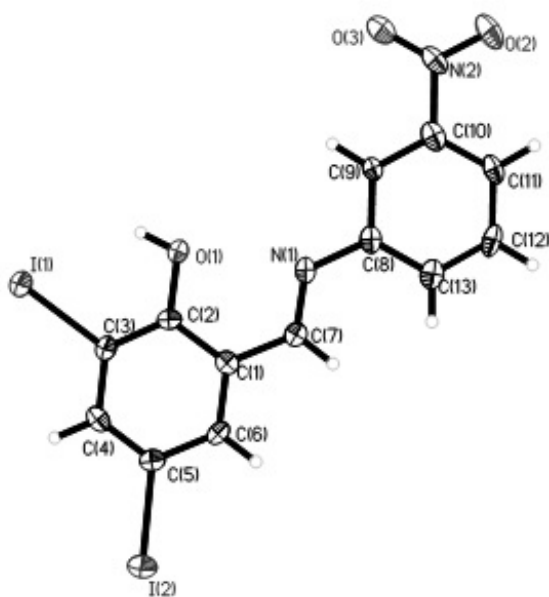


Microwave solid phase synthesis and crystal structure of 2,4-diiodo-6-[(3-nitro-phenylimino)-methyl]-phenol, $C_{13}H_8I_2N_2O_3$

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Received June 27, 2012, accepted September 26, 2012, available online November 15, 2012, CCDC no. 1267/3872



Abstract

$C_{13}H_8I_2N_2O_3$, orthorhombic, *Pbcm* (no. 57), $a = 9.104(2)$ Å, $b = 23.654(3)$ Å, $c = 6.696(2)$ Å, $V = 1442.0$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0392$, $wR_{\text{ref}}(F^2) = 0.1508$, $T = 298$ K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.17×0.20×0.20 mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	43.70 cm ^{−1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{\text{max}}$:	51°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	9719, 1459
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 1186
$N(\text{param})_{\text{refined}}$:	121
Programs:	SHELX [9]

Source of material

3,5-diiodo-salicylaldehyde (4 mmol) and an equivalent amount of 3-nitro-phenylamine were mixed together and microwave radiated for 3 min at 150W. The yellow powder was dissolved in ethanol and kept untouched in air for 3 days. Large block-shaped crystals were precipitated. They were filtered, washed with ethanol and dried in air (yield 89%).

Discussion

The halogen substituted salicylaldehyde Schiff base were widely applied in biologic technology, chemical analysis, organic syn-

thesis, and for other purposes [1–7]. The rapid growth of this field has afforded numerous chiral ligands as molds that impart asymmetry to the reactants. The vanadium-chiral Schiff base complex, readily prepared from 3,5-diiodo-salicylaldehyde and (*S*)-tert-leucinol, was found to be highly enantioselective [8]. From these former investigations we concluded that compounds of 3,5-diiodosalicylaldehyde could possess potential biological activities and antimicrobial properties. In this paper, the microwave solid phase synthesis and crystal structure of 2,4-diiodo-6-[(3-nitro-phenylimino)-methyl]-phenol was reported. The molecular structure of $C_{13}H_8I_2N_2O_3$ crystallizes orthorhombically with space group *Pbcm*. The I(1)–C(3), I(2)–C(5), N(1)–C(7) and N(1)–C(8) bonds are 2.098(10) Å, 2.094(10) Å, 1.275(12) Å and 1.423(13) Å, respectively. The bond angles of C(7)–N(1)–C(8) and N(1)–C(7)–C(1) are 123.7(9)° and 120.9(10)°, respectively. The dihedral angles between two benzene rings are 0.0°. The X-ray analysis reveals that intermolecular C(6)–H(6)⋯O(3) (length C(6)–O(3) = 3.427(8) Å), O(1)–H(1A)⋯I(1) (length O(1)–I(1) = 3.162(7) Å) and O(1)–H(1A)⋯I(2) (length O1–I2 = 3.005(7) Å) hydrogen bonds occur in the crystal lattice.

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}
H(1A)	4d	0.2664	0.4282	¼	0.078
H(4)	4d	0.7085	0.3565	¼	0.047
H(6)	4d	0.7600	0.5240	¼	0.046
H(7)	4d	0.5489	0.5882	¼	0.045
H(9)	4d	0.0751	0.5879	¼	0.043
H(11)	4d	0.0785	0.7559	¼	0.080
H(12)	4d	0.3355	0.7544	¼	0.084
H(13)	4d	0.4581	0.6707	¼	0.068

