Crystal structure of (2*E*)-2-[3-(1*H*-imidazol-1-yl)-1-phenylpropylidene]-hydrazinecarboxamide monohydrate, $C_{13}H_{17}N_5O_2$

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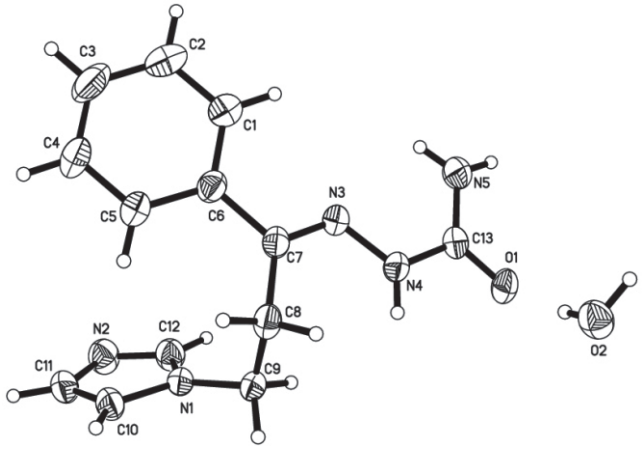
Received January 23, 2015, accepted June 25, 2015, available online June 30, 2015, CCDC no. 1267/4347



Abstract

$C_{13}H_{17}N_5O_2$, monoclinic, $P2_1/c$ (no. 14), a = 11.1953(3) Å, b = 5.5215(2) Å, c = 22.6998(5) Å, β = 99.929(2)°, V = 1382.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.0373, $wR_{ref}(F^2)$ = 0.1104, T = 296 K.

Table 1. Data collection and handling.

Crystal:	colourless plates, size 0.06×0.31×0.92 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	7.68 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	143.87°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2594, 2594
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2260
$N(param)_{refined}$:	204
Programs:	SHELX [1]

Source of material

A solution containing 3-(1*H*-imidazol-1-yl)-1-phenylpropan-1-one [2] (0.86 g, 4.3 mmol), semicarbazide hydrochloride (0.32 g, 4.3 mmol), and anhydrous sodium acetate (0.35 g, 4.3 mmol) in absolute ethanol (40 mL) was stirred at ambient temperature for 18 h. The reaction mixture was filtered and the filtrate was evaporated under vacuum. The residue was crystallized from ethanol to give 0.44 g (40%) of the title compound as colourless crystals (**m.p.**: 412-414 K), which were suitable for single crystal x-ray analysis.

Discussion

Epilepsy is the most prevalent neurological disorder, affecting about 50 million people worldwide [3]. Although there are recent significant advances in pharmacotherapy of epilepsy, nearly 20-30% of epileptic patients cannot be adequately controlled by the clinically available anti-epileptic drugs [4]. Therefore, there is an essential demand for more effective and safer new anti-epileptics. The title compound displayed 50% seizure inhibition at a dose level of 972 μmol/kg in animal model mice using subcutaneous pentylenetetrazole (scPTZ) convulsive screen without any neurotoxicity [5]. The crystal structure of title compound contains one title molecule and one additional water molecule in the asymmetric unit. The six-membered ring (C1–C6) is nearly planar. The dihedral angles between the six-membered ring and imidazole ring (N1/C10/C11/N2/C12) is 61.68 (5)°. All geometric parameters are in the expected ranges and close to the values reported for a related structure [6]. The molecules packing in the crystal structure is stabilized by four intermolecular hydrogen bonds, of which O1, O2 and N2 work as hydrogen bond acceptors and N4, N5, and O2 work as hydrogen bond donors. The distance of the interactions between N4–H1N4···O1 is 2.181(16) Å, N5–H2N5···O2 is 2.068(17) Å, O2–H2OW···N2 is 1.99(2) Å, and O2–H1OW···O1 is 2.036 Å and the angles are 170.1(15)°, 173.0(16)°, 169(2)°, 173.11(21)°, respectively.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	0.5395	–0.0175	0.2836	0.065
H(2A)	4*e*	0.3805	–0.1751	0.3232	0.084
H(3A)	4*e*	0.3379	–0.0357	0.4130	0.086
H(4A)	4*e*	0.4561	0.2654	0.4633	0.091
H(5A)	4*e*	0.6137	0.4300	0.4237	0.073
H(8A)	4*e*	0.7009	0.6489	0.3633	0.047
H(8B)	4*e*	0.7781	0.6402	0.3121	0.047
H(9A)	4*e*	0.9332	0.4332	0.3790	0.049
H(9B)	4*e*	0.9132	0.7014	0.3977	0.049
H(10A)	4*e*	0.7866	0.7498	0.4922	0.055
H(11A)	4*e*	0.7799	0.4734	0.5754	0.060
H(12A)	4*e*	0.9105	0.1116	0.4541	0.054
H(1N4)	4*e*	0.888(1)	0.360(3)	0.2761(7)	0.046(4)
H(1N5)	4*e*	0.747(2)	–0.114(3)	0.2165(8)	0.056(5)
H(2N5)	4*e*	0.841(2)	–0.184(3)	0.1776(8)	0.059(5)
H(1OW)	4*e*	0.946(2)	0.456(4)	0.1563(9)	0.076(6)
H(2OW)	4*e*	0.902(2)	0.503(4)	0.0980(9)	0.071(6)

