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Crystal structure of 1,2-bis(4-methoxyphenyl)-2-((3-(trifluoromethyl)phenyl)amino)ethan-1-one, $C_{23}H_{20}F_3NO_3$

DOI 10.1515/ncrs-2016-0130

Received June 1, 2016; accepted August 22, 2016; available online September 9, 2016

Abstract

$C_{23}H_{20}F_3NO_3$, monoclinic, $C2/c$ (no. 15), $a = 48.3041(18)$ Å, $b = 6.0540(2)$ Å, $c = 28.5445(11)$ Å, $\beta = 105.519(2)^\circ$, $V = 8043.0(5)$ Å³, $Z = 16$, $R_{\text{gt}}(F) = 0.056$, $wR_{\text{ref}}(F^2) = 0.140$, $T = 293(2)$ K.

CCDC no.: 1482601

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The asymmetric unit, containing two crystallographically independent molecules, 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:	Yellow blocks
Wavelength:	Size $0.32 \times 0.30 \times 0.28$ mm Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.1 cm^{-1}
Diffraction, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{\text{max}}$, completeness:	55° , $>99\%$
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	150225, 9206, 0.063
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 6485
$N(\text{param})_{\text{refined}}$:	553
Programs:	SHELX [13], Bruker programs [14]

Source of material

A mixture of 2-hydroxy-1,2-bis(4-methoxyphenyl)ethanone (2.72 g, 0.01 mol), and 3-(trifluoromethyl)aniline (1.23 g, 0.01 mol), in toluene (50 mL) in the presence of conc.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
F1A	0.40735(3)	0.7377(3)	0.68791(7)	0.1048(7)
F2A	0.40077(3)	0.7598(3)	0.75785(6)	0.0878(5)
F3A	0.38748(3)	1.0215(3)	0.70875(7)	0.0833(5)
F1B	0.36085(3)	0.3083(3)	0.53939(7)	0.0946(6)
F2B	0.38413(4)	0.0827(3)	0.59078(6)	0.0901(5)
F3B	0.38329(3)	0.0478(3)	0.51697(6)	0.0972(6)
O1A	0.24565(3)	1.0526(3)	0.71442(6)	0.0602(4)
O2A	0.20536(4)	0.8344(4)	0.47251(6)	0.0798(6)
O3A	0.12347(3)	0.6008(3)	0.67964(6)	0.0555(4)
N1A	0.28518(4)	0.8101(3)	0.69215(7)	0.0495(5)
C1A	0.33570(4)	0.8007(3)	0.70016(7)	0.0401(4)
H1AA	0.3376	0.9376	0.7155	0.048*
C2A	0.35973(4)	0.6954(3)	0.69304(7)	0.0426(4)
C3A	0.35753(5)	0.4903(4)	0.67110(8)	0.0526(5)
H3AA	0.3737	0.4191	0.6668	0.063*
C4A	0.33096(5)	0.3943(4)	0.65585(9)	0.0583(6)
H4AA	0.3292	0.2563	0.6410	0.070*
C5A	0.30670(5)	0.4972(4)	0.66197(8)	0.0511(5)
H5AA	0.2889	0.4289	0.6509	0.061*
C6A	0.30868(4)	0.7032(3)	0.68460(7)	0.0400(4)
C7A	0.25617(4)	0.7287(3)	0.67538(7)	0.0412(4)
H7AA	0.2555	0.5748	0.6857	0.049*
C8A	0.23708(4)	0.8708(3)	0.69862(7)	0.0416(4)
C9A	0.20795(4)	0.7920(3)	0.69717(6)	0.0375(4)
C10A	0.18835(4)	0.9418(3)	0.70635(7)	0.0435(5)
H10A	0.1945	1.0842	0.7160	0.052*
C11A	0.16000(4)	0.8869(3)	0.70160(7)	0.0450(5)
H11A	0.1473	0.9906	0.7080	0.054*
C12A	0.15075(4)	0.6747(3)	0.68710(7)	0.0406(4)
C13A	0.17024(4)	0.5195(3)	0.67952(8)	0.0465(5)
H13A	0.1642	0.3760	0.6707	0.056*
C14A	0.19844(4)	0.5762(3)	0.68494(7)	0.0438(5)
H14A	0.2114	0.4698	0.6804	0.053*
C15A	0.24412(4)	0.7440(3)	0.62012(7)	0.0402(4)
C16A	0.22872(5)	0.5750(4)	0.59318(8)	0.0514(5)
H16A	0.2271	0.4417	0.6084	0.062*
C17A	0.21546(5)	0.5972(4)	0.54364(8)	0.0573(6)
H17A	0.2049	0.4810	0.5263	0.069*
C18A	0.21815(5)	0.7914(4)	0.52068(8)	0.0548(6)
C19A	0.23451(5)	0.9600(4)	0.54663(9)	0.0589(6)
H19A	0.2370	1.0901	0.5309	0.071*
C20A	0.24719(5)	0.9368(4)	0.59575(8)	0.0517(5)
H20A	0.2580	1.0525	0.6128	0.062*
C21A	0.38842(5)	0.8024(4)	0.71106(8)	0.0527(5)
C22A	0.10187(5)	0.7518(4)	0.68533(9)	0.0586(6)
H22A	0.0836	0.6779	0.6781	0.088*
H22B	0.1008	0.8740	0.6635	0.088*
H22C	0.1067	0.8048	0.7182	0.088*
C23A	0.18596(7)	0.6756(7)	0.44595(10)	0.1041(12)
H23A	0.1779	0.7279	0.4134	0.156*
H23B	0.1708	0.6518	0.4614	0.156*
H23C	0.1959	0.5393	0.4450	0.156*
O1B	0.52837(3)	0.1381(3)	0.52035(6)	0.0573(4)
O2B	0.64609(3)	0.6569(3)	0.58820(6)	0.0583(4)
O3B	0.53620(4)	0.1301(3)	0.76564(5)	0.0668(5)

