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Crystal structure of 2-((1*H*-benzo[*d*]imidazol-2-ylimino)methyl)-4,6-di-*tert*-butylphenol, C₂₂H₂₇N₃O

Table 1: Data collection and handling.

Crystal:	Yellow prism
Size:	0.2 × 0.15 × 0.13 mm
Wavelength:	Cu K α radiation (1.54184 Å)
μ :	0.55 mm ⁻¹
Diffractometer, scan mode:	Xcalibur, ω -scans
$\theta_{\max}$, completeness:	67°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	18885, 3686, 0.049
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2825
$N(\text{param})_{\text{refined}}$:	261
Programs:	CrysAlis ^{PRO} [1], SHELX [2], OLEX2 [3]

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Abstract

C₂₂H₂₇N₃O, monoclinic, $P2_1/c$ (no. 14), $a = 18.4984(4)$ Å, $b = 11.4221(3)$ Å, $c = 9.79600(19)$ Å, $\beta = 90.9652(19)^\circ$, $V = 2069.51(8)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0605$, $wR_{\text{ref}}(F^2) = 0.1773$, $T = 293(2)$ K.

CCDC no.: 1900012

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.

Source of material

In a typical experiment 2-aminobenzimidazole (1.3315 g, 10 mmol) and 3,5-bis(1,1-dimethylethyl)-2-hydroxybenzaldehyde (2.3433 g, 10 mmol) were mixed in 25 mL ethanol and refluxed for 3 h. When the solution was cooled to room temperature, a light yellow solid was obtained.

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Crystallization from methanol at room temperature gave the title compound as colorless crystals of good diffraction quality. ¹H NMR (DMSO-*d*₆, 500 MHz) δ (p.p.m.): 13.34 (s, 1H), 12.79 (s, 1H), 9.63 (s, 1H), 7.67 (d, $J = 2.4$ Hz, 1H), 7.59 (s, 1H), 7.48 (d, $J = 2.4$ Hz, 2H), 7.23–7.18 (m, 2H), 1.44 (s, 9H), 1.31 (s, 9H). ¹³C NMR (DMSO-*d*₆, 126 MHz) δ (p.p.m.): 168.55, 158.54, 141.38, 136.54, 129.46, 129.37, 118.41, 35.15, 34.46, 31.65, 29.71. IR (cm⁻¹, KBr): 3406 (br), 2096 (m), 2431 (m), 1910 (w), 1674 (s), 1603 (m), 1584 (m), 1513 (m), 1483 (m), 1452 (m), 1439 (m), 1397 (s), 1362 (w), 1320 (s), 1289 (s), 1249 (m), 1175 (s), 1072 (m), 1025 (w), 1001 (w), 933 (m), 882 (w), 800 (m), 766 (w), 739 (w), 729 (m), 715 (s), 685 (w), 664 (w), 616 (w), 523 (m).

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms. There is a disorder of one of the *tert*-butyl groups (Table 2, not shown in the figure).

Discussion

The structures of benzimidazole moiety have been widely studied, since they possess significant biological activity such as anticancer, and antiproliferative [4, 5]. Schiff bases are also a very important family of the compounds because of their applications in biological and analytical chemistry [6–10]. The hybrid molecules composed by the combination of benzimidazole and Schiff base may exert potential biological