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4*e*	0.97783(8)	0.1484(2)	0.20599(4)	0.0467(5)	0.0461(5)	0.0440(5)	0.0037(4)	0.0215(4)	0.0002(4)
N(1)	4*e*	0.85659(9)	0.4586(2)	0.45199(4)	0.0418(6)	0.0375(6)	0.0349(5)	–0.0071(4)	0.0135(4)	–0.0043(4)
N(2)	4*e*	0.8497(1)	0.2111(2)	0.52756(5)	0.0668(8)	0.0497(7)	0.0428(6)	–0.0020(5)	0.0147(5)	0.0062(5)
N(3)	4*e*	0.74261(9)	0.1755(2)	0.28808(4)	0.0368(5)	0.0450(6)	0.0352(5)	–0.0025(4)	0.0116(4)	–0.0021(4)
N(4)	4*e*	0.8415(1)	0.2463(2)	0.26399(5)	0.0390(6)	0.0428(6)	0.0373(6)	–0.0048(5)	0.0157(4)	–0.0061(4)
N(5)	4*e*	0.8092(1)	–0.0833(2)	0.20066(5)	0.0495(7)	0.0457(6)	0.0431(6)	–0.0004(5)	0.0139(5)	–0.0085(5)
C(1)	4*e*	0.5238(1)	0.0404(3)	0.32000(7)	0.0443(8)	0.065(1)	0.0542(8)	–0.0080(6)	0.0115(6)	–0.0074(7)
C(2)	4*e*	0.4281(2)	–0.0538(4)	0.34372(9)	0.0475(9)	0.079(1)	0.084(1)	–0.0219(8)	0.0126(8)	–0.0048(9)
C(3)	4*e*	0.4023(2)	0.0291(4)	0.39724(9)	0.0431(8)	0.098(1)	0.079(1)	–0.0141(8)	0.0240(8)	0.012(1)
C(4)	4*e*	0.4726(2)	0.2085(4)	0.42699(9)	0.054(1)	0.118(2)	0.063(1)	–0.017(1)	0.0317(8)	–0.011(1)
C(5)	4*e*	0.5680(1)	0.3057(4)	0.40340(7)	0.0463(8)	0.086(1)	0.0555(9)	–0.0159(7)	0.0235(7)	–0.0172(8)
C(6)	4*e*	0.5969(1)	0.2213(2)	0.34999(6)	0.0327(6)	0.0484(7)	0.0408(7)	0.0010(5)	0.0095(5)	0.0021(5)
C(7)	4*e*	0.7041(1)	0.3135(2)	0.32648(5)	0.0350(6)	0.0423(7)	0.0320(6)	0.0024(5)	0.0091(4)	0.0013(5)
C(8)	4*e*	0.7603(1)	0.5542(2)	0.34683(6)	0.0470(7)	0.0355(6)	0.0391(6)	0.0020(5)	0.0178(5)	0.0028(5)
C(9)	4*e*	0.8765(1)	0.5418(2)	0.39341(5)	0.0466(7)	0.0428(7)	0.0367(6)	–0.0120(5)	0.0181(5)	–0.0027(5)
C(10)	4*e*	0.8106(1)	0.5884(3)	0.49445(6)	0.0585(8)	0.0400(7)	0.0425(7)	–0.0067(6)	0.0203(6)	–0.0085(5)
C(11)	4*e*	0.8073(1)	0.4338(3)	0.54016(6)	0.0621(9)	0.0544(8)	0.0359(7)	–0.0121(7)	0.0173(6)	–0.0068(6)
C(12)	4*e*	0.8784(1)	0.2357(2)	0.47422(6)	0.0527(8)	0.0394(7)	0.0452(7)	0.0002(5)	0.0146(6)	–0.0011(5)
C(13)	4*e*	0.8809(1)	0.0993(2)	0.22271(5)	0.0403(6)	0.0389(6)	0.0301(6)	0.0060(5)	0.0089(5)	0.0027(4)
O(2)	4*e*	0.9317(1)	0.5707(2)	0.13245(5)	0.0684(7)	0.0409(5)	0.0463(6)	0.0011(5)	0.0118(5)	–0.0035(5)

Acknowledgments. The authors would like to extend their sincere appreciation to the Deanship of Scientific Research at King Saud University for its funding of this research through the Research Group Project no. RGP-VPP-196.

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