Crystal structure of 5,6-dimethyl-1*H*-benzo[*d*]imidazol-3-ium 2-(2,4-dichloro-phenoxy)acetate, $C_{17}H_{16}Cl_2N_2O_3$

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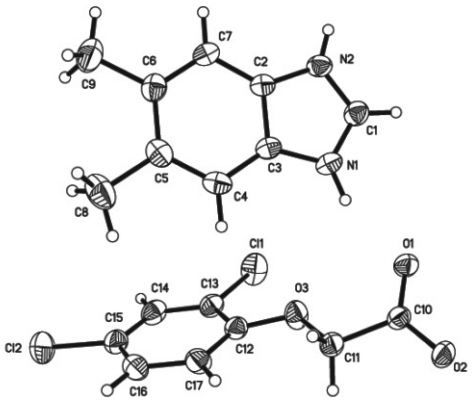

Abstract

$C_{17}H_{16}Cl_2N_2O_3$, monoclinic, $P2_1/c$ (no. 14), a = 10.900(1) Å, b = 15.311(2) Å, c = 10.605(1) Å, β = 101.844(2)°, V = 1732.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.0444, $wR_{ref}(F^2)$ = 0.1144, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.39×0.40×0.43 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.92 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ and ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13092, 3032
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2708
$N(param)_{refined}$:	219
Programs:	SHELXS-97 [5]

Source of material

The title compound (I) was synthesized by the following procedure. A mixture of 2,4-Dichlorophenoxyacetic acid 2.21g (0.01 mol) and 5,6-dimethyl-1*H*-benzo[d]imidazole 1.46g (0.02 mol) was stirred with ethanol (50mL) at 367K for 3h. An ethanol solution of the title compound was slowly evaporated and colourless block crystals were obtained after a week.

Discussion

2,4-Dichlorophenoxyacetic acid and its derivatives have attracted considerable attention bcause of diverse biomedical applications in herbicides and plant-growth substances [1]. Also, 2,4-Dichlorophenoxyacetic acid are extensively used as synthetic auxiliaries in organic chemistry and versatile ligands in coordination chemistry [2,3]. In previous papers, we reported the crystal structure of benzimidazolium 2-(2,4-dichlorophenoxy)- acetate monohydrate [4]. As part of this ongoing search for new 2,4-Dichlorophenoxyacetic acid compounds, the title compound has been synthesized and its structure is reported here. In the title compound, bond lengths and angles fall in the usual ranges. In the crystal structure, there is one weak N–H···O intramolecular hydrogen bond.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.1155	0.6311	0.3668	0.057
H(2)	4*e*	0.0093	0.4272	0.1711	0.056
H(1A)	4*e*	0.0201	0.5804	0.1603	0.060
H(4)	4*e*	0.2114	0.5507	0.6091	0.060
H(7)	4*e*	0.0881	0.3001	0.3633	0.056
H(8A)	4*e*	0.2895	0.4690	0.7903	0.122
H(8B)	4*e*	0.3528	0.3816	0.7605	0.122
H(8C)	4*e*	0.2216	0.3796	0.7994	0.122
H(9A)	4*e*	0.1755	0.2095	0.5329	0.114
H(9B)	4*e*	0.1658	0.2458	0.6687	0.114
H(9C)	4*e*	0.2947	0.2474	0.6687	0.114
H(11A)	4*e*	0.2541	0.8217	0.6257	0.057
H(11B)	4*e*	0.1665	0.7416	0.6372	0.057
H(14)	4*e*	0.6321	0.5529	0.6426	0.067
H(16)	4*e*	0.4767	0.6253	0.9363	0.070
H(17)	4*e*	0.3282	0.7067	0.7967	0.063