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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
I(1)	4d	0.37680(8)	0.32821(3)	¼	0.0422(5)	0.0315(5)	0.1021(7)	−0.0050(3)	0	0
I(2)	4d	0.98603(8)	0.42935(3)	¼	0.0326(4)	0.0573(6)	0.0670(6)	0.0032(3)	0	0
N(1)	4d	0.347(1)	0.5653(3)	¼	0.044(5)	0.027(4)	0.059(5)	0.000(4)	0	0
N(2)	4d	−0.106(1)	0.6729(4)	¼	0.048(6)	0.045(7)	0.081(7)	0.021(5)	0	0
O(1)	4d	0.3070(8)	0.4592(3)	¼	0.035(4)	0.027(4)	0.094(6)	−0.004(3)	0	0
O(2)	4d	−0.167(1)	0.7186(4)	¼	0.074(7)	0.048(6)	0.18(1)	0.035(5)	0	0
O(3)	4d	−0.171(1)	0.6285(4)	¼	0.047(5)	0.054(6)	0.131(9)	0.016(5)	0	0
C(1)	4d	0.547(1)	0.5001(4)	¼	0.036(5)	0.036(5)	0.042(5)	0.003(4)	0	0
C(2)	4d	0.453(1)	0.4519(4)	¼	0.026(5)	0.039(5)	0.044(5)	−0.001(4)	0	0
C(3)	4d	0.516(1)	0.3990(4)	¼	0.039(5)	0.028(5)	0.048(6)	−0.004(4)	0	0
C(4)	4d	0.668(1)	0.3926(4)	¼	0.041(6)	0.036(5)	0.042(5)	0.011(5)	0	0
C(5)	4d	0.757(1)	0.4377(5)	¼	0.030(5)	0.044(6)	0.036(5)	−0.002(4)	0	0
C(6)	4d	0.698(1)	0.4927(4)	¼	0.034(5)	0.033(5)	0.049(6)	−0.011(4)	0	0
C(7)	4d	0.486(1)	0.5573(4)	¼	0.037(6)	0.034(5)	0.043(6)	−0.003(4)	0	0
C(8)	4d	0.281(1)	0.6198(4)	¼	0.049(6)	0.030(5)	0.047(6)	0.001(5)	0	0
C(9)	4d	0.129(1)	0.6213(4)	¼	0.033(5)	0.026(5)	0.047(6)	0.004(4)	0	0
C(10)	4d	0.058(2)	0.6720(4)	¼	0.056(7)	0.040(6)	0.045(6)	0.011(5)	0	0
C(11)	4d	0.130(1)	0.7219(5)	¼	0.053(8)	0.027(6)	0.12(1)	0.015(5)	0	0
C(12)	4d	0.283(2)	0.7207(5)	¼	0.069(9)	0.025(6)	0.12(1)	−0.014(6)	0	0
C(13)	4d	0.356(2)	0.6708(5)	¼	0.057(8)	0.037(6)	0.075(8)	−0.003(5)	0	0

Acknowledgments. The work was co-financed by the Priority Academic Program Development of Jiangsu Higher Education Institutions (PAPD), China.

References

- Nielsen, M.; Dauksaite, V.; Kjems, J.; Gothelf, K. V.: DNA-directed Coupling of Organic Modules by Multiple Parallel Reductive Aminations and Subsequent Cleavage of Selected DNA Sequences. *Bioconjugate Chem.* **16** (2005) 981-985.
- You, Z.-L.; Zhu, H.-L.: Syntheses, Crystal Structures, and Antibacterial Activities of Four Schiff Base Copper(II), Zinc(II), and Cadmium(II) Complexes Derived from 2-[(2-Dimethylaminoethylimino) methyl]phenol. *Z. Kristallogr.* **632** (2004) 140-146.
- Xu, S.-P.; Pei, Y.; Xu, G.; Su, W.-X.; Shu, J.-X.: Crystal Structures and Antibacterial Activities of 3,5-Diiodosalicylaldehyde-2-aminoethanol. *J. Xuzhou Norm. Univ.: Nat. Sci. Ed.* **28** (2010) 57-60.
- Liu, J.; Zhang, T.-X.; Lu T.-B.; Qu, L.-H.; Zhou, H.; Zhang, Q.-L.; Ji, L.-N.: DNA-binding and Cleavage Studies of Macrocyclic Copper (II) Complexes. *J. Inorg. Biochem.* **91** (2002) 269-276.
- Roth, A.; Spielberg, E. T.; Plass, W.: Kit for Unsymmetric Dinucleating Double-Schiff-Base Ligands: Facile Access to a Versatile New Ligand System and Its First Heterobimetallic Copper-Zinc Complex. *Inorg. Chem. Comm.* **46** (2007) 4362-4364.
- Li, C.-H.; Wang, Q.; Xu, Y.-Q.; Hu, C.-W.: Synthesis and Crystal Structure of a New Three-dimensional Supramolecule of Copper(II) Schiff Base Complex. *Chinese J. Struct. Chem.* **27** (2008) 187-190.
- Peng, L.-L.; Ye, J.-C.: The Microwave Synthesis in Liquid State of Salicylaldehyde Schiff Base and Metal Complexes. *Journal of Hubei Normal University (Natural Science)*. **24** (2004) 86-88.
- Pelotier, B.; Anson, M. S.; Campbell, I. B.; Macdonald, S. J. F.; Priem, G.; Jackson, R. F. W.: Enantioselective Sulfide Oxidation with H₂O₂: A Solid Phase and Array Approach for the Optimisation of Chiral Schiff Base-Vanadium Catalysts. *Synlett.* (2002) 1055-1060.
- Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr.* **A64** (2008) 112-122.