

See discussions, stats, and author profiles for this publication at: <https://www.researchgate.net/publication/340018076>

# Structure and Conformational Analysis of 5,5-Bis(bromomethyl)-2,2-diphenyl-1,3-dioxane

Article in Russian Journal of Organic Chemistry · January 2020

DOI: 10.1134/S1070428020010017

CITATION

1

READS

19

8 authors, including:



R. A. Khalikov

Bashkir State Medical University

6 PUBLICATIONS 3 CITATIONS

SEE PROFILE



V. A. Kataev

43 PUBLICATIONS 50 CITATIONS

SEE PROFILE



Tatyana V. Tyumkina

Ufa Scientific Center of the Russian Academy of Science

103 PUBLICATIONS 464 CITATIONS

SEE PROFILE



Ekaterina S. Meshcheryakova

Russian Academy of Sciences

68 PUBLICATIONS 143 CITATIONS

SEE PROFILE

Some of the authors of this publication are also working on these related projects:



Conformational analysis of six member cyclic compounds [View project](#)



Oligomerization of alkenes, catalysed by metallocenes and post-metallocenes, in one-pot synthesis of practically important functionalized stereoregular derivatives [View project](#)

# Structure and Conformational Analysis of 5,5-Bis(bromomethyl)-2,2-diphenyl-1,3-dioxane

Sh. Yu. Khazhiev<sup>a</sup>, M. A. Khusainov<sup>b</sup>, R. A. Khalikov<sup>c</sup>, V. A. Kataev<sup>c</sup>, T. V. Tyumkina<sup>d</sup>,  
E. S. Meshcheryakova<sup>d</sup>, L. M. Khalilov<sup>d</sup>, and V. V. Kuznetsov<sup>b, e,\*</sup>

<sup>a</sup> Bashneft Public Corporation, Ufa, 450077 Russia

<sup>b</sup> Ufa State Petroleum Technological University, Ufa, 450062 Russia

<sup>c</sup> Bashkir State Medical University, Ufa, 450008 Russia

<sup>d</sup> Institute of Petrochemistry and Catalysis, Russian Academy of Sciences, Ufa, 450075 Russia

<sup>e</sup> Ufa State Aviation Technical University, Ufa, 450008 Russia

\*e-mail: kuzmaggy@mail.ru

Received June 4, 2019; revised November 19, 2019; accepted November 22, 2019

**Abstract**—The structure of 5,5-bis(bromomethyl)-2,2-diphenyl-1,3-dioxane was studied by <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy and X-ray analysis. Its molecules in crystal adopt a *chair* conformation, whereas equilibrium between energy-degenerate *chair* invertomers exists in solution at room temperature. According to the low-temperature NMR data, the interconversion barrier amounts to 8.9 kcal/mol. The ring inversion path was simulated by DFT quantum chemical calculations using PBE/3ξ and (in some cases) RI-MP2/λ2 approximations, and the potential barrier to interconversion in different solvents was estimated by the cluster model. The calculated geometric parameters of the title compound were consistent with the experimental X-ray diffraction data, and the calculated interconversion barrier matched a cluster containing 5 molecules of methylene chloride in the nearest solvation shell of the substituted 1,3-dioxane molecule.

**Keywords:** 1,3-dioxane, X-ray analysis, conformer, interconversion barrier, cluster model, computer simulation.

**DOI:** 10.1134/S1070428020010017

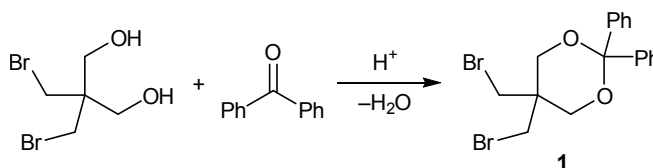
Substituted 1,3-dioxanes are classical models for conformational analysis; they are also used as reagents in fine organic synthesis [1–4]. Interest in 5,5-bis(halomethyl)-1,3-dioxanes is determined by the presence in their molecules of additional reaction centers, i.e., halogen atoms that can be replaced via nucleophilic substitution reactions [5]. The conformational behavior of 5,5-bis(halomethyl)-1,3-dioxanes at room temperature is characterized by fast (on the NMR time scale) inversion of the six-membered 1,3-dioxane ring [6, 7]; 2-substituted 5,5-bis(halomethyl)-1,3-dioxanes preferentially exist as *chair* conformers with the equatorial substituent on C<sup>2</sup> [8–11].

