodium-glucose co-transporter 2 (SGLT2) inhibitors, see: Arakawa *et al.* (2001); Meng *et al.* (2008). For bond-length data, see: Allen *et al.* (1987).

Experimental

Crystal data

$C_{10}H_{12}N_4O$	V = 1003.2 (3) Å^3
M_r = 204.24	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 4.9291 (10) Å	μ = 0.09 mm^{-1}
b = 18.145 (4) Å	T = 113 K
c = 11.363 (2) Å	0.34 × 0.06 × 0.04 mm
β = 99.19 (3)°	

Data collection

Rigaku Saturn CCD area-detector diffractometer	6870 measured reflections
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2007)	1768 independent reflections
T_{min} = 0.969, T_{max} = 0.996	1487 reflections with $I > 2\sigma(I)$
	R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.103	
S = 1.10	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
1768 reflections	$\Delta\rho_{min}$ = −0.34 e Å^{-3}
142 parameters	
1 restraint	

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯N4^i	0.91 (1)	1.90 (1)	2.7897 (16)	166 (1)

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

Data collection: *CrystalClear* (Rigaku, 2007); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

References

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S1. Comment

Sodium-Glucose Cotransporter 2 (SGLT2) inhibitors constitute a new class of antidiabetic agents (Arakawa *et al.*, 2001; Meng *et al.*, 2008). The title compound, (I), was prepared as an intermediate of a new class of SGLT2 inhibitors designed in our laboratories.

In (I) (Fig. 1), all bond lengths in the molecular are normal (Allen *et al.*, 1987). Atoms O1/C2/C9/C10 lie in the benzene ring (C3—C8) plane with a maximun deviation of 0.045 (2) Å for O1 . The tetrazole ring (N1—N4/C1) forms the dihedral angle of 67.52 (2) ° with the benzene ring (C3—C8).

In the crystal structure, relatively short distance of 3.760 (3) Å between the centroids of tetrazole rings suggests an existence of π—π interactions. Intermolecular N—H···N hydrogen bonds (Table 1) link the molecules related by translation along axis *a* into chains.

S2. Experimental

A round-bottomed flask was charged with 1.61 g (10 mmol) of 4-ethoxyphenylacetonitrile, 3.25 g (50 mmol) of sodium azide and 2.67 g (50 mmol) of ammonium chloride and 50 ml of DMF, and the resulting mixture was stirred at 120 ° C for 15 h. On complete cooling, the mixture was filtered to remove the existing solid and the filtrate was evaporated on a rotary evaporator equipped with an oil pump, and the residue was dissolved in 100 ml of water. The aqueous solution thus obtained was adjusted to pH = 2 with concentrated hydrochloric acid, when it turned turbid. This turbid mixture was cooled with ice-water bath and stirred for complete crystallization. The precipitated crystals were collected *via* suction filtration and dried at 60° C *in vacuo* to afford the title product as white crystals 1.68 g (yield 82.3%). Crystals suitable for single-crystal X-ray diffraction were obtained *via* slow evaporation at room temperature of a solution of the pure title compound in dichloromethane/petroleum ether.

S3. Refinement

All H atoms were found on difference maps. C-bound H atoms were placed in idealized positions (C—H 0.93 - 0.97 Å), and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) for aryl and methylene and 1.5U_{eq}(C) for the methyl H atoms. The N-bound H atom was refined isotropically.
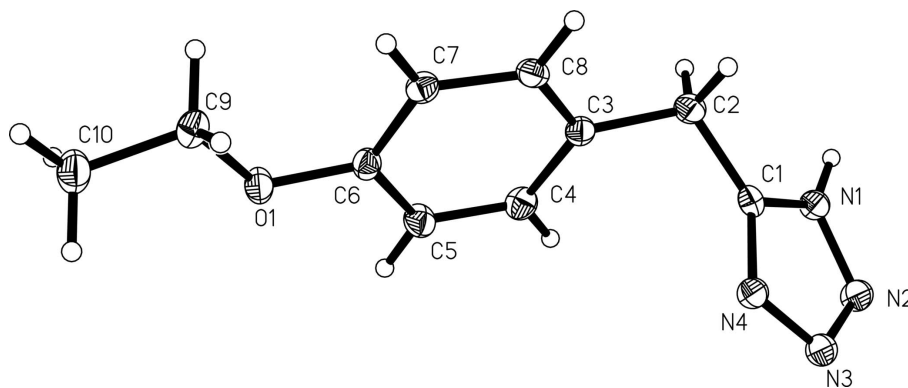
Figure 1

The molecular structure of (I) with the atomic labels and 40% probability displacement ellipsoids.

