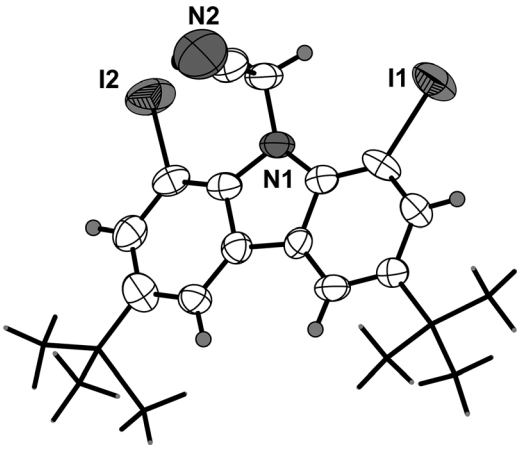
Lin-Li Tang and Fei Zeng*

The crystal structure of 2-(3,6-di-*tert*-butyl-1,8-diiodo-9*H*-carbazol-9-yl)acetonitrile, $C_{22}H_{24}I_2N_2$



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Abstract

$C_{22}H_{24}I_2N_2$, orthorhombic, *Pbca* (no. 33), a = 21.069(4) Å, b = 8.7963(17) Å, c = 23.757(5) Å, V = 4402.8(15) Å^3, Z = 8, $R_{gt}(F)$ = 0.0530, w$R_{ref}(F^2)$ = 0.1569, T = 296(2) K.

CCDC no.: 2153325

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.

Table 1: Data collection and handling.

Crystal:	Brown block
Size:	0.38 × 0.32 × 0.16 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	2.87 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	47,153, 9974, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 7966
$N(param)_{refined}$:	463
Programs:	Bruker [1], SHELX [2]

Source of material

Five grams of 3,6-di-*tert*-butylcarbazole was dissolved in 60 ml of chloroform, and after stirring for half an hour, 5 g iodine and 3 ml of concentrated sulfuric acid were added, and the mixture was stirred at room temperature for 24 h. The reaction solution was quenched by adding 100 ml of water, and then dichloromethane (DCM) was added to extract and separate the layers. The retained organic layer (lower layer) was placed in a 100 ml round-bottomed flask, and then spin-dried using a rotary evaporator to obtain 3,6-di-*tert*-butyl-1,8-diiodo-9*H*-carbazole as white solid. At room temperature 0.5 ml of KOH (50%, 8.95 mmol) was added to a stirred solution of 1.9 g of 3,6-di-*tert*-butyl-1,8-diiodo-9*H*-carbazole (3.58 mmol) and 0.115 g of TBAB in 14 ml of DMSO. After stirred for half an hour, 0.7 ml of $BrCH_2CN$ (5.37 mmol) was added dropwise. The mixture was warmed to 80 °C and stirred for 5 h. The reaction was quenched by ice water, and extracted by DCM (3 × 50 ml). The organic layer was dried with anhydride Na_2SO_4. The solvent was removed *in vacuo*. The organic layer was dried with anhydride Na_2SO_4. The residue was purified by purified by recrystallization using ethanol to yield 2-(3,6-di-*tert*-butyl-1,8-diiodo-9*H*-carbazol-9-yl)acetonitrile as a brown solid. Crystals of the title compound were obtained by slow evaporation of 2-(3,6-di-*tert*-butyl-1,8-diiodo-9*H*-carbazol-9-yl)acetonitrile in CH_2Cl_2 within one weeks.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

***Corresponding author: Fei Zeng**, Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou, Hunan 425199, People's Republic of China,
E-mail: zengfei@iccas.ac.cn. https://orcid.org/0000-0002-1555-1812
Lin-Li Tang, Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou, Hunan 425199, People's Republic of China

