Crystal structure of *N*-butyl-2-(3,5-dimethyl-1,1-dioxido-2*H*-1,2,6-thiadiazin-4-yl)benzamide, C₁₆H₂₁N₃O₃S

Nilay Bhatt^I, Pralav Bhatt^{II}, Thavendran Govender^{II}, Hendrik G. Kruger^{II} and Glenn E. M. Maguire^{*, III}

^I Chemistry Department, JJT University, Rajasthan, India

^{II} School of Pharmacy and Pharmacology, University of KwaZulu-Natal, Durban 4000, South Africa

^{III} School of Chemistry and Physics, University of KwaZulu-Natal, Durban 4000, South Africa

Received October 21, 2013, accepted January 14, 2014, available online January 28, 2014, CCDC no. 983713

Abstract

C₁₆H₂₁N₃O₃S, monoclinic, *P*2₁/*n* (no. 14), *a* = 7.7608(2) Å, *b* = 15.3483(4) Å, *c* = 13.6538(3) Å, β = 94.283(2)°, *V* = 1621.8 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0370, *wR*_{ref}(*F*²) = 0.1038, *T* = 173 K.

Source of material

2-(3,5-dimethyl-1,1-dioxido-2*H*-1,2,6-thiadiazin-4-yl)benzoic acid (0.017 mol) and carbonyldiimidazole (0.017 mol) in dry tetrahydrofuran (50 ml) was stirred for 30 mins at room temperature. To this solution *N*-butyl amine (0.021 mol) was slowly added and the contents stirred for 12 hrs at room temperature until the reaction was completed (TLC *R*_f = 0.8 using 80% ethyl acetate/hexane). The solvent was then completely evaporated and the residual mass was treated with 5% HCl (25 ml) and stirred for 1 hr.

The pale yellow product was filtered giving a 65% yield. **m.p.** = 466 K. Crystals suitable for X-ray analysis were grown in methanol/ethyl acetate at room temperature.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.19×0.21×0.21 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	2.18 cm ⁻¹
Diffractometer, scan mode:	Nonius Kappa-CCD diffractometer, φ and ω
2θ _{max} :	56.56°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	7916, 4033
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3257
<i>N</i> (<i>param</i>) _{refined} :	219
Programs:	SHELX [4], OLEX2 [5], DIAMOND [6]

Experimental details

All hydrogen atoms, except H1 and H3, were placed in idealised positions and refined in riding models with *U*_{iso} assigned the values to be 1.2 or 1.5 times *U*_{eq} of the atoms to which they are attached and the constraint distances of C–H ranging from 0.95 Å to 0.99 Å. The hydrogens H1 and H3 were located in the difference electron density maps and refined independently.

Discussion

The synthesis of 1,2,6-thiadiazine-1,1-dioxides derivatives was first reported using sulfamide with alpha and beta diketones [1]. We previously have reported the 3,5-dimethyl based structure with an aromatic ring at position 4 of the thiadiazine ring containing an ethyl ester function [2]. We have reported the synthesis of *N*-alkyl analogues of 1,2,6-thiadiazine-1,1-dioxides as potential antimicrobial agents [3]. Herein we report the crystal structure of a *N*-butyl analogues from the same class of 1,2,6-thiadiazine-1,1-dioxides. The sulfur atom deviates from the plane of the ring by 0.105 Å. There is a classic hydrogen bond for N1...O3' = 2.8134(16) Å and N3...O2'' = 2.9624(16) Å. These connections create a 2-dimensional, hydrogen bonded network.

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	4e	0.228(2)	0.160(1)	0.645(1)	0.041(5)
H(3)	4e	0.311(2)	0.356(1)	0.910(1)	0.037(5)
H(1A)	4e	0.4438	0.1611	0.7584	0.041
H(1B)	4e	0.5072	0.2501	0.8103	0.041
H(1C)	4e	0.5833	0.2208	0.7097	0.041

* Correspondence author (e-mail: maguireg@ukzn.ac.za)

Table 2. continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(5A)	4e	0.3059	0.5066	0.5572	0.050
H(5B)	4e	0.2257	0.5268	0.6594	0.050
H(5C)	4e	0.1009	0.5135	0.5619	0.050
H(7)	4e	0.6499	0.3792	0.6318	0.032
H(8)	4e	0.8777	0.4640	0.6988	0.034
H(9)	4e	0.8416	0.5514	0.8357	0.034
H(10)	4e	0.5770	0.5529	0.9066	0.030
H(13A)	4e	0.0086	0.4444	0.9418	0.036

Table 2. continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(13B)	4e	0.0991	0.3780	1.0201	0.036
H(14A)	4e	−0.1560	0.3213	0.9283	0.033
H(14B)	4e	−0.0486	0.3217	0.8328	0.033
H(15A)	4e	0.0452	0.2195	1.0038	0.042
H(15B)	4e	0.1448	0.2178	0.9052	0.042
H(16A)	4e	−0.2061	0.1632	0.9124	0.061
H(16B)	4e	−0.0467	0.0970	0.9135	0.061
H(16C)	4e	−0.0958	0.1553	0.8183	0.061