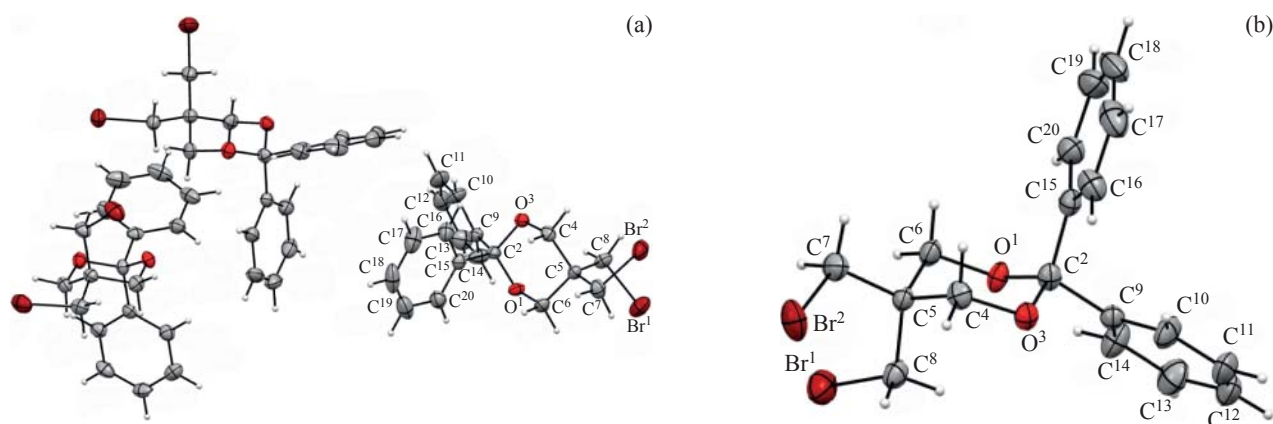
The present article reports the structure and conformational transformations of previously unknown 5,5-bis(bromomethyl)-2,2-diphenyl-1,3-dioxane (**1**), which were studied by <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy and X-ray analysis, as well as by DFT PBE/3ξ quantum chemical method (PRIRODA software [12]). This method showed good results for structurally related

compounds [11]. In addition, the effect of the solvent nature and the number of its molecules in the solvation shell on the relative energies of minima and transition state on the potential energy surface (PES) of compound **1** was analyzed in the framework of the cluster model. A sample of dioxane **1** was synthesized by condensation of 2,2-bis(bromomethyl)propane-1,3-diol with benzophenone (Scheme 1).

The X-ray diffraction data for a single crystal of **1** are given in Experimental and were deposited to the Cambridge Crystallographic Data Centre (CCDC entry no. 1880875). Compound **1** crystallizes in the non-centrosymmetric *P1* space group of the triclinic crystal

**Scheme 1.**





**Fig. 1.** (a) Asymmetric unit cell of 5,5-bis(bromomethyl)-2,2-diphenyl-1,3-dioxane (**1**) containing three independent molecules; (b) molecule **1** with non-hydrogen atoms shown as thermal vibration ellipsoids with a probability of 50%.

system with a unit cell containing three independent molecules with similar geometric parameters (Fig. 1, Table 1). Molecules **1** in crystal have a *chair* conformation. The heteroatom moiety of the 1,3-dioxane ring is characterized by expected C–O bond lengths (1.410–1.431 Å) and bond angles in the range 110–114°. The torsion angles are also consistent with those typical of *chair* conformers (Table 1) [2, 11]. It should be noted that the bromomethyl substituents are oriented *gauche* with respect to each other (Fig. 1); as shown previously by quantum chemical calculations [13], this structure is more energetically favorable than alternative conformations of carbon–halogen bonds in the substituents on C<sup>5</sup> of 1,3-dioxanes.