5-(4-Ethoxybenzyl)-1*H*-tetrazole

Crystal data

$C_{10}H_{12}N_4O$	$F(000) = 432$
$M_r = 204.24$	$D_x = 1.352$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 3598 reflections
$a = 4.9291 (10)$ Å	$\theta = 1.1–27.9°$
$b = 18.145 (4)$ Å	$\mu = 0.09$ mm^{-1}
$c = 11.363 (2)$ Å	$T = 113$ K
$\beta = 99.19 (3)°$	Needle, colourless
$V = 1003.2 (3)$ Å^3	$0.34 \times 0.06 \times 0.04$ mm
$Z = 4$	

Data collection

Rigaku Saturn CCD area-detector diffractometer	6870 measured reflections
Radiation source: rotating anode	1768 independent reflections
Multilayer monochromator	1487 reflections with $I > 2\sigma(I)$
Detector resolution: 14.63 pixels mm^{-1}	$R_{int} = 0.048$
ω and φ scans	$\theta_{max} = 25.0°$, $\theta_{min} = 2.1°$
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2007)	$h = -5 \rightarrow 5$
	$k = -21 \rightarrow 19$
$T_{min} = 0.969$, $T_{max} = 0.996$	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.043$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.103$	
$S = 1.10$	$w = 1/[\sigma^2(F_o^2) + (0.067P)^2]$
1768 reflections	where $P = (F_o^2 + 2F_c^2)/3$
142 parameters	$(\Delta/\sigma)_{max} = 0.004$
1 restraint	$\Delta\rho_{max} = 0.24$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.34$ e Å^{-3}
	Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.354 (18)

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	$U_{iso}*/U_{eq}$
O1	0.09591 (18)	0.17311 (5)	0.48558 (8)	0.0241 (3)
N1	1.0194 (2)	0.48750 (6)	0.33955 (10)	0.0214 (3)
N2	0.9379 (2)	0.55573 (6)	0.36698 (11)	0.0252 (3)
N3	0.6749 (2)	0.55229 (5)	0.36251 (11)	0.0242 (3)
N4	0.5824 (2)	0.48270 (5)	0.33271 (10)	0.0211 (3)
C1	0.8013 (2)	0.44332 (7)	0.31894 (11)	0.0187 (3)
C2	0.8099 (3)	0.36438 (7)	0.28353 (12)	0.0225 (4)
H2A	0.9937	0.3456	0.3100	0.027*
H2B	0.7744	0.3613	0.1972	0.027*
C3	0.6058 (3)	0.31536 (7)	0.33333 (12)	0.0195 (3)
C4	0.5715 (3)	0.32048 (7)	0.45271 (12)	0.0234 (3)
H4	0.6676	0.3562	0.5012	0.028*
C5	0.3974 (3)	0.27333 (7)	0.49957 (12)	0.0243 (4)
H5	0.3739	0.2781	0.5788	0.029*
C6	0.2566 (2)	0.21864 (7)	0.42871 (12)	0.0198 (3)
C7	0.2858 (2)	0.21280 (7)	0.30957 (11)	0.0204 (3)
H7	0.1916	0.1766	0.2614	0.024*
C8	0.4583 (3)	0.26196 (7)	0.26324 (12)	0.0206 (3)
H8	0.4748	0.2588	0.1830	0.025*
C9	−0.0624 (3)	0.11821 (7)	0.41507 (12)	0.0244 (4)
H9A	0.0578	0.0838	0.3832	0.029*
H9B	−0.1819	0.1410	0.3490	0.029*
C10	−0.2311 (3)	0.07851 (8)	0.49534 (14)	0.0319 (4)
H10A	−0.1106	0.0560	0.5601	0.048*
H10B	−0.3407	0.0412	0.4505	0.048*
H10C	−0.3489	0.1131	0.5264	0.048*
H1	1.197 (2)	0.4782 (8)	0.3340 (13)	0.030 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0280 (6)	0.0230 (5)	0.0223 (6)	−0.0068 (4)	0.0069 (4)	0.0006 (4)
N1	0.0145 (6)	0.0227 (6)	0.0272 (7)	0.0000 (5)	0.0042 (5)	0.0004 (5)
N2	0.0213 (6)	0.0226 (6)	0.0322 (7)	−0.0013 (5)	0.0056 (5)	−0.0012 (5)
N3	0.0212 (6)	0.0227 (6)	0.0292 (7)	−0.0017 (5)	0.0062 (5)	−0.0001 (5)
N4	0.0174 (6)	0.0204 (6)	0.0255 (7)	−0.0009 (5)	0.0037 (5)	−0.0005 (4)

