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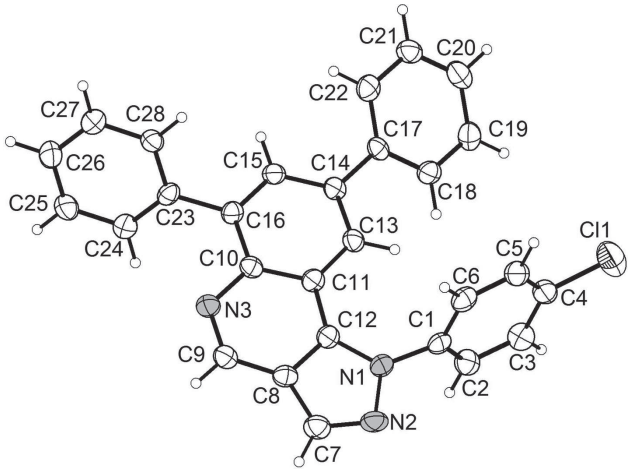
Crystal structure of 1-(4-chlorophenyl)-6,8-diphenyl-1*H*-pyrazolo[4,3-*c*]quinoline, $C_{28}H_{18}ClN_3$



DOI 10.1515/ncrs-2016-0235
Received August 1, 2016; accepted January 13, 2017; available online February 10, 2017

Abstract

$C_{28}H_{18}ClN_3$, monoclinic, $P2_1/c$, (no. 14) $a = 5.9519(2)$ Å, $b = 8.0511(3)$ Å, $c = 43.4071(14)$ Å, $\beta = 90.001(2)°$, $V = 2080.04(12)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.066$, $wR_{ref}(F^2) = 0.190$, $T = 173$ K.

CCDC no.: 1433469

Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless rod
Size:	$0.47 \times 0.19 \times 0.04$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.206 mm^{-1}
Diffractometer, scan mode:	Bruker D8 Venture, ω (0.5° width)
θ_{max}, completeness:	25.49°, 96%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10748,3720, 0.0980
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3009
$N(param)_{refined}$:	290
Programs:	XPREP [2], WinGX [4], SHELXS [3]

Source of material

The title compound was previously prepared by the reaction of 6,8-dibromo-4-chloroquinoline-3-carbaldehyde with 4-chlorophenylhydrazine hydrochloride (1.5 equiv.) in ethanol in the presence of triethylamine (1.5 equiv.) to afford the corresponding (6,8-dibromo-4-chloroquinolin-3-yl) methylene]-2-(4-chlorophenyl)hydrazide. This hydrazide derivative was, in turn, cyclized with ethanolic KOH (5% in ethanol) to afford the corresponding 6,8-dibromo-1-(2-chlorophenyl)-1*H*-pyrazolo[4,3-*c*]quinoline. The 1,6,8-triaryl-1*H*-pyrazolo[4,3-*c*]quinoline used in this investigation was prepared *via* Suzuki-Miyaura cross-coupling of the corresponding 1-aryl-6,8-dibromo-1*H*-pyrazolo[4,3-*c*]quinoline with arylboronic acids [1]. Crystals of the title compound were obtained by recrystallization in dimethylformamide.

Experimental details

Data reduction was carried out using *SAINT+* software, and *SADABS* was used to make empirical absorption corrections [2]. The crystal structure was solved through direct methods using *SHELXS-97* [3]. Hydrogen atoms were positioned geometrically and allowed to ride on their respective parent atoms with d(C–H) = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. Diagrams and publication material were generated using *WinGX* [4] and *PLATON* [5].

