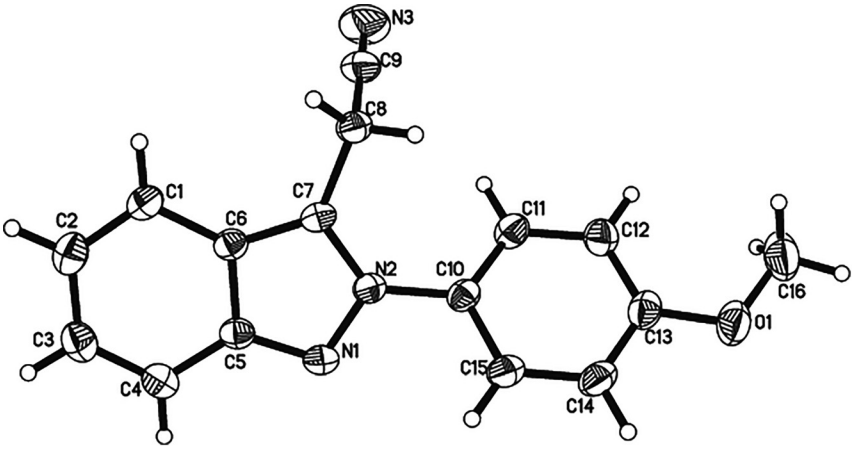
Beibei Cui, Xia Mi* and Jingyu Zhang*

Crystal structure of 2-(2-(4-methoxyphenyl)-2*H*-indazol-3-yl)acetonitrile, $C_{16}H_{13}N_3O$



https://doi.org/10.1515/ncrs-2023-0025
Received January 12, 2023; accepted February 1, 2023;
published online February 14, 2023

Abstract

$C_{16}H_{13}N_3O$, orthorhombic, $P2_12_12_1$ (no. 19), a = 7.6087(4) Å, b = 9.6681(4) Å, c = 18.6600(9) Å, V = 1372.66(11) Å^3, Z = 4, $R_{gt}(F)$ = 0.0420, $wR_{ref}(F^2)$ = 0.1093, T = 293 K.

CCDC no.: 2235769

The asymmetric unit of the molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.17 × 0.12 × 0.10 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	0.66 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω
θ_{max}, completeness:	71.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5203, 2594, 0.028
Criterion for I_{obs}, $N(hkl)_{gt}$:	2212
$N(param)_{refined}$:	183
Programs:	CrysAlis^{Pro} [1], Olex2 [2], Shelx [3, 4]

Source of materials

The title compound was prepared by the reaction of 2-(4-methoxyphenyl)-2*H*-indazole (0.2 mmol) and bromoacetonitrile (0.4 mmol) in the presence of $Ir(ppy)_3$ (0.004 mmol, 2 mol%) as a catalyst, K_2HPO_4 (0.4 mmol) as a base, DMSO (1.0 mL) as a solvent under argon atmosphere. The reaction mixture was stirred for 24 h with a 5 W blue LED irradiation at room temperature, followed by filtering through a pad of celite and then washed with ethyl acetate (10 mL × 3). The residue was purified by chromatography on silica gel (eluent: EA/PE) and the product was obtained in 61% yield. The compound was crystallized as colourless crystals by slow evaporation from a solution of ethyl acetate at room temperature.

Experimental details

All hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Indazoles, an important class of N-heterocycles, play an important role in organic synthesis and medical chemistry [5, 6].

***Corresponding authors: Xia Mi and Jingyu Zhang**, College of Pharmacy, Henan University of Chinese Medicine, Zhengzhou 450046, P. R. China, E-mail: mixia@hactcm.edu.cn (X. Mi), jyzhang2004@126.com (J. Zhang). https://orcid.org/0000-0002-5824-6930 (X. Mi). https://orcid.org/0000-0001-9822-0615 (J. Zhang)
Beibei Cui, College of Pharmacy, Henan University of Chinese Medicine, Zhengzhou 450046, P. R. China

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.4133 (3)	0.9572 (3)	0.73803 (13)	0.0715 (8)
N1	0.3250 (3)	0.7969 (2)	0.41251 (13)	0.0450 (6)
N2	0.3875 (3)	0.7178 (2)	0.46781 (12)	0.0407 (5)
N3	0.3008 (5)	0.3044 (3)	0.5388 (2)	0.0913 (12)
C1	0.4543 (4)	0.4859 (3)	0.31992 (16)	0.0491 (7)
H1	0.5034	0.4007	0.3315	0.059*
C2	0.4142 (4)	0.5192 (4)	0.25077 (18)	0.0583 (8)
H2	0.4393	0.4561	0.2146	0.070*
C3	0.3357 (4)	0.6471 (4)	0.23256 (17)	0.0574 (8)
H3	0.3082	0.6650	0.1849	0.069*
C4	0.2996 (4)	0.7444 (3)	0.28302 (16)	0.0521 (7)
H4	0.2470	0.8278	0.2705	0.063*
C5	0.3442 (4)	0.7153 (3)	0.35493 (15)	0.0420 (6)
C6	0.4184 (4)	0.5855 (3)	0.37325 (15)	0.0404 (6)
C7	0.4457 (3)	0.5914 (3)	0.44621 (15)	0.0399 (6)
C8	0.5274 (4)	0.4855 (3)	0.49453 (15)	0.0457 (7)
H8A	0.6208	0.4385	0.4689	0.055*
H8B	0.5794	0.5318	0.5355	0.055*
C9	0.4003 (5)	0.3830 (3)	0.51996 (18)	0.0580 (8)
C10	0.3909 (3)	0.7765 (3)	0.53805 (15)	0.0414 (6)
C11	0.3338 (4)	0.7024 (3)	0.59619 (16)	0.0501 (7)
H11	0.2897	0.6135	0.5898	0.060*
C12	0.3412 (4)	0.7588 (3)	0.66444 (16)	0.0539 (8)
H12	0.3049	0.7073	0.7038	0.065*
C13	0.4027 (4)	0.8920 (3)	0.67353 (17)	0.0536 (8)
C14	0.4563 (4)	0.9676 (3)	0.61448 (18)	0.0562 (8)
H14	0.4954	1.0580	0.6205	0.067*
C15	0.4523 (4)	0.9102 (3)	0.54704 (17)	0.0487 (7)
H15	0.4905	0.9610	0.5077	0.058*
C16	0.3641 (6)	0.8822 (5)	0.80045 (19)	0.0874 (14)
H16A	0.3774	0.9402	0.8419	0.131*
H16B	0.4379	0.8022	0.8053	0.131*
H16C	0.2437	0.8536	0.7964	0.131*

2*H*-indazole is a common structure of indazole. Many strategies have been developed to synthesize such molecules [7, 8]. Herein, we firstly report a new C3-cyanomethyl substituted 2*H*-indazole compound obtained.

The asymmetric unit of the title structure consists of one molecule of the title compound (cf. the figure). The molecule consists of a 2*H*-indazolyl group (N1, N2, C1–C7) and a *p*-methoxyphenyl group (O1, C10–C16). In the structure, the atoms of 2H-indazole ring are coplanar. The p-methoxyphenyl group and indazole ring are not in the same plane, which is similar to several reported structures of 2H-indazols derivatives [9–13], and the torsion angles of C16–O1–C13–C12, C6–C7–C8–C9 and N1–N2–C10–C15 are 2.4(5), −86.2(4) and −44.8(4)°, respectively.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was funded by the National Natural Science Foundation of China (No. 21602046) and the Foundation for University Key Teachers from the Education Department of Henan Province (No. 2020GGJS107).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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