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Crystal structure of 13-(4-fluorophenyl)-11, 13-dihydro-1*H*-benzo[*h*]indazolo[6,7-*b*] [1, 6] naphthyridin-12(6*H*)-one — dimethylformamide — water (1/2/1), $C_{29}H_{31}FN_6O_4$

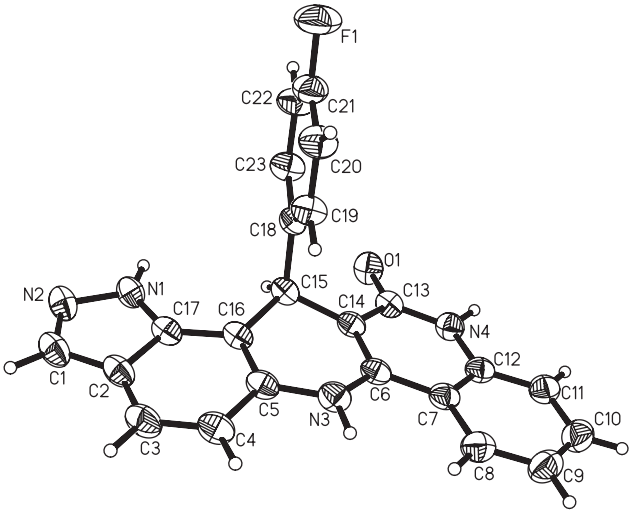

Figure 1: The molecule structure of the title compound (solvate DMF and H_2O omission) showing 30% probability displacement ellipsoids and the atom-numbering scheme.

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Abstract

$C_{29}H_{31}FN_6O_4$, monoclinic, $P2_1/n$, $a = 12.405(2)$ Å, $b = 11.5703(18)$ Å, $c = 19.459(3)$ Å, $\beta = 96.358(2)°$, $V = 2775.9(8)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0457$, $wR_{ref}(F^2) = 0.1329$, $T = 296(2)$ K.

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Table 1: Data collection and handling.

Crystal:	Colourless, block, size 0.102×0.156×0.198 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.94 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω scans
$2\theta_{max}$:	50.4°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	19427, 4962
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3948
$N(param)_{refined}$:	434
Programs:	SHELX [4, 5]

Table 2: Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1B)	4*e*	0.696(2)	0.773(2)	0.204(1)	0.109(9)
H(1C)	4*e*	0.728(2)	0.666(2)	0.182(1)	0.098(8)
H(1)	4*e*	0.185(2)	0.473(2)	−0.086(1)	0.074(6)
H(3)	4*e*	0.212(2)	0.965(1)	0.023(1)	0.077(6)
H(4)	4*e*	−0.141(1)	0.782(2)	0.1317(9)	0.072(6)
H(1A)	4*e*	0.3950	0.5961	−0.1975	0.087
H(3A)	4*e*	0.4098	0.8311	−0.1382	0.089
H(4A)	4*e*	0.3373	0.9420	−0.0578	0.084
H(8A)	4*e*	0.1388	1.0915	0.0765	0.082
H(9A)	4*e*	0.0427	1.2181	0.1364	0.094
H(10A)	4*e*	−0.1077	1.1575	0.1864	0.093
H(11A)	4*e*	−0.1660	0.9695	0.1731	0.082
H(15A)	4*e*	0.0818	0.6007	−0.0053	0.058
H(19A)	4*e*	0.3056	0.7112	0.1072	0.075
H(20A)	4*e*	0.4020	0.6139	0.1977	0.088
H(22A)	4*e*	0.1988	0.3480	0.1670	0.094
H(23A)	4*e*	0.1038	0.4442	0.0749	0.079
H(24A)	4*e*	−0.0968	0.4849	0.2272	0.128
H(25A)	4*e*	0.1180	0.3899	0.3302	0.158
H(25B)	4*e*	0.1158	0.5044	0.3731	0.158
H(25C)	4*e*	0.1930	0.4937	0.3148	0.158
H(26A)	4*e*	−0.0100	0.6514	0.2218	0.135
H(26B)	4*e*	0.1149	0.6285	0.2245	0.135
H(26C)	4*e*	0.0674	0.6842	0.2882	0.135
H(27A)	4*e*	0.7083	0.7301	−0.0475	0.111
H(27B)	4*e*	0.6085	0.8783	0.0603	0.101
H(28A)	4*e*	0.6064	0.5783	−0.0240	0.133
H(28B)	4*e*	0.4866	0.6205	−0.0226	0.133
H(28C)	4*e*	0.5561	0.5846	0.0463	0.133
H(28D)	4*e*	0.4929	0.7415	0.0834	0.096

