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Crystal structure of 1-(4-bromophenyl)-2-(2-methyl-4-nitro-1*H*-imidazol-1-yl)ethanone oxime, $C_{12}H_{11}BrN_4O_3$

Yong-De Liu[I], Lu-Bing Yan[I], Xin-Yi Wang[II] and Juan Sun[*, II]

[I] School of Chemistry and Chemical Engineering, Henan University of Technology, Zhengzhou 450001, Henan Province, P. R. China
[II] School of Life Sciences, Shandong University of Technology, ZiBo 255049, Shandong Province, P. R. China

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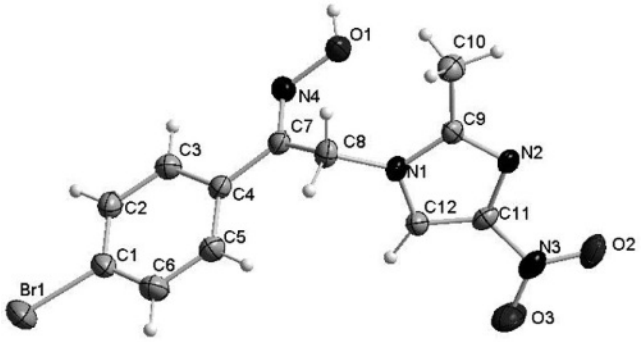

Abstract

$C_{12}H_{11}BrN_4O_3$, orthorhombic, *Pbca* (no. 61), $a = 8.2167(7)$ Å, $b = 11.419(1)$ Å, $c = 28.750(2)$ Å, $V = 2697.5$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0455$, $wR_{ref}(F^2) = 0.1054$, $T = 273$ K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.18×0.19×0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	30.61 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, ω
$2\theta_{max}$:	51.34°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	21490, 2542
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1542
$N(param)_{refined}$:	183
Programs:	SHELX [8]

Source of material

2-Bromo-1-(4-bromophenyl)ethanone (0.278 g, 1 mmol) and 2-methyl-4-nitro-1*H*-imidazole (0.127g, 1 mmol) were mixed in ethanol (20 ml). The mixture was stirred for a few minutes at 85 °C, in the catalytic conditions of K_2CO_3 and tetrabutylammoniumbromide (TBAB). The formed precipitate was filtered and purified by recrystallization obtaining a white powder. The white product was mixed with with six equivalents of hydroxylamine hydrochloride in the solution of ethanol, and two times K_2CO_3 as the catalyst was added. The mixture was stirred for 2 hours in the reflux conditions. Colourless block-shaped single crystals were obtained by slow evaporation of the solution in air.

Experimental details

H-atoms were positioned geometrically and refined using a riding model, with C–H = 0.93-0.97 Å, O–H = 0.82 Å, and with U_{iso}(H) set to 1.7U_{eq}(C) and 1.4U_{eq}(O).

Discussion

Nitroimidazoles are a common drugs that can be used against anaerobic bacterias. It derivatives are important pharmaceutical and nuclear materials intermediates [1–4]. In addition, nitroimidazoles have also been widely used as ligands in the preparation of complexes [5, 6]. In this paper, we introduced a new nitroimidazoles compound, and report on its antibacterial activity. In fact, it has good antibacterial activities to four common bacterias (*E. Coli*, *P. Aeruginosa*, *B. Subtilis*, *S. Aureus*), and the values of IC_{50} are 1.0-10 μg/ml. In the structure two molecules are connected to form chains along the *b* direction by intermolecular O–H···O hydrogen bonding interaction. There are also some weaker intramolecular hydrogen bonds involved in the composition of a spatial structure.The dihedral angle between benzene ring C1-C6 and imidazole ring N1-C12 is 68.1(2)°. All bond lengths in the molecules are within normal values [7].