Discussion

In recent years there has been growing interest in the design and synthesis of pyrazolo[4,3-*c*]quinoline-based compounds [6] due to their wide-range of biological properties. Pyrazolo[4,3-*c*]quinoline derivatives serve as photosensitizing anti-cancer agents [7], benzodiazepine antagonists [8], selective cyclooxygenase-2 (COX-2) inhibitors [9] and others exhibit anti-inflammatory properties [6]. Interest in pyrazolo[4,3-*c*]quinoline-based compounds is

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7902(7)	0.5667(5)	0.10136(10)	0.0326(9)
C2	0.8760(9)	0.5319(5)	0.07259(10)	0.0398(10)
H2	1.0169	0.4777	0.0704	0.048*
C3	0.7519(9)	0.5778(6)	0.04686(11)	0.0432(11)
H3	0.8082	0.5568	0.0268	0.052*
C4	0.5448(8)	0.6546(6)	0.05075(11)	0.0409(11)
C5	0.4628(8)	0.6884(5)	0.07956(11)	0.0406(11)
H5	0.3206	0.7406	0.0818	0.049*
C6	0.5849(8)	0.6470(5)	0.10514(11)	0.0375(10)
H6	0.5304	0.6727	0.1252	0.045*
C7	1.1560(8)	0.5054(6)	0.16335(10)	0.0395(10)
H7	1.2833	0.5328	0.1756	0.047*
C8	1.0106(8)	0.3707(5)	0.16969(10)	0.0326(9)
C9	0.9907(8)	0.2425(5)	0.19190(10)	0.0334(9)
H9	1.0971	0.2399	0.2082	0.040*
C10	0.6785(7)	0.1312(5)	0.16763(9)	0.0293(9)
C11	0.6773(7)	0.2527(5)	0.14389(9)	0.0302(9)
C12	0.8507(7)	0.3747(5)	0.14645(9)	0.0317(9)
C13	0.5199(7)	0.2438(5)	0.11996(9)	0.0307(9)
H13	0.5198	0.3269	0.1044	0.037*
C14	0.3657(7)	0.1171(5)	0.11852(9)	0.0297(9)
C15	0.3600(7)	−0.0006(5)	0.14265(9)	0.0301(9)
H15	0.2485	−0.0850	0.1424	0.036*
C16	0.5144(7)	0.0044(5)	0.16673(9)	0.0296(9)
C17	0.2097(7)	0.1035(5)	0.09169(9)	0.0286(9)
C18	0.2744(8)	0.1631(5)	0.06268(10)	0.0338(10)
H18	0.4170	0.2145	0.0603	0.041*
C19	0.1340(9)	0.1483(5)	0.03749(10)	0.0401(11)
H19	0.1810	0.1880	0.0179	0.048*
C20	−0.0772(8)	0.0748(6)	0.04083(11)	0.0420(11)
H20	−0.1753	0.0650	0.0237	0.050*
C21	−0.1407(8)	0.0171(5)	0.06916(10)	0.0384(10)
H21	−0.2847	−0.0321	0.0716	0.046*
C22	0.0019(8)	0.0292(5)	0.09456(10)	0.0360(10)
H22	−0.0443	−0.0139	0.1139	0.043*
C23	0.4975(7)	−0.1273(5)	0.19095(9)	0.0300(9)

not only limited to the synthesis or development of more diverse and complex bioactive derivatives for structure-activity relationship evaluations, but has since been extended to focus on their photophysical properties as potential photosensitizing anti-cancer agents and fluorescence probes. The crystallographic study of the 1-(4-chlorophenyl)-6,8-diphenyl-1*H*-pyrazolo[4,3-c]quinolone was carried out to determine its molecular structure and the geometry of the pyrazolo[4,3-c]quinoline tricyclic system. The 1-aryl ring is highly deformed out of plane of the pyrazoloquinazoline framework (average torsion angle *ca.* 75.2°) compared to other aryl substituents to avoid steric interaction between its *ortho*-hydrogen atoms and H-9 (C13). The slight twist of the 6-aryl ring from co-planarity (average torsion angle *ca.* −59.2°), on the other hand, minimizes steric interaction between its *ortho*-hydrogen atoms on C24 and C28 with the lone pair electrons on N3 and C15, respectively.

Acknowledgements: The authors are grateful to the University of South Africa and the National Research Foundation for financial assistance.

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