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Table 2: (continued)

Atom	Site	x	y	z	U_{iso}
H(28E)	4e	0.5502	0.6210	0.0810	0.096
H(28F)	4e	0.4577	0.6550	0.0231	0.096
H(29A)	4e	0.5903	0.8706	0.0720	0.146
H(29B)	4e	0.5331	0.7609	0.0983	0.146
H(29C)	4e	0.4762	0.8329	0.0363	0.146
H(29D)	4e	0.6521	0.7162	−0.0755	0.124
H(29E)	4e	0.5803	0.6118	−0.0568	0.124
H(29F)	4e	0.6990	0.6248	−0.0207	0.124

The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound, was prepared by the reaction of 4-fluorobenzaldehyde, 4-hydroxyquinolin-2(1*H*)-one, and 1*H*-indazol-6-amine, with the method described in literature [1]. Crystals suitable for X-ray diffraction were obtained by slow evaporation of a DMF/H_2O solution.

Table 3: Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F(1)	4e	0.3661(1)	0.4136(1)	0.24148(7)	0.127(1)	0.0993(9)	0.0853(8)	0.0157(8)	−0.0309(8)	0.0245(7)
O(1)	4e	−0.07062(9)	0.6190(1)	0.06957(7)	0.0561(7)	0.0689(8)	0.0807(8)	−0.0176(6)	0.0191(6)	−0.0072(6)
O(2)	4e	−0.0602(2)	0.3565(2)	0.2802(2)	0.153(2)	0.114(2)	0.242(3)	−0.053(2)	0.023(2)	0.008(2)
O(3)	4e	0.7510(2)	0.7359(2)	0.1923(1)	0.090(1)	0.093(1)	0.116(1)	−0.010(1)	0.047(1)	−0.020(1)
O(5)	4e	0.7147(5)	0.8750(4)	−0.0096(3)	0.083(3)	0.072(2)	0.109(3)	−0.021(2)	0.026(2)	0.002(2)
O(5')	4e	0.6781(7)	0.8943(6)	−0.0175(5)	0.105(6)	0.093(4)	0.156(5)	−0.036(4)	0.048(4)	0.005(3)
N(1)	4e	0.2329(1)	0.5279(1)	−0.09868(7)	0.0509(8)	0.0714(9)	0.0540(8)	−0.0101(7)	0.0106(6)	−0.0014(7)
N(2)	4e	0.2918(1)	0.4986(2)	−0.15147(8)	0.0610(9)	0.090(1)	0.0594(9)	−0.0041(8)	0.0166(7)	−0.0042(8)
N(3)	4e	0.1947(1)	0.8923(1)	0.02349(8)	0.0608(8)	0.0577(8)	0.0628(9)	−0.0167(7)	0.0087(7)	0.0033(7)
N(4)	4e	−0.0827(1)	0.8023(1)	0.10895(7)	0.0516(8)	0.0654(9)	0.0615(8)	−0.0009(7)	0.0101(6)	0.0048(7)
N(5)	4e	0.0396(1)	0.5170(2)	0.27893(8)	0.072(1)	0.081(1)	0.0623(9)	−0.0048(8)	0.0044(8)	0.0116(8)
N(6)	4e	0.595(2)	0.736(1)	0.011(1)	0.060(4)	0.066(4)	0.078(4)	−0.013(4)	0.010(4)	0.001(3)
N(6')	4e	0.596(3)	0.733(1)	0.014(2)	0.066(5)	0.066(5)	0.076(5)	−0.006(4)	0.009(4)	0.002(4)
C(1)	4e	0.3471(2)	0.5920(2)	−0.1641(1)	0.061(1)	0.098(2)	0.061(1)	−0.008(1)	0.0188(9)	0.008(1)
C(2)	4e	0.3253(1)	0.6846(2)	−0.12114(9)	0.0528(9)	0.083(1)	0.0531(9)	−0.0085(8)	0.0112(7)	0.0101(9)
C(3)	4e	0.3589(2)	0.7999(2)	−0.1118(1)	0.064(1)	0.086(1)	0.076(1)	−0.016(1)	0.0235(9)	0.018(1)
C(4)	4e	0.3160(2)	0.8653(2)	−0.0638(1)	0.067(1)	0.070(1)	0.075(1)	−0.0188(9)	0.0156(9)	0.0119(9)
C(5)	4e	0.2392(1)	0.8186(2)	−0.02246(8)	0.0498(9)	0.065(1)	0.0537(9)	−0.0113(7)	0.0034(7)	0.0091(8)
C(6)	4e	0.1032(1)	0.8625(1)	0.05282(8)	0.0509(9)	0.0589(9)	0.0440(8)	−0.0078(7)	−0.0026(6)	0.0073(7)