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	4*e*	0.47922(6)	0.62952(4)	0.42711(6)	0.0839(4)	0.0832(4)	0.0660(4)	0.0251(3)	0.0368(3)	0.0071(3)
Cl(2)	4*e*	0.67793(6)	0.51875(5)	0.90488(7)	0.0786(4)	0.0773(4)	0.0869(4)	0.0247(3)	–0.0094(3)	0.0109(3)
N(1)	4*e*	0.1020(2)	0.57693(9)	0.3472(2)	0.0586(9)	0.0349(7)	0.0526(9)	–0.0046(6)	0.0175(7)	–0.0046(6)
N(2)	4*e*	0.0414(1)	0.4602(1)	0.2351(1)	0.0585(9)	0.0433(8)	0.0395(7)	–0.0074(7)	0.0109(7)	–0.0044(6)
O(1)	4*e*	0.1032(1)	0.75596(8)	0.3610(1)	0.0658(8)	0.0424(7)	0.0426(6)	0.0051(6)	0.0077(6)	–0.0062(5)
O(2)	4*e*	0.0669(2)	0.87272(8)	0.4719(1)	0.084(1)	0.0452(7)	0.0456(7)	0.0237(7)	0.0022(7)	–0.0047(5)
O(3)	4*e*	0.2995(1)	0.71484(9)	0.5419(1)	0.0585(8)	0.0660(8)	0.0471(7)	0.0244(7)	0.0119(6)	–0.0006(6)
C(1)	4*e*	0.0487(2)	0.5461(1)	0.2328(2)	0.060(1)	0.045(1)	0.046(1)	–0.0022(8)	0.0149(8)	0.0040(8)
C(2)	4*e*	0.0945(2)	0.4324(1)	0.3585(2)	0.0414(9)	0.0412(9)	0.0423(9)	–0.0048(7)	0.0146(7)	–0.0041(7)
C(3)	4*e*	0.1324(2)	0.5068(1)	0.4304(2)	0.0428(9)	0.0390(9)	0.0462(9)	–0.0047(7)	0.0166(8)	–0.0044(7)
C(4)	4*e*	0.1889(2)	0.5007(1)	0.5600(2)	0.050(1)	0.056(1)	0.046(1)	–0.0119(8)	0.0133(8)	–0.0117(8)
C(5)	4*e*	0.2109(2)	0.4193(1)	0.6140(2)	0.045(1)	0.069(1)	0.047(1)	–0.0064(9)	0.0111(8)	0.0041(9)
C(6)	4*e*	0.1754(2)	0.3428(1)	0.5389(2)	0.046(1)	0.055(1)	0.057(1)	–0.0013(8)	0.0168(8)	0.0106(9)
C(7)	4*e*	0.1148(2)	0.3496(1)	0.4119(2)	0.051(1)	0.0386(9)	0.055(1)	–0.0046(7)	0.0173(8)	–0.0030(8)
C(8)	4*e*	0.2745(2)	0.4117(2)	0.7537(2)	0.077(2)	0.112(2)	0.052(1)	–0.008(1)	0.005(1)	0.012(1)
C(9)	4*e*	0.2056(2)	0.2532(2)	0.5966(3)	0.074(2)	0.065(1)	0.089(2)	0.004(1)	0.016(1)	0.027(1)
C(10)	4*e*	0.1205(2)	0.8019(1)	0.4595(2)	0.051(1)	0.0362(9)	0.0409(9)	0.0030(7)	0.0120(8)	–0.0006(7)
C(11)	4*e*	0.2114(2)	0.7718(1)	0.5801(2)	0.054(1)	0.045(1)	0.0438(9)	0.0116(8)	0.0091(8)	–0.0049(7)
C(12)	4*e*	0.3870(2)	0.6755(1)	0.6344(2)	0.0416(9)	0.0412(9)	0.051(1)	0.0027(7)	0.0075(8)	–0.0023(7)
C(13)	4*e*	0.4800(2)	0.6298(1)	0.5903(2)	0.047(1)	0.0432(9)	0.057(1)	0.0017(7)	0.0157(8)	–0.0011(8)
C(14)	4*e*	0.5700(2)	0.5829(1)	0.6732(2)	0.044(1)	0.047(1)	0.077(1)	0.0055(8)	0.014(1)	–0.0013(9)
C(15)	4*e*	0.5664(2)	0.5812(1)	0.8020(2)	0.050(1)	0.046(1)	0.066(1)	0.0024(8)	–0.0005(9)	0.0006(9)
C(16)	4*e*	0.4773(2)	0.6266(1)	0.8488(2)	0.060(1)	0.061(1)	0.051(1)	0.0031(9)	0.0024(9)	–0.0034(9)
C(17)	4*e*	0.3878(2)	0.6748(1)	0.7649(2)	0.051(1)	0.056(1)	0.050(1)	0.0081(8)	0.0083(8)	–0.0077(8)

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