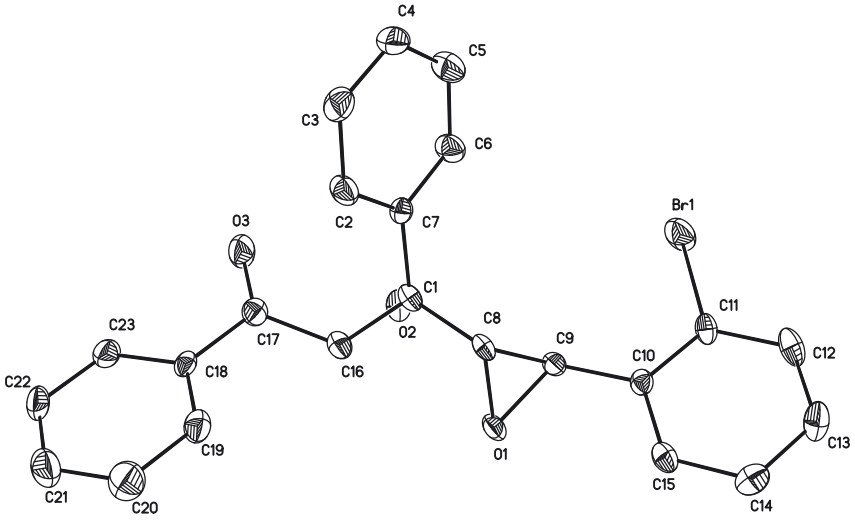
Chengjuan Li, Yingjie Li, Aoqi Wang, Shiqi Xi and Yalin Zhang*

The crystal structure of (3′-(2-bromophenyl)-2-phenyl-[2,2′-bioxiran]-3-yl)(phenyl)methanone, $C_{92}H_{68}O_{12}Br_4$



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Abstract

$C_{92}H_{68}O_{12}Br_4$, monoclinic, $P2_1/c$ (no. 14), a = 12.3604(11) Å, b = 9.8658(9) Å, c = 15.2010(13) Å, β = 93.773(2)°, V = 1849.7(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0697, $wR_{ref}(F^2)$ = 0.1790, T = 298.15 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	White bulk
Size:	0.21 × 0.20 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	2.24 mm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX II, ϕ and ω
θ_{max}, completeness:	24.7°, 91 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5,527, 3,044, 0.067
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 21\ \sigma(I_{obs})$, 1,841
$N(param)_{refined}$:	244
Programs:	Bruker,[1] SHELX[2]

1 Source of materials

2–Bromoacetophenone (0.8 mmol), 2-bromobenzaldehyde (0.4 mmol) and $NaHCO_3$ (0.6 mmol) were successively added to 10 mL water in a 25 mL flask. The mixture was stirred at room temperature for 6 h and then concentrated in vacuo. The residue was purified through column chromatography on silica gel to give the title compound. The crystals were obtained (yield: 37 %) by dissolving the title compound (0.02 g) in ethyl acetate (2 mL) and evaporating slowly at room temperature for about 1 day.

2 Experimental details

All hydrogen atomic positions were taken from a difference Fourier map. They were refined with variable isotropic displacement parameters. Their U_{iso} values were set to 1.2U_{eq} (C, of benzene ring) of the parent atoms, respectively. All the H atoms were refined as riding on their parent atom.

