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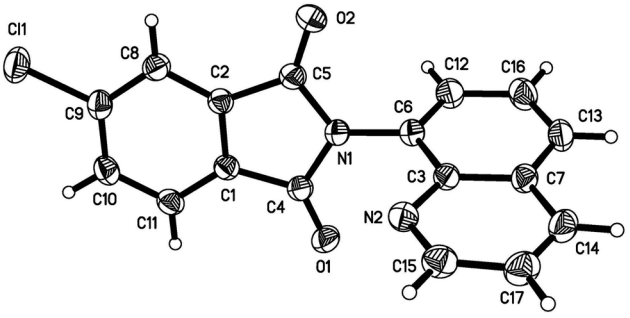
The crystal structure of 5-chloro-2-(quinolin-8-yl) isoindoline-1,3-dione, $C_{17}H_9ClN_2O_2$



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Abstract

$C_{17}H_9ClN_2O_2$, triclinic, $P\bar{1}$ (no. 2), a = 7.8944(4) Å, b = 8.0678(5) Å, c = 12.6285(9) Å, α = 74.627(6)°, β = 78.654(5)°, γ = 63.569(5)°, V = 691.58(8) Å^3, Z = 2, $R_{gt}(F)$ = 0.0431, $wR_{ref}(F^2)$ = 0.1309, T = 293(2) 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

All of reagents were purchased with analysis grade. 2.82 g 3-chloro-*N*-(quinolin-8-yl)benzamide (10 mmol), 0.38 g anhydrous copper nitrate (2 mmol), 4.15 g 2,2′-azobisisobutyronitrile (25 mmol), and 1.34 g silver acetate (8 mmol) were added to 40 mL distilled acetonitrile in a borosil sealed tube. The tube was heated at 403 K under oxygen atmosphere for 8 h, then cooled to room temperature and filtered. The filtrate evaporated slowly in air. A few days later, colourless crystals were harvested, yield 38% (based on 3-chloro-*N*-(quinolin-8-yl)benzamide).

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.21 × 0.15 × 0.11 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	2.52 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	73.6°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4378, 2684, 0.012
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2519
$N(param)_{refined}$:	199
Programs:	CrysAlis^{PRO} [1], Olex2 [2], SHELX [3, 4]

Experimental details

Single crystal X-ray diffraction data were collected at 293 K using an Agilent Super Nova diffractometer equipped with a Dual source (Cu at Home/Near) and an Atlas S2 detector. The structure was solved by direct methods with the SHELXS-2018 program. All H-atoms attached to C atoms were positioned with idealized geometry and refined isotropically (U_{iso}(H) = 1.2U_{eq}(C)) using a riding model with C–H = 0.930 Å. Structural refinement suggests the possible presence of a small amount (< 5%) of disorder

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

Atom	x	y	z	U_{iso}*/U_{eq}
Cl1	−0.09394 (8)	0.82222 (8)	0.74406 (4)	0.0689 (2)
O1	0.09789 (19)	0.6497 (2)	0.21944 (12)	0.0584 (4)
O2	0.51686 (19)	0.6988 (2)	0.39965 (13)	0.0654 (4)
N1	0.34452 (19)	0.6585 (2)	0.28782 (12)	0.0443 (3)
N2	0.5263 (2)	0.2934 (2)	0.24958 (13)	0.0492 (4)
C1	0.0678 (2)	0.7107 (2)	0.40240 (14)	0.0413 (3)
C2	0.1957 (2)	0.7235 (2)	0.45732 (14)	0.0415 (4)
C3	0.5698 (2)	0.4297 (2)	0.17598 (14)	0.0417 (4)
C4	0.1609 (2)	0.6690 (2)	0.29257 (14)	0.0433 (4)
C5	0.3747 (2)	0.6934 (2)	0.38365 (15)	0.0448 (4)
C6	0.4822 (2)	0.6172 (2)	0.19531 (14)	0.0439 (4)
C7	0.7000 (2)	0.3942 (3)	0.08110 (15)	0.0468 (4)
C8	0.1516 (2)	0.7578 (3)	0.56276 (15)	0.0478 (4)

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Table 2: (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H8	0.237623	0.766199	0.599639	0.057*
C9	−0.0294 (3)	0.7790 (2)	0.61070 (15)	0.0483 (4)
C10	−0.1596 (3)	0.7666 (3)	0.55747 (16)	0.0510 (4)
H10	−0.278957	0.781186	0.592914	0.061*
C11	−0.1116 (2)	0.7324 (3)	0.45076 (16)	0.0489 (4)
H11	−0.197367	0.724525	0.413400	0.059*
C12	0.5290 (3)	0.7556 (3)	0.12776 (17)	0.0556 (4)
H12	0.473255	0.876498	0.142816	0.067*
C13	0.7431 (3)	0.5407 (3)	0.01106 (16)	0.0557 (5)
H13	0.827275	0.517372	−0.051652	0.067*
C14	0.7854 (3)	0.2064 (3)	0.06340 (18)	0.0581 (5)
H14	0.869949	0.176299	0.001588	0.070*
C15	0.6128 (3)	0.1211 (3)	0.22912 (19)	0.0581 (5)
H15	0.585777	0.026217	0.278956	0.070*
C16	0.6618 (3)	0.7165 (3)	0.03485 (18)	0.0619 (5)
H16	0.694102	0.811649	−0.010451	0.074*
C17	0.7428 (3)	0.0711 (3)	0.1373 (2)	0.0630 (5)
H17	0.798944	−0.053042	0.127378	0.076*

of the Cl atom between the 5- and 6- positions on the isoindoline ring.

Comment

The aerobic carbonylation of C(sp^2)–H with less toxic 2,2′-azobisisobutyronitrile using transition metal salts as the effective catalysts has attracted a lot of interest. Through this way many N-quinolyl substituted phthalimide derivatives have been studied and some of their crystal structures have also been reported [5–8]. However, the crystal of the title compound, 5-chloro-2-(quinolin-8-yl)isoindoline-1,3-dione (CQDD), has not been reported. CQDD crystalizes in the triclinic space group $P\bar{1}$ (no. 2). The angle subtended by the two aromatic moieties is greater than 90° [the C5–N1–C6–C3 and C5–N1–C6–C12 torsion angles are 107.62(17)° and 107.85(18)°, respectively]. C–H–N and C–H···O hydrogen bonds [namely C8–H8···and C17–H17–O1] link CQDD molecules to generate a three-dimensional structure. All of the bond lenths of CQDD are comparable to those found in its analogues [5, 7, 8].

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