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
N1B	0.48367(3)	0.3352(3)	0.54094(6)	0.0400(4)
C1B	0.43535(4)	0.2680(3)	0.54693(6)	0.0367(4)
H1BA	0.4351	0.1326	0.5315	0.044*
C2B	0.41119(4)	0.3383(3)	0.56005(7)	0.0395(4)
C3B	0.41101(4)	0.5378(4)	0.58372(7)	0.0475(5)
H3BA	0.3947	0.5846	0.5923	0.057*
C4B	0.43549(5)	0.6651(4)	0.59421(8)	0.0519(5)
H4BA	0.4357	0.7988	0.6103	0.062*
C5B	0.45960(4)	0.5975(3)	0.58121(7)	0.0455(5)
H5BA	0.4759	0.6859	0.5888	0.055*
C6B	0.45992(4)	0.3985(3)	0.55674(6)	0.0354(4)
C7B	0.51215(4)	0.4050(3)	0.56808(6)	0.0353(4)
H7BA	0.5133	0.5662	0.5661	0.042*
C8B	0.53439(4)	0.3037(3)	0.54508(7)	0.0375(4)
C9B	0.56347(4)	0.4023(3)	0.55641(6)	0.0335(4)
C10B	0.58559(4)	0.2776(3)	0.54733(7)	0.0409(4)
H10B	0.5815	0.1374	0.5340	0.049*
C11B	0.61341(4)	0.3546(4)	0.55743(7)	0.0458(5)
H11B	0.6278	0.2671	0.5512	0.055*
C12B	0.61959(4)	0.5633(3)	0.57691(7)	0.0407(4)
C13B	0.59793(4)	0.6922(3)	0.58617(7)	0.0412(4)
H13B	0.6021	0.8326	0.5994	0.049*
C14B	0.57017(4)	0.6134(3)	0.57585(7)	0.0376(4)
H14B	0.5558	0.7018	0.5819	0.045*
C15B	0.51966(4)	0.3380(3)	0.62178(6)	0.0350(4)
C16B	0.53133(4)	0.4831(3)	0.65889(7)	0.0424(4)
H16B	0.5355	0.6265	0.6512	0.051*
C17B	0.53712(4)	0.4222(4)	0.70746(7)	0.0470(5)
H17B	0.5449	0.5243	0.7318	0.056*
C18B	0.53134(4)	0.2104(4)	0.71944(7)	0.0459(5)
C19B	0.51970(5)	0.0623(4)	0.68267(8)	0.0543(5)
H19B	0.5156	−0.0810	0.6904	0.065*
C20B	0.51411(5)	0.1245(3)	0.63470(8)	0.0484(5)
H20B	0.5065	0.0218	0.6105	0.058*
C21B	0.38527(4)	0.1951(4)	0.55145(8)	0.0497(5)
C22B	0.66912(5)	0.5357(5)	0.57837(9)	0.0705(7)
H22D	0.6865	0.6199	0.5887	0.106*
H22E	0.6650	0.5073	0.5441	0.106*
H22F	0.6714	0.3981	0.5957	0.106*
C23B	0.54729(6)	0.2778(6)	0.80505(8)	0.0816(9)
H23D	0.5491	0.2019	0.8352	0.122*
H23E	0.5344	0.4007	0.8028	0.122*
H23F	0.5658	0.3303	0.8037	0.122*
H1NB	0.4833(5)	0.209(4)	0.5286(8)	0.049(6)*
H1NA	0.2869(5)	0.920(4)	0.7082(8)	0.056(7)*