***Corresponding author: Yalin Zhang**, School of Chemistry and Chemical Engineering, Liaocheng University, Liaocheng 252000, Shandong, China, E-mail: zhangyalin@lcu.edu.cn. https://orcid.org/0009-0002-0592-6200
Chengjuan Li, Yingjie Li, Aoqi Wang and Shiqi Xi, School of Chemistry and Chemical Engineering, Liaocheng University, Liaocheng 252000, Shandong, China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.53846 (5)	0.64349 (8)	0.07687 (6)	0.0564 (3)
C1	0.8868 (4)	0.4611 (6)	0.1432 (3)	0.0236 (13)
C2	0.9075 (5)	0.3681 (7)	0.2958 (4)	0.0374 (16)
H2	0.9810	0.3889	0.2975	0.045*
C3	0.8649 (6)	0.3131 (7)	0.3681 (4)	0.0443 (18)
H3	0.9096	0.2969	0.4187	0.053*
C4	0.7570 (6)	0.2816 (7)	0.3671 (5)	0.0477 (18)
H4	0.7275	0.2450	0.4166	0.057*
C5	0.6925 (6)	0.3054 (7)	0.2905 (5)	0.0502 (19)
H5	0.6192	0.2837	0.2880	0.060*
C6	0.7362 (5)	0.3598 (7)	0.2200 (4)	0.0385 (16)
H6	0.6919	0.3751	0.1691	0.046*
C7	0.8433 (4)	0.3931 (6)	0.2207 (4)	0.0253 (14)
C8	0.8663 (4)	0.6099 (6)	0.1384 (4)	0.0257 (14)
H8	0.8754	0.6596	0.1942	0.031*
C9	0.7895 (4)	0.6676 (6)	0.0712 (4)	0.0261 (14)
H9	0.7507	0.6020	0.0323	0.031*
C10	0.7337 (4)	0.7979 (6)	0.0826 (4)	0.0259 (14)
C11	0.6218 (4)	0.8039 (6)	0.0842 (4)	0.0308 (15)
C12	0.5681 (5)	0.9251 (8)	0.0893 (4)	0.0457 (18)
H12	0.4928	0.9272	0.0888	0.055*
C13	0.6265 (5)	1.0425 (8)	0.0949 (4)	0.0471 (18)
H13	0.5909	1.1248	0.1005	0.057*
C14	0.7376 (5)	1.0401 (7)	0.0924 (4)	0.0431 (17)
H14	0.7769	1.1206	0.0943	0.052*
C15	0.7895 (5)	0.9184 (7)	0.0871 (4)	0.0323 (15)
H15	0.8646	0.9171	0.0864	0.039*
C16	0.9812 (5)	0.4088 (7)	0.0979 (4)	0.0315 (15)
H16	1.0249	0.4772	0.0698	0.038*
C17	1.0385 (5)	0.2815 (7)	0.1244 (4)	0.0331 (15)
C18	1.1570 (4)	0.2941 (6)	0.1452 (4)	0.0266 (14)
C19	1.2050 (5)	0.4154 (7)	0.1677 (4)	0.0410 (17)
H19	1.1625	0.4925	0.1726	0.049*
C20	1.3157 (6)	0.4236 (9)	0.1828 (5)	0.058 (2)
H20	1.3481	0.5065	0.1972	0.070*
C21	1.3781 (6)	0.3099 (9)	0.1769 (5)	0.058 (2)
H21	1.4529	0.3157	0.1872	0.069*
C22	1.3310 (6)	0.1880 (8)	0.1559 (5)	0.049 (2)
H22	1.3737	0.1107	0.1526	0.059*
C23	1.2211 (5)	0.1795 (7)	0.1396 (4)	0.0391 (16)
H23	1.1893	0.0965	0.1247	0.047*
O1	0.9033 (3)	0.6769 (4)	0.0626 (3)	0.0345 (11)
O2	0.8720 (3)	0.3927 (4)	0.0607 (3)	0.0342 (11)
O3	0.9932 (4)	0.1748 (5)	0.1261 (4)	0.0568 (14)

3 Comment

Catalysis is one of the fastest-growing and most crucial areas in organic synthetic and industrial chemistry, where nearly 90 % of new chemicals are produced via the important catalytic reactions.[3] In recent years, alkali catalysis, photocatalysis and electrocatalysis have developed rapidly and lots of excellent results have been reported successively in these fields.[4,5] As a typical class of base-catalyzed reactions, Darzens reaction is an exceptionally efficient method for generating new stereogenic centers with high diastereoselectivity.[6,7] This reaction is of particular significance for the synthesis of epoxy carbonyls and analogous compounds, and is thus regarded as one of the most pivotal C–C bond forming transformations in synthetic organic chemistry. Epoxides are important functional groups in organic synthesis, acting as both as targets and as intermediates, which could produce bifunctional compounds through nucleophilic attack. Particularly, the field of epoxy carbonyl chemistry has seen substantial advancements over recent decades.[8,9] Nevertheless, there is limited works involving epoxy componds with two carbonyl groups.[10,11] Based on the above results, the compound of (3′-(2-bromophenyl)-2-phenyl-[2,2′-bioxiran]-3-yl)(phenyl)methanone was synthesized and its detailed crystal structural analysis was presented as follows.[12,13]

The asymmetric unit contains one molecule of the title compound. The bond lengths of Br1–C11, O1–C8 and C7–C6 are 1.888(6) Å, 1.430(7) Å, 1.364(8) Å, respectively. The bond angle of C9–O1–C8 and C1–O2–C16 is 61.7(3)°, 62.5(3)°, respectively, and the bond lengths and bond angles of the title molecule are within a reasonable range.[14–16] In addition, the bond angles in the phenyl ring is in the range of 117.4(6)°–121.7(6)°, and the bond angle at three membered ring (C8/O1/C9) substitutions site is the smallest case, indicating that the substitution on the benzene ring may reduce the bond angle. The dihedral angle between the C2\C3\C4\C5\C6\C7 plane and the C10\C11\C12\C13\C14\C15 plane is 74.5°, and the dihedral angle between the C10\C11\C12\C13\C14\C15 ring plane and the C18\C19\C20\C21\C22\C23 plane is 12.0°.

Conflict of interest: The authors declare no conflicts of interest regarding this article.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

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