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	8*c*	–0.0466	0.2135	0.5790	0.063
H(3A)	8*c*	–0.0470	0.3354	0.6428	0.058
H(5)	8*c*	0.2033	0.5734	0.5717	0.067
H(6)	8*c*	0.2048	0.4519	0.5075	0.077
H(8A)	8*c*	0.2776	0.6259	0.6880	0.056
H(8B)	8*c*	0.2735	0.6427	0.6340	0.056
H(10A)	8*c*	0.2453	0.8678	0.7669	0.084
H(10B)	8*c*	0.3591	0.7886	0.7364	0.084
H(10C)	8*c*	0.2068	0.7341	0.7611	0.084
H(12)	8*c*	–0.0082	0.7776	0.6087	0.052
H(1)	8*c*	0.0002	0.5396	0.7546	0.087

* Correspondence author (e-mail: sunreiyi86@163.com)

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)	8*c*	0.09494(7)	0.21495(5)	0.48709(2)	0.1155(5)	0.0754(4)	0.0496(3)	0.0092(3)	–0.0106(3)	–0.0168(2)
N(4)	8*c*	0.0046(4)	0.5039(3)	0.6924(1)	0.063(2)	0.044(2)	0.041(2)	0.009(2)	0.001(2)	–0.003(2)
O(2)	8*c*	–0.1254(4)	1.0925(3)	0.6588(1)	0.082(2)	0.043(2)	0.091(2)	0.017(2)	0.007(2)	0.007(2)
O(3)	8*c*	–0.1574(4)	0.9880(3)	0.5968(1)	0.100(3)	0.071(2)	0.074(2)	0.006(2)	–0.035(2)	0.016(2)
N(1)	8*c*	0.1274(4)	0.7540(2)	0.6678(1)	0.047(2)	0.030(2)	0.043(2)	–0.000(2)	0.000(2)	–0.000(1)
N(2)	8*c*	0.0675(4)	0.9267(2)	0.6980(1)	0.045(2)	0.029(2)	0.041(2)	–0.005(2)	0.002(2)	0.002(1)
N(3)	8*c*	–0.1040(4)	1.0036(3)	0.6361(1)	0.054(2)	0.042(2)	0.065(2)	–0.004(2)	0.003(2)	0.014(2)
C(1)	8*c*	0.0853(5)	0.3205(4)	0.5378(1)	0.066(3)	0.047(3)	0.041(2)	0.009(2)	–0.008(2)	–0.006(2)
C(2)	8*c*	0.0061(5)	0.2855(3)	0.5778(1)	0.066(3)	0.039(2)	0.053(2)	–0.002(2)	–0.008(2)	–0.001(2)
C(3)	8*c*	0.0060(5)	0.3588(3)	0.6158(1)	0.054(2)	0.048(3)	0.043(2)	–0.001(2)	0.001(2)	0.002(2)
C(4)	8*c*	0.0834(4)	0.4669(3)	0.6148(1)	0.043(2)	0.032(2)	0.044(2)	0.003(2)	–0.000(2)	0.003(2)
C(5)	8*c*	0.1546(5)	0.5000(4)	0.5737(1)	0.073(3)	0.045(3)	0.049(2)	–0.006(2)	0.005(2)	0.004(2)
C(6)	8*c*	0.1560(6)	0.4275(4)	0.5350(1)	0.084(3)	0.063(3)	0.045(2)	–0.005(3)	0.008(2)	0.002(2)
C(7)	8*c*	0.0901(4)	0.5399(3)	0.6575(1)	0.042(2)	0.033(2)	0.045(2)	0.005(2)	0.001(2)	0.002(2)
C(8)	8*c*	0.2064(5)	0.6400(3)	0.6617(1)	0.047(2)	0.036(2)	0.056(2)	0.003(2)	0.004(2)	–0.003(2)
C(9)	8*c*	0.1485(4)	0.8287(3)	0.7045(1)	0.041(2)	0.032(2)	0.039(2)	–0.009(2)	0.003(2)	0.001(2)
C(10)	8*c*	0.2487(6)	0.8025(3)	0.7459(1)	0.062(3)	0.049(2)	0.057(2)	–0.005(2)	–0.016(2)	0.002(2)
C(11)	8*c*	–0.0070(5)	0.9116(3)	0.6557(1)	0.042(2)	0.037(2)	0.046(2)	–0.004(2)	0.002(2)	0.012(2)
C(12)	8*c*	0.0282(5)	0.8072(3)	0.6370(1)	0.056(2)	0.037(2)	0.038(2)	–0.004(2)	–0.003(2)	–0.000(2)
O(1)	8*c*	0.0304(4)	0.5743(2)	0.73119(8)	0.083(2)	0.043(2)	0.049(2)	–0.005(2)	0.011(2)	–0.000(1)

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