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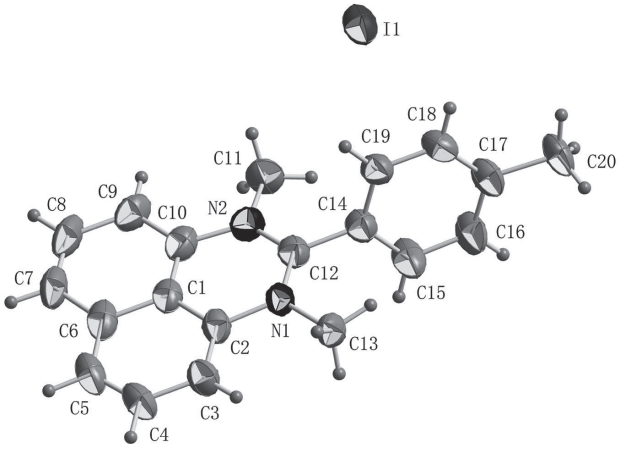
Crystal structure of 1,3-dimethyl-2-(*p*-tolyl)-1*H*-perimidin-3-ium iodide 1.5 hydrate, $C_{20}H_{22}IN_2O_{1.5}$



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Abstract

$C_{20}H_{22}IN_2O_{1.5}$, monoclinic, $C2/c$ (no. 15), $a = 38.828(4)$ Å, $b = 9.5689(9)$ Å, $c = 26.186(2)$ Å, $\beta = 127.719(1)°$, $Z = 8$, $V = 7696.0(12)$ Å^3, $R_{gt}(F) = 0.0379$, $wR_{ref}(F^2) = 0.1058$, $T = 296(2)$ 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.

Source of material

Iodomethane (3.02 g, 0.021 mol) was added dropwise to a solution of 1-methyl-2-(p-tolyl)-1H-perimidine (2.72 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 ethanol and

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Table 1: Data collection and handling.

Crystal:	Block, colorless
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.68 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	35774, 6781, 0.018
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5614
$N(param)_{refined}$:	448
Programs:	Bruker programs [1], SHELX [2, 3]