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	0.19352(9)	0.51881(16)	1.07728(16)	0.0699(5)
H1	0.1548(14)	0.572(2)	1.071(3)	0.074(7)*
N1	0.11123(8)	0.68777(17)	0.98631(16)	0.0550(4)
N2	0.01050(8)	0.77048(17)	1.09666(16)	0.0539(4)
H2	0.0224(14)	0.741(2)	1.184(3)	0.085(8)*
N3	0.02033(8)	0.80758(17)	0.87379(15)	0.0555(5)
C1	0.23929(10)	0.54324(19)	0.97461(18)	0.0516(5)
C2	0.30229(11)	0.47638(18)	0.96015(19)	0.0511(5)
C3	0.34794(10)	0.50796(18)	0.85516(19)	0.0514(5)
H3	0.3897	0.4640	0.8440	0.062*
C4	0.33563(10)	0.60122(18)	0.76487(18)	0.0495(5)
C5	0.27301(10)	0.66354(19)	0.78051(18)	0.0518(5)
H5	0.2627	0.7253	0.7214	0.062*
C6	0.22386(10)	0.63616(19)	0.88412(18)	0.0506(5)
C7	0.15851(10)	0.7041(2)	0.89249(19)	0.0539(5)
H7	0.1499	0.7621	0.8277	0.065*
C8	0.04890(10)	0.75469(19)	0.98109(18)	0.0505(5)
C9	−0.04668(10)	0.84225(19)	1.06247(19)	0.0516(5)
C10	−0.04024(10)	0.8647(2)	0.92268(19)	0.0536(5)
C11	−0.09086(12)	0.9348(3)	0.8566(2)	0.0703(6)
H11	−0.0879	0.9494	0.7635	0.084*
C12	−0.14530(13)	0.9820(2)	0.9319(3)	0.0761(7)
H12	−0.1795	1.0298	0.8891	0.091*
C13	−0.15056(13)	0.9600(2)	1.0716(3)	0.0744(7)
H13	−0.1881	0.9936	1.1198	0.089*
C14	−0.10200(11)	0.8904(2)	1.1390(2)	0.0626(6)
H14	−0.1056	0.8756	1.2320	0.075*
C15	0.32094(12)	0.3748(2)	1.0574(2)	0.0627(6)
C16	0.39179(14)	0.3151(2)	1.0206(3)	0.0770(7)
H16A	0.3885	0.2864	0.9286	0.115*
H16B	0.4007	0.2509	1.0818	0.115*
H16C	0.4307	0.3705	1.0282	0.115*
C17	0.33066(17)	0.4232(3)	1.2034(2)	0.0843(8)
H17A	0.3693	0.4794	1.2053	0.127*
H17B	0.3421	0.3602	1.2648	0.127*
H17C	0.2867	0.4604	1.2309	0.127*
C18	0.26111(18)	0.2820(3)	1.0520(4)	0.0988(10)
H18A	0.2167	0.3157	1.0828	0.148*
H18B	0.2744	0.2174	1.1099	0.148*
H18C	0.2547	0.2548	0.9598	0.148*
C19	0.38883(11)	0.6285(2)	0.6500(2)	0.0566(5)
C20 ^a	0.46174(17)	0.5695(5)	0.6708(4)	0.1077(16)
H20A ^a	0.4934	0.5922	0.5987	0.162*
H20B ^a	0.4556	0.4861	0.6697	0.162*
H20C ^a	0.4824	0.5931	0.7570	0.162*
C20A ^b	0.4632(13)	0.659(4)	0.726(3)	0.1077(16)
H20D ^b	0.5015	0.6591	0.6605	0.162*
H20E ^b	0.4732	0.6017	0.7948	0.162*
H20F ^b	0.4599	0.7351	0.7668	0.162*
C21 ^a	0.3559(2)	0.5822(4)	0.5156(3)	0.1001(13)
H21A ^a	0.3112	0.6219	0.4964	0.150*
H21B ^a	0.3471	0.4996	0.5237	0.150*
H21C ^a	0.3889	0.5959	0.4428	0.150*
C21A ^b	0.3943(18)	0.517(2)	0.563(2)	0.1001(13)
H21D ^b	0.3474	0.4974	0.5268	0.150*

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H21E ^b	0.4122	0.4542	0.6189	0.150*
H21F ^b	0.4268	0.5309	0.4895	0.150*
C22 ^a	0.3982(2)	0.7594(3)	0.6332(5)	0.1040(14)
H22A ^a	0.3516	0.7958	0.6239	0.156*
H22B ^a	0.4259	0.7748	0.5531	0.156*
H22C ^a	0.4231	0.7906	0.7119	0.156*
C22A ^b	0.3561(14)	0.719(2)	0.552(3)	0.1040(14)
H22D ^b	0.3508	0.7926	0.5986	0.156*
H22E ^b	0.3097	0.6927	0.5197	0.156*
H22F ^b	0.3875	0.7296	0.4757	0.156*

Occupancies: ^a = 0.880(4), ^b = 0.120(4).

activities. Thus we synthesized the Schiff base containing benzimidazole moiety.

The title compound crystallizes in the monoclinic system with a *P*2₁/*c* space group with one 2-((1*H*-benzo[*d*]imidazol-2-ylimino)methyl)-4,6-di-*tert*-butylphenol molecule in the asymmetric unit. As shown in the figure, the molecule structure consists of a 2,4-di-*tert*-butylphenol group and a benzimidazole group linked by the –C=N– group. The 2,4-di-*tert*-butylphenol ring is nearly planar excluding the methyl groups. The angle between two planes containing the benzimidazole ring/imine N atom and the phenol ring/imine C atom, respectively is 25.91°. The C7–N1 bonding distance of 1.293(2) Å is consistent with a C=N double bond, as in other structurally characterized Schiff base compounds [11, 12]. Intermolecular hydrogen bonds are found through the imidazole nitrogen atoms [N2...N3A: 2.860(2) Å] forming an infinite chain.

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