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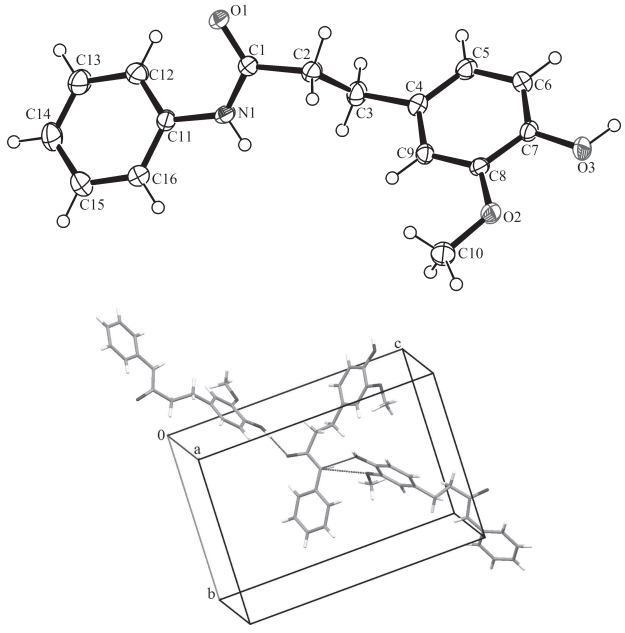
Crystal structure of 3-(4-hydroxy-3-methoxyphenyl)-*N*-phenylpropanamide, $C_{16}H_{17}NO_3$



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Abstract

$C_{16}H_{17}NO_3$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 7.9882$ (2) Å, $b = 11.4053$ (3) Å, $c = 16.2381$ (5) Å, $V = 1479.42$ (7) Å^3, $Z = 4$, $R_{gt}(F) = 0.0480$, $wR_{ref}(F^2) = 0.0991$, $T = 173$ K.

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Table 1: Data collection and handling.

Crystal:	Colorless, plate, size 0.09×0.24×0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.84 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, φ and ω scan
$2\theta_{max}$:	54.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3394, 3394
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2516
$N(param)_{refined}$:	190
Programs:	SIR2011 [16], WinGX [17], Nonius data collection and reduction software [18], SHELX [19]

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The crystal structure is shown in the figure (upper part). The hydrogen bonded connection of adjacent molecules is shown in the lower part of the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

3-(4-Hydroxy-3-methoxyphenyl)-*N*-phenylpropanamide was obtained by a method which is similar to a known procedure [1]: 3-(4-hydroxy-3-methoxyphenyl)-*N*-phenylprop-2(*E*)-enamide (50 mg, 0.2 mmol) and hydrazine hydrate (0.1 mL, 2 mmol) were refluxed in ethanol (2 mL) for 16 h. When the reaction was completed the mixture was

poured into ice and acidified with concentrated hydrochloric acid. The water solution was extracted with ethyl acetate (4×6 mL); the organic layers were combined, dried over $MgSO_4$ and evaporated. The title compound was obtained as colorless crystals (21 mg, 39%) with a melting temperature of 402 K (129 °C). The compound is known and its spectral data correspond to those reported in literature [2]. Single crystals were obtained by slow evaporation from ethyl acetate.

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(3)	4*a*	0.306(4)	−0.172(2)	0.873(2)	0.061(8)
H(1)	4*a*	0.383(3)	0.425(2)	0.560(2)	0.041(7)
H(2A)	4*a*	0.4491	0.2042	0.5567	0.038
H(2B)	4*a*	0.2962	0.1425	0.5149	0.038
H(3A)	4*a*	0.2570	0.3018	0.6475	0.041
H(3B)	4*a*	0.1124	0.2262	0.6094	0.041
H(5)	4*a*	0.0517	0.0418	0.6754	0.038
H(6)	4*a*	0.1150	−0.1013	0.7726	0.036
H(9)	4*a*	0.5042	0.1851	0.7085	0.030
H(10A)	4*a*	0.7645	0.1256	0.7472	0.064
H(10B)	4*a*	0.8354	0.1064	0.8362	0.064
H(10C)	4*a*	0.6905	0.1967	0.8214	0.064
H(12)	4*a*	0.1496	0.4938	0.3931	0.043
H(13)	4*a*	0.1556	0.6706	0.3239	0.051
H(14)	4*a*	0.3418	0.8150	0.3603	0.057
H(15)	4*a*	0.5315	0.7811	0.4657	0.059
H(16)	4*a*	0.5359	0.6011	0.5323	0.046

Experimental details

Hydrogen atoms at the amino and hydroxy group were located in a difference Fourier map and freely refined. The C-bound hydrogen atoms were positioned geometrically with C—H distances ranging from 0.93 to 0.97 Å and refined as riding on their parent atoms with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl groups and $U_{iso}(H) = 1.2U_{eq}(C)$ for other H atoms.

