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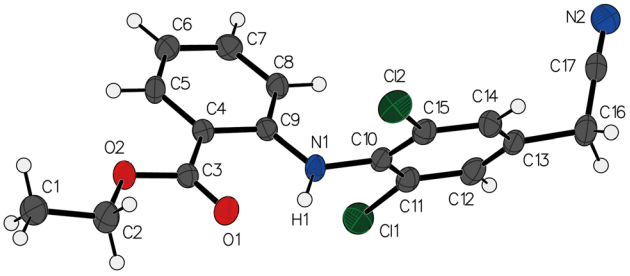
Crystal structure of ethyl 2-((2,6-dichloro-4-(cyanomethyl)phenyl) amino)benzoate, $C_{17}H_{14}Cl_2N_2O_2$



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Abstract

$C_{17}H_{14}Cl_2N_2O_2$, monoclinic, $P2_1/n$ (no. 14), a = 8.4863(17) Å, b = 12.654(2) Å, c = 15.088(3) Å, β = 103.612(5)°, V = 1574.8(5) Å^3, Z = 4, $R_{gt}(F)$ = 0.0423, $wR_{ref}(F^2)$ = 0.1472, T = 205 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:	0.25 × 0.18 × 0.11 mm
Wavelength:	MoKα radiation (0.71073 Å)
μ:	0.42 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13,957, 3607, 0.043
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2787
$N(param)_{refined}$:	209
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

1 Source of materials

To a magnetically stirred mixture of ethyl 2-((4-(bromomethyl)-2,6-dichlorophenyl)amino)benzoate (2.0 g, 5 mmol) in acetonitrile (15 mL) was added potassium cyanide (650 mg, 10 mmol) and then the reaction mixture was heated in an oil bath at 50 °C for 4 h. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was evaporated to give residue under reduced pressure. The title compound was separated by silica-gel column chromatography with ethyl acetate-petroleum ether (20 %) gradient solvent system. The target product was obtained as a white solid. Yield: 77.1 %.

2 Experimental details

During the refinement process, all hydrogen atom positions were assumed to be in idealized positions and treated as riding atoms. The U_{iso} values for the hydrogen atoms were set to 1.2 times the U_{eq} of the parent atoms. The structure solution was achieved using ShelXT [2], and the refinement was performed using ShelXL [3] incorporated in the Olex2 software [4].

3 Comment

Diphenylamine derivatives have emerged as promising compounds due to their versatile applications and fascinating properties [5]. The investigation of the crystal structure of diphenylamine derivatives holds great importance in understanding their molecular packing and intermolecular interactions [6–9]. This paper provided the determination of the crystal structure of ethyl 2-((2,6-dichloro-4-(cyanomethyl)phenyl) amino) benzoate, which provides valuable insights into the spatial arrangement and intermolecular interactions within the crystal lattice.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.9592 (3)	−0.13504 (18)	0.65290 (16)	0.0400 (5)
H1A	0.9793	−0.1667	0.5980	0.060*
H1B	0.8512	−0.1539	0.6586	0.060*
H1C	1.0386	−0.1608	0.7056	0.060*
C2	0.9723 (3)	−0.01672 (17)	0.64744 (14)	0.0366 (5)
H2A	0.9526	0.0161	0.7027	0.044*
H2B	1.0812	0.0033	0.6419	0.044*
C3	0.8474 (2)	0.12306 (14)	0.55199 (12)	0.0235 (4)
C4	0.7209 (2)	0.15386 (14)	0.47080 (12)	0.0224 (4)
C5	0.6120 (2)	0.07797 (15)	0.42411 (13)	0.0276 (4)
H5	0.6202	0.0080	0.4457	0.033*
C6	0.4935 (2)	0.10264 (16)	0.34757 (14)	0.0298 (4)
H6	0.4216	0.0505	0.3174	0.036*
C7	0.4822 (2)	0.20601 (16)	0.31589 (13)	0.0290 (4)
H7	0.4032	0.2236	0.2630	0.035*
C8	0.5848 (2)	0.28275 (15)	0.36068 (12)	0.0266 (4)
H8	0.5736	0.3524	0.3383	0.032*
C9	0.7067 (2)	0.25983 (14)	0.43945 (12)	0.0225 (4)
C10	0.7830 (2)	0.44600 (14)	0.46833 (13)	0.0252 (4)
C11	0.8144 (2)	0.49651 (16)	0.39189 (13)	0.0270 (4)
C12	0.7884 (2)	0.60391 (17)	0.37647 (14)	0.0331 (5)
H12	0.8102	0.6355	0.3242	0.040*
C13	0.7302 (2)	0.66440 (15)	0.43856 (15)	0.0322 (5)
C14	0.7035 (2)	0.61783 (16)	0.51648 (15)	0.0334 (5)
H14	0.6689	0.6591	0.5601	0.040*
C15	0.7276 (2)	0.50996 (16)	0.53029 (13)	0.0269 (4)
C16	0.6950 (3)	0.78210 (17)	0.42125 (19)	0.0484 (6)
H16A	0.7113	0.8188	0.4799	0.058*
H16B	0.7720	0.8114	0.3885	0.058*
C17	0.5297 (3)	0.80153 (15)	0.36817 (14)	0.0297 (4)
Cl1	0.89415 (7)	0.42363 (5)	0.31527 (4)	0.04031 (19)
Cl2	0.68855 (7)	0.45284 (5)	0.62735 (4)	0.04230 (19)
N1	0.8096 (2)	0.33719 (12)	0.48438 (11)	0.0295 (4)
H1	0.8957	0.3177	0.5248	0.035*
N2	0.4025 (2)	0.81887 (16)	0.32684 (14)	0.0436 (5)
O1	0.94209 (18)	0.18384 (11)	0.59988 (9)	0.0327 (3)
O2	0.85155 (17)	0.01858 (10)	0.56769 (9)	0.0302 (3)

The ethyl 2-((2,6-dichloro-4-(cyanomethyl)phenyl)amino) benzoate consists of several key chemical groups. The ethyl ester group is connected to the benzoate moiety. The bond length of C2–O2 is 1.455(3) Å and the bond length of C3–O2 is 1.342(3) Å. The bond angle of C2–O2–C3 is 115.63(14) °. Two chlorine atoms replace the two hydrogen atoms on cyanomethyl benzene, and the bond lengths of Cl1–C11 and Cl2–C15 are 1.736(3) and 1.734(3) Å. In addition, the bond length of C17–N2 is 1.134(3) Å in the cyanomethyl group, and the bond angle of N2–C17–C16 is 178.5(3) °. The substituated phenyl rings are not coplanar, exhibiting a distinct dihedral angle of 14.9°. They are consistent with crystal structures reported elsewhere [10–12].

The aromatic rings of adjacent molecules stack together through π–π interactions [13, 14].

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