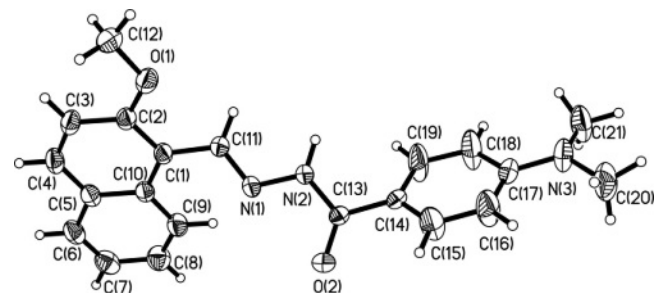


Crystal structure of *N'*-(2-methoxynaphthylidene)-4-dimethylamino benzohydrazide, C₂₁H₂₁N₃O₂

Shi-Yong Liu^{*,1} and Mei Zhang^{II}^I College of Chemistry & Pharmacy, Taizhou University, Taizhou 317000, Zhejiang Province, P. R. China^{II} Department of Chemistry, Liaoning Normal University, Dalian 116029, Liaoning Province, P. R. China

Received February 17, 2012, accepted July 19, 2012, available online October 05, 2012, CCDC no. 1267/3827



Abstract

C₂₁H₂₁N₃O₂, monoclinic, *P*2₁/*c* (no. 14), *a* = 5.0165(5) Å, *b* = 13.148(1) Å, *c* = 26.705(2) Å, β = 90.198(1)°, *V* = 1761.4 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0656, *wR*_{ref}(*F*²) = 0.1768, *T* = 298 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.15×0.17×0.17 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	0.86 cm ⁻¹
Diffractometer, scan mode:	Bruker SMART CCD area-detector, ω
2θ _{max} :	52.96°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	13359, 3614
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2193
<i>N</i> (<i>param</i>) _{refined} :	241
Programs:	SADABS [13], SMART, SAINT [14] SHELX [15]

Source of material

The compound was prepared by the condensation reaction of 2-methoxy-1-naphthaldehyde (1 mmol, 0.186 g) and 4-dimethylaminobenzohydrazide (1 mmol, 0.179 g) in anhydrous methanol (30 ml) at ambient temperature. Colourless block-shaped single crystals suitable for X-ray structural determination were obtained by slow evaporation of the solution for a week.

Experimental details

H(2) was located from a difference Fourier map and refined isotropically, with the N–H distance restrained to 0.90(1) Å. The remaining H atoms were positioned geometrically and constrained to ride on their parent atoms, with C–H distances of 0.93–0.96 Å, and with *U*_{iso}(H) = 1.2*U*_{eq}(C) and 1.5*U*_{eq}(C_{methyl}).

Discussion

Considerable attention has been focused on hydrazones and their medicinal applications [1–5]. The study on the crystal structures of such compounds is of particular interest [6–8]. As a continuation of our work on such compounds, we report herein the crystal structure of the title hydrazone derivative. The molecular struc-

ture of the title compound is shown in the figure. The dihedral angle between the C(1)–C(10) naphthyl ring and the C(14)–C(19) benzene ring is 90.0(3)°. The dihedral angle between the C(20)–N(3)–C(21) plane and the benzene ring is 12.3(3)°. All bond lengths are within normal values [9], and comparable to those observed in similar compounds [10–12]. In the crystal structure of the compound, molecules are linked through N–H⋯O hydrogen bonds, to form one-dimensional chains running along the *a*-axis.

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(3)	4e	0.9340	0.5071	0.2908	0.068
H(4)	4e	0.6249	0.6226	0.2649	0.072
H(6)	4e	0.2699	0.6770	0.2072	0.077
H(7)	4e	0.0147	0.6498	0.1376	0.086
H(8)	4e	0.0746	0.5017	0.0912	0.086
H(9)	4e	0.3739	0.3818	0.1154	0.072
H(11)	4e	0.9225	0.2564	0.1682	0.059
H(12A)	4e	1.3075	0.4019	0.2875	0.103
H(12B)	4e	1.3031	0.2853	0.2997	0.103
H(12C)	4e	1.0853	0.3573	0.3223	0.103
H(15)	4e	0.2699	−0.1126	0.1203	0.110
H(16)	4e	0.4171	−0.2583	0.0835	0.121
H(18)	4e	0.9252	−0.1000	0.0048	0.147
H(19)	4e	0.8071	0.0415	0.0477	0.139
H(20A)	4e	0.6297	−0.3902	0.0590	0.173
H(20B)	4e	0.7468	−0.4322	0.0087	0.173
H(20C)	4e	0.4699	−0.3769	0.0088	0.173
H(21A)	4e	0.8575	−0.2450	−0.0545	0.162
H(21B)	4e	1.0064	−0.3436	−0.0368	0.162
H(21C)	4e	1.1023	−0.2366	−0.0181	0.162
H(2)	4e	0.803(3)	0.126(3)	0.124(1)	0.080