The data of 1D and 2D <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy (including NOESY, <sup>1</sup>H–<sup>1</sup>H COSY, and HSQC techniques; Table 2) indicated that cyclic ketal **1** in different solvents at room temperature exists as an equilibrium mixture of invertomers. This followed from the fact that methylene protons of the dibromomethyl substituents and on the magnetically equivalent C<sup>4</sup> and C<sup>6</sup> atoms resonated as singlets. In the low-tem-

perature <sup>1</sup>H NMR spectrum in CD<sub>2</sub>Cl<sub>2</sub>, the C<sup>4</sup>H<sub>2</sub> and C<sup>6</sup>H<sub>2</sub> protons appeared as a doublet with a coalescence temperature of 213 K. The corresponding signal splitting was observed in the <sup>13</sup>C NMR spectrum. On the basis of these data, the interconversion barrier was estimated at  $\Delta G_{298}^\ddagger = 8.9$  kcal/mol.

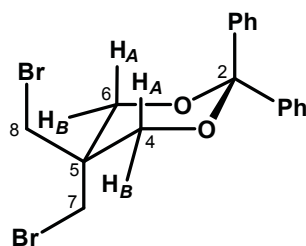
The experimental results were confirmed by conformational analysis of dioxane **1** in the PBE/3 $\zeta$  and (for the isolated molecule) RI-MP2/ $\lambda/2$  approximations. Two minima corresponding to *chair* (*C*) and 2,5-*twist* conformers (2,5-*T*), as well as transition state (TS) with a half-*chair* conformation, were localized on the potential energy surface (Scheme 2). The calculated bond length and bond and torsion angles for structure *C* were fairly similar to those found experimentally by X-ray analysis (Table 1).

Table 3 contains the relative energies of all species corresponding to stationary points on the PES for the isolated molecule of **1**, as well as for some clusters with several solvents. The differences in the calculated energies of *C* and 2,5-*T* in clusters with solvents (PBE/3 $\zeta$ ,  $\Delta G_{298}^0$ ) are larger than that found for the gas

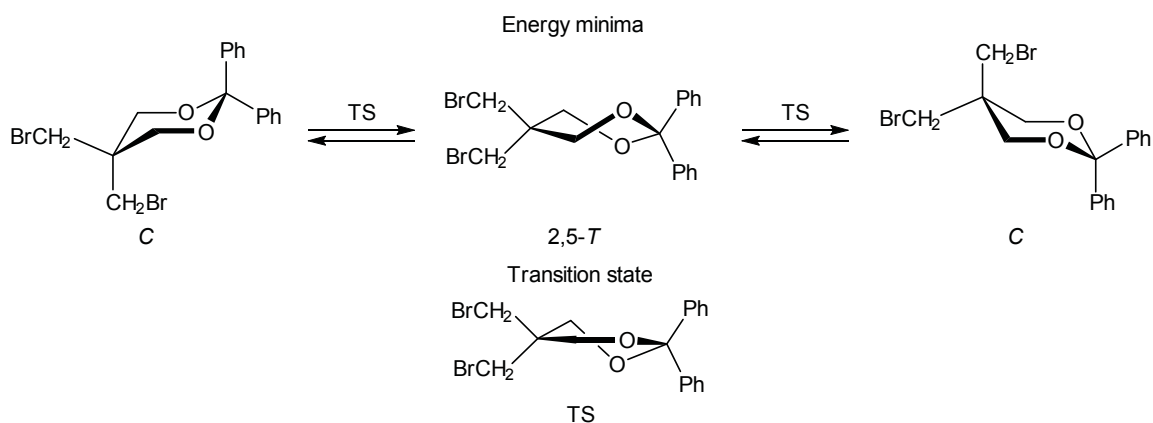
**Table 1.** Selected bond lengths and bond and torsion angles in the molecule of 5,5-bis(bromomethyl)-2,2-diphenyl-1,3-dioxane (**1**)<sup>a</sup>

