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NOTE

Crystal Structure of an Unexpected Oxime-Type Polymer $(C_{10}H_{11}BrNO)_n$

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An oxime-type polymer, $(C_{10}H_{11}BrNO)_n$, has been unexpectedly obtained in the process of synthesis of *bis*(3-bromobenzaldehyde)O,O'-(hexane-1,6-diyl)dioxime. The crystal structure of the title compound has been determined by single-crystal X-ray diffraction method. The title compound crystallizes in a monoclinic crystal system, space group P2(1), with a = 4.1460(5), b = 5.4641(7), c = 22.030(2) Å, β = 91.230(1)° and Z = 2. And the polymer chains take a herringbone fashion.

Key Words: Oxime-type compound, Synthesis, Crystal structure.

Synthesis and crystal structure of oxime-type compounds and their complexes have been a hot topic in recent years[1]. Oximes are easily prepared from the corresponding carbonyl compounds with hydroxylamine and its analogues are stable to hydrolysis compared with the corresponding imines. Thus, the electrophilicity of the imino carbon of oximes is lower and addition of nucleophiles to O-substituted oximes does not readily proceed[2]. Because the oxime-type compounds are more stable to resist the metathesis of C=N bonds[3], the introduction of some functional groups or substitutions of some parts with appropriate ones are effective and inevitable. Here, an unexpected oxime-type polymer $(C_{10}H_{11}BrNO)_n$ was synthesized and the single crystal structure was also determined.

The title compound was synthesized according to an analogous method reported earlier[4]. To an ethanolic solution (5 mL) of 3-bromobenzaldehyde (370 mg, 2 mmol) was added an ethanol solution (5 mL) of 1,6-*bis*(aminooxy)hexane (148.2 mg, 1 mmol). The mixture solution was stirred at 328 K for 4 h. When cooled to room temperature, the precipitate was filtered and washed successively with ethanol and hexane, respectively. The product was dried under vacuum and purified with recrystallization from ethanol to yield 339.9 mg of the title compound, Yield, 65.6 %. Anal. calcd. (%) for $(C_{10}H_{11}BrNO)_n$: C, 49.5; H, 5.55; N, 7.07; Found: C, 48.10; H, 5.62; N, 7.32.

X-Ray structure determination: Colourless block-shaped single crystals suitable for X-ray diffraction studies were obtained after several weeks by slow evaporation from an ethanol-tetrahydrofuran mixed solution of the title compound. A crystal of dimensions 0.35 mm × 0.31 mm × 0.10 mm was used to determine the crystal structures by X-ray diffraction technique on Bruker SMART CCD area-detector diffractometer using Mo-K_α radiation (λ = 0.71073 Å, T = 298(2) K) graphite monochromation radiation. All calculations were performed using the SHELXL-97 crystal graphic software package[5]. All the non-hydrogen atoms were refined anisotropically. The hydrogen atoms were located by difference synthesis and refined isotropically. The selected bond lengths and bond angles are listed in Table-1. CCDC: 908284.

The synthesis of the title compound was used the similar synthetic route according to previously reported work[4]. However, it is remarkable that it did not form the desired symmetrical bisoxime compound, *bis*(3-bromobenzaldehyde) O,O'-(hexane-1,6-diyl)dioxime, but obtained a unexpected oxime-type polymer $(C_{10}H_{11}BrNO)_n$, which was formed in the course of the the Schiff base reaction of 3-bromobenzaldehyde and 1,6-*bis*(aminooxy) hexane. The results indicate the reaction maybe resulted in the cleavage and rearrangement of 1,6-*bis*(aminooxy)hexane molecular giving a unexpected oxime-type polymer $(C_{10}H_{11}BrNO)_n$ instead of the usually desired bisoxime compound[6].