* Correspondence author (e-mail: liushiyong2010@yahoo.cn)

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

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
N(1)	4e	0.5587(5)	0.2323(2)	0.14990(9)	0.047(1)	0.042(1)	0.061(2)	0.002(1)	0.005(1)	-0.013(1)
N(2)	4e	0.6295(5)	0.1409(2)	0.1277(1)	0.040(1)	0.043(1)	0.066(2)	0.000(1)	0.001(1)	-0.017(1)
N(3)	4e	0.7735(7)	-0.2842(2)	0.0152(1)	0.114(3)	0.045(2)	0.080(2)	-0.005(2)	0.009(2)	-0.018(2)
O(1)	4e	1.0304(4)	0.3268(2)	0.25128(9)	0.067(1)	0.052(1)	0.069(2)	-0.002(1)	-0.009(1)	-0.011(1)
O(2)	4e	0.1991(4)	0.0910(2)	0.12477(8)	0.039(1)	0.064(1)	0.074(2)	0.001(1)	-0.003(1)	-0.009(1)
C(1)	4e	0.7043(6)	0.3801(2)	0.1946(1)	0.046(2)	0.039(2)	0.054(2)	-0.007(1)	0.012(1)	-0.008(1)
C(2)	4e	0.8581(6)	0.4017(2)	0.2363(1)	0.050(2)	0.044(2)	0.054(2)	-0.010(1)	0.010(1)	-0.007(1)
C(3)	4e	0.8287(6)	0.4937(2)	0.2628(1)	0.065(2)	0.052(2)	0.053(2)	-0.015(2)	0.008(2)	-0.012(2)
C(4)	4e	0.6447(7)	0.5624(2)	0.2471(1)	0.072(2)	0.043(2)	0.066(2)	-0.009(2)	0.019(2)	-0.015(2)
C(5)	4e	0.4837(6)	0.5458(2)	0.2049(1)	0.055(2)	0.040(2)	0.065(2)	-0.007(1)	0.019(2)	-0.004(1)
C(6)	4e	0.2925(7)	0.6174(2)	0.1889(1)	0.067(2)	0.046(2)	0.080(3)	0.002(2)	0.022(2)	-0.004(2)
C(7)	4e	0.1405(7)	0.6018(3)	0.1475(2)	0.070(2)	0.055(2)	0.089(3)	0.011(2)	0.015(2)	0.013(2)
C(8)	4e	0.1760(7)	0.5123(3)	0.1200(1)	0.077(2)	0.061(2)	0.076(2)	0.006(2)	-0.001(2)	0.002(2)
C(9)	4e	0.3558(6)	0.4406(2)	0.1344(1)	0.063(2)	0.049(2)	0.067(2)	0.002(2)	0.003(2)	-0.006(2)
C(10)	4e	0.5159(6)	0.4535(2)	0.1775(1)	0.047(2)	0.042(2)	0.053(2)	-0.008(1)	0.015(1)	-0.005(1)
C(11)	4e	0.7503(6)	0.2825(2)	0.1697(1)	0.045(2)	0.045(2)	0.058(2)	-0.003(1)	0.003(1)	-0.012(1)
C(12)	4e	1.1948(7)	0.3442(3)	0.2936(1)	0.060(2)	0.077(2)	0.070(2)	-0.007(2)	-0.007(2)	-0.007(2)
C(13)	4e	0.4334(5)	0.0748(2)	0.1152(1)	0.038(2)	0.046(2)	0.041(2)	-0.001(1)	-0.006(1)	-0.002(1)
C(14)	4e	0.5255(5)	-0.0181(2)	0.0893(1)	0.039(2)	0.044(2)	0.041(2)	-0.006(1)	-0.008(1)	-0.002(1)
C(15)	4e	0.4109(8)	-0.1089(3)	0.0978(2)	0.106(3)	0.054(2)	0.116(3)	-0.018(2)	0.065(3)	-0.007(2)
C(16)	4e	0.4951(9)	-0.1968(3)	0.0744(2)	0.129(4)	0.043(2)	0.130(4)	-0.021(2)	0.062(3)	-0.008(2)
C(17)	4e	0.6852(6)	-0.1971(2)	0.0391(1)	0.067(2)	0.044(2)	0.051(2)	-0.005(2)	-0.006(2)	-0.006(1)
C(18)	4e	0.795(1)	-0.1050(3)	0.0294(2)	0.158(4)	0.060(2)	0.149(4)	-0.039(3)	0.104(4)	-0.043(3)
C(19)	4e	0.719(1)	-0.0191(3)	0.0548(2)	0.147(4)	0.052(2)	0.150(4)	-0.045(2)	0.097(4)	-0.038(2)
C(20)	4e	0.645(1)	-0.3785(3)	0.0236(2)	0.144(4)	0.045(2)	0.159(5)	-0.002(2)	0.014(4)	-0.015(3)
C(21)	4e	0.950(1)	-0.2767(3)	-0.0270(2)	0.144(4)	0.076(3)	0.104(4)	0.012(3)	0.027(3)	-0.038(3)

Acknowledgments. We thank Zhejiang Provincial Natural Science Foundation of China for research support (Project no. LY12B02004).

