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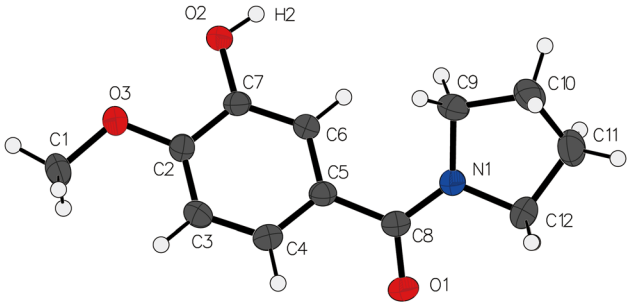
Crystal structure of (3-hydroxy-4-methoxyphenyl)(pyrrolidin-1-yl)methanone, $C_{12}H_{15}NO_3$



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Abstract

$C_{12}H_{15}NO_3$, monoclinic, $P2_1/c$ (no. 14), a = 6.7230(2) Å, b = 11.1182(3) Å, c = 14.4996(5) Å, β = 94.8870(10)°, V = 1079.87(6) Å^3, Z = 4, $R_{gt}(F)$ = 0.0511, $wR_{ref}(F^2)$ = 0.1525, T = 170 K.

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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:	Colourless block
Size:	0.15 × 0.12 × 0.08 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker D8 Venture, φ and ω
θ_{max}, completeness:	27.5°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11,456, 2464, 0.075
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1826
$N(param)_{refined}$:	147
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

1 Source of materials

A mixture of 3-hydroxy-4-methoxybenzoic acid (1.68 g, 10 mmol), pyrrolidine (2.13 g, 30 mmol), 2-(7-azabenzotriazol-1-yl)-N,N,N′,N′-tetramethyluronium hexafluorophosphate (HATU) (4.18 g, 11 mmol) and N,N-diisopropylethylamine (2.58 g, 20 mmol) was dissolved in N,N-dimethylformamide (20 mL). The mixture was stirred for 4 h at 40 °C, until the TLC indicated the reaction was completed. The mixture was diluted with brine, and then extracted with ethyl acetate (3 × 30 mL). The organic phase was washed with brine (30 mL), dried with anhydrous sodium sulphate, and then concentrated under pressure. The title compound was separated by silica-gel column chromatography with ethyl acetate–petroleum ether (20 %) gradient solvent system. The target product was obtained as a white solid. Yield: 91.4 %.

2 Experimental details

The intensity data were processed using the Bruker Saint software [1]. All hydrogen atoms were treated as riding atoms, with their positions idealized and their U_{iso} values set to 1.2 times the U_{eq} of their respective parent atoms. The crystal structure was solved using ShelXT [2] and refined with ShelXL [3]. The entire refinement process was conducted using the Olex2 software [4].

3 Comment

Phenyl(pyrrolidin-1-yl)methanone derivatives have exhibited notable bioactivity, thereby resulting in extensive investigation and reporting of numerous associated crystal structures

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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	−0.2490 (3)	0.53479 (18)	0.91431 (14)	0.0388 (5)
H1A	−0.323975	0.559521	0.856501	0.058*
H1B	−0.168916	0.602558	0.939835	0.058*
H1C	−0.342298	0.509485	0.958918	0.058*
C2	0.0249 (3)	0.45953 (15)	0.83822 (12)	0.0282 (4)
C3	0.0327 (3)	0.56059 (15)	0.78249 (12)	0.0303 (4)
H3	−0.069081	0.619808	0.782949	0.036*
C4	0.1873 (3)	0.57615 (16)	0.72614 (12)	0.0298 (4)
H4	0.190937	0.646010	0.688599	0.036*
C5	0.3368 (3)	0.49017 (15)	0.72424 (11)	0.0261 (4)
C6	0.3298 (3)	0.38858 (15)	0.78090 (12)	0.0281 (4)
H6	0.432736	0.330018	0.781001	0.034*
C7	0.1752 (3)	0.37228 (14)	0.83674 (12)	0.0273 (4)
C8	0.5060 (3)	0.51677 (15)	0.66691 (11)	0.0269 (4)
C9	0.5491 (3)	0.30011 (16)	0.61949 (14)	0.0393 (5)
H9A	0.405120	0.289784	0.600635	0.047*
H9B	0.581753	0.258411	0.679190	0.047*
C10	0.6761 (3)	0.25258 (18)	0.54536 (14)	0.0417 (5)
H10A	0.714406	0.167674	0.557383	0.050*
H10B	0.604099	0.258747	0.483085	0.050*
C11	0.8579 (3)	0.33404 (19)	0.55372 (15)	0.0436 (5)
H11A	0.926715	0.333648	0.496003	0.052*
H11B	0.953434	0.309333	0.606028	0.052*
C12	0.7694 (3)	0.45682 (17)	0.57096 (13)	0.0337 (4)
H12A	0.868337	0.509628	0.605437	0.040*
H12B	0.721566	0.496397	0.512046	0.040*
N1	0.6027 (2)	0.42863 (13)	0.62650 (10)	0.0287 (4)
O1	0.5551 (2)	0.62399 (11)	0.65738 (9)	0.0368 (4)
O2	0.1632 (2)	0.27443 (11)	0.89271 (9)	0.0360 (4)
H2	0.248337	0.223156	0.879859	0.054*
O3	−0.1202 (2)	0.43677 (11)	0.89632 (10)	0.0385 (4)

[5–9]. In the present investigation, we report the elucidation of the crystal structure of a newly synthesized compound, namely (3-hydroxy-4-methoxyphenyl)(pyrrolidin-1-yl)methanone, which belongs to the class of phenyl(pyrrolidin-1-yl) methanone derivatives.

In the context of this molecular configuration, the hydrogen moieties in the benzene ring have been substituted by hydroxyl and methoxy functional groups. Notably, the hydroxyl moiety reveals a carbon-oxygen bond (C7–O2) length measuring 1.364(3) Å, whereas the methoxy moiety exhibits a carbon-oxygen bond (C2–O3) length of 1.366(3) Å. Furthermore, the carbon–oxygen–carbon bond angle (C2–O3–C1) within the methoxy group is determined to be 116.54 (15)°, precisely adhering to the predetermined range for bond lengths and angles [10–12].

In the crystalline structure, an intermolecular hydrogen bonding interaction between the hydroxyl and ketone groups (O2–H2···O1) has been identified. Notably, the O2–H2···O1 hydrogen bond exhibits a bond length of 1.8371(14) Å and a bond angle of 173.4 (1)°. These observations suggest the pivotal role of hydrogen bonding in conferring structural stability among the molecular entities.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

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