C(7)	4e	0.0461(1)	0.9494(1)	0.08799(8)	0.0592(9)	0.0581(9)	0.0470(8)	−0.0023(8)	−0.0056(7)	0.0064(7)
C(8)	4e	0.0780(2)	1.0657(2)	0.0959(1)	0.075(1)	0.063(1)	0.066(1)	−0.0065(9)	−0.0016(9)	0.0048(9)
C(9)	4e	0.0204(2)	1.1415(2)	0.1319(1)	0.093(2)	0.058(1)	0.081(1)	0.002(1)	−0.007(1)	−0.0054(9)
C(10)	4e	−0.0702(2)	1.1056(2)	0.1614(1)	0.084(1)	0.073(1)	0.074(1)	0.015(1)	−0.000(1)	−0.002(1)
C(11)	4e	−0.1044(2)	0.9935(2)	0.1538(1)	0.068(1)	0.071(1)	0.064(1)	0.0120(9)	0.0036(9)	0.0066(9)
C(12)	4e	−0.0471(1)	0.9151(1)	0.11712(8)	0.0570(9)	0.0598(9)	0.0488(9)	0.0035(7)	−0.0034(7)	0.0075(7)
C(13)	4e	−0.0319(1)	0.7191(1)	0.07549(8)	0.0463(8)	0.062(1)	0.0488(8)	−0.0060(7)	0.0024(6)	0.0032(7)
C(14)	4e	0.0673(1)	0.7501(1)	0.04901(7)	0.0468(8)	0.0566(9)	0.0425(8)	−0.0067(7)	−0.0002(6)	0.0051(6)
C(15)	4e	0.1322(1)	0.6542(1)	0.02074(7)	0.0423(8)	0.0566(9)	0.0449(8)	−0.0126(6)	0.0026(6)	0.0010(6)
C(16)	4e	0.2066(1)	0.7044(1)	−0.02801(7)	0.0426(8)	0.0620(9)	0.0456(8)	−0.0098(7)	0.0013(6)	0.0069(7)
C(17)	4e	0.2501(1)	0.6393(2)	−0.07932(8)	0.0431(8)	0.069(1)	0.0481(8)	−0.0083(7)	0.0019(6)	0.0078(7)
C(18)	4e	0.1954(1)	0.5877(1)	0.08018(8)	0.0462(8)	0.0547(9)	0.0455(8)	−0.0064(6)	0.0067(6)	0.0010(6)
C(19)	4e	0.2842(1)	0.6372(2)	0.11841(9)	0.061(1)	0.062(1)	0.060(1)	−0.0136(8)	−0.0057(8)	0.0061(8)
C(20)	4e	0.3421(2)	0.5797(2)	0.1728(1)	0.069(1)	0.078(1)	0.068(1)	−0.0091(9)	−0.0153(9)	0.0022(9)
C(21)	4e	0.3093(2)	0.4727(2)	0.1887(1)	0.081(1)	0.075(1)	0.058(1)	0.008(1)	−0.0088(9)	0.0096(9)
C(22)	4e	0.2208(2)	0.4209(2)	0.1541(1)	0.097(2)	0.062(1)	0.074(1)	−0.010(1)	−0.004(1)	0.0186(9)
C(23)	4e	0.1640(2)	0.4790(2)	0.09920(9)	0.067(1)	0.062(1)	0.067(1)	−0.0173(8)	−0.0037(8)	0.0070(8)
C(24)	4e	−0.0442(2)	0.4536(3)	0.2596(2)	0.091(2)	0.119(2)	0.105(2)	−0.015(2)	−0.010(1)	0.003(2)
C(25)	4e	0.1235(2)	0.4726(3)	0.3283(2)	0.098(2)	0.177(3)	0.114(2)	−0.012(2)	−0.018(2)	0.054(2)
C(26)	4e	0.0542(3)	0.6295(2)	0.2511(2)	0.141(2)	0.095(2)	0.110(2)	0.002(2)	0.055(2)	0.024(2)
C(27)	4e	0.6764(4)	0.7787(3)	−0.0176(2)	0.107(3)	0.075(2)	0.102(3)	−0.010(2)	0.040(2)	−0.014(2)
C(27')	4e	0.6271(5)	0.8397(4)	0.0215(3)	0.102(3)	0.073(3)	0.083(3)	−0.025(3)	0.034(3)	−0.010(2)
C(28)	4e	0.5581(4)	0.6205(3)	0.0020(3)	0.106(3)	0.081(3)	0.136(4)	−0.024(2)	−0.026(3)	0.032(3)
C(28')	4e	0.5179(4)	0.6836(5)	0.0536(3)	0.073(3)	0.076(3)	0.092(3)	−0.016(2)	0.015(2)	0.008(3)
C(29)	4e	0.5447(4)	0.8057(5)	0.0582(3)	0.093(3)	0.161(5)	0.116(4)	−0.026(3)	0.033(3)	−0.020(3)
C(29')	4e	0.6350(6)	0.6660(5)	−0.0390(3)	0.119(4)	0.085(4)	0.113(4)	0.017(3)	0.036(3)	−0.009(3)

