Synthesis and Structural Characterization of *N*-(2-Hydroxyphenyl)tetrachlorophthalimide

JIAN LI

Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, P.R. China

Corresponding author: Tel: +86 536 8877561; E-mail: ljwfu@163.com

(Received: 27 October 2011;

Accepted: 22 August 2012)

AJC-11994

N-(2-Hydroxyphenyl)tetrachlorophthalimide ($C_{14}H_5NO_3Cl_4$, Mr = 376.99) was synthesized and characterized by elemental analysis, 1H NMR spectra and single crystal X-ray diffraction. The crystal belongs to monoclinic, space group $P2_1/c$, with $a = 12.2501(16)$, $b = 14.3426(18)$, $c = 20.092(3)$ Å, $\beta = 55.038(2)^\circ$, $V = 2893.0(6)$ Å³, $Z = 8$, $D_c = 1.731$ g/cm³, $\lambda = 0.71073$ Å, $\mu(MoK\alpha) = 0.828$ mm⁻¹, $F(000) = 1504$. The final refinement gave $R = 0.0351$, $wR = 0.0847$ for 5, 102 observed reflections with $I > 2\sigma(I)$. The asymmetric unit of the title compound contains two independent molecules. The dihedral angles between the phthalimide and 2-hydroxyphenyl groups are $64.6(2)^\circ$ and $64.5(2)^\circ$, respectively, in two independent molecules of the asymmetric unit. The molecules of the crystal are connected, via the intermolecular O-H...O hydrogen bonds interactions, to form network.

Key Words: *N*-(2-Hydroxyphenyl)tetrachlorophthalimide, Synthesis, Structure characterization.

INTRODUCTION

Phthalimides and *N*-substituted phthalimides are an important class of compounds because of their interesting biological activities¹. Phthalimides have also served as starting materials and intermediates for the synthesis of pharmacophores² and alkaloids³. Mahapatra *et al.*⁴ have synthesized several *N*-substituted tetrachlorophthalimides and the study revealed *N*-(2,4-dinitrophenyl)tetrachlorophthalimide could be a representative of a new group of α -glucosidase inhibitors and exhibit significant antihyperglycemic effect. In our previous work several *N*-substituted tetrachlorophthalimides have been synthesized⁵⁻⁸. In this paper, the synthesis and the structural characterization of the *N*-(2-hydroxyphenyl)tetrachlorophthalimide is reported.

EXPERIMENTAL

Synthesis of the *N*-(2-hydroxyphenyl)tetrachlorophthalimide: All the reagents were of AR grade and used without further purification. A mixture of 4,5,6,7-tetrachloroisobenzofuran-1,3-dione (2.86 g, 0.01 mol) and 2-aminophenol (1.09 g, 0.01 mol) in acetic acid (10 mL) was refluxed for 2 h. After cooling, filtration and drying, *N*-(2-hydroxyphenyl)tetrachlorophthalimide was obtained. 10 mg of the present compound were dissolved in 15 mL acetone and the solution was kept at room temperature for 8 d. Natural evaporation gave colourless single crystals of the title compound, suitable for X-ray analysis. Elemental analysis: calcd. (%) for

$C_{14}H_5NO_3Cl_4$: C 44.56, H 1.33, N 3.71. Found (%): C 44.65, H 1.38, N 3.70. 1H NMR data (CD_3COCD_3 , ppm): $\delta = 7.45$ (d, 1H), 6.81 (m, 2H), 6.70 (d, H), 4.99 (s, H).

Data collection and structure determination: A selected crystal of the title compound was mounted on a SMART CCD diffractometer. The reflection data were measured at 298 K, using a graphite monochromator $MoK\alpha$ ($\lambda = 0.71073$ Å) radiation with an ω - 2θ scan mode. The total reflections were 11,953 with 5,102 independent ones ($R_{int} = 0.0189$), of which 399 were observed with $I > 2\sigma(I)$. Intensities were corrected for Lorentz and polarization effects and empirical absorption and all data were corrected using SADAB⁹ program.