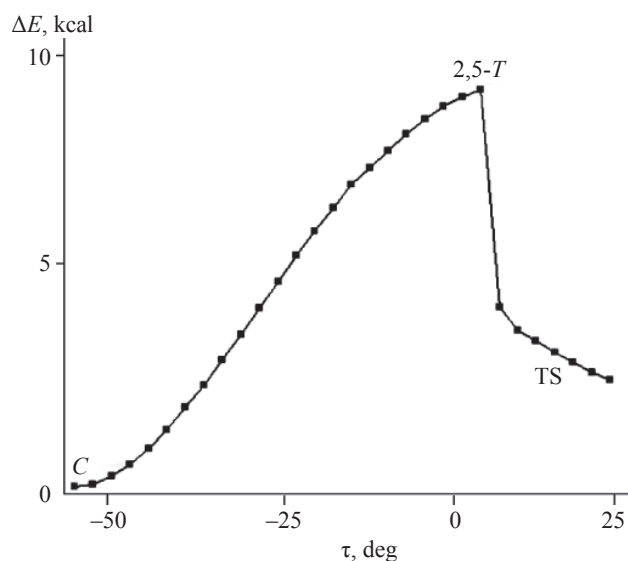
Bond	Bond length, Å		Bond angle	$\phi$ , deg		Torsion angle	$\tau$ , deg	
	calcd.	exptl.		calcd.	exptl.		calcd.	exptl.
Br <sup>1</sup> –C <sup>8</sup>	1.987	1.944(4)	O <sup>1</sup> C <sup>2</sup> O <sup>3</sup>	110.6	110.3(3)	C <sup>4</sup> C <sup>5</sup> C <sup>6</sup> O <sup>1</sup>	54.9	55.3(4)
C <sup>2</sup> –O <sup>1</sup>	1.432	1.410(4)	C <sup>2</sup> O <sup>1</sup> C <sup>6</sup>	113.5	114.0(3)	C <sup>6</sup> C <sup>5</sup> C <sup>4</sup> O <sup>3</sup>	–54.2	–55.3(4)
C <sup>2</sup> –O <sup>3</sup>	1.432	1.431(4)	C <sup>2</sup> O <sup>3</sup> C <sup>4</sup>	113.7	114.1(2)	C <sup>2</sup> O <sup>3</sup> C <sup>4</sup> C <sup>5</sup>	56.9	58.1(4)
C <sup>4</sup> –O <sup>3</sup>	1.432	1.420(4)	C <sup>5</sup> C <sup>6</sup> O <sup>1</sup>	111.2	110.4(3)	C <sup>2</sup> O <sup>1</sup> C <sup>6</sup> C <sup>5</sup>	–58.7	–57.3(4)
C <sup>2</sup> –C <sup>9</sup>	1.529	1.516(5)	C <sup>5</sup> C <sup>4</sup> O <sup>3</sup>	111.9	111.7(3)	C <sup>4</sup> O <sup>3</sup> C <sup>2</sup> O <sup>1</sup>	–55.3	–56.1(4)
C <sup>5</sup> –C <sup>8</sup>	1.529	1.511(5)	C <sup>4</sup> C <sup>5</sup> C <sup>6</sup>	105.2	104.8(3)	C <sup>6</sup> O <sup>1</sup> C <sup>2</sup> O <sup>3</sup>	56.4	55.5(4)

<sup>a</sup> Calculations were performed for the *chair* conformer in the gas phase using PBE/3 $\zeta$  approximation.