hydrochloric acid (6–8 drops) was heated under reflux for 6 h, then cooled to room temperature. To the obtained solution, a catalytic amount of pyridine (1.5 mL) was added portion wise. The reaction mixture was heated under reflux to precipitate the crude product. The solvent was evaporated under reduced pressure and the residue was recrystallized from methanol to give the title compound with a yield of 95%, m.p. = 97–99 °C.

Experimental details

Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with $U_{iso}(H)$ set to $1.2U_{eq}(C)$. The H atoms of the methyl group were allowed to rotate with a fixed angle around the C—C bond to best fit the experimental electron density with $U_{iso}(H)$ set to $1.5U_{eq}(C)$.

Discussion

The functionalization of the carboxamide fragment is one of the most efficient approaches to obtain potentially bioactive substances [1, 2]. For some substituted carboxamides their lipid-lowering effects were studied in hyperlipidemic rats [3]. Also, several carboxamide derivatives are known to show antimicrobial, antiviral and antitumor activity [4–8]. On the other hand, some of heterocyclic derivatives containing a carboxamide moiety possess wide-spread use because of their anti-microbial [9], anti-arrhythmic, serotonin antagonist and anti-anxiety activities [10] and monoamino oxidase inhibitors activities [11] and are also well known as expected potential pharmaceutical carriers for catatonic agents [12].

The unit cell of $C_{23}H_{20}F_3NO_3$ contains two independent molecules, the phenyl ring (C1–C6/C1A–C6A) was nearly parallel to phenyl ring (C9–C14/C9A–C14A) in both molecules with dihedral angles between the two planes equal to 11.64° and 10.48° , respectively. On other hand the phenyl ring (C1–C6/C1A–C6A) makes dihedral angles with (C15–C20/C15A–C20A) 82.65° and 87.95° , respectively. The molecules were arranged in the crystal structure via one classical intermolecular hydrogen bond $N1B \cdots H1NB \cdots O1B^i$. The $D \cdots A$ distance is $2.50(2)$ Å and the angle is $167.0(2)^\circ$ with symmetry code: (i) $-x + 1, -y, -z + 1$.

Acknowledgements: The project was financially supported by King Saud University, Vice Deanship of Research Chairs.

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