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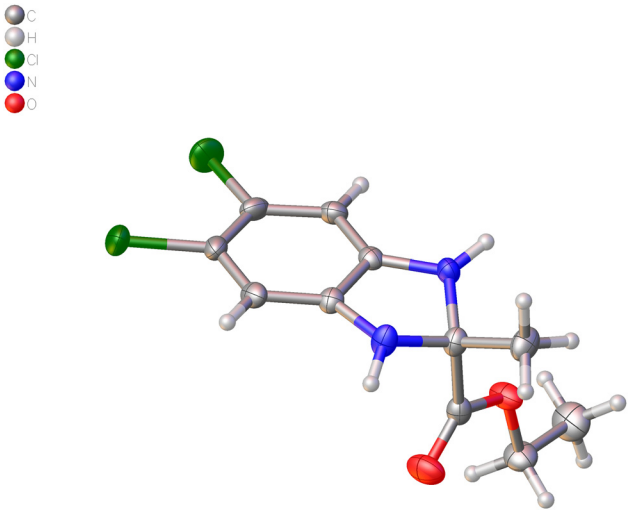
Crystal structure of ethyl 5,6-dichloro-2-methyl-2,3-dihydro-1 *H*-benzo[*d*]imidazole-2-carboxylate, $C_{11}H_{12}Cl_2N_2O_2$



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Abstract

$C_{11}H_{12}Cl_2N_2O_2$, monoclinic, $P2_1/c$ (no. 14), a = 19.937(2) Å, b = 5.7080(8) Å, c = 23.406(3) Å, β = 108.521(4)°, V = 2525.7(6) Å^3, Z = 8, $R_{gt}(F)$ = 0.0483, $wR_{ref}(F^2)$ = 0.1171, T = 200 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.

1 Source of material

To an oven-dried tube were sequentially added 4,5-dichlorobenzene-1,2-diamine (105.6 mg, 0.6 mmol), toluene (1 mL), ethyl-

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.45 × 0.32 × 0.13 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.51 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}:	13,598, 2900, 0.072
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1942
$N(param)_{refined}$:	160
Programs:	CrysAlis^{PRO} (*1*), Olex2 (*2*), SHELX (*3*, *4*)

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.52171 (3)	0.23970 (13)	0.66578 (4)	0.0463 (2)
Cl2	0.54234 (3)	0.70377 (13)	0.60081 (4)	0.0484 (2)
O1	0.81396 (10)	−0.0916 (3)	0.59900 (8)	0.0413 (5)
O2	0.85050 (9)	0.2559 (3)	0.57668 (7)	0.0307 (4)
N1	0.79140 (10)	0.1126 (4)	0.70260 (9)	0.0261 (5)
H1	0.7927 (13)	−0.040 (5)	0.7040 (11)	0.033 (8)*
N2	0.80730 (9)	0.4614 (3)	0.66323 (8)	0.0229 (4)
H2	0.830110	0.592167	0.661858	0.027*
C1	0.72572 (11)	0.2250 (4)	0.68192 (10)	0.0211 (5)
C2	0.66105 (12)	0.1623 (4)	0.68586 (10)	0.0253 (5)
H2A	0.654858	0.019577	0.704452	0.030*
C3	0.60437 (12)	0.3157 (4)	0.66156 (11)	0.0265 (5)
C4	0.61333 (12)	0.5220 (4)	0.63437 (11)	0.0278 (6)
C5	0.67997 (11)	0.5896 (4)	0.63193 (10)	0.0240 (5)
H5	0.686426	0.733545	0.613956	0.029*
C6	0.73560 (11)	0.4397 (4)	0.65654 (9)	0.0201 (5)
C7	0.83747 (11)	0.2252 (4)	0.67297 (10)	0.0231 (5)
C8	0.91481 (12)	0.2211 (5)	0.71169 (11)	0.0314 (6)
H8A	0.931387	0.058599	0.717894	0.047*
H8B	0.919825	0.293901	0.750749	0.047*
H8C	0.943054	0.308215	0.691379	0.047*
C9	0.83162 (11)	0.1068 (4)	0.61219 (10)	0.0233 (5)
C10	0.85219 (14)	0.1732 (5)	0.51817 (11)	0.0338 (6)
H10A	0.805461	0.110110	0.494222	0.041*
H10B	0.888091	0.048343	0.523278	0.041*
C11	0.87082 (16)	0.3817 (5)	0.48766 (12)	0.0437 (7)
H11A	0.874053	0.335147	0.448318	0.066*
H11B	0.916444	0.444925	0.512568	0.066*
H11C	0.834188	0.501914	0.482074	0.066*

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2-oxopropanoate (23.2 mg, 0.2 mmol), and boron trifluoride diethyl etherate (8.5 mg, 30 mol%). The reaction mixture was open to the air and stirred at room temperature for 20 min. After completion of the reaction, the solution was concentrated in vacuum. The residue was purified by silica gel column chromatography using a mixture of petroleum ether and ethyl acetate (5:1) as eluent to give the desired product.

2 Experimental details

The carbon-bound hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

3 Comment

2,3–Dihydro-1*H*-benzo[*d*]imidazole and its derivatives are important bioactive substances, which are the basic structures of many natural products and important intermediates in the synthesis of a variety of drugs (*5*–*7*). Therefore, synthesis and modification of imidazoles has been a hot topic in the field of medicine and pharmacy.

The basic structure of the title compound is imidazole, containing a 2,3-dihydro-1*H*-benzo[*d*]imidazole, an acid ester, and two chlorine atoms. The C=O bond on the ester group was confirmed by the distance of 1.197(3) Å (O1–C9). The key lengths and angles obtained from the title structure are within the normal range and are consistent with those previously reported in similar structures (*8*, *9*).

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