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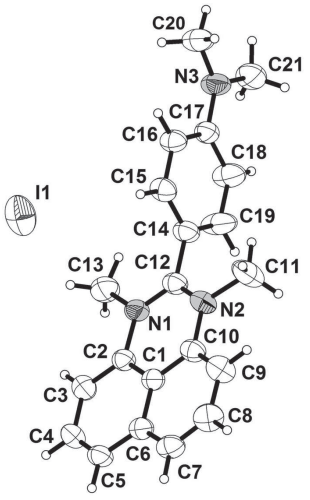
Crystal structure of 2-(4-(dimethylamino)phenyl)-1,3-dimethyl-1*H*-perimidin-3-ium iodide, $C_{21}H_{22}I_1N_3$



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Abstract

$C_{21}H_{22}I_1N_3$, monoclinic, $C2/c$, $a = 21.829(7)$ Å, $b = 10.741(3)$ Å, $c = 17.564(5)$ Å, $\beta = 109.446(3)°$, $V = 3883(2)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0260$, $wR_{ref}(F^2) = 0.0687$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. 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:	Yellow block
Size:	0.38 × 0.35 × 0.30 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	16.6 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	50°, >98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	17887, 3382, 0.017
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2855
$N(param)_{refined}$:	230
Programs:	SHELX [8, 9], Bruker programs [10], OLEX2 [11]

Source of material

Iodomethane (3.02 g, 0.021 mol) was added in one portion to a solution of *N*,*N*-dimethyl-4-(1*H*-perimidin-2-yl)aniline (2.87 g, 0.01 mol) in dimethylformamide (15 mL) at 373 K. The mixture was heated for 10 h, giving a yellow precipitate. After cooling, the yellow solid was filtered and dried under vacuum to give *N*,*N*-dimethyl-4-(1-methyl-1*H*-perimidin-2-yl)aniline (2.11 g, 70%), which was used in the next reaction without purification. Iodomethane (3.02 g, 0.021 mol) was added dropwise to a solution of *N*,*N*-dimethyl-4-(1-methyl-1*H*-perimidin-2-yl)aniline (3.01 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 title compound (2.88 g, 65%). Light yellow needle crystals were obtained by recrystallization from anhydrous ethanol.

Experimental details

The methyl groups were idealized and refined using rigid groups allowed to rotate about the N–C bond (AFIX 137 option of the SHELXL program [9]). The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{eq}$(C) and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{eq}$(C).

Discussion

Multi-nuclear *N*-heterocyclic compounds like perimidines have drawn extensive examinations of many researchers for a long time [1–3]. There are several preparative methods for the synthesis of perimidine derivatives [4–7]. However,

