X-ray Structure Analysis Online

Crystal Structure of (3-Hydroxy-4-methoxyphenyl)-(1,4,5-triphenyl)-1*H*-imidazole

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The crystal structure of the title compound was solved in the monoclinic space group $P2_1/c$, with a = 18.8290(8)Å, b = 24.515(2)Å, c = 9.6301(18)Å, β = 99.245(7)°, V = 4387.5(9)Å^3, Z = 8, R_1 = 0.0618, for 4752 reflections with $I > 2\sigma$(I), with two unique molecules in the asymmetric unit. Relatively strong inter-molecular hydrogen bonds connect these two molecules, forming a dimer structure. Weak hydrogen bonds between these dimers together with C–H···π interactions stabilize the crystal structure.

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Increasing interest in imidazole compounds arises from their biological and pharmaceutical importance.[1] There are several methods for the synthesis of highly substituted imidazoles. Several substituted imidazoles are known to be as inhibitors of P 38 kinase.[2] Among these compounds, lepidilines A and B exhibit micromolar cytotoxicity against several human cancer cell lines.[3] Thus, the prevalence of the imidazole moiety in biologically active compounds has received an increased attention in obtaining tri- and tetra-substituted imidazoles *via* regiocontrolled process. A highly efficient solvent-free one-pot synthesis of tetrasubstituted imidazoles was effected using SBA-R-SO_3H as nano-catalyst.[4]

The title compound was synthesized by heating a mixture of Benzil (2.5 mmol), 3-hydroxy-4-methoxybenzaldehyde (2.5 mmol), aniline (2.5 mmol), ammonium acetate (5 mmol) and activated SBA-sulfonic acid (0.02 g) at 140°C for 6 min. The progress of the reaction was monitored by thin-layer chromatography method. After cooling to room temperature, the mixture was dissolved in hot ethyl acetate, and the catalyst was removed by filtration. The filtrate was left for crystallization. Suitable crystals for crystallographic analysis were obtained by recrystallization from ethyl acetate.

Crystal structure of the title compound was solved by direct methods, and refined by full-matrix least-squares on F^2 by means of SHELXL-97.[5] Hydroxyl H atoms were located in difference density maps, and their coordinates were refined freely, with U_{iso}(H) = 1.5U_{eq}(O). The rest of the H atoms were placed in at calculated positions with C–H = 0.93 and 0.96 Å, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 and 1.5U_{eq}(C), for aromatic and methane, respectively. Selected crystallographic and experimental details and the bond length

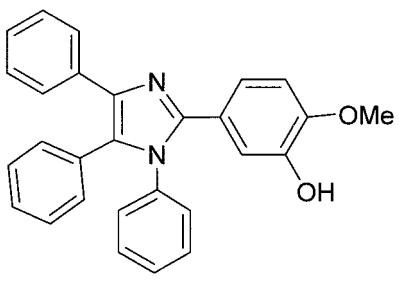

Fig. 1 Chemical diagram of the title compound.

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Table 1 Crystal data and structure refinement for the title compound

Chemical formula: $C_{28}H_{22}N_2O_2$	
Formula weight = 418.48	
T = 298 K	
Crystal system: monoclinic	
Space group: $P2_1/c$	
a = 18.8290(8)Å	
b = 24.515(2)Å	β = 99.245(7)°
c = 9.6301(18)Å	
V = 4387.5(9)Å^3	
Z = 8	
D_x = 1.267 g/cm^3	
Radiation: Mo K_α (λ = 0.71073 Å)	
λ(Mo K_α) = 0.080 mm^{-1}	
F(0 0 0) = 1760	
Crystal size = 0.34 × 0.08 × 0.06 mm	
No. of reflections collected = 34560	
No. of independent reflections = 8614	
θ range for data collection: 2.00 to 29.25°	
Data/Restraints/Parameters = 8614/0/585	
Goodness-of-fit on F^2 = 0.958	
R indices [$I > 2\sigma(I)$]: $R1$ = 0.0618, $wR2$ = 0.1064	
R indices (all data): $R1$ = 0.1305, $wR2$ = 0.1224	
$(\Delta/\sigma)_{max}$ = 0.000	
$(\Delta\rho)_{max}$ = 0.14 eÅ^{-3}	
$(\Delta\rho)_{min}$ = –0.18 eÅ^{-3}	
Measurement: STOE IPDS II	
Program system: X-Area	
Structure determination: SHELXS-97	
Refinement: full matrix	
CCDC deposition number: 693630	

Table 2 Selected bond distances (Å) and torsion angles (˚)

C1-N2	1.392(3)	N3-C30	1.316(3)
N2-C3	1.373(3)	C30-N4	1.372(3)
N1-C3	1.323(3)	N4-C28	1.396(3)
C2-N1	1.392(3)	N3-C29	1.389(3)
N2-C10	1.447(3)	O3-C33	1.377(3)
C6-O1	1.371(3)	O4-C34	1.369(3)
C7-O2	1.365(3)	O4-C56	1.431(4)
O2-C55	1.418(3)		
C6-C7-O2-C55	171.6(3)	C33-C34-O4-C56	178.3(3)
C8-C7-O2-C55	-9.6(4)	C35-C34-O4-C56	-1.2(4)

