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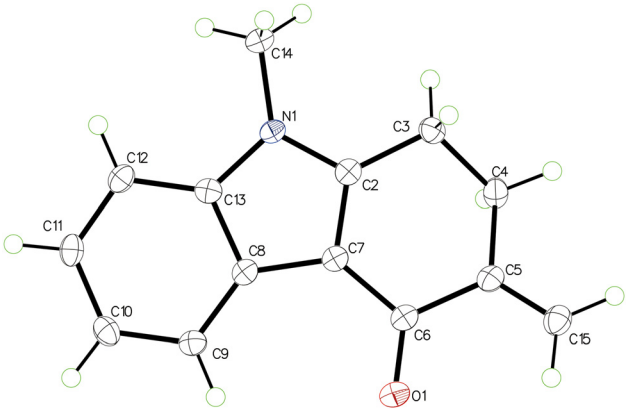
Crystal structure of 9-methyl-3-methylene-1,2,3,9-tetrahydro-4*H*-carbazol-4-one, $C_{14}H_{13}NO$



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Abstract

$C_{14}H_{13}NO$, monoclinic, $P2_1/n$ (no. 14), a = 9.2770(7) Å, b = 8.4582(6) Å, c = 13.4430(12) Å, β = 98.199(8)°, V = 1044.05(14) Å^3, Z = 4, $R_{gt}(F)$ = 0.0484, $wR_{ref}(F^2)$ = 0.1151, T = 100 K.

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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.

Source of material

1,2,3,9-Tetrahydro-9-methyl-4H-carbazol-4-one was purchased from Shanghai Bepharm Science & Technology Co., Ltd., Shanghai, China. 1,2,3,9-Tetrahydro-9-methyl-4*H*-carbazol-4-one (1.00 g, 5 mmol), dimethylamine hydrochloride (380 mg, 4.6 mmol), paraformaldehyde (150 mg, 5 mmol) and acetic acid (25 ml) were added, and refluxed for 4 h. The reaction was monitored by TLC (PE:EA = 2:1). After the reaction was complete, the reaction solution was cooled to room temperature, water was added to precipitate a solid, and the 9-methyl-3-methylene-1,2,3,9-tetrahydro-4*H*-carbazol-4-one, a white solid, was obtained by negative-pressure filtration. The title compound was dissolved in hot ethanol. Then the solution was cooled naturally to crystallize at room temperature.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.13 × 0.12 × 0.11 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	29.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5167, 2462, 0.028
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 1972
$N(param)_{refined}$:	154
Programs:	CrysAlis^{PRO} [1], SHELX [2]

Experimental details

The H atoms were positioned geometrically and were included in the refinement in the riding-model approximation. The crystal structure was solved by direct method and refined with SHELXT [2].

Comment

9-Methyl-3-methylene-1,2,3,9-tetrahydro-4H-carbazol-4-one, an impurity of Ondansetron, belongs to carbazole ketone compouds. Carbazole ketone compouds are important scaffolds for the synthesis of some natural products and bioactive substances [3]. Ondansetron, a serotonin 5-HT3 receptor selective antagonist [4, 5, 6], is effective in

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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}
O1	0.61090 (11)	−0.00383 (12)	0.24068 (8)	0.0211 (3)
N1	0.46948 (12)	0.30457 (14)	−0.03918 (9)	0.0155 (3)
C2	0.58035 (15)	0.21320 (16)	0.00686 (11)	0.0153 (3)
C3	0.71485 (16)	0.17327 (18)	−0.03569 (12)	0.0183 (3)
H3A	0.7434	0.2611	−0.0750	0.022*
H3B	0.6984	0.0815	−0.0791	0.022*
C4	0.83483 (16)	0.13829 (18)	0.05277 (12)	0.0210 (4)
H4A	0.9193	0.0959	0.0270	0.025*
H4B	0.8637	0.2363	0.0875	0.025*
C5	0.78621 (15)	0.02261 (17)	0.12629 (11)	0.0174 (3)
C6	0.64345 (15)	0.05667 (17)	0.16308 (11)	0.0160 (3)
C7	0.54889 (15)	0.16382 (17)	0.10003 (11)	0.0153 (3)
C8	0.40946 (15)	0.23055 (17)	0.11246 (11)	0.0154 (3)
C9	0.32278 (15)	0.22991 (18)	0.18935 (12)	0.0176 (3)
H9	0.3501	0.1721	0.2479	0.021*
C10	0.19524 (16)	0.31725 (18)	0.17644 (12)	0.0205 (4)
H10	0.1371	0.3191	0.2274	0.025*
C11	0.15221 (15)	0.40280 (18)	0.08799 (12)	0.0204 (3)
H11	0.0663	0.4609	0.0818	0.025*
C12	0.23392 (15)	0.40323 (17)	0.00979 (12)	0.0184 (3)
H12	0.2039	0.4580	−0.0495	0.022*
C13	0.36371 (15)	0.31760 (17)	0.02408 (11)	0.0158 (3)
C14	0.45844 (16)	0.37296 (18)	−0.13946 (11)	0.0187 (3)
H14A	0.5526	0.4095	−0.1511	0.028*
H14B	0.3916	0.4602	−0.1448	0.028*
H14C	0.4239	0.2942	−0.1886	0.028*
C15	0.86253 (17)	−0.1047 (2)	0.15992 (13)	0.0223 (4)
H15A	0.956 (2)	−0.128 (2)	0.1372 (14)	0.028 (5)*
H15B	0.8270 (19)	−0.177 (2)	0.2101 (14)	0.029 (5)*

preventing acute nausea and vomiting associated with emetogenic chemotherapy and/or radiotherapy in children and is now the most clinically used antiemetic drug [7, 8]. Because the impurities in a drug have potential toxicity and biological activity, the characterisation of each impurity is essential for the safety evaluation of the drug. The crystal was obtained by slow evaporation from an ethanol solution.

The title compound possesses a 6/5/6 ring system consisting of the benzene ring, pyrrole ring and cyclohexanone. The bonds lengths of C=C, C—N, C=O and C—C are 1.381(2)–1.411(2), 1.3625(18)–1.4570(19), 1.2374(18), 1.4427(19)–1.537(2) Å, respectively, all of which derived from the title structure are in reasonable ranges [9].

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

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