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Crystal structure of 2, 3-Bis (5-bromo-4-fluoro-2-nitrophenyl) oxirane

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Abstract

Single crystals of 2, 3-Bis (5-bromo-4-fluoro-2-nitrophenyl) oxirane (BrFOXI) were grown by slow evaporation method at room temperature. Crystallographic data set was collected using ref. [1] Smart Apexx II x-ray diffraction. Single crystal x-ray diffraction analysis reveals that crystallizes in the monoclinic, space group P2 (1)/c with unit cell dimensions, a=14.3073(19) Å, b=7.0042(9) Å, c=15.682(2) Å and β=106.664(4)°. The crystal structure was controlled by C-H…O types of intra and intermolecular interaction.

Keywords: Epoxy; Molecular structure; Crystal packing; Dimer formation

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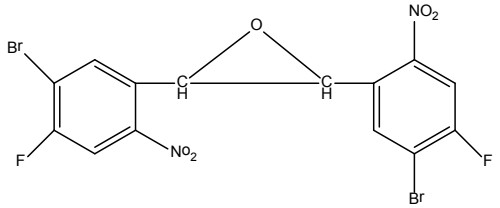
Introduction

Epoxy compounds are found to be useful in paint manufacturing, composite formations, development of adhesives as well as in many microelectronic applications such epoxy molding, bio medical applications [2-4]. Epoxides are organic three-membered compounds, where an oxygen atom is attached to two adjacent carbon atoms arise from oxidative metabolism of endogenous, as well as xenobiotic compounds. The resultant epoxides are typically unstable in aqueous environments and chemically reactive. The xenobiotics and certain endogenous substances which include epoxide intermediates have been implicated in genetic mutations and as cancer causing agents [5,6]. It is of vital importance for us to study the structure of epoxide compounds to understand the relationship in the above mentioned biological process. In view of the above said importance the properties of epoxides, the crystal structural studies of the compound mentioned is carried out.

Experimental

Synthesis of BrFOXI

The reaction of 5-bromo-4-fluoro-2-nitrobenzaldehyde (0.5 g, 2.01 mmol) with triethyl phosphite (0.67 g, 4.03 mmol) in the presence of $ZnBr_2$ (0.05 g, 0.20 mmol) at room temperature for 10 min followed by different procedures using the above using mentioned general procedure afforded trans-epoxide as a colorless solid. Single crystals suitable for X-ray diffraction studies were obtained by slow evaporation of the compound in chloroform/ ethyl acetate.



Data Collection

X-ray diffraction intensity data were collected for all three compounds on Bruker Kappa Apex II single crystal X-ray diffractometer equipped with graphite mono- chromate CuKα

(λ=1.54178 Å) radiation and CCD detector. Crystals were cut to suitable size and mounted on a glass fiber using cyano acrylate adhesive. The unit cell parameters were determined from 36 frames measured (0.5° phi-scan) from three different crystallographic zones and using the method of difference vectors. The intensity data were collected with an average four-fold redundancy per reflection and optimum resolution (0.75 Å). The intensity data collection, frames integration, Lorentz and polarization correction and decay correction were done using SAINT-NT (version7.06a) software. Empirical absorption correction (multi-scan) was performed using SADABS program.

Results and Discussion

Structure Solving and Refinement

Crystal structure was solved by Direct Methods using *SHELXS-97*. All the non-hydrogen atoms were located without any difficulty. The structure was then refined by full-matrix least-squares method using *SHELXL- 97*. They arrived model was refined using isotropic thermal parameters followed by anisotropic thermal parameters refinements. After completion of the refinement where R factor is converged with negligible shift/e.s.d and agreeable Goof and other parameters, hydrogen atoms were positioned geometrically C—H=0.93–0.98 Å and allowed to ride on their parent atoms, with U_{iso}(H) =1.5U_{eq}(C) for methyl H 1.2U_{eq}(C) for other H atoms. The relevant crystallographic detail is given in (**Table 1)**.

The molecular structure (ORTEP diagram) of BrFOXY is shown in (**Figure 1)**. The bond lengths and bond angles are listed in (**Table 2)**. The epoxy ring (O3/C4/C5), its looks like a triangle shape it form planar conformation and it is oriented axially with two halogen substituted phenyl rings (C1/C2C3/C10/C11/C12/Br1/F2) and (C6/C7/C8/C9/C13/C14/Br2/F1) makes dihedral angles of 64.5(3)° and 66.5(3)°, respectively. The dihedral angle between the two halogen substituted phenyl rings is 60.5(1)°.