C1	0.0161 (7)	0.0232 (7)	0.0170 (7)	−0.0019 (5)	0.0033 (5)	0.0031 (5)
C2	0.0184 (7)	0.0232 (7)	0.0270 (8)	0.0011 (5)	0.0066 (6)	−0.0001 (5)
C3	0.0165 (7)	0.0185 (7)	0.0239 (8)	0.0030 (5)	0.0042 (6)	0.0025 (5)
C4	0.0241 (8)	0.0220 (7)	0.0232 (8)	−0.0034 (5)	0.0009 (6)	−0.0033 (5)
C5	0.0292 (8)	0.0262 (7)	0.0177 (8)	−0.0028 (6)	0.0047 (6)	−0.0009 (5)
C6	0.0184 (7)	0.0182 (7)	0.0228 (8)	0.0006 (5)	0.0034 (6)	0.0033 (5)
C7	0.0200 (7)	0.0195 (7)	0.0211 (8)	−0.0007 (5)	0.0017 (6)	−0.0020 (5)
C8	0.0222 (7)	0.0220 (7)	0.0183 (8)	0.0036 (5)	0.0055 (6)	−0.0001 (5)
C9	0.0237 (7)	0.0204 (7)	0.0284 (9)	−0.0029 (6)	0.0022 (6)	0.0011 (5)
C10	0.0268 (8)	0.0277 (8)	0.0418 (10)	−0.0040 (6)	0.0077 (7)	0.0055 (6)

Geometric parameters (Å, º)

O1—C6	1.3753 (15)	C4—C5	1.3778 (18)
O1—C9	1.4296 (16)	C4—H4	0.9300
N1—C1	1.3313 (16)	C5—C6	1.3914 (18)
N1—N2	1.3533 (15)	C5—H5	0.9300
N1—H1	0.906 (9)	C6—C7	1.3881 (18)
N2—N3	1.2908 (16)	C7—C8	1.3922 (18)
N3—N4	1.3666 (14)	C7—H7	0.9300
N4—C1	1.3244 (16)	C8—H8	0.9300
C1—C2	1.4903 (18)	C9—C10	1.5113 (18)
C2—C3	1.5184 (17)	C9—H9A	0.9700
C2—H2A	0.9700	C9—H9B	0.9700
C2—H2B	0.9700	C10—H10A	0.9600
C3—C8	1.3845 (18)	C10—H10B	0.9600
C3—C4	1.3965 (18)	C10—H10C	0.9600
C6—O1—C9	117.34 (10)	C4—C5—H5	119.9
C1—N1—N2	109.29 (10)	C6—C5—H5	119.9
C1—N1—H1	129.7 (10)	O1—C6—C7	124.72 (12)
N2—N1—H1	120.9 (10)	O1—C6—C5	115.34 (11)
N3—N2—N1	106.26 (10)	C7—C6—C5	119.94 (12)
N2—N3—N4	110.35 (10)	C6—C7—C8	118.98 (12)
C1—N4—N3	106.37 (10)	C6—C7—H7	120.5
N4—C1—N1	107.74 (11)	C8—C7—H7	120.5
N4—C1—C2	127.53 (11)	C3—C8—C7	121.81 (12)
N1—C1—C2	124.71 (11)	C3—C8—H8	119.1
C1—C2—C3	114.42 (10)	C7—C8—H8	119.1
C1—C2—H2A	108.7	O1—C9—C10	107.31 (11)
C3—C2—H2A	108.7	O1—C9—H9A	110.3
C1—C2—H2B	108.7	C10—C9—H9A	110.3
C3—C2—H2B	108.7	O1—C9—H9B	110.3
H2A—C2—H2B	107.6	C10—C9—H9B	110.3
C8—C3—C4	118.14 (11)	H9A—C9—H9B	108.5
C8—C3—C2	120.93 (12)	C9—C10—H10A	109.5
C4—C3—C2	120.86 (12)	C9—C10—H10B	109.5
C5—C4—C3	120.89 (12)	H10A—C10—H10B	109.5

C5—C4—H4	119.6	C9—C10—H10C	109.5
C3—C4—H4	119.6	H10A—C10—H10C	109.5
C4—C5—C6	120.20 (13)	H10B—C10—H10C	109.5
C1—N1—N2—N3	−0.13 (14)	C2—C3—C4—C5	−176.44 (11)
N1—N2—N3—N4	0.10 (14)	C3—C4—C5—C6	1.3 (2)
N2—N3—N4—C1	−0.03 (14)	C9—O1—C6—C7	−3.22 (17)
N3—N4—C1—N1	−0.05 (14)	C9—O1—C6—C5	177.21 (11)
N3—N4—C1—C2	−178.47 (12)	C4—C5—C6—O1	177.84 (11)
N2—N1—C1—N4	0.12 (15)	C4—C5—C6—C7	−1.75 (19)
N2—N1—C1—C2	178.59 (12)	O1—C6—C7—C8	−179.16 (11)
N4—C1—C2—C3	−36.43 (19)	C5—C6—C7—C8	0.39 (18)
N1—C1—C2—C3	145.41 (13)	C4—C3—C8—C7	−1.88 (18)
C1—C2—C3—C8	138.01 (13)	C2—C3—C8—C7	175.05 (11)
C1—C2—C3—C4	−45.14 (17)	C6—C7—C8—C3	1.45 (19)
C8—C3—C4—C5	0.49 (19)	C6—O1—C9—C10	−177.01 (10)

Hydrogen-bond geometry (Å, º)

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$N4[i]	0.91 (1)	1.90 (1)	2.7897 (16)	166 (1)

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