Crystal structure of (2*E*)-2-[1-(4-bromophenyl)-3-(1*H*-imidazol-1-yl) propylidene]-*N*-(2,4-dichlorophenyl)hydrazinecarboxamide, $C_{19}H_{16}BrCl_2N_5O$

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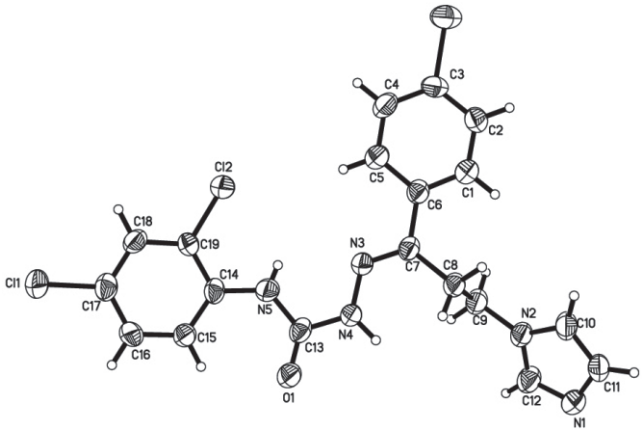

Abstract

$C_{19}H_{16}BrCl_2N_5O$, triclinic, $P\overline{1}$ (no. 2), a = 7.4661(3) Å, b = 10.9874(4) Å, c = 12.8619(5) Å, α = 109.979(3)°, β = 92.053(3)°, γ = 95.677(3)°, V = 984.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.0653, $wR_{ref}(F^2)$ = 0.1906, T = 296 K.

Table 1. Data collection and handling.

Crystal:	colourless plates, size 0.06×0.32×0.34 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	55.22 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	139.36°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12681, 3565
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2032
$N(param)_{refined}$:	262
Programs:	SHELX [1]

Source of material

A solution of *N*-(2,4-dichlorophenyl)hydrazinecarboxamide [2] (2.2 g, 10 mmol), 1-(4-bromophenyl)-3-(1*H*-imidazol-1-yl)-propan-1-one [3] (2.79 g, 10 mmol) and few drops of glacial acetic acid in ethanol (15 mL) was stirred at ambient temperature for 18 h. The reaction mixture was evaporated under reduced pressure and the residue was crystallized from ethanol to give 2.84 g (59%) of the title compound as colourless crystals which were suitable for single crystal x-ray analysis. **m.p.** 494-496 K.

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Discussion

Epilepsy is one of the most common disorders of the brain, affecting more than 50 million individuals worldwide [4]. However, only about 70% of patients suffering from epilepsy are adequately controlled by the currently available anti-epileptic drugs (AEDs) [5]. Therefore, the search for more effective and safer new AEDs should have a considerable attention. The title compound showed 16 and 50% seizure inhibition in animal model mice using maximal electroshock (MES) and subcutaneous pentylenetetrazole (scPTZ) convulsive screens, respectively, at a dose level of 520 μmol/kg [6]. X-ray crystallography is a decisive analytical tool which can confirm the configuration of the title compound. Fortunately, we have succeeded to get single crystals of the title compound which were suitable for x-ray crystallography. The crystal structure of the target compound contains one molecule in the asymmetric unit. The two six-membered rings (C1-C6 and C14-C19) are nearly planner. The dihedral angle between the two six-membered rings is 8.7(10)°. The dihedral angles between the six-membered rings (C1-C6 and C14-C19) and the imidazole ring (N1/N2/C10-C12) are 44.62(13)° and 48.82(13)°, respectively. The packing in the crystal structure is stabilized by two inter-molecular hydrogen bonds, of which N1 works as hydrogen bond acceptor and N4 and C4 work as hydrogen bond donors. The distance of the interactions between N46–H1N4···N1 is 2.12(6) Å and C8–H8A···N1 is 2.53(1) Å and the angles are 165.00(6)° and 141.00(1)°, respectively.

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

Atom	Site	x	y	z	U_{iso}
H(1A)	2*i*	–0.0852	0.6360	0.4412	0.072
H(2A)	2*i*	–0.3586	0.5156	0.4346	0.072
H(4A)	2*i*	–0.1839	0.2122	0.2017	0.076
H(5A)	2*i*	0.0863	0.3349	0.2099	0.074
H(8A)	2*i*	0.3816	0.7223	0.4400	0.063
H(8B)	2*i*	0.1964	0.7023	0.4894	0.063
H(9A)	2*i*	0.2496	0.8309	0.3336	0.069
H(9B)	2*i*	0.0607	0.8064	0.3774	0.069
H(10A)	2*i*	0.1244	0.9068	0.6242	0.084
H(11A)	2*i*	0.2434	1.1294	0.7331	0.076
H(12A)	2*i*	0.3621	1.0683	0.4289	0.078
H(15A)	2*i*	0.8326	0.4468	0.0186	0.065
H(16A)	2*i*	0.9820	0.2830	–0.0914	0.069
H(18A)	2*i*	0.5473	0.0247	–0.1190	0.069
H(1N4)	2*i*	0.502(8)	0.685(6)	0.299(5)	0.06(2)
H(1N5)	2*i*	0.425(8)	0.404(5)	0.104(4)	0.04(2)

