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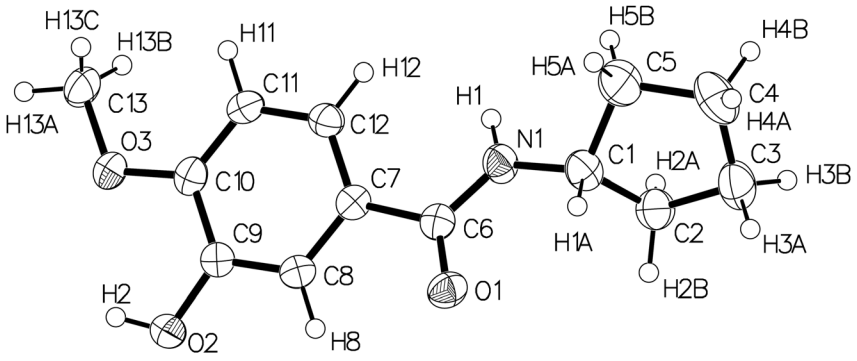
The crystal structure of *N*-cyclopentyl-3-hydroxy-4-methoxybenzamide, $C_{13}H_{17}NO_3$



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Abstract

$C_{13}H_{17}NO_3$, monoclinic, *Cc* (no. 9), a = 11.6502(8) Å, b = 13.8752(8) Å, c = 7.9644(5) Å, β = 106.051(2)°, V = 1237.25(14) Å^3, Z = 4, $R_{gt}(F)$ = 0.0427, $wR_{ref}(F^2)$ = 0.1023, T = 170 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.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.28 × 0.15 × 0.12 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	D8 VENTURE, φ and ω
θ_{max}, completeness:	26.4°, 98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5924, 2141, 0.045
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 1747
$N(param)_{refined}$:	157
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Source of material

A mixture of 10 mmol 3-hydroxy-4-methoxybenzoic acid, 10.4 mmol cyclopentylamine, and 20 mmol *N*,*N*-diisopropylethylamine (DIEA) were dissolved in 20 mL DMF. Then, 10.4 mmol of 2-(7-Azabenzotriazol-1-yl)-*N*,*N*,*N*′,*N*′-tetramethyluronium hexafluorophosphate (HATU) was added to the above solution. After 5 h stirring, the reaction was complete as monitored by Thin Layer Chromatography. Hundred milliliters of brine was added to the mixture, and the product was extracted with ethyl acetate (3 times with 500 mL). This product was washed with 5% citric acid, 5% $NaHCO_3$, and H_2O respectively. The mixture was dried over $MgSO_4$, filtered, and concentrated under vacuum. The crystals were achieved through recrystallization in ethyl acetate, and dried at room temperature for seven days.

Experimental details

All H atoms bonded to their parent C atoms were treated as riding atoms in their geometrically idealized positions, with d(C–H) = 0.95 Å and U_{iso} = 1.2 U_{eq}.

Comment

Benzamide derivatives were studied to exhibit several biological properties, like antiviral [5], antipsychotic [6],

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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	0.7121 (3)	0.5779 (2)	0.8326 (4)	0.0368 (9)
H1A	0.708766	0.551263	0.947884	0.044*
C2	0.8348 (3)	0.5581 (3)	0.8098 (5)	0.0426 (9)
H2A	0.836531	0.570422	0.688133	0.051*
H2B	0.859790	0.490786	0.841437	0.051*
C3	0.9146 (4)	0.6297 (3)	0.9369 (5)	0.0503 (11)
H3A	0.942702	0.601663	1.055539	0.060*
H3B	0.984837	0.647802	0.896745	0.060*
C4	0.8349 (4)	0.7172 (3)	0.9366 (6)	0.0570 (12)
H4A	0.839466	0.736907	1.057803	0.068*
H4B	0.860384	0.772170	0.876208	0.068*
C5	0.7080 (4)	0.6871 (3)	0.8407 (6)	0.0526 (11)
H5A	0.651019	0.708429	0.905182	0.063*
H5B	0.683580	0.715006	0.721744	0.063*
C6	0.5604 (3)	0.4543 (2)	0.7103 (4)	0.0301 (8)
C7	0.4537 (3)	0.4270 (2)	0.5658 (4)	0.0286 (8)
C8	0.4214 (3)	0.3299 (2)	0.5526 (4)	0.0337 (8)
H8	0.468580	0.284715	0.631995	0.040*
C9	0.3223 (3)	0.2986 (2)	0.4263 (5)	0.0339 (8)
C10	0.2517 (3)	0.3656 (2)	0.3104 (4)	0.0319 (8)
C11	0.2831 (3)	0.4617 (2)	0.3241 (4)	0.0321 (8)
H11	0.235661	0.507130	0.245544	0.039*
C12	0.3835 (3)	0.4925 (2)	0.4517 (4)	0.0311 (8)
H12	0.404043	0.558965	0.460637	0.037*
C13	0.0842 (3)	0.3921 (3)	0.0620 (5)	0.0464 (10)
H13A	0.018946	0.356452	−0.017842	0.070*
H13B	0.134297	0.421863	−0.004277	0.070*
H13C	0.050918	0.442449	0.121220	0.070*
N1	0.6129 (3)	0.5381 (2)	0.6962 (3)	0.0357 (7)
H1	0.586283	0.571266	0.599029	0.043*
O1	0.5970 (2)	0.40001 (16)	0.8395 (3)	0.0357 (6)
O2	0.2991 (2)	0.20202 (16)	0.4160 (4)	0.0509 (8)
H2	0.225880	0.192950	0.370114	0.076*
O3	0.1550 (2)	0.32725 (16)	0.1895 (3)	0.0384 (6)

anti-cancer [7], anti-oxidant [8], anti-inflammatory [9], anti-Alzheimer, anti-fatigue [10], and antidiabetic effects [11]. For example, several sulfamoylbenzamides and benzamide derivatives were verified to exhibit antiviral *effects via* significantly reducing intracellular HBV DNA in the cytoplasm [5, 12]. Some nitro benzamide analogs could significantly inhibit the expression of cyclooxygenase-2, interleukin-1β and tumor necrosis factor, and exhibited anti-inflammatory activities [13]. Additionally, some benzamide derivatives, like metoclopramide, exhibited antiemetic properties through blocking dopaminergic and serotonergic receptors [6], and were also used for the treatment of gastroesophageal reflux disease in infants [14].

The molecular structure of the title compound is displayed in figure. All bond lengths and angles are normal, and agree with structural reports of 4-methoxybenzamide derivatives [15–17]. The methoxy-substituted benzene [C7–C12] forms a dihedral angle of 22.9° with the amide group [–N1–C6=O1–]. The cyclopentyl ring adopts a conformation between envelope and half-chair. The hydroxyl group donates its H atom to O1, which acts as acceptor in the strong intermolecular hydrogen bond (O2–H2···O1, 1.943(3) Å).

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