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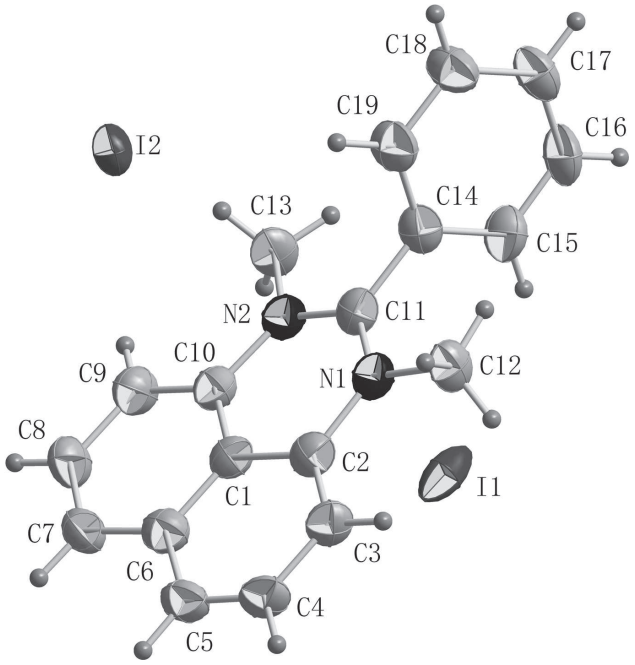
Crystal structure of 1,3-dimethyl-2-phenyl-1*H*-perimidin-3-ium iodide, $C_{19}H_{17}IN_2$



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Abstract

$C_{19}H_{17}IN_2$, monoclinic, $C2/c$, $a = 18.701(4)$ Å, $b = 7.9230(16)$ Å, $c = 24.069(6)$ Å, $\beta = 111.168(2)°$, $V = 3325.6(13)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0201$, $wR_{ref}(F^2) = 0.0462$, $T = 296(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.38 × 0.36 × 0.32 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	19.2 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	50°, 98.6%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14906, 2887, 0.015
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2763
$N(param)_{refined}$:	203
Programs:	Bruker programs [1], SHELX [2, 3], PLATON [17]

Source of material

(1) Iodomethane (3.02 g, 0.021 mol) was added in one portion to a solution of 2-phenyl-1*H*-perimidine (2.44 g, 0.01 mol) in dimethylformamide (15 mL) at 373 K. The mixture was heated for 8 h, giving a yellow precipitate. After cooling, the yellow solid was filtered and dried under vacuum to give 1-methyl-2-phenyl-1*H*-perimidine (1.83 g, 71%).

(2) Iodomethane (3.02 g, 0.021 mol) was added dropwise to a solution of 1-methyl-2-phenyl-1*H*-perimidine (2.58 g, 0.01 mol) in dimethylformamide (20 mL) at 373 K. The mixture was heated for 8 h, giving a yellow precipitate. After cooling, the yellow solid was filtered, washed with anhydrous ethanol and dried under vacuum to give the desired salt 1,3-dimethyl-2-phenyl-1*H*-perimidin-3-ium iodide (3.04 g, 76%). A light yellow crystal was obtained by recrystallization from anhydrous ethanol.

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The methyl groups were idealized and refined using rigid groups allowed to rotate about the N—C bond (AFIX 137 option of the SHELXL-2013 program [2, 3]). The U_{iso} values of the hydrogen atoms of the methyl groups were set to $1.5U_{eq}$(C) and the U_{iso} values of all other hydrogen atoms were set to 1.2 U_{eq}(C).