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}
Br(1)	2*i*	–0.5265(1)	0.25364(7)	0.30556(6)	0.0710(6)	0.0736(5)	0.0778(5)	–0.0135(4)	0.0033(4)	0.0327(3)
Cl(1)	2*i*	0.8942(2)	0.0103(1)	–0.1929(1)	0.077(1)	0.0582(8)	0.0747(9)	0.0120(8)	0.0302(9)	0.0056(7)
Cl(2)	2*i*	0.2972(2)	0.1709(1)	0.0021(1)	0.064(1)	0.0547(8)	0.0692(8)	–0.0033(7)	0.0200(8)	0.0101(6)
O(1)	2*i*	0.7224(6)	0.6088(3)	0.1615(3)	0.075(3)	0.049(2)	0.063(2)	–0.003(2)	0.022(2)	0.010(2)
N(1)	2*i*	0.3407(8)	1.1465(4)	0.5905(4)	0.071(4)	0.046(2)	0.069(3)	–0.002(2)	0.015(3)	0.007(2)
N(2)	2*i*	0.2296(7)	0.9438(4)	0.4905(4)	0.067(4)	0.042(2)	0.051(2)	0.004(2)	0.013(2)	0.011(2)
N(3)	2*i*	0.3036(7)	0.5373(4)	0.2486(3)	0.054(3)	0.044(2)	0.046(2)	0.001(2)	0.011(2)	0.010(2)
N(4)	2*i*	0.4630(7)	0.6069(4)	0.2448(4)	0.058(4)	0.041(2)	0.055(2)	–0.002(2)	0.018(3)	0.006(2)
N(5)	2*i*	0.5109(9)	0.4294(4)	0.0944(4)	0.066(4)	0.046(2)	0.056(3)	–0.002(3)	0.018(3)	0.016(2)
C(1)	2*i*	–0.1058(9)	0.5507(5)	0.3917(5)	0.065(5)	0.047(3)	0.061(3)	0.002(3)	0.013(3)	0.010(2)
C(2)	2*i*	–0.2704(9)	0.4791(5)	0.3883(5)	0.066(5)	0.056(3)	0.058(3)	0.009(3)	0.021(3)	0.019(3)
C(3)	2*i*	–0.3013(8)	0.3523(5)	0.3147(5)	0.048(4)	0.059(3)	0.055(3)	–0.008(3)	0.001(3)	0.023(3)
C(4)	2*i*	–0.165(1)	0.2986(5)	0.2493(5)	0.081(5)	0.041(3)	0.058(3)	–0.001(3)	0.008(3)	0.006(2)
C(5)	2*i*	–0.0035(9)	0.3720(5)	0.2544(5)	0.072(5)	0.046(3)	0.059(3)	–0.001(3)	0.014(3)	0.010(2)
C(6)	2*i*	0.0282(8)	0.5013(5)	0.3252(4)	0.061(4)	0.044(3)	0.042(2)	0.006(3)	0.009(3)	0.015(2)
C(7)	2*i*	0.2021(8)	0.5819(4)	0.3289(4)	0.057(4)	0.037(2)	0.044(2)	0.009(2)	0.007(3)	0.011(2)
C(8)	2*i*	0.2518(9)	0.7079(5)	0.4239(4)	0.066(4)	0.039(2)	0.049(3)	0.003(3)	0.018(3)	0.013(2)
C(9)	2*i*	0.1897(9)	0.8228(5)	0.3970(4)	0.075(5)	0.044(3)	0.049(3)	0.006(3)	0.015(3)	0.011(2)
C(10)	2*i*	0.188(1)	0.9662(5)	0.5978(5)	0.097(6)	0.046(3)	0.063(3)	0.005(3)	0.038(4)	0.011(2)
C(11)	2*i*	0.2541(9)	1.0890(5)	0.6576(5)	0.073(5)	0.052(3)	0.057(3)	0.008(3)	0.025(3)	0.006(2)
C(12)	2*i*	0.3191(9)	1.0559(5)	0.4919(5)	0.089(6)	0.045(3)	0.057(3)	0.002(3)	0.016(3)	0.014(2)
C(13)	2*i*	0.5783(9)	0.5512(5)	0.1649(4)	0.072(5)	0.037(3)	0.045(3)	0.006(3)	0.012(3)	0.014(2)
C(14)	2*i*	0.6033(8)	0.3372(5)	0.0219(4)	0.063(4)	0.045(3)	0.039(2)	0.013(3)	0.016(3)	0.014(2)
C(15)	2*i*	0.7756(9)	0.3617(5)	–0.0073(4)	0.060(4)	0.047(3)	0.050(3)	0.000(3)	0.017(3)	0.011(2)
C(16)	2*i*	0.8654(9)	0.2641(5)	–0.0736(4)	0.055(4)	0.063(3)	0.049(3)	0.002(3)	0.019(3)	0.014(2)
C(17)	2*i*	0.7815(9)	0.1376(5)	–0.1136(4)	0.070(5)	0.053(3)	0.049(3)	0.015(3)	0.017(3)	0.015(2)
C(18)	2*i*	0.6055(9)	0.1094(5)	–0.0904(4)	0.073(5)	0.040(3)	0.050(3)	–0.003(3)	0.015(3)	0.006(2)
C(19)	2*i*	0.5192(8)	0.2092(5)	–0.0242(4)	0.049(4)	0.048(3)	0.046(3)	0.006(3)	0.014(3)	0.013(2)

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