The nitro groups (N1/O1/O2) and (N2/O4/O5) are equatorially oriented with the halogen substituted phenyl ring (C1/C2C3/C10/C11/C12/Br1/F2) and (C6/C7/C8/C9/C13/C14/Br2/F1) which are evidenced by the torsion angle values are [O1/N1/C11/C12=] -153.3(4)°; [O2/N1/C11/C3=] -156.1(4)°; [O4/N2/C14/C6=] 7.2(7)° and [O4/N2/C14/C13=] -171.4(4)°, respectively. C-H…O types of intermolecular interaction makes C(10) chain running along a-direction (**Figure 2)**. Relevant hydrogen bond details are given in (**Table 3)**.

Table 1 Crystal data for BrFOXY.

Parameters	Values
Empirical formula	$C_{14}H_6Br_2F_2N_2O_5$
Formula weight	480.03
Temperature	296(2) K
Wavelength	1.54178 Å
Crystal system, space group	monoclinic, P2(1)/c
Unit cell dimensions	a=14.3073(19) Å
	b=7.0042(9) Å, β=106.664(4)°
	c=15.682(2) Å
Volume	1505.5(3)Å^3
Z, Calculated density	4, 2.118 Mg/m^3
Absorption coefficient	7.366 mm^{-1}
F (000)	928
Crystal size	0.29 × 0.28 × 0.27 mm
Theta range for data collection	3.22 to 64.93°
Limiting indices	-16<=h<=13, -8<=k<=8, -16<=l<=18
Reflections collected/unique	8652/2463 [R(int)=0.0541]
Completeness to theta=64.93	95.90%
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2463 / 0 / 226
Goodness-of-fit on F^2	1.616
Final R indices [I>2sigma(I)]	R1=0.0628, wR2=0.1856
R indices (all data)	R1=0.0636, wR2=0.1874
Largest diff. peak and hole	1.416 and -1.088 e. Å^{-3}

Acknowledgements

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Additional Information

Crystallographic data for the structures reported here have been deposited with CCDC Deposition No's CCDC 1408773, 1408774 and 1408775. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK, Fax: (+44) 1223 336 033; E-mail: deposit@ccdc.cam.ac.uk

Table 2 Atomic coordinates (× 10^4) and equivalent isotropic displacement parameters (Å^2 × 10^3) for the non-hydrogen atoms of BrFOXY.

Atoms	x	y	z	U(eq)
Br1	6565(1)	3928(1)	4916(1)	22(1)
Br2	-951(1)	1468(1)	3747(1)	21(1)
F1	-1559(2)	1236(4)	1743(2)	24(1)
F2	7214(2)	194(4)	4300(2)	23(1)
O1	3289(2)	-2510(6)	2592(2)	29(1)
O2	4534(3)	-4355(6)	3116(2)	28(1)
O3	2827(2)	2083(5)	4090(2)	19(1)
O4	2441(2)	1337(5)	1470(2)	24(1)
O5	1110(3)	1433(6)	375(3)	32(1)
N1	4121(3)	-2798(6)	3044(3)	18(1)
N2	1548(3)	1434(6)	1174(3)	18(1)
C1	5787(3)	1867(7)	4333(3)	15(1)
C2	4791(3)	1909(7)	4170(3)	14(1)
C3	4212(3)	403(7)	3747(3)	14(1)
C4	3145(3)	487(7)	3645(3)	15(1)
C5	2516(3)	2043(7)	3129(3)	14(1)
C6	1441(3)	1745(6)	2738(3)	14(1)
C7	859(3)	1704(6)	3307(3)	14(1)
C8	-150(3)	1512(6)	2974(3)	16(1)
C9	-584(3)	1382(7)	2061(4)	19(1)
C10	6236(3)	254(7)	4115(3)	17(1)
C11	4695(3)	-1158(6)	3520(3)	15(1)
C12	5688(4)	-1269(7)	3703(3)	16(1)
C13	-37(4)	1379(7)	1475(3)	19(1)
C14	976(3)	1540(7)	1826(3)	15(1)

