Crystal structure of 5',7'-dibromo-1'-(2-bromoethyl)spiro[[1,3]dioxolane-2,3'-indolin]-2'-one, $C_{12}H_{10}Br_3NO_3$

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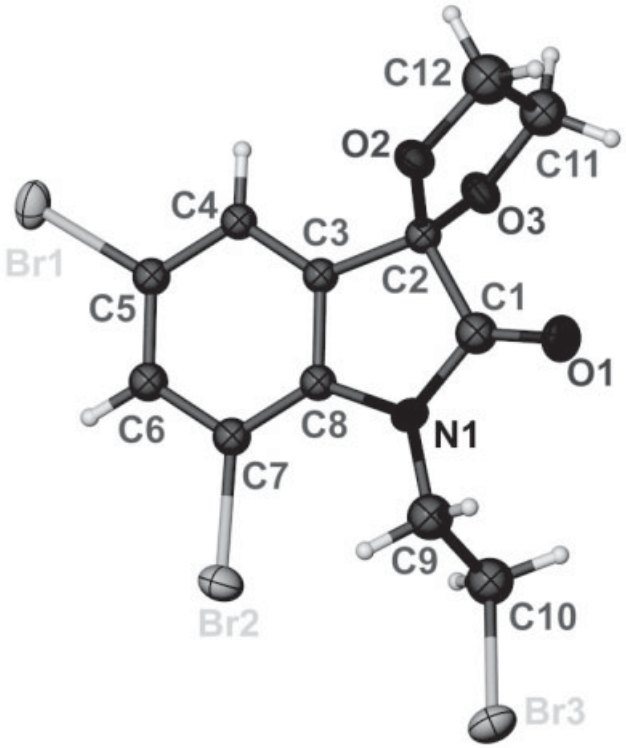

Abstract

$C_{12}H_{10}Br_3NO_3$, orthorhombic, *Pbca* (no. 61), a = 12.942(2) Å, b = 10.304(2) Å, c = 21.527(3) Å, V = 2870.5 Å^3, Z = 8, $R_{gt}(F)$ = 0.0385, $wR_{ref}(F^2)$ = 0.0868, T = 296 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.15×0.20×0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	84.34 cm^{-1}
Diffractometer, scan mode:	Bruker APEXII CCD, φ and ω
$2\theta_{max}$:	56.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	21944, 3460
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2159
$N(param)_{refined}$:	173
Programs:	SHELX [6]

Source of material

A mixture of 5,7-dibromoindoline-2,3-dione (1.52 g, 5.0 mmol), potassium carbonate (1.00 g, 7.2 mmol) and 1,2-dibromoethane (2.82 g, 15.0 mmol) in *N,N*-dimethylformamide (7 ml) was stirred at 333-343 K for 18 h. After cooling to room temperature, the reaction mixture was poured into ice water (80 ml) and extracted with ethyl acetate (20 ml × 3). The combined organic phases were dried over anhydrous sodium sulfate and the solvent was removed by rotary evaporation. The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate = 6:1 (*v/v*) as an eluent. The residue included the prevailing product 5,7-dibromo-1-(2-bromoethyl)indoline-2,3-dione (R_f = 0.40, m.p. 375-377 K ; yield 40%) and the title product 5',7'-dibromo-1'-(2-bromoethyl)spiro[[1,3]dioxolane-2,3'-indolin]-2'-one (R_f = 0.5, m.p. 382-383 K; yield 38%). The prevailing product, 5,7-dibromo-1-(2-bromoethyl)indoline-2,3-dione, was determined by MS and ^{1}H-NMR. The yellow crystals of the title compound were obtained by slow evaporation of the solution of petroleum ether/ethyl acetate = 6:1 (*v/v*).

Discussion

1*H*-indole-2,3-dione and its derivatives demonstrate a broad spectrum of biological and pharmacological activities including anti-cancer activities [1, 2]. It has been reported that the introduction of alkyl group or aryl group into the N atom of indolones can significantly increase its cytotoxicity against a variety of cancer cell lines [1-3]. To obtain *N*-alkylated 5,7-dibromoindoline-2,3-dione analogues, we designed the reaction of 5,7-dibromoindoline-2,3-dione with an excess of 1,2-dibromoethane. Interestingly, an unexpected nucleophilic addition of 1,2-dibromoethane to one of the carbonyl groups on 5,7-dibromoindoline-2,3-dione occurred to gave the title compound in 38% yield. Herein, we report the crystal structure of this new compound. The indol-2-one ring is approximately perpendicular to the O2–C2–O3–C11–C12 plane with a dihedral angle of 89.18(3)°. The C–O bond lengths (1.408(4) Å, 1.404(5) Å) and the angle of O3–C2–O2 (107.4(3)°) are well in agreement with those analogous compounds reported [4, 5].

