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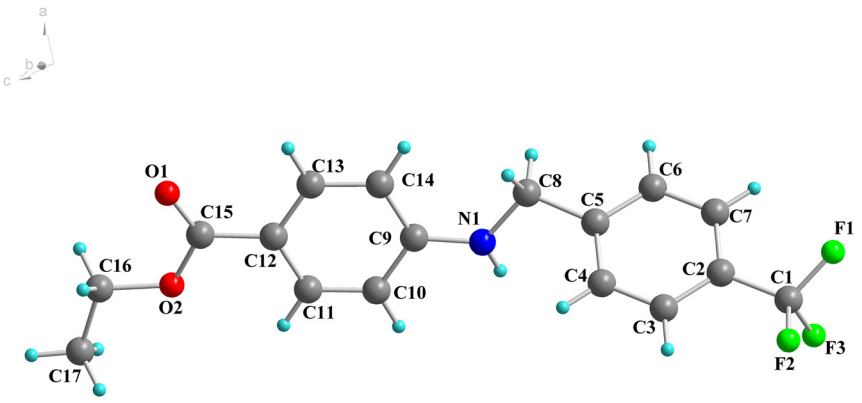
Synthesis and crystal structure of ethyl 4-((4-trifluoromethylbenzyl)amino)benzo, $C_{17}H_{16}F_3NO_2$



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Abstract

$C_{17}H_{16}F_3NO_2$, monoclinic, $P2_1/n$, a = 5.7291(4) Å, b = 37.994(3) Å, c = 6.9806(5) Å, β = 100.575(7)°, V = 1493.7(2) Å^3, Z = 4, $R_{gt}(F)$ = 0.0598, w$R_{ref}(F^2)$ = 0.1310, T = 100 K.

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Table 1: Data collection and handling.

Crystal:	colourless block
Size:	0.15 × 0.12 × 0.11 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.12 mm^{-1}
Diffractometer, scan mode:	Rigaku SuperNova, ω scans
θ_{max}, completeness:	29.7°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7665, 3549, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2,374
$N(param)_{refined}$:	213
Programs:	Rigaku,[1] SHELX[2,3]

1 Source of materials

The title compound was prepared according to the following synthetic protocol. A *p*-aminobenzoate (165 mg, 1.0 mmol), *p*-toluenesulfonic acid monohydrate 100 mg and 4-(trifluoromethyl)benzaldehyde (226 mg, 1.3 mmol) in dry toluene (20 mL) were heated at reflux temperature under nitrogen atmosphere for 48 h. The mixture was cooled to room temperature and filtered. The filtrate was washed with 5 % aqueous sodium hydroxide and water, and dried over anhydrous magnesium sulfate. The crude product was purifed on a silica-gel column using a mixture of petroleum ether and ethyl acetate, yielding light yellow solid (yield: 50 %). The yellow crystals of the title compound were obtained by slow evaporation of dichloromethane/hexane at room temperature and the suitable single crystal was studied by X-ray diffraction method (Table 1).

2 Experimental details

The hydrogen atoms were refined with constraints or restraints.

3 Comment

Diarylamines are important building blocks in organic synthesis and are present as privileged structures in numerous pharmaceuticals and biologically active compounds. Due to the importance to medicinal chemistry, many methods exist for diarylamine synthesis, with transition metal-catalyzed C–N bond formation being especially prominent in recent years.[4–7] Up to date, there are only a few reports on diarylamine compounds especially their crystal structures.[8–10]

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There is one molecule in the asymmetric unit of the title structure which is shown in the figure. The *p*-methyltrifluorophenyl group is attached to the 4-position of the ethyl benzoate. The CCN bond distance is 1.442(3) Å for C8···N1, which is normal for arylamine. The three bond angles around the amino group are 124.44(18)° for C8–N1–C9, 111.07(17)° and 123.0(2)° for N1–C9–C14, respectively. The dihedral angel between the benzene rings is 57.56°. Molecules are linked by NH···O hydrogen bonds to form one-dimensional supramolecular structure. These chains are linked by CHC interactions with 2.967 Å for nitrogen (N3) atom to the nearest oxygen (O1) atom.

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