References

- Hillmer, A. S.; Putcha, P.; Levin, J.; Hogen, T.; Hyman, B. T.; Kretschmar, H.; McLean, P. J.; Giese, A.: Converse modulation of toxic alpha-synuclein oligomers in living cells by *N'*-benzylidene-benzohydrazide derivatives and ferric iron. *Biochem. Biophys. Res. Commun.* **391** (2010) 461-466.
- Zhong, X.; Wei, H.-L.; Liu, W.-S.; Wang, D.-Q.; Wang, X.: The crystal structures of copper(II), manganese(II), and nickel(II) complexes of a (Z)-2-hydroxy-*N'*-(2-oxoindolin-3-ylidene) benzohydrazide - potential antitumor agents. *Bioorg. Med. Chem. Lett.* **17** (2007) 3774-3777.
- Zhu, Q.-Y.; Wei, Y.-J.; Wang, F.-W.: Synthesis, structures, and antibacterial activities of 3-methoxy-*N'*-(3-bromo-5-chloro-2-hydroxybenzylidene)benzohydrazide dimethanol solvates and 3-hydroxy-*N'*-(3-bromo-5-chloro-2-hydroxybenzylidene)benzohydrazide methanol solvate. *Pol. J. Chem.* **83** (2009) 1233-1240.
- Jimenez-Pulido, S. B.; Linares-Ordóñez, F. M.; Martínez-Martos, J. M.; Moreno-Carretero, M. N.; Quiros-Olozabal, M.; Ramirez-Exposito, M. J.: Metal complexes with the ligand derived from 6-acetyl-1,3,7-trimethyl-lumazine and benzohydrazide. Molecular structures of two new Co(II) and Rh(III) complexes and analysis of in vitro antitumor activity. *J. Inorg. Biochem.* **102** (2008) 1677-1683.
- Raj, K. K. V.; Narayana, B.; Ashalatha, B. V.; Kumari, N. S.; Sarojini, B. K.: Synthesis of some bioactive 2-bromo-5-methoxy-*N'*-[4-(aryl)-1,3-thiazol-2-yl]benzohydrazide derivatives. *Eur. J. Med. Chem.* **42** (2007) 425-429.
- Kolev, T.; Yancheva, D.; Glavcheva, Z.; Schurmann, M.; Kleb, D. C.; Preut, H.; Bleckmann, P.: Crystal structure of 4-hydroxy-3-methoxybenzaldehyde-4-nitrophenyl-hydrazone, C₁₄H₁₃N₃O₄. *Z. Kristallogr. NCS* **216** (2001) 237-238.
- Zhao, L.-F.: Crystal structure of (E)-4-methoxy-nitrophenone N-(2,4-dinitrophenyl)hydrazide, C₁₄H₁₁N₅O₇. *Z. Kristallogr. NCS* **221** (2006) 29-30.
- Li, Z.-P.: Crystal structure of 3,5-diiodosalicylaldehyde (2-hydroxybenzoyl)hydrazide, C₁₄H₁₀I₂N₂O₃. *Z. Kristallogr. NCS* **222** (2007) 253-254.
- Allen, F. H.; Kennard, O.; Watson, D. G.; Brammer, L.; Orpen, A. G.; Taylor, R.: Tables of bond lengths determined by X-ray and neutron-diffraction. .1. Bond lengths in organic-compounds. *J. Chem. Soc. Perkin Trans. 2* (1987) S1-S19.
- Liu, S.-Y.; Wang, X.-L.: (E)-*N'*-(2-Chloro-5-nitrobenzylidene)-3-methoxybenzohydrazide monohydrate. *Acta Crystallogr.* **E67** (2011) o1625.
- Sun, S.-S.; Liu, S.-Y.; Zheng, T.-T.; Wang, X.-L.: (E)-*N'*-(2-Hydroxy-3,5-diiodobenzylidene)nicotinohydrazide acetonitrile monosolvate. *Acta Crystallogr.* **E67** (2011) o1624.
- Guo, H.-C.; Liu, S.-Y.; Wang, X.-L.: (E)-*N'*-(5-Bromo-2-hydroxybenzylidene)-3-methylbenzohydrazide. *Acta Crystallogr.* **E67** (2011) o1878.
- Bruker. *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA, 2001.
- Bruker. *SMART* (Version 5.625), *SAINT* (Version 6.01). Bruker AXS Inc., Madison, Wisconsin, USA, 2007.
- Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr.* **A64** (2008) 112-122.