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

Atom	Site	x	y	z	U_{iso}
H(4A)	8*c*	0.4879	0.4319	0.3223	0.048
H(6A)	8*c*	0.4198	0.6623	0.4659	0.056
H(9A)	8*c*	0.0205	0.4084	0.3858	0.076
H(9B)	8*c*	0.0755	0.4984	0.4347	0.076
H(10A)	8*c*	–0.0188	0.5806	0.3243	0.081
H(10B)	8*c*	0.0519	0.6737	0.3639	0.081
H(11A)	8*c*	0.3514	0.3332	0.1541	0.081
H(11B)	8*c*	0.2329	0.3132	0.1684	0.081
H(12A)	8*c*	0.2800	0.1379	0.2142	0.094
H(12B)	8*c*	0.3963	0.1829	0.2176	0.094

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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}
Br(1)	8*c*	0.61166(3)	0.56301(5)	0.41727(2)	0.0402(2)	0.0808(3)	0.0530(3)	–0.0132(2)	–0.0087(2)	0.0043(2)
Br(2)	8*c*	0.20171(4)	0.69567(5)	0.47256(2)	0.0562(3)	0.0827(4)	0.0648(3)	0.0080(3)	0.0043(2)	–0.0304(3)
Br(3)	8*c*	–0.11112(3)	0.63359(6)	0.42299(3)	0.0397(3)	0.1048(4)	0.0768(4)	0.0131(3)	0.0087(2)	–0.0154(3)
N(1)	8*c*	0.1615(2)	0.4722(3)	0.3562(2)	0.030(2)	0.063(2)	0.053(2)	0.005(2)	0.000(2)	–0.013(2)
O(1)	8*c*	0.0943(2)	0.3459(4)	0.2783(2)	0.044(2)	0.110(3)	0.082(3)	–0.007(2)	–0.012(2)	–0.036(2)
O(2)	8*c*	0.3109(2)	0.2477(2)	0.2876(1)	0.059(2)	0.042(2)	0.045(2)	0.004(1)	–0.001(1)	–0.001(1)
O(3)	8*c*	0.3033(2)	0.4296(2)	0.2286(1)	0.068(2)	0.042(2)	0.038(2)	0.004(1)	–0.005(1)	–0.001(1)
C(1)	8*c*	0.1676(3)	0.3943(4)	0.3048(2)	0.042(2)	0.059(3)	0.055(3)	0.002(2)	–0.007(2)	–0.007(2)
C(2)	8*c*	0.2825(3)	0.3796(4)	0.2880(2)	0.038(2)	0.043(2)	0.037(2)	0.003(2)	–0.005(2)	–0.005(2)
C(3)	8*c*	0.3354(3)	0.4562(4)	0.3377(2)	0.036(2)	0.042(2)	0.035(2)	0.001(2)	–0.002(2)	–0.000(2)
C(4)	8*c*	0.4393(3)	0.4713(4)	0.3479(2)	0.035(2)	0.048(2)	0.036(2)	–0.001(2)	0.003(2)	0.001(2)
C(5)	8*c*	0.4691(3)	0.5471(4)	0.3977(2)	0.034(2)	0.053(2)	0.040(2)	–0.006(2)	–0.002(2)	0.006(2)
C(6)	8*c*	0.3976(3)	0.6094(4)	0.4336(2)	0.050(3)	0.052(2)	0.036(2)	–0.011(2)	–0.006(2)	–0.003(2)
C(7)	8*c*	0.2924(3)	0.5950(4)	0.4226(2)	0.043(2)	0.050(2)	0.039(2)	0.003(2)	0.001(2)	–0.007(2)
C(8)	8*c*	0.2606(3)	0.5128(4)	0.3753(2)	0.034(2)	0.049(2)	0.039(2)	0.002(2)	–0.003(2)	–0.002(2)
C(9)	8*c*	0.0619(3)	0.4861(5)	0.3908(2)	0.052(3)	0.067(3)	0.072(3)	–0.001(2)	–0.007(2)	–0.005(3)
C(10)	8*c*	0.0064(4)	0.5989(5)	0.3659(2)	0.064(3)	0.076(3)	0.061(3)	0.000(3)	0.008(2)	0.009(3)
C(11)	8*c*	0.3009(4)	0.3222(4)	0.1869(2)	0.095(4)	0.059(3)	0.048(3)	–0.007(3)	0.008(3)	–0.013(2)
C(12)	8*c*	0.3254(5)	0.2097(4)	0.2245(2)	0.133(5)	0.048(3)	0.055(3)	0.003(3)	0.004(3)	–0.007(2)

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