X-Ray crystallographic analysis revealed the crystal structure of title compound. And the structure is shown in Fig. 1. Selected bond distances and angles are listed in Table-2. The title compound crystallizes in a monoclinic crystal system,

TABLE-2
SELECTED BOND LENGTHS (Å) AND ANGLES (º) FOR THE TITLE COMPOUND

Atom	Distance	Atom	Distance	Atom	Distance
Br(1)-C(8)	1.78(2)	C(2)-C(3)	1.53(6)	C(6)-C(11)	1.39(4)
N(1)-C(5)	1.34(4)	C(2)-C(4)$^{\#1}$	1.53(6)	C(6)-C(7)	1.39(3)
N(1)-O(1)	1.41(2)	C(3)-C(4)	1.53(5)	C(7)-C(8)	1.39(3)
O(1)-C(1)	1.23(3)	C(4)-C(2)$^{\#2}$	1.53(6)	C(8)-C(9)	1.38(3)
C(1)-C(2)	1.53(3)	C(5)-C(6)	1.48(3)	C(9)-C(10)	1.38(3)
Atom	Angle	Atom	Angle	Atom	Angle
C(5)-N(1)-O(1)	118(2)	C(2)-C(3)-C(4)	113(4)	C(6)-C(7)-C(8)	123.0(2)
C(1)-O(1)-N(1)	124(2)	C(2)$^{\#2}$-C(4)-C(3)	112(4)	C(9)-C(8)-C(7)	114.0(2)
O(1)-C(1)-C(2)	114(2)	N(1)-C(5)-C(6)	117(2)	C(9)-C(8)-Br(1)	123.6(2)
C(3)-C(2)-C(1)	113(3)	C(11)-C(6)-C(7)	118.8(2)	C(7)-C(8)-Br(1)	122.0(2)
C(3)-C(2)-C(4)$^{\#1}$	125(3)	C(11)-C(6)-C(5)	116(2)	C(8)-C(9)-C(10)	125.7(2)
C(1)-C(2)-C(4)$^{\#1}$	110(3)	C(7)-C(6)-C(5)	125(2)	C(11)-C(10)-C(9)	117.0(2)

Symmetry transformations used to generate equivalent atoms: $^{\#1}$ -x+2,y-1/2,-z+1; $^{\#2}$ -x+2,y+1/2,-z+1

TABLE-1
CRYSTAL DATA AND REFINEMENT PARAMETERS FOR THE TITLE COMPOUND

Empirical formula	$(C_{10}H_{11}BrNO)_n$
Crystal colour	Colourless, block-shaped
Formula weight	241.10
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2(1)
Cell dimensions	a = 4.1460(5) Å, b = 5.4641(7) Å, c = 22.030(2) Å β =91.230(1) º
Volume	498.96(1) Å^3
Z	2
Density (calculated)	1.605 mg/m^3
Absorption coefficient	4.078 mm^{-1}
F(000)	242
Index ranges	-4 ≤ h ≤ 4, -6 ≤ k ≤ 5, -26 ≤ l ≤ 25
Reflections collected/ unique	2186/968 [R_{int}= 0.0840]
Data/restraints/parameters	968/1/128
Goodness of fit indicator	1.085
Final R indices [I>2σ(I)]	R_1 = 0.1044, wR_2 = 0.2484
R indices (all data)	R_1 = 0.1315, wR_2 = 0.2714
Largest diff. peak and hole	0.957 and -1.262 e.Å^{-3}

space group P2(1), with a = 4.1460(5), b = 5.4641(7), c = 22.030(2) Å, β = 91.230(1)º and Z = 2. And the title compound shows a monomers molecular order dominated by edge-to-face interactions, leading to an interlocking herringbone arrangement. These interactions afford a one-dimensional infinite chain of $(C_{10}H_{11}BrNO)_n$ along the b-axis.

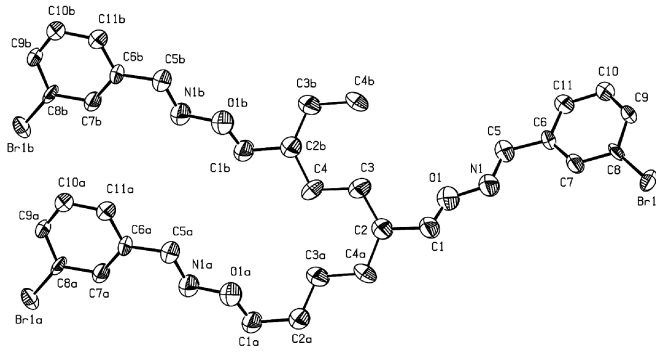

Fig. 1. Molecular structure of the title compound

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