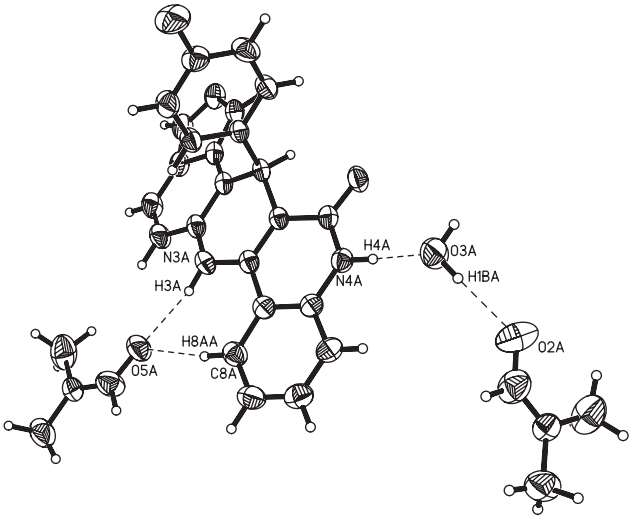

Figure 2: The hydrogen bonds between target molecule and solvent molecules.

Experimental details

The H atoms were calculated geometrically and refined as riding, with C—H = 0.93–0.98 Å, except for H1A, H1B, H1, H3 and H4, and with $U_{iso}(H) = 1.2U_{eq}$(parent atom, $1.5U_{eq}$ for methyl groups).

Discussion

Imidazoles and naphthyridines are important pharmaceutical molecules [2, 3]. In the crystal structure of the title compound, the central pyridyl ring adopts a skew-boat confirmation. The atoms of C5, C6, C14 and C16 are coplanar, with atoms N3 and C15 deviating from the defined plane by −0.144(2) and −0.308(3) Å, respectively. The adjacent quinoline and indazole moieties are planar (Figure 1), with mean deviation being 0.023(1) and 0.012(1) Å, respectively. The pyridine ring is nearly perpendicular to the phenyl ring (C18-C23), making a dihedral angle of 88.1(1)°.

There are four inter molecular hydrogen bonds between the hexacyclic molecule and the solvent molecules DMF and H_2O. The atom O5 in DMF forms two hydrogen bonds of N3—H3···O5 and C8—H8A···O5, while the target molecule is conneted to a molecule of H_2O via the hydrogen bond of N4—H4···O3. This molecule of H_2O links another molecule of DMF though the fourth hydrogen bond as shown in Figure 2.

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