The structure was solved by direct methods using SHELXS-97¹⁰ program. All the non-hydrogen atoms were refined on F^2 anisotropically by full-matrix least squares method. All hydrogen atoms were placed in the geometrically calculated positions. The contributions of these hydrogen atoms were included in the structure factor calculations. The atomic scattering factors and anomalous dispersion corrections were taken from international table for X-ray crystallography¹¹. The final least-square cycle gave $R = 0.0351$ and $wR = 0.0847$ ($w = 1/[\sigma^2(F_o^2) + (0.0440P)^2 + 1.0177P]$, where $P = (F_o^2 + 2F_c^2)/3$). $S = 1.023$, $(\Delta\rho)_{min} = -0.178$ and $(\Delta\rho)_{max} = 0.272$ e/Å³. CIF file containing complete information on the studied structure was deposited with CCDC, deposition number 849424 and is freely available upon request from the following web site: www.ccdc.cam.ac.uk/data_request/cif.

RESULTS AND DISCUSSION

The atomic coordinates and equivalent isotropic thermal parameters for the non-H atoms in the title compound are given in Table-1. The selected bond lengths and bond angles are listed in Table-2. The hydrogen bond schemes are seen in Table-3. A displacement ellipsoid plot with atomic numbering scheme is shown in Fig. 1 and a perspective view of the crystal packing in the unit cell is shown in Fig. 2.

TABLE-1
ATOMIC COORDINATES ($\times 10^4$) AND THERMAL
PARAMETERS ($\text{\AA}^2 \times 10^3$)

Atom	x	Y	Z	U(eq)
C(1)	-484(2)	6288(2)	5683(2)	42(1)
C(2)	-1651(2)	6643(2)	5825(2)	50(1)
C(3)	-2631(3)	6976(2)	6580(2)	60(1)
C(4)	-2479(3)	6965(2)	7205(2)	62(1)
C(5)	-1345(3)	6593(2)	7080(2)	53(1)
C(6)	-355(2)	6253(2)	6326(1)	41(1)
C(7)	1168(2)	4925(2)	6041(1)	38(1)
C(8)	2398(2)	4825(2)	6027(1)	37(1)
C(9)	3147(2)	4053(2)	5911(1)	42(1)
C(10)	4262(2)	4163(2)	5922(1)	44(1)
C(11)	4586(2)	5043(2)	6066(1)	45(1)
C(12)	3795(2)	5815(2)	6209(1)	45(1)
C(13)	2706(2)	5689(2)	6181(1)	38(1)
C(14)	1683(2)	6370(2)	6310(1)	43(1)
C(15)	3560(2)	10057(2)	6232(1)	39(1)
C(16)	4302(2)	10121(2)	6552(2)	55(1)
C(17)	3951(3)	9604(2)	7220(2)	65(1)
C(18)	2864(3)	9027(2)	7591(2)	63(1)
C(19)	2148(3)	8929(2)	7260(1)	50(1)
C(20)	2505(2)	9434(2)	6578(1)	38(1)
C(21)	459(2)	9448(2)	6581(1)	40(1)
C(22)	181(2)	9139(2)	5982(1)	37(1)
C(23)	-972(2)	9176(2)	6015(1)	42(1)
C(24)	-924(2)	8876(2)	5339(2)	44(1)
C(25)	256(2)	8539(2)	4654(2)	43(1)
C(26)	1416(2)	8496(2)	4633(1)	39(1)
C(27)	1353(2)	8805(1)	5301(1)	35(1)
C(28)	2418(2)	8882(1)	5443(1)	34(1)
N(1)	821(2)	5864(1)	6203(1)	40(1)
N(2)	1816(2)	9291(1)	6206(1)	36(1)
O(1)	582(2)	4337(1)	5938(1)	47(1)
O(2)	1566(2)	7181(1)	6476(1)	61(1)
O(3)	3566(2)	8655(1)	5009(1)	42(1)
O(4)	-284(2)	9779(1)	7243(1)	61(1)
O(5)	553(2)	5985(1)	4947(1)	52(1)
O(6)	3805(2)	10580(1)	5596(1)	49(1)
Cl(1)	2724(1)	2969(1)	5760(1)	64(1)
Cl(2)	5249(1)	3223(1)	5755(1)	64(1)
Cl(3)	5974(1)	5164(1)	6070(1)	66(1)
Cl(4)	4144(1)	6886(1)	6421(1)	70(1)
Cl(5)	-2430(1)	9582(1)	6862(1)	63(1)
Cl(6)	-2331(1)	8933(1)	5345(1)	60(1)
Cl(7)	294(1)	8169(1)	3831(1)	65(1)
Cl(8)	2875(1)	8094(1)	3785(1)	56(1)