Discussion

3-Aryl-*N*-arylpropanamides rise interest both due to synthetic applications and wide range of biological activity (e. g., they act as antitubercular [3] and antibiotic [4] agents, as well as HCA2 receptor agonists [5]). These compounds serve as starting materials for synthesis of 3-arylpropanols [6], 1,3-diarylpropan-1-ones [7], quinolines [8], β-l lactams [9], 4-aryl-2-quinolinones [10] and 1-indanones [11], as well as ligands in iridium complexes [12]. Despite the wide application of these compounds only few structures have been analyzed by X-ray diffraction method: the closest examples include 1,3-di-(3-phenylpropanoylamino)benzene [13], *N*-ethyl-3-phenyl-*N*-[2-[(3-phenyl-5-isoxazolyl)methylamino] phenyl]propanamide [14] and 3-aryl-*N*-phenylpropanamides containing nitrogen heterocycle in *para*-position of dihydrocinnamoyl moiety [15]. However this is the first example, of a crystal structure of dihydroferulic acid anilide. The title compound is achiral, but it crystallizes in chiral space group ($P2_1\,2_1\,2_1$). Secondary amino group participates in bifurcated hydrogen bond, where acceptors are oxygen atoms of hydroxy and methoxy

Table 3: Fractional atomic coordinate and displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4*a*	0.2809(2)	0.2988(1)	0.40942(8)	0.0417(9)	0.0282(7)	0.0199(7)	−0.0032(6)	−0.0044(6)	−0.0007(5)
O(2)	4*a*	0.6158(2)	0.0323(1)	0.81828(8)	0.0290(7)	0.0354(7)	0.0292(7)	−0.0038(7)	−0.0069(6)	0.0073(6)
O(3)	4*a*	0.3950(2)	−0.1221(1)	0.85765(9)	0.0400(9)	0.0292(7)	0.0338(8)	−0.0033(7)	−0.0066(7)	0.0114(7)
N(1)	4*a*	0.3473(3)	0.4219(1)	0.5129(1)	0.056(1)	0.0267(9)	0.0226(9)	−0.0064(8)	−0.0131(9)	0.0006(7)
C(1)	4*a*	0.3165(3)	0.3147(2)	0.4827(1)	0.030(1)	0.026(1)	0.026(1)	−0.0009(9)	−0.0018(9)	0.0004(8)
C(2)	4*a*	0.3319(3)	0.2137(2)	0.5425(1)	0.041(1)	0.027(1)	0.027(1)	−0.000(1)	−0.005(1)	0.0016(8)
C(3)	4*a*	0.2309(3)	0.2269(2)	0.6223(1)	0.030(1)	0.038(1)	0.034(1)	0.002(1)	−0.0039(9)	0.0115(9)
C(4)	4*a*	0.2687(3)	0.1295(2)	0.6829(1)	0.030(1)	0.030(1)	0.023(1)	0.0035(9)	0.0019(9)	0.0020(8)
C(5)	4*a*	0.1551(3)	0.0429(2)	0.7017(1)	0.029(1)	0.038(1)	0.028(1)	−0.001(1)	−0.0054(9)	0.0039(9)
C(6)	4*a*	0.1933(3)	−0.0437(2)	0.7600(1)	0.032(1)	0.027(1)	0.030(1)	−0.0046(9)	0.0005(9)	0.0020(9)
C(7)	4*a*	0.3469(3)	−0.0433(2)	0.7986(1)	0.035(1)	0.0218(9)	0.0211(9)	0.003(1)	−0.0014(8)	0.0022(8)
C(8)	4*a*	0.4654(2)	0.0423(2)	0.7779(1)	0.027(1)	0.026(1)	0.0200(9)	−0.0004(9)	−0.0022(8)	−0.0016(8)
C(9)	4*a*	0.4257(2)	0.1277(2)	0.7214(1)	0.029(1)	0.0247(9)	0.022(1)	−0.0009(9)	0.0032(8)	0.0017(8)
C(10)	4*a*	0.7364(3)	0.1227(2)	0.8047(1)	0.033(1)	0.051(1)	0.042(1)	−0.010(1)	−0.009(1)	0.009(1)
C(11)	4*a*	0.3436(3)	0.5294(2)	0.4688(1)	0.042(1)	0.025(1)	0.023(1)	−0.003(1)	0.0003(9)	0.0010(8)
C(12)	4*a*	0.2282(3)	0.5506(2)	0.4071(1)	0.044(1)	0.028(1)	0.034(1)	−0.002(1)	−0.006(1)	−0.0009(9)
C(13)	4*a*	0.2310(3)	0.6572(2)	0.3665(1)	0.054(2)	0.034(1)	0.040(1)	0.005(1)	−0.011(1)	0.0070(9)
C(14)	4*a*	0.3425(4)	0.7434(2)	0.3877(1)	0.065(2)	0.031(1)	0.046(1)	−0.004(1)	−0.001(1)	0.012(1)
C(15)	4*a*	0.4562(4)	0.7228(2)	0.4505(1)	0.068(2)	0.036(1)	0.043(1)	−0.019(1)	−0.002(1)	0.004(1)
C(16)	4*a*	0.4583(3)	0.6154(2)	0.4908(1)	0.051(1)	0.037(1)	0.029(1)	−0.010(1)	−0.006(1)	0.002(1)

groups (N—H···O type, $d_{D\cdots A} = 2.984(3)$ and 3.031(2) Å, respectively). Meanwhile the hydroxy group is the donor of O—H···O type hydrogen bond ($d_{D\cdots A} = 2.597(2)$ Å), where acceptor is oxygen atom of carbonyl group. By means of these hydrogen bonds the three-dimensional molecular networks are formed (lower part of the figure showing one molecule with two adjacent ones). The 4-hydroxy-3-methoxyphenyl fragment is almost planar.

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