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

Atom	x	y	z	U_{iso}*/U_{eq}
I2	−0.079488(9)	0.98965(3)	0.020967(15)	0.07124(12)
I1	0.164420(10)	0.36573(4)	0.309302(15)	0.07974(13)
O2	−0.03783(19)	0.2095(5)	0.1576(2)	0.1342(17)
H2A	−0.0136	0.2091	0.1953	0.201*
H2B	−0.0363	0.1559	0.1332	0.201*
N1	0.01707(9)	0.7558(3)	0.07866(14)	0.0492(7)
N2	−0.02088(11)	0.6172(4)	0.10198(16)	0.0590(8)
N3	−0.18228(9)	0.8937(3)	−0.15015(14)	0.0463(7)
C5	−0.06228(15)	0.6206(6)	−0.1137(2)	0.0773(14)
H5	−0.0798	0.5913	−0.1566	0.093*
C6	−0.06783(12)	0.5627(5)	−0.0705(2)	0.0662(12)
C1	−0.04127(11)	0.6118(4)	−0.00493(18)	0.0519(9)
C2	−0.00965(11)	0.7131(4)	0.01290(17)	0.0509(8)
C12	0.01034(11)	0.7107(4)	0.11965(18)	0.0515(8)
C14	0.03907(12)	0.7594(4)	0.18777(18)	0.0542(9)
C15	0.03324(17)	0.8884(6)	0.2047(2)	0.0874(16)
H15	0.0107	0.9463	0.1737	0.105*
C16	0.06129(18)	0.9309(6)	0.2685(2)	0.0947(18)
H16	0.0568	1.0169	0.2801	0.114*
C17	0.09582(14)	0.8488(5)	0.3153(2)	0.0687(12)
C20	0.12821(18)	0.9053(8)	0.3836(2)	0.0993(19)
H20A	0.1424	0.9864	0.3828	0.149*
H20B	0.1495	0.8349	0.4105	0.149*
H20C	0.1131	0.9302	0.4006	0.149*
C13	0.05114(14)	0.8595(5)	0.0980(2)	0.0667(11)
H13A	0.0683	0.8273	0.0853	0.100*
H13B	0.0695	0.8717	0.1439	0.100*
H13C	0.0378	0.9471	0.0772	0.100*
C11	−0.03097(19)	0.5807(7)	0.1463(3)	0.0955(17)
H11A	−0.0206	0.4881	0.1628	0.143*
H11B	−0.0619	0.5843	0.1234	0.143*
H11C	−0.0170	0.6460	0.1815	0.143*
C10	−0.04721(12)	0.5593(4)	0.0390(2)	0.0585(10)
C9	−0.07704(15)	0.4560(5)	0.0215(3)	0.0837(15)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H9	−0.0807	0.4198	0.0509	0.100*
C8	−0.10241(15)	0.4057(6)	−0.0435(3)	0.0947(19)
H8	−0.1223	0.3342	−0.0559	0.114*
C7	−0.09848(15)	0.4576(6)	−0.0869(3)	0.0870(16)
H7	−0.1164	0.4238	−0.1289	0.104*
C18	0.10090(14)	0.7214(5)	0.2975(2)	0.0663(11)
H18	0.1238	0.6641	0.3285	0.080*
C19	0.07292(14)	0.6756(5)	0.23470(19)	0.0615(10)
H19	0.0768	0.5877	0.2238	0.074*
C3	−0.00445(14)	0.7657(5)	−0.0302(2)	0.0659(11)
H3	0.0169	0.8321	−0.0174	0.079*
C4	−0.03175(17)	0.7183(6)	−0.0940(2)	0.0821(14)
H4	−0.0286	0.7559	−0.1237	0.099*
C25	−0.17093(13)	1.2183(5)	−0.24712(19)	0.0614(10)
H25	−0.1688	1.2895	−0.2692	0.074*
C24	−0.15646(14)	1.0879(5)	−0.2457(2)	0.0651(11)
H24	−0.1445	1.0717	−0.2669	0.078*
C23	−0.15906(13)	0.9769(4)	−0.21310(19)	0.0570(9)
H23	−0.1496	0.8880	−0.2136	0.068*
C22	−0.17574(11)	1.0019(4)	−0.18056(17)	0.0462(8)
C32	−0.19358(10)	0.9213(4)	−0.11245(16)	0.0453(8)
C34	−0.20099(12)	0.8063(4)	−0.08252(17)	0.0502(8)
C39	−0.24237(13)	0.7961(5)	−0.0990(2)	0.0643(11)
H39	−0.2634	0.8627	−0.1259	0.077*
C38	−0.25224(15)	0.6872(5)	−0.0756(2)	0.0739(12)
H38	−0.2801	0.6809	−0.0871	0.089*
C37	−0.22143(16)	0.5866(5)	−0.0351(2)	0.0670(11)
C40	−0.2330(2)	0.4671(6)	−0.0103(3)	0.0951(17)
H40A	−0.2295	0.4964	0.0277	0.143*
H40B	−0.2142	0.3892	0.0000	0.143*
H40C	−0.2627	0.4399	−0.0429	0.143*
C27	−0.20642(12)	1.3776(4)	−0.21622(19)	0.0599(10)
H27	−0.2063	1.4512	−0.2393	0.072*
C26	−0.18931(11)	1.2466(4)	−0.21521(17)	0.0521(9)
C33	−0.17779(17)	0.7502(4)	−0.1652(2)	0.0686(11)
H33A	−0.1918	0.7425	−0.2105	0.103*
H33B	−0.1912	0.6866	−0.1537	0.103*
H33C	−0.1475	0.7277	−0.1411	0.103*
C35	−0.16969(13)	0.7085(4)	−0.0410(2)	0.0604(10)
H35	−0.1417	0.7155	−0.0289	0.072*
C36	−0.18009(15)	0.6008(5)	−0.0176(2)	0.0663(11)
H36	−0.1587	0.5362	0.0106	0.080*
N4	−0.20240(9)	1.0517(3)	−0.10570(14)	0.0473(7)
C31	−0.20382(15)	1.0908(5)	−0.05240(19)	0.0626(10)
H31A	−0.2334	1.1090	−0.0694	0.094*
H31B	−0.1866	1.1732	−0.0314	0.094*
H31C	−0.1924	1.0156	−0.0218	0.094*
C30	−0.20565(10)	1.1629(4)	−0.14404(16)	0.0454(8)
C21	−0.19002(10)	1.1365(4)	−0.17953(16)	0.0446(8)
C29	−0.22229(12)	1.2903(4)	−0.1464(2)	0.0565(9)
H29	−0.2330	1.3063	−0.1235	0.068*
C28	−0.22303(13)	1.3972(4)	−0.1839(2)	0.0629(11)
H28	−0.2352	1.4832	−0.1866	0.076*
O1	0.04859(19)	0.3069(7)	0.2480(3)	0.158(2)
H1A	0.0758	0.3422	0.2917	0.236*
H1B	0.0526	0.2371	0.2735	0.236*
O3	−0.1282(3)	0.3201(10)	0.0871(4)	0.251(4)
H3A	−0.1082	0.2597	0.1011	0.376*
H3B	−0.1444	0.2960	0.0973	0.376*

dried under vacuum to give the title compound (3.11 g, 75%). Yellow needle crystals were obtained by recrystallization from ethyl acetate.

Experimental details

The methyl groups refined using rigid groups allowed to rotate about the N—C bond (AFIX 137 option of the SHELX program). The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{eq}$(C) and the U_{iso} values of other hydrogen atons were set to $1.2U_{eq}$(C).

Discussion

Perimidines have drawn extensive examinations [4, 5]. There are several preparative methods for the synthesis of perimidine derivatives [6, 7].

There are two cations, two anions and three water molecules in the asymmetric unit. In its structure, the bond angles of N(1)—C(12)—N(2), N(1)—C(12)—C(14) and N(2)—C(12)—C(14) are 121.5(3), 119.7(3) and 118.7(3) in turn, which indicates the sp^2 hybridization state of C(12) atom and the new-formed three-membered heterocyclic ring is essentially planar. Geometric parameteres are in accord with those of other compounds of this class reported by us [8].

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