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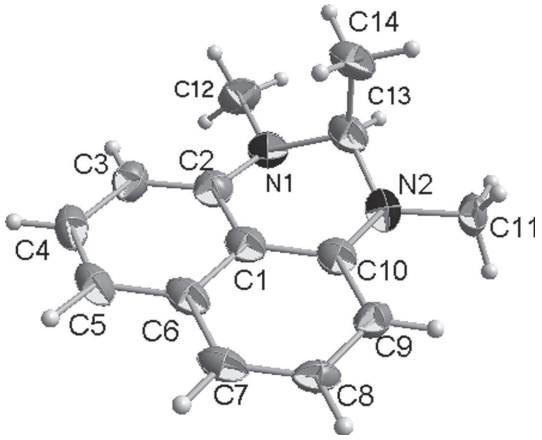
Crystal structure of 1,2,3-trimethyl-2,3-dihydro-1*H*-perimidine, $C_{14}H_{16}N_2$



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Abstract

$C_{14}H_{16}N_2$, monoclinic, $C2/c$ (no. 15), $a = 32.300(3)$ Å, $b = 7.2653(6)$ Å, $c = 10.4316(10)$ Å, $\beta = 102.133(5)°$, $V = 2393.3(4)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0464$, $wR_{ref}(F^2) = 0.1411$, $T = 296(2)$ 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:	Colourless blocks
Size:	0.41 × 0.38 × 0.29 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.7 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7687, 2111, 0.015
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1593
$N(param)_{refined}$:	148
Programs:	SHELX [10, 11], Bruker programs [12]

Source of material

Iodomethane (3.02 g, 0.021 mol) was added in one portion to a solution of 1,2-dimethyl-1*H*-perimidine (1.96 g, 0.01 mol) in dimethylformamide (15 mL) at 373 K. The mixture was heated for 6 h, giving a yellow precipitate. After cooling, the yellow solid was filtered and dried under vacuum to give 1,2,3-trimethyl-1*H*-perimidin-3-ium iodide (2.37 g, 70%), which was used in the next reaction without purification. Sodium borohydride (0.57 g, 0.015 mol) was added batch-wise to a solution of 1,2,3-trimethyl-1*H*-perimidin-3-ium iodide (3.38 g, 0.01 mol) in methanol (25 mL) under nitrogen atmosphere. The mixture was stirred at room temperature for 30 min, giving a white precipitate. After the completion of the reaction, the white solid was filtered, washed with water and dried under vacuum to yield the desired product (1.38 g, 65%). Colorless prisms of the product were obtained upon recrystallization from anhydrous ethanol.

Experimental details

All hydrogen atoms were identified in difference Fourier synthesis. The methyl groups were idealized and refined using rigid groups allowed to rotate about the N—C bond and C—C bond (AFIX 137 option of the SHELXL program [11]). 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 atons 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, because they exhibit a diverse range of biological

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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	0.09162(4)	0.09647(16)	0.68751(12)	0.0530(4)
N2	0.16555(4)	0.10822(17)	0.70616(12)	0.0566(4)
C1	0.12033(4)	0.28185(17)	0.53648(12)	0.0431(4)
C5	0.07324(6)	0.4657(2)	0.37256(16)	0.0701(5)
H5	0.0689	0.5475	0.3024	0.084*
C6	0.11503(5)	0.40982(19)	0.43173(14)	0.0547(4)
C2	0.08428(4)	0.21484(18)	0.58082(13)	0.0458(4)
C13	0.12998(5)	−0.0142(2)	0.70547(15)	0.0557(4)
H13	0.1341	−0.0710	0.7925	0.067*
C14	0.12678(6)	−0.1691(2)	0.60560(18)	0.0780(6)
H14A	0.1523	−0.2409	0.6240	0.117*
H14B	0.1230	−0.1178	0.5191	0.117*
H14C	0.1030	−0.2462	0.6107	0.117*
C10	0.16197(4)	0.22733(18)	0.60095(14)	0.0486(4)
C11	0.20685(6)	0.0385(3)	0.7683(2)	0.0974(7)
H11A	0.2253	0.1397	0.7999	0.146*
H11B	0.2184	−0.0309	0.7058	0.146*
H11C	0.2042	−0.0395	0.8405	0.146*
C12	0.05608(6)	0.0140(3)	0.73012(19)	0.0796(6)
H12A	0.0402	−0.0600	0.6606	0.119*
H12B	0.0382	0.1091	0.7525	0.119*
H12C	0.0662	−0.0619	0.8055	0.119*
C3	0.04454(5)	0.2754(2)	0.52104(16)	0.0614(4)
H3	0.0209	0.2328	0.5497	0.074*
C4	0.03967(6)	0.4008(2)	0.41734(17)	0.0721(5)
H4	0.0126	0.4408	0.3781	0.086*
C9	0.19636(5)	0.3015(2)	0.55970(18)	0.0673(5)
H9	0.2236	0.2670	0.6009	0.081*
C8	0.19039(7)	0.4281(3)	0.4563(2)	0.0787(6)
H8	0.2139	0.4773	0.4303	0.094*
C7	0.15122(7)	0.4804(2)	0.39331(18)	0.0720(5)
H7	0.1481	0.5635	0.3241	0.086*

activities [1–3]. Perimidines also can be used as dyes and have a wide application in industrial field, and the famous product was reported as solvent black 3 [4]. There are several preparative methods for the synthesis of perimidine derivatives [5–9]. A new 1,2-dimethyl-1*H*-perimidine was synthesized and characterized by single-crystal X-ray diffraction.

All bond lengths and angles are in the expected ranges. The whole molecule shows almost mirror symmetry.

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