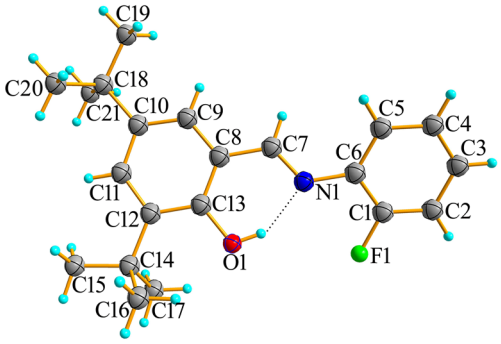
Wen-Ze Zhao* and Huan Tian

Crystal structure of (*E*)-2,4-di-tert-butyl-6-(((2-fluorophenyl)imino) methyl)phenol, $C_{21}H_{26}FNO$



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Abstract

$C_{21}H_{26}FNO$, orthorhombic, $Pna2_1$ (no. 33), a = 12.329(3) Å, b = 9.048(3) Å, c = 16.881(5) Å, β = 90°, V = 1883.2(9) Å^3, Z = 4, $R_{gt}(F)$ = 0.0500, $wR_{ref}(F^2)$ = 0.1184, T = 273(2) K.

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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:	Yellow block
Size:	0.20 × 0.19 × 0.15 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.08 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13,925, 3287, 0.039
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2975
$N(param)_{refined}$:	224
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

1 Source of materials

A solution of 3,5-di-tert-butylsalicylaldehyde (0.47 g, 2 mmol) in ethanol (25 mL) was added to a stirred ethanol solution (30 mL) of 2-fluoroaniline (0.22 g, 2 mmol) in a round-bottomed flask. The mixed solution was heated and reflowed for 12 h and cooled to room temperature. The light yellow solid was filtered, and washed with ethanol for 4 times. 0.52 g of light yellow solid was achieved. The yield was 80 %. Dissolve 0.1 g of the compound in 20 mL of chloroform. 15 mL of methanol was added. After slow volatilization for 4 days crystals were obtained.

2 Experimental details

Using Olex2 [1], the structure was solved using Charge Flipping and refined with the ShelXL [3] refinement. All hydrogen atoms were positioned geometrically, with the d(C–H) = 0.97–0.99 Å, U_{iso}(H) = 1.2 times U_{eq}(C) and U_{iso}(H) = 1.5 times U_{eq}(O).

3 Comment

Schiff base compounds constructed from aldehydes containing salicylaldehyde structures [5, 6] are vulnerable to intramolecular proton transfer, resulting in larger Stokes shift, under photoexcitation, the molecule absorbs energy and transitions from enol form to ketone form; hence, the compound based on the Excess State Intramolecular Proton Transfer mechanism typically has two emission bands [7]. Some of these compounds present excellent photochromic and mechanochromic properties [8, 9]. Here we report the crystal structure of (*E*)-2,4-di-*tert*-butyl-6-(((2-fluorophenyl) imino) methyl)phenol, which shows that 3,5-di-*tert*-butylsalicylaldehyde has undergone condensation reaction with *o*-fluoroaniline. The bond length of imine bond C7=N1 is

***Corresponding author: Wen-Ze Zhao**, Shandong Vocational College of Industry, Zibo, Shandong, 550025, People's Republic of China, E-mail: zhaowenze2022@126.com. https://orcid.org/0000-0002-9854-1766
Huan Tian, Shandong Vocational College of Industry, Zibo, Shandong, 550025, People's Republic of China

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5428 (3)	0.3509 (4)	0.7151 (2)	0.0407 (9)
C2	0.6134 (3)	0.2829 (5)	0.7658 (2)	0.0499 (10)
H2	0.6229	0.3175	0.8172	0.060*
C3	0.6701 (3)	0.1617 (5)	0.7388 (3)	0.0526 (11)
H3	0.7185	0.1132	0.7721	0.063*
C4	0.6553 (4)	0.1133 (5)	0.6637 (3)	0.0591 (12)
H4	0.6945	0.0320	0.6458	0.071*
C5	0.5829 (3)	0.1823 (5)	0.6128 (2)	0.0488 (10)
H5	0.5739	0.1473	0.5614	0.059*
C6	0.5237 (3)	0.3040 (4)	0.6386 (2)	0.0309 (8)
C7	0.4005 (3)	0.3346 (4)	0.5325 (2)	0.0331 (8)
H7	0.4132	0.2369	0.5181	0.040*
C8	0.3247 (3)	0.4214 (4)	0.4860 (2)	0.0314 (8)
C9	0.2737 (3)	0.3575 (4)	0.4208 (2)	0.0332 (8)
H9	0.2921	0.2615	0.4065	0.040*
C10	0.1973 (3)	0.4313 (4)	0.3768 (2)	0.0316 (8)
C11	0.1707 (3)	0.5743 (4)	0.40212 (19)	0.0336 (8)
H11	0.1172	0.6247	0.3742	0.040*
C12	0.2186 (3)	0.6459 (4)	0.46579 (19)	0.0300 (8)
C13	0.2984 (3)	0.5671 (4)	0.50785 (17)	0.0279 (7)
C14	0.1847 (3)	0.8026 (4)	0.4909 (2)	0.0395 (9)
C15	0.0951 (4)	0.8645 (5)	0.4375 (3)	0.0573 (12)
H15A	0.1215	0.8721	0.3841	0.086*
H15B	0.0741	0.9605	0.4561	0.086*
H15C	0.0336	0.7995	0.4387	0.086*
C16	0.2829 (4)	0.9074 (4)	0.4869 (3)	0.0630 (13)
H16A	0.3394	0.8710	0.5208	0.094*
H16B	0.2615	1.0043	0.5041	0.094*
H16C	0.3090	0.9124	0.4334	0.094*
C17	0.1394 (4)	0.7977 (5)	0.5760 (2)	0.0558 (11)
H17A	0.0734	0.7415	0.5767	0.084*
H17B	0.1251	0.8965	0.5939	0.084*
H17C	0.1916	0.7521	0.6104	0.084*
C18	0.1390 (3)	0.3631 (4)	0.3050 (2)	0.0391 (9)
C19	0.1923 (4)	0.2189 (5)	0.2794 (3)	0.0565 (11)
H19A	0.1882	0.1485	0.3218	0.085*
H19B	0.2670	0.2369	0.2664	0.085*
H19C	0.1554	0.1805	0.2337	0.085*
C20	0.1421 (5)	0.4687 (5)	0.2346 (3)	0.0661 (13)
H20A	0.2159	0.4847	0.2189	0.099*
H20B	0.1098	0.5613	0.2494	0.099*
H20C	0.1023	0.4267	0.1913	0.099*
C21	0.0208 (3)	0.3307 (7)	0.3279 (3)	0.0727 (15)
H21A	−0.0168	0.2901	0.2831	0.109*
H21B	−0.0139	0.4207	0.3441	0.109*
H21C	0.0193	0.2611	0.3708	0.109*
F1	0.4880 (3)	0.4692 (3)	0.74103 (17)	0.0840 (10)
N1	0.4504 (2)	0.3873 (3)	0.59261 (16)	0.0330 (7)
O1	0.3485 (2)	0.6316 (3)	0.57005 (14)	0.0382 (6)
H1	0.3892	0.5718	0.5912	0.057*

1.279 Å, and all bond lengths and bond angles fall within the normal range [10]. The dihedral angle between the two benzene rings is 158.57°, and there is an intramolecular hydrogen bond O1–H1···N1 in the molecule. Recently, for the title compound a structure described in a trigonal space group symmetry was reported [11].

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