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

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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, β = 55.038(2)°, V = 2893.0(6) Å^3, Z = 8, D_c = 1.731 g/cm^3, λ = 0.71073 Å, μ(MoK_α) = 0.828 mm^{-1}, F(000) = 1504. The final refinement gave R = 0.0351, wR = 0.0847 for 5, 102 observed reflections with I > 2σ(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)° and 64.5 (2)°, 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 activities[1]. Phthalimides have also served as starting materials and intermediates for the synthesis of pharmacophores[2] and alkaloids[3]. Mahapatra *et al.*[4] have synthesized several *N*-substituted tetrachlorophth-alimides and the study revealed *N*-(2,4-dinitrophenyl)tetra-chlorophthalimide could be a representative of a new group of α-glucosidase inhibitors and exhibit significant antihyperglycemic effect. In our previous work seveval *N*-substituted tetrachlorophthalimides have been synthesized[5-8]. 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): δ = 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_α (λ = 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 > 2σ(I). Intensities were corrected for Lorentz and polarization effects and empirical absorption and all data were corrected using SADABB[9] program.

The structure was solved by direct methods using SHELXS-97[10] 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 structurefactor calculations. The atomic scattering factors and anomalous dispersion corrections were taken from international table for X-ray crystallography[11]. The final least-square cycle gave R = 0.0351 and ωR = 0.0847 ($w = 1/[\sigma^2(Fo^2) + (0.0440P)^2 + 1.0177P]$, where $P = (Fo^2 + 2Fc^2)/3$). S = 1.023, $(\Delta\rho)_{min}$ = -0.178 and $(\Delta\rho)_{max}$ = 0.272 e/Å^3. 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 (× 10^4) AND THERMAL PARAMETERS (Å^2 × 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)º and 64.5 (2)º, 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*-substituted tetrachlorophthalimides[5-8]. 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 (Å) AND BOND ANGLES (º)

Bond	Length (Å)	Bond	Angle (º)
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 (Åº)

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

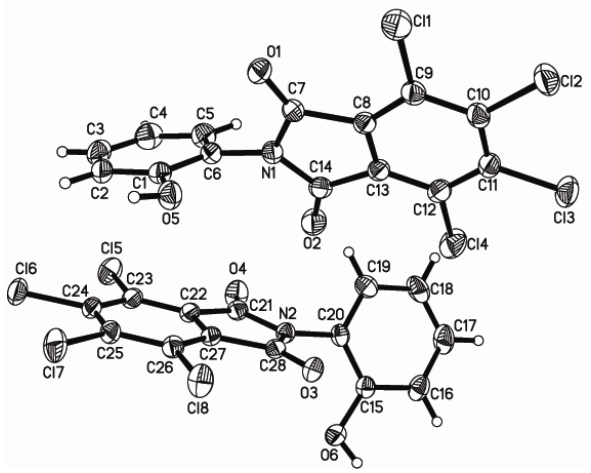

Fig. 1. Molecular structure with atomic numbering scheme

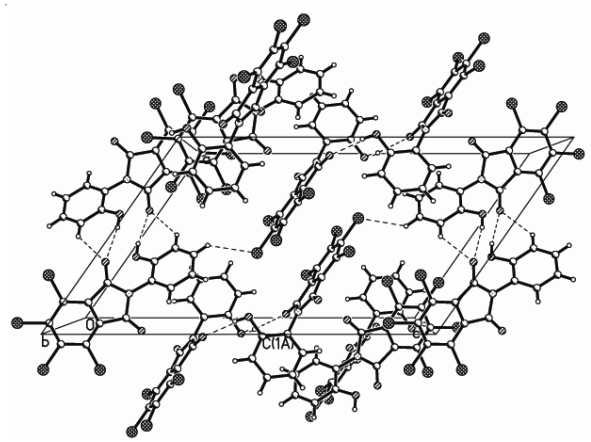

Fig. 2. View of crystal packing down the b-axis

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