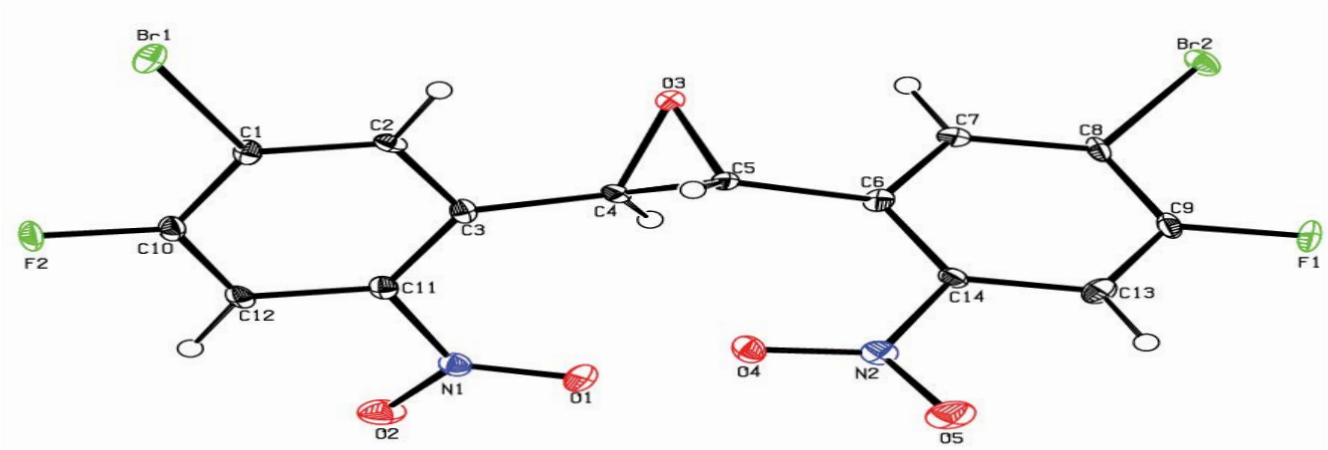

Figure 1 The molecular structure of BrFOXy, showing the atomic numbering and displacement ellipsoids drawn at the 30% probability level.

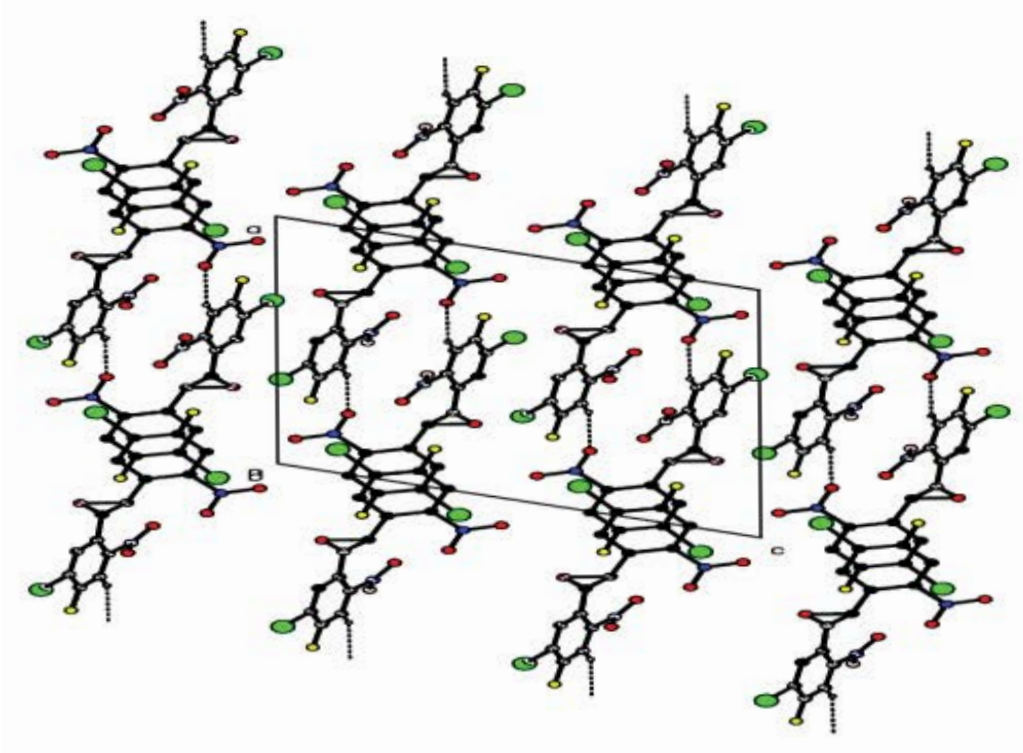

Figure 2 The packing of the molecules BrFOXY viewed down b-axis.

Table 3 Hydrogen bond interactions for **BrFOXY** [Å and ≡]. Symmetry code: 1-x,-1/2+y, 1/2-z.

D-H...A	D-H	H...A	D...A	DHA
C12-H1...O4^{i}	0.93	2.44	3.235(7)	143

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