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
S(1)	4e	0.03548(4)	0.25573(2)	0.58470(2)	0.0198(2)	0.0215(2)	0.0247(2)	−0.0032(1)	−0.0019(1)	−0.0030(1)
O(1)	4e	−0.1051(1)	0.24416(7)	0.64496(9)	0.0280(6)	0.0368(6)	0.0443(7)	0.0028(5)	0.0108(5)	0.0056(5)
O(2)	4e	0.0189(1)	0.21265(7)	0.49148(8)	0.0358(6)	0.0352(6)	0.0293(6)	−0.0042(5)	−0.0064(5)	−0.0099(5)
O(3)	4e	0.2021(1)	0.53932(7)	0.85729(9)	0.0282(5)	0.0188(5)	0.0528(7)	0.0052(4)	0.0063(5)	0.0046(5)
N(1)	4e	0.2142(2)	0.21766(8)	0.64345(9)	0.0253(6)	0.0168(6)	0.0317(6)	−0.0011(5)	−0.0051(5)	−0.0044(5)
N(2)	4e	0.0716(2)	0.35709(8)	0.57024(9)	0.0270(6)	0.0220(6)	0.0319(6)	−0.0032(5)	−0.0091(5)	0.0009(5)
N(3)	4e	0.2361(2)	0.39791(8)	0.90247(9)	0.0265(6)	0.0181(6)	0.0291(6)	0.0017(5)	0.0028(5)	0.0014(5)
C(1)	4e	0.4803(2)	0.22115(9)	0.7471(1)	0.0277(7)	0.0207(7)	0.0333(8)	0.0023(5)	−0.0062(6)	−0.0013(6)
C(2)	4e	0.3375(2)	0.26922(9)	0.6903(1)	0.0206(6)	0.0206(6)	0.0218(6)	−0.0001(5)	0.0001(5)	−0.0034(5)
C(3)	4e	0.3312(2)	0.35852(9)	0.6826(1)	0.0194(6)	0.0190(6)	0.0240(6)	0.0002(5)	−0.0020(5)	−0.0036(5)
C(4)	4e	0.2006(2)	0.39859(9)	0.6183(1)	0.0231(7)	0.0197(6)	0.0255(7)	−0.0007(5)	−0.0019(5)	−0.0004(5)
C(5)	4e	0.2090(2)	0.4948(1)	0.5974(1)	0.0340(8)	0.0212(7)	0.0432(9)	−0.0032(6)	−0.0114(7)	0.0053(6)
C(6)	4e	0.4754(2)	0.41278(8)	0.7297(1)	0.0204(6)	0.0156(6)	0.0265(7)	0.0000(5)	−0.0037(5)	−0.0006(5)
C(7)	4e	0.6344(2)	0.41386(9)	0.6881(1)	0.0245(7)	0.0226(7)	0.0317(7)	0.0011(5)	0.0000(6)	−0.0039(6)
C(8)	4e	0.7700(2)	0.4647(1)	0.7275(1)	0.0200(6)	0.0264(7)	0.0391(8)	−0.0012(5)	0.0009(6)	0.0008(6)
C(9)	4e	0.7487(2)	0.51640(9)	0.8089(1)	0.0228(7)	0.0238(7)	0.0372(8)	−0.0050(5)	−0.0067(6)	0.0004(6)
C(10)	4e	0.5917(2)	0.51701(9)	0.8511(1)	0.0272(7)	0.0192(6)	0.0272(7)	−0.0017(5)	−0.0049(6)	−0.0024(5)
C(11)	4e	0.4552(2)	0.46515(8)	0.8126(1)	0.0213(6)	0.0155(6)	0.0246(6)	0.0012(5)	−0.0040(5)	0.0011(5)
C(12)	4e	0.2866(2)	0.47047(9)	0.8596(1)	0.0220(6)	0.0175(6)	0.0228(6)	0.0000(5)	−0.0041(5)	−0.0022(5)
C(13)	4e	0.0735(2)	0.3890(1)	0.9490(1)	0.0360(8)	0.0249(7)	0.0290(7)	−0.0016(6)	0.0101(6)	−0.0041(6)
C(14)	4e	−0.0386(2)	0.3155(1)	0.9052(1)	0.0274(7)	0.0252(7)	0.0303(7)	0.0004(6)	0.0040(6)	0.0012(6)
C(15)	4e	0.0301(2)	0.2252(1)	0.9314(1)	0.0341(8)	0.0261(8)	0.0442(9)	−0.0002(6)	−0.0032(7)	0.0029(7)
C(16)	4e	−0.0903(2)	0.1539(1)	0.8903(1)	0.0390(9)	0.0263(8)	0.057(1)	−0.0039(7)	0.0054(8)	−0.0030(7)

Acknowledgments. The authors wish to thank Dr. Hong Su from the University of Cape Town for assistance with the data collection and refinement. We thank the editor for providing the figure.

References

- Wright, J. B.: The reaction of Sulfamide with α - & β -diketones. The preparation of 1,2,5-thiadiazoles-1,1-dioxide & 1,2,6-thiadiazine-1,1-dioxides. *J. Org. Chem.* **29** (1964) 1905-1909.
- Bhatt, N.; Bhatt, P.; Vyas, K. B.; Nimavat, K.; Govendran, T.; Kruger, H. G.; Maguire, G. E. M.: 2-Ethyl-(3,5-dimethyl-1,1-dioxo-2*H*-1,2,6-thiadiazine-4-yl)-benzoic acid. *Acta Crystallogr.* **E68** (2012) o2160.
- Bhatt, N.; Nimavat, K.; Govendran, T.; Kruger, H. G.; Maguire, G. E. M.; Bhatt, P.: *N*-alkyl-2-(3,5)-dimethyl-1,1-dioxido-2*H*-1,2,6-thiadiazin-4-yl)benzamides as potential antimicrobial agents. *J. Pharm. Res.* **6** (2013) 355-360.
- Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr.* **A64** (2008) 112-122.
- Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: A complete structure solution, refinement and analysis program (2009). *J. Appl. Cryst.* **42** 339-341.
- Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Version 3.2i. Crystal Impact, Bonn, Germany 2012.