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Crystal structure of 2,2-dichloro-1-(4-chloro-1*H*-indol-1-yl)ethan-1-one, $C_{10}H_6Cl_3NO$

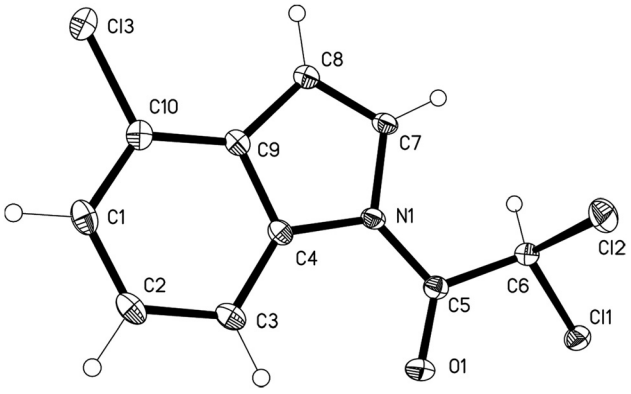

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.21 × 0.19 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.85 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ_{max}, completeness:	31.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	25,606, 3460, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3271
$N(param)_{refined}$:	137
Programs:	Bruker programs [1], OLEX2 [2, 3], SHELX [4]

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Abstract

$C_{10}H_6Cl_3NO$, monoclinic, $P2_1/n$ (no. 14), a = 9.7097(10) Å, b = 10.6037(11) Å, c = 10.1735(10) Å, β = 96.580(4)°, V = 1040.55(18) Å^3, Z = 4, $R_{gt}(F)$ = 0.0214, $wR_{ref}(F^2)$ = 0.0574, T = 101 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.

Source of material

The title compound was synthesized according to the literature [5]. About 0.61 g 4-chloroindole (4.0 mmol) was dissolved in dichloroethane (40 mL) at room temperature. About 0.59 g dichloracetyl chloride (4.0 mmol) was added dropwise to this stirred solution. Then, the mixture was heated to reflux for 4 h. The crude product was purified by column chromatography (petroleum ether: EtOAc = 50:1, *v*/*v*) to give the title compound. Crystals were grown using a mixed solvent of EtOAc and petroleum ether at room temperature.

Experimental details

The structures was solved by direct methods with the SHELXS program [4]. All hydrogen atoms were placed in geometrically calculated positions and constrained to ride on their parent atoms (Table 2).

Comment

N-dichloroacetamides herbicide safeners have generated considerable commercial interest and research activity for their application to crops to alleviate the injury from chloroacetanilide, thiocarbamate and imidazolinone herbicides [6, 7]. Indole derivatives have significant physiological activity and occupy an important position in agriculture, medicine and materials science [8–13]. In view of the principle of fragment splicing, the indole derivative and dichloroacetyl were spliced together to design novel potential *N*-dichloroacetyl indole derivatives herbicide safeners [14].

There is one molecule in the asymmetric unit (see the Figure). Bond lengths and angles are all in normal ranges

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.66735 (2)	0.44793 (2)	0.93686 (2)	0.01812 (6)
Cl2	0.50769 (3)	0.58554 (2)	0.72222 (3)	0.02354 (7)
Cl3	0.97674 (2)	0.57050 (2)	0.16074 (2)	0.01820 (6)
O1	0.69760 (7)	0.31633 (6)	0.69040 (7)	0.01742 (13)
N1	0.76683 (8)	0.48313 (7)	0.57268 (7)	0.01227 (13)
C1	0.90682 (10)	0.33479 (9)	0.23380 (9)	0.01738 (17)
H1	0.9402	0.3061	0.1548	0.021*
C2	0.85569 (10)	0.24892 (9)	0.32038 (10)	0.01780 (17)
H2	0.8553	0.1617	0.2991	0.021*
C3	0.80533 (10)	0.28680 (8)	0.43660 (9)	0.01566 (16)
H3	0.7706	0.2276	0.4947	0.019*
C4	0.80815 (9)	0.41569 (8)	0.46412 (9)	0.01235 (15)
C5	0.71410 (9)	0.42902 (8)	0.67961 (9)	0.01283 (15)
C6	0.67366 (9)	0.52315 (8)	0.78359 (9)	0.01435 (15)
H6	0.7431	0.5931	0.7941	0.017*
C7	0.79327 (9)	0.61296 (8)	0.55275 (9)	0.01327 (15)
H7	0.7748	0.6791	0.6113	0.016*
C8	0.84864 (9)	0.62818 (8)	0.43817 (9)	0.01327 (15)
H8	0.8759	0.7059	0.4025	0.016*
C9	0.85910 (9)	0.50510 (8)	0.37950 (8)	0.01250 (15)
C10	0.90847 (9)	0.46241 (9)	0.26412 (9)	0.01451 (16)

[15–18]. Obviously, there is π–π conjugation effect in the amide moiety, which causes a short N(1)–C(5) (1.3792(11) Å) bond. The torsion angle of C(4)–N(1)–C(5)–O(1) and C(7)–N(1)–C(5)–O(1) are –0.23(14)° and –178.22(9)° respectively, which indicate the amide moiety and indole ring are almost coplanar. Weak intermolecular C–H···O and C–H···Cl hydrogen bonds are observed, which connect the molecules into a three dimensional network.

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