As seen from Fig. 1, the asymmetric unit of the title compound contains two independent molecules. The dihedral angles between the phthalimide and 2-hydroxyphenyl groups are 64.6° (2°) and 64.5° (2°), respectively, in two independent molecules of the asymmetric unit. The bond lengths and angles (Table-2) are in agreement with those in the synthesized previously *N*-substi-

tuted tetrachlorophthalimides⁵⁻⁸. As seen from Fig. 2 and Table-3, the crystal structure of the title compound is stabilized by O-H...O hydrogen bonds interactions. Fig. 2 reveals that in the crystal the molecules of the title compound are connected, *via* the intermolecular hydrogen bonds interactions, to form network.

TABLE-2
SELECTED BOND LENGTHS ($\text{\AA}$) AND BOND ANGLES ($^\circ$)

Bond	Length ($\text{\AA}$)	Bond	Angle ($^\circ$)
C(1)-O(5)	1.356(3)	C(5)-C(6)-N(1)	119.6(2)
C(6)-N(1)	1.428(3)	O(1)-C(7)-N(1)	126.0(2)
C(7)-O(1)	1.201(3)	N(1)-C(7)-C(8)	105.44(19)
C(7)-N(1)	1.392(3)	O(2)-C(14)-N(1)	124.6(2)
C(14)-O(2)	1.196(3)	O(4)-C(21)-N(2)	125.1(2)
C(14)-N(1)	1.397(3)	O(3)-C(28)-N(2)	125.0(2)
C(15)-O(6)	1.358(3)	C(7)-N(1)-C(14)	112.47(18)
C(20)-N(2)	1.427(3)	C(7)-N(1)-C(6)	124.12(19)
C(21)-O(4)	1.198(3)	C(14)-N(1)-C(6)	123.14(19)
C(21)-N(2)	1.396(3)	C(28)-N(2)-C(21)	112.09(18)
C(28)-O(3)	1.200(2)	C(28)-N(2)-C(20)	122.58(17)
C(28)-N(2)	1.394(3)	C(21)-N(2)-C(20)	125.03(18)

TABLE-3
HYDROGEN BOND SCHEMES ($\text{\AA}^\circ$)

D-H...A	D-H	H...A	D-A	D-H...A
O5-H5...O1 ^a	0.82	2.04	2.852	171
O6-H6...O3 ^b	0.82	2.12	2.927	169

Symmetry codes: a) $-x, -y+1, -z+1$; b) $-x+1, -y+2, -z+1$

Fig. 1. Molecular structure with atomic numbering scheme

Fig. 2. View of crystal packing down the b-axis

ACKNOWLEDGEMENTS

This work was supported by Shandong Provincial Natural Science Foundation, China (No. ZR2009BL027).

REFERENCES

1. L.M. Lima, P. Castro, A.L. Machado, C.A.M. Frage, C. Lugniur, V.L.G. Moraes and E. Barreiro, *J. Biol. Org. Med. Chem.*, **21**, 3067 (2002).
2. A. Couture, E. Deniau, P. Woisel and P. Grandclaoudon, *Synthesis*, 1439 (1997).
3. A. Couture, E. Deniau, P. Grandclaoudon and C. Hoarau, *J. Org. Chem.*, **63**, 3128 (1998).
4. S. Mahapatra, J. Singh, R. Raju, B.C. Maiti and T.K. Maity, *Asian J. Chem.*, **23**, 1581 (2011).
5. J. Li, *Acta Cryst.*, **E63**, o3331 (2007).
6. J. Li, *Acta Cryst.*, **E63**, o3332 (2007).
7. J. Li, *Acta Cryst.*, **E63**, o3333 (2007).
8. J. Li, *Acta Cryst.*, **E63**, o3515 (2007).
9. G.M. Sheldrick, SADABS, Program for Empirical Absorption Correction of Area Detector Data, University of Gottingen, Germany (1996).
10. G.M. Sheldrick, SHELXTL V 5.1 Software Reference Manual, Bruker AXS, Inc., Madison, Wisconsin, USA (1997).
11. Siemens, SMART and SAINT, Area Detector Control and Integration Software. Siemens Analytical X-Ray Systems, Inc., Madison, Wisconsin, USA (1996).