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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	0.19375(11)	0.1936(2)	0.41275(13)	0.0410(5)
C2	0.23068(12)	0.2553(2)	0.37200(14)	0.0443(5)
C3	0.28508(13)	0.2008(3)	0.36377(16)	0.0557(6)
H3	0.3094	0.2420	0.3370	0.067*
C4	0.30325(14)	0.0818(3)	0.39640(16)	0.0584(7)
H4	0.3400	0.0446	0.3908	0.070*
C5	0.26907(13)	0.0197(2)	0.43570(14)	0.0518(6)
H5	0.2826	−0.0590	0.4567	0.062*
C6	0.21287(12)	0.0729(2)	0.44523(13)	0.0454(5)
C7	0.17423(13)	0.0124(2)	0.48431(15)	0.0542(6)
H7	0.1862	−0.0659	0.5069	0.065*
C8	0.11971(14)	0.0676(3)	0.48915(17)	0.0613(7)
H8	0.0946	0.0253	0.5144	0.074*
C9	0.10001(13)	0.1862(3)	0.45725(18)	0.0595(7)
H9	0.0625	0.2222	0.4613	0.071*
C10	0.13725(12)	0.2486(2)	0.41971(15)	0.0463(5)
C11	0.06461(15)	0.4315(3)	0.3995(2)	0.0771(9)
H11A	0.0262	0.3833	0.3742	0.116*
H11B	0.0593	0.5133	0.3762	0.116*
H11C	0.0717	0.4377	0.4563	0.116*
C12	0.15587(12)	0.4273(2)	0.34743(14)	0.0469(6)
C13	0.24644(15)	0.4374(3)	0.29498(18)	0.0670(8)
H13A	0.2910	0.4440	0.3289	0.100*
H13B	0.2291	0.5191	0.2792	0.100*
H13C	0.2437	0.3897	0.2477	0.100*
C14	0.13451(12)	0.5516(2)	0.31087(15)	0.0508(6)
C15	0.09106(12)	0.5596(2)	0.23325(15)	0.0501(6)
H15	0.0760	0.4870	0.2041	0.060*
C16	0.06958(12)	0.6732(2)	0.19824(15)	0.0497(6)
H16	0.0397	0.6761	0.1462	0.060*
C17	0.09188(13)	0.7843(2)	0.23960(15)	0.0477(6)
C18	0.13655(19)	0.7745(3)	0.31749(18)	0.0778(10)
H18	0.1529	0.8464	0.3466	0.093*
C19	0.15675(18)	0.6603(3)	0.35194(18)	0.0851(11)
H19	0.1861	0.6565	0.4042	0.102*
C20	0.02107(15)	0.9075(3)	0.12874(17)	0.0691(8)
H20A	0.0313	0.8583	0.0890	0.104*
H20B	0.0156	0.9929	0.1116	0.104*
H20C	−0.0184	0.8775	0.1350	0.104*
C21	0.09575(18)	1.0116(3)	0.24875(19)	0.0732(8)
H21A	0.0814	1.0147	0.2948	0.110*
H21B	0.0787	1.0819	0.2145	0.110*
H21C	0.1424	1.0137	0.2665	0.110*
I1	0.09819(2)	0.27074(2)	0.08068(2)	0.08093(11)
N1	0.20884(10)	0.37508(18)	0.33968(12)	0.0470(5)
N2	0.12126(10)	0.36977(19)	0.38674(13)	0.0498(5)
N3	0.07296(12)	0.8984(2)	0.20445(13)	0.0628(6)

the characters of their corresponding salts have not been reported. The bond lengths and angles in the title structure are all in the expected ranges. There are no hydrogen bonds in this salt compound, as the cation does not own hydrogen bond donating groups.

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References

1. Bu, X.; Deady, L. W.; Finlay, G. J.; Baguley, B. C.; Denny, W. A.: Synthesis and cytotoxic activity of 7-Oxo-7*H*-dibenz[f,ij]isoquinoline and 7-oxo-7*H*-benzo[e]perimidine derivatives. J. Med. Chem. **44** (2001) 2004–2014.
2. Fu, C. R.; Pei, J.; Ning, Y.; Liu, M.; Shan, P. C.; Liu, J.; Li, Y. Q.; Hu, F. Z.; Zhu, Y. Q.; Yang, H. Z.; Zou, X. M.: Synthesis and insecticidal activities of novel pyrazole oxime ether derivatives with different substituted pyridyl rings. Pest Manag. Sci. **70** (2013) 1207–1214.
3. Hashim, J. A.; Kezhal, M. S.: Synthesis, characterization and biological activity of 2-Aryl-2,3-dihydro-1*H*-perimidine. Res. Pharm. Biotech. **5** (2014) 1–6.
4. Hendrickson, J. B.; Hussoin, M. S.: Seeking the ideal dehydrating reagent. J. Org. Chem. **52** (1987) 4137–4139.
5. Vanden, E. J. J.; Delfosse, F.; Mayence, A.; Haverbeke, Y. V.: Old reagents, new results-aromatization of Hantzsch 1,4-dihydropyridines with manganese-dioxide and 2,3-dichloro-5,6-dicyano-1,4-benzoquinone. Tetrahedron **51** (1995) 5813–5818.
6. Deady, L. W.; Rodemann, T. J.: Synthesis of perimidine and fused perimidine derivatives from reaction of 1,8-naphthalenediamine with an iminoisocoumarin. Heterocycl. Chem. **35** (1998) 1417–1419.
7. Starshikov, M.; Pozharskii, F. T.: Synthesis of 2-(5-halogeno-2-f uryl)-2,3-dihydroperimidines. Chem. Heterocycl. Compd. **9** (1973) 922–924.
8. Sheldrick, G. M.: SHELXS-97: Program for the Solution of Crystal Structures. University of Göttingen, Germany, (1997).
9. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
10. Bruker. APEX2, SAINT and SADABS. Brucker AXS Inc., Madison, Wisconsin, USA, (2009).
11. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. J. Appl. Cryst. **42** (2009) 339–341.