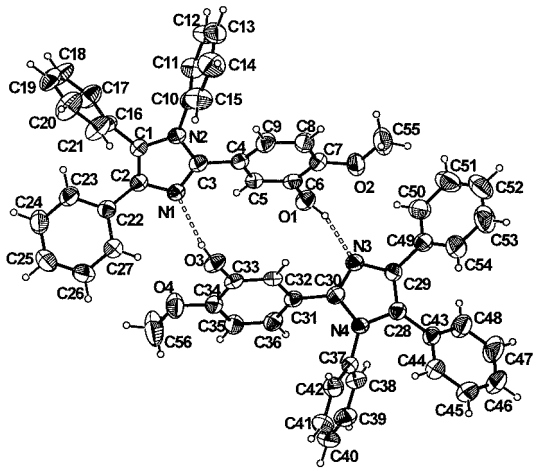

Fig. 2 ORTEP structure of the title compound, showing 50% probability ellipsoids. H atoms are shown as circles of arbitrary radii. Inter-molecular hydrogen bonds are indicated by dashed lines.

together with torsion angles are given in Tables 1 and 2.

The title compound crystallizes with two unique molecules in the asymmetric unit, which are connected *via* relatively strong inter-molecular hydrogen bonds, between the hydroxyl group from one molecule to the nitrogen atom in the imidazole ring of another one, making these two molecules to be a dimer form (Figs. 1 & 2).

Hydroxy-substituted tetraphenyl imidazole was previously described in a triclinic setting with two molecules in the asymmetric unit.[6] In the previously reported structure two phenyl rings at the 2 and 4 positions made a planar conformation to the central imidazole ring while the other phenyl rings at the 1 and 5 positions were located almost perpendicular to the imidazole ring. However, in the current study all the phenyl rings are twisted from the central imidazole plane. Rings A(C10–C15), B(C4–C9), C(C22–C27) and D(C16–C21) corresponding to positions 1, 2, 4 and 5 make dihedral angles

Table 3 Hydrogen-bond D–H···A for the compound [Å and ˚]

D-H···A	D-H(Å)	H···A(Å)	D···A(Å)	D-H···A(˚)
O1-H1…N3	0.92(3)	1.94(3)	2.815(3)	160(3)
O3-H3…N1	0.82(3)	2.07(3)	2.824(3)	153(3)
C41-H41…O1	0.93(1)	2.56(1)	3.411(3)	151(1)
C35-H35…O3	0.93(1)	2.51(1)	3.397(3)	159(1)

with the central imidazole ring (N2, C3, N1, C2, C1) by 87.2˚, 28.7˚, 25.6˚ and 88.6˚, respectively. However, in the other molecule, the corresponding rings E(C37–C42), F(C31–C36), G(C49–C54) and H(C43–C48) are twisted with dihedral angles of 71.5˚, 47.1˚, 43.0˚ and 45.3˚, respectively, with regards to the imidazole ring (N4, C30, N3, C29, C28).

Rings B(C4–C9) and F(C31–C36) in two molecules in the asymmetric unit are almost parallel with dihedral angle of 5.3˚. However, two imidazole and other corresponding phenyl rings (A(C10–C15) & E(C37–C42), C(C22–C27) & G(C49–C54), and D(C16–C21) & H(C43–C48)) in two molecules in the asymmetric unit make dihedral angles of 24.4˚, 29.8˚, 40.4˚, and 31.5˚, respectively. The torsion angle of metoxy-phenyl (C55–O2–C7–C6), 171.6(3)˚ shows more deviation from planarity compared to the corresponding value in the other molecule (C56–O4–C34–C33), 178.3(3)˚ (Table 2).

The packing of the structure is partially stabilized by weak C–H···O hydrogen bonds between carbons from benzyl rings to the oxygen from hydroxyl groups and also by C–H···π interactions [C–H–centroid = 2.61 – 2.92 Å] (Table 3).

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References

1. S. Balalaie, M. M. Hashemi, and M. Akhbari, *Tetrahedron Lett.*, **2003**, *44*, 1709.
2. C. F. Claiborne, N. J. Liverton, and K. T. Nguyen, *Tetrahedron Lett.*, **1998**, *39*, 8939.
3. B. Cui, B. L. Zheng, K. He, and Q. Y. Zheng, *J. Nat. Prod.*, **2003**, *66*, 1101.
4. M. Ghoranneviss, G. Mohammadi Ziarani, A. Abbasi, M. R. Hantehzadeh, and Z. Farahani, *Acta Cryst.*, **2008**, *E64*, o1233.
5. G. M. Sheldrick, *Acta Cryst.*, **2008**, *A64*, 112.
6. S. Park, O-H. Kwon, S. Kim, S. Park, M.-G. Choi, M. Cha, S. Y. Park, and D.-J. Jang, *J. Am. Chem. Soc.*, **2005**, *127*, 10070.