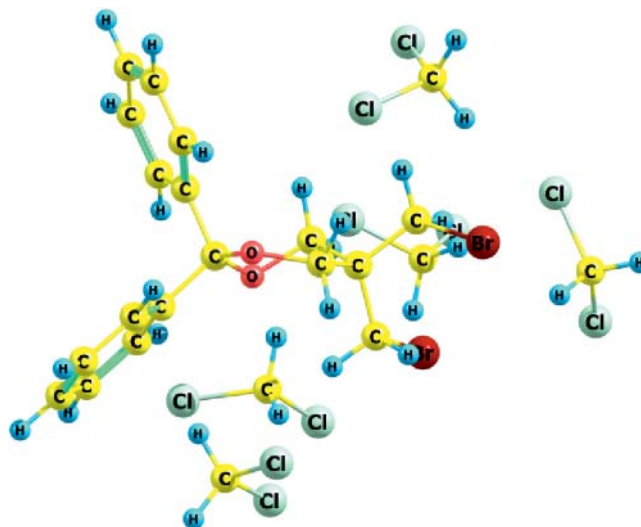
**Table 2.**  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of 5,5-bis(bromomethyl)-2,2-diphenyl-1,3-dioxane (**1**)

Solvent	Proton	$\delta$ , ppm	Carbon atom	$\delta_{\text{C}}$ , ppm
$\text{CDCl}_3$	$\text{CH}_2\text{Br}$	3.6 s	$\text{C}^2$	101.8
	$\text{CH}_2\text{O}$	4.0 s	$\text{C}^4, \text{C}^6$	66.5
	$\text{C}_6\text{H}_5$	7.3–7.6 m	$\text{C}^5$	38.0
			$\text{C}^7, \text{C}^8$	35.5
			$\text{C}_{\text{arom}}$	126.4–140.9
$\text{CDCl}_2$ , 298 K	$\text{CH}_2\text{Br}$	3.6 s	$\text{C}^2$	101.6
	$\text{CH}_2\text{O}$	4.0 s	$\text{C}^4, \text{C}^6$	66.4
	$\text{C}_6\text{H}_5$	7.3–7.5 m	$\text{C}^5$	37.9
			$\text{C}^7, \text{C}^8$	35.6
			$\text{C}_{\text{arom}}$	126.2–141.2
$\text{CDCl}_2$ , 213 K	$\text{CH}_2\text{Br}$	3.6 d	$\text{C}^2$	101.2
	$\text{CH}_2\text{O}$	3.9 s	$\text{C}^4, \text{C}^6$	66.2
	$\text{C}_6\text{H}_5$	7.3–7.5 m	$\text{C}^5$	37.9
			$\text{C}^7, \text{C}^8$	35.3, 36.7
			$\text{C}_{\text{arom}}$	124.6–128.3
$\text{DMSO}-d_6$	$\text{CH}_2\text{Br}$	3.4 s	$\text{C}^2$	100.9
	$\text{CH}_2\text{O}$	3.9 s	$\text{C}^4, \text{C}^6$	66.6
	$\text{C}_6\text{H}_5$	7.3–7.5 m	$\text{C}^5$	37.3
			$\text{C}^7, \text{C}^8$	36.2
			$\text{C}_{\text{arom}}$	125.9–141.1
$\text{C}_6\text{D}_6$	$\text{CH}_2\text{Br}$	3.1 s	$\text{C}^2$	101.9
	$\text{CH}_2\text{O}$	3.6 s	$\text{C}^4, \text{C}^6$	66.2
	$\text{C}_6\text{H}_5$	7.0–7.5 m	$\text{C}^5$	37.8
			$\text{C}^7, \text{C}^8$	35.5
			$\text{C}_{\text{arom}}$	126.6–141.9

**Scheme 2.**



**Fig. 2.** Plot of the energy of the cluster  $1@5\text{CH}_2\text{Cl}_2$  versus torsion angle  $\text{O}^3\text{C}^4\text{C}^5\text{C}^6$  at 0 K.