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2988 (5)	1.1112 (12)	0.7439 (5)	0.049 (2)
C2	0.2633 (5)	1.0092 (12)	0.7744 (4)	0.049 (2)
H2	0.260475	1.021876	0.813220	0.059*
C3	0.2311 (5)	0.8869 (11)	0.7495 (4)	0.045 (2)
C4	0.2383 (5)	0.8681 (11)	0.6921 (5)	0.048 (2)
H4	0.218779	0.786719	0.674079	0.058*
C5	0.2749 (5)	0.9706 (10)	0.6608 (4)	0.043 (2)
C6	0.3049 (4)	1.0954 (10)	0.6865 (4)	0.043 (2)
C7	0.1918 (6)	0.7711 (12)	0.7837 (5)	0.058 (3)
C8	0.2316 (8)	0.6221 (18)	0.7847 (8)	0.102 (5)
H8A	0.249009	0.603819	0.747959	0.153*
H8B	0.204834	0.538378	0.795208	0.153*
H8C	0.265421	0.632047	0.811468	0.153*
C9	0.1306 (8)	0.739 (2)	0.7526 (9)	0.091 (4)
H9A	0.110003	0.832883	0.743365	0.136*
H9B	0.103089	0.678903	0.776020	0.136*
H9C	0.139734	0.683806	0.718618	0.136*
C10	0.1759 (11)	0.828 (2)	0.8428 (7)	0.114 (5)
H10A	0.214278	0.858473	0.861571	0.170*
H10B	0.155693	0.748771	0.863824	0.170*
H10C	0.147830	0.914084	0.840176	0.170*
C11	0.2931 (4)	0.9717 (10)	0.6017 (4)	0.043 (2)
C12	0.3323 (4)	1.1001 (10)	0.5927 (4)	0.045 (2)
C13	0.3576 (5)	1.1217 (11)	0.5404 (5)	0.048 (2)
C14	0.3443 (5)	1.0189 (12)	0.4976 (5)	0.053 (2)
H14	0.361740	1.036449	0.462257	0.064*
C15	0.3070 (6)	0.8930 (14)	0.5048 (5)	0.061 (3)
C16	0.2809 (5)	0.8706 (11)	0.5581 (4)	0.047 (2)
H16	0.255072	0.786705	0.564513	0.056*
C17	0.2941 (12)	0.784 (3)	0.4593 (9)	0.118 (3)
C18	0.2229 (9)	0.747 (2)	0.4519 (10)	0.118 (3)
H18A	0.207875	0.692613	0.484231	0.177*
H18B	0.217185	0.685928	0.418808	0.177*
H18C	0.199494	0.840114	0.447947	0.177*
C19	0.3202 (9)	0.609 (2)	0.4821 (8)	0.118 (3)
H19A	0.341881	0.620579	0.517377	0.177*
H19B	0.348851	0.567049	0.454740	0.177*
H19C	0.284636	0.542248	0.486785	0.177*
C20	0.3283 (10)	0.795 (2)	0.4052 (8)	0.118 (3)
H20A	0.304058	0.856031	0.379426	0.177*
H20B	0.334138	0.695400	0.389696	0.177*
H20C	0.368944	0.841729	0.411285	0.177*
C21	0.6037 (5)	0.4129 (12)	0.4676 (4)	0.050 (2)
C22	0.5900 (5)	0.5201 (12)	0.5081 (5)	0.054 (3)
H22	0.608171	0.508104	0.543478	0.065*
C23	0.5504 (6)	0.6463 (13)	0.4995 (5)	0.056 (2)
C24	0.5234 (5)	0.6644 (12)	0.4459 (4)	0.050 (2)
H24	0.498147	0.748274	0.437672	0.060*
C25	0.5354 (5)	0.5529 (11)	0.4051 (4)	0.046 (2)
C26	0.5740 (5)	0.4277 (11)	0.4147 (4)	0.046 (2)
C27	0.5395 (7)	0.7673 (14)	0.5481 (5)	0.060 (3)
C28	0.5405 (11)	0.9226 (19)	0.5213 (8)	0.120 (5)
H28A	0.581225	0.940026	0.504471	0.180*
H28B	0.532619	0.998444	0.549509	0.180*
H28C	0.508142	0.928108	0.492896	0.180*
C29	0.4729 (11)	0.757 (2)	0.5635 (11)	0.124 (5)
H29A	0.447246	0.760968	0.530118	0.186*
H29B	0.462030	0.840130	0.587768	0.186*
H29C	0.465411	0.662613	0.582737	0.186*
C30	0.5948 (11)	0.761 (2)	0.5866 (9)	0.108 (5)
H30A	0.593987	0.667740	0.607360	0.163*
H30B	0.592980	0.845445	0.612290	0.163*
H30C	0.633291	0.766948	0.565050	0.163*
C31	0.5167 (4)	0.5462 (11)	0.3459 (4)	0.044 (2)
C32	0.4813 (5)	0.6454 (11)	0.3143 (4)	0.046 (2)