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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}
N1	0.02337(10)	0.0880(2)	0.10919(7)	0.0404(4)
N2	0.00485(9)	0.3438(2)	0.14921(7)	0.0412(4)
C2	−0.05664(12)	0.0579(3)	0.08300(9)	0.0415(4)
C14	0.13526(12)	0.2481(3)	0.16972(10)	0.0430(5)
C11	0.05110(11)	0.2267(2)	0.14101(9)	0.0392(4)
C1	−0.10546(11)	0.1761(2)	0.09493(8)	0.0396(4)
C6	−0.18568(12)	0.1482(3)	0.07256(9)	0.0483(5)
C10	−0.07554(11)	0.3222(3)	0.12880(9)	0.0410(4)
C9	−0.12331(14)	0.4357(3)	0.14080(11)	0.0569(6)
H9	−0.1035	0.5329	0.1626	0.068*
C16	0.25785(14)	0.3241(3)	0.16719(13)	0.0608(6)
H16	0.2876	0.3660	0.1466	0.073*
C3	−0.08599(14)	−0.0823(3)	0.04876(11)	0.0569(6)
H3	−0.0537	−0.1605	0.0410	0.068*
C17	0.29231(13)	0.2778(3)	0.22568(12)	0.0583(6)
H17	0.3452	0.2870	0.2445	0.070*
C12	0.07474(14)	−0.0462(3)	0.10397(12)	0.0593(6)
H12A	0.0642	−0.1485	0.1210	0.089*
H12B	0.1270	−0.0134	0.1248	0.089*
H12C	0.0666	−0.0646	0.0627	0.089*
C15	0.17931(13)	0.3095(3)	0.13828(11)	0.0539(5)
H15	0.1563	0.3402	0.0985	0.065*
C8	−0.20211(15)	0.4040(4)	0.11994(12)	0.0677(7)
H8	−0.2342	0.4796	0.1292	0.081*
C19	0.17027(13)	0.2034(3)	0.22913(10)	0.0535(6)
H19	0.1409	0.1638	0.2504	0.064*
C13	0.03612(15)	0.5060(3)	0.17746(13)	0.0628(7)
H13A	0.0310	0.5131	0.2157	0.094*
H13B	0.0084	0.5970	0.1527	0.094*
H13C	0.0893	0.5136	0.1825	0.094*
C18	0.24860(13)	0.2178(3)	0.25649(12)	0.0599(6)
H18	0.2721	0.1865	0.2962	0.072*
C5	−0.21444(14)	0.0039(3)	0.03718(11)	0.0607(6)
H5	−0.2670	−0.0164	0.0217	0.073*
C7	−0.23314(14)	0.2659(4)	0.08657(11)	0.0615(6)
H7	−0.2859	0.2488	0.0729	0.074*
C4	−0.16575(16)	−0.1051(3)	0.02568(12)	0.0677(7)
H4	−0.1859	−0.1983	0.0016	0.081*
I2[a]	0.0000	0.92943(3)	0.2500	0.04629(7)
I1[a]	0.0000	0.5000	0.0000	0.08320(11)

[a]Occupancy: 0.5.

Comment

Perimidines have long attracted the attention of researchers because they exhibit a diverse range of biological activities [4–7]. Perimidines can also be used as dyes and have a wide application in industrial field with solvent black 3 being a notable example 3 [8]. There are several preparative methods available for the synthesis of perimidine derivatives [9–13]. The most common method is the condensation reaction of 1,8-diaminonaphthalene with a carbonyl group [14–16]. Herein, a perimidine iodate was synthesized and characterized by single-crystal X-ray diffraction [14, 15]. In the structure of the title compound, the bond angles N(1)—C(11)—N(2), N(1)—C(11)—C(14) and N(2)—C(11)—C(14) are 121.40(18), 119.33(18) and 119.23(17)°, respectively, indicative of the sp^2 hybridization state of the C(11) atom. The newly-formed three-membered heterocyclic ring is essentially planar, as demonstrated by the fact that the angle between the naphthalene and *N*-heterocyclic rings is 4.065°. As revelaed by PLATON analysis [17], the maximum deviation from the least-squares plane through the 13 atoms of the perimidine ring is 0.060(2) Å. Two half-occupied iodide anions are present in the asymmetric unit and sit on special positions.

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