H32	0.461978	0.728906	0.331102	0.055*
C33	0.4745 (5)	0.6193 (12)	0.2555 (5)	0.049 (2)
C34	0.5051 (5)	0.4937 (11)	0.2333 (5)	0.049 (2)
H34	0.500751	0.474853	0.194982	0.059*
C35	0.5416 (5)	0.3945 (11)	0.2648 (4)	0.046 (2)
C36	0.5462 (4)	0.4195 (11)	0.3226 (4)	0.045 (2)
C37	0.4390 (6)	0.7355 (12)	0.2171 (5)	0.052 (2)
C38	0.4766 (8)	0.866 (2)	0.2061 (9)	0.123 (8)
H38A	0.496572	0.898783	0.240379	0.185*
H38B	0.450178	0.945986	0.191839	0.185*
H38C	0.508586	0.841316	0.178830	0.185*
C39	0.3751 (7)	0.7773 (19)	0.2487 (7)	0.080 (4)
H39A	0.348842	0.688361	0.251544	0.120*
H39B	0.352953	0.854518	0.227960	0.120*
H39C	0.384727	0.814245	0.285736	0.120*
C40	0.4159 (12)	0.670 (3)	0.1626 (8)	0.141 (10)
H40A	0.451459	0.635656	0.140890	0.212*
H40B	0.393403	0.747088	0.141960	0.212*
H40C	0.387994	0.586430	0.170118	0.212*
C41	0.5865 (6)	0.1775 (13)	0.3654 (6)	0.058 (3)
H41A	0.612961	0.146757	0.396781	0.070*
H41B	0.606632	0.142933	0.330991	0.070*
C42	0.5218 (7)	0.1029 (13)	0.3709 (5)	0.065 (3)
C43	0.3427 (6)	1.3408 (11)	0.6461 (6)	0.055 (3)
H43A	0.374995	1.373885	0.619711	0.066*
H43B	0.355638	1.373824	0.683393	0.066*
C44	0.2832 (8)	1.4134 (14)	0.6319 (6)	0.074 (4)
I1	0.35550 (5)	1.25975 (11)	0.79165 (4)	0.0791 (3)
I2	0.42659 (5)	1.29108 (11)	0.52107 (5)	0.0867 (3)
I3	0.67336 (5)	0.25233 (10)	0.48930 (5)	0.0813 (3)
I4	0.59634 (5)	0.23584 (11)	0.22030 (4)	0.0786 (3)
N1	0.3395 (4)	1.1771 (9)	0.6453 (4)	0.0471 (19)
N2	0.2381 (8)	1.4726 (18)	0.6200 (8)	0.134 (7)
N3	0.5816 (4)	0.3413 (9)	0.3645 (4)	0.0441 (18)
N4	0.4727 (8)	0.0498 (15)	0.3744 (7)	0.105 (5)

Comment

Carbazole-based compounds have been widely utilized in prominent optoelectronic applications such as organic light

emitting diodes (OLEDs) and photovoltaic cells, because of their excellent thermal and electronic properties [3–8]. Biologically active carbazole alkaloids have been isolated from diverse natural sources and exhibit a broad range of different frameworks and functional groups. Therefore, a large number of classical and nonclassical methods have been developed for their synthesis [9, 10]. The synthesis of carbazole derivatives is a hot topic in organic chemistry. Previously, Zhang reported the synthesis and crystal structure of 3,6-di-*tert*-butyl-1,8-diiodo-9-methyl-9*H*-carbazole [11]. Herein, we reported the synthesis of 2-(3,6-di-*tert*-butyl-1,8-diiodo-9*H*-carbazol-9-yl)acetonitrile, which may enriched the application of carbazoles in materials chemistry.

The title compound, built up by the $C_{22}H_{24}I_2N_2$ molecules, has been synthesized. There are two crystallographically independent molecules in the asymmetric unit. Both molecules are very similar. The single-crystal structure verifies that all bond lengths are in normal ranges [11].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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