**Fig. 3.** Transition state for the interconversion of the cluster  $1@5\text{CH}_2\text{Cl}_2$ .

phase. The barrier to interconversion ( $\Delta G_{298}^\ddagger$ ) also increases in going to cluster models. The calculated and experimental  $\Delta G_{298}^\ddagger$  values for the cluster  $1@5\text{CH}_2\text{Cl}_2$  are fairly similar (8.9 and 8.33 kcal/mol, respectively). Figure 2 shows the energy of  $1@5\text{CH}_2\text{Cl}_2$  plotted against the torsion angle  $\text{O}^3\text{C}^4\text{C}^5\text{C}^6$ . Thus, it is highly

likely that the first solvation shell includes 5–6 methylene chloride molecules (Fig. 3). We previously showed that a probable number of solvent molecules in clusters with saturated heterocyclic molecules such as 1,3-dioxanes [14] and tetrahydro-1,3-oxazines [15] should not exceed 10 under standard conditions. Ac-

**Table 3.** Energy parameters of conformational transformations of 5,5-bis(bromomethyl)-2,2-diphenyl-1,3-dioxane (**1**), calculated by the PBE/3 $\zeta$  method

Solvent	Conformer	$-E_0$ , <sup>a</sup> hartree	$\Delta E_0^0$ ( $\Delta E_0^\ddagger$ ), kcal/mol	$\Delta H_{298}^0$ ( $\Delta H_{298}^\ddagger$ ), kcal/mol	$\Delta G_{298}^0$ ( $\Delta G_{298}^\ddagger$ ), kcal/mol	$\Delta S_{298}^0$ ( $\Delta S_{298}^\ddagger$ ), kal mol <sup>-1</sup> K <sup>-1</sup>
–	<i>C</i>	5993.778232	0	0	0	0
–	<i>2,5-T</i>	5993.776440	1.12	1.21	0.41	2.67
–	(TS)	5993.766142	(7.59)	(7.37)	(7.06)	(1.04)
–	<i>C</i> <sup>b</sup>	5990.487591	0	0	0	0
–	<i>2,5-T</i> <sup>b</sup>	5990.484313	2.06	2.14	1.23	3.04
–	(TS) <sup>b</sup>	5990.472571	(9.04)	(9.22)	(8.60)	(2.10)
5 $\text{CHCl}_3$	<i>C</i>	13087.561564	0	0	0	0
	<i>2,5-T</i>	13087.560206	0.85	0.91	0.78	0.43
	(TS)	13087.550008	(7.25)	(6.98)	(7.72)	(–2.48)
5 $\text{CH}_2\text{Cl}_2$	<i>C</i>	10790.348269	0	0	0	0
	<i>2,5-T</i>	10790.344978	2.07	2.16	0.86	4.38
	(TS)	10790.335995	(7.70)	(7.41)	(8.33)	(–3.07)
10 $\text{CH}_2\text{Cl}_2$	<i>C</i>	15586.918992	0	0	0	0
	<i>2,5-T</i>	15586.916788	1.38	1.38	1.78	–1.37
	(TS)	15586.906675	(7.73)	(7.31)	(9.67)	(–7.91)
5 $\text{C}_6\text{H}_6$	<i>C</i>	7153.408643	0	0	0	0
	<i>2,5-T</i>	7153.406458	1.37	1.36	2.45	–3.67
	(TS)	7153.394712	(8.74)	(8.36)	(10.57)	(–7.44)

<sup>a</sup> With a correction for zero-point vibrational energy.

<sup>b</sup> RI-MP2/ $\lambda$ 2 calculation.

cording to both calculated (Table 3) and experimental data, there is some effect of the solvent on the  $\Delta G_{298}^\ddagger$  value. In particular, the  $\Delta G_{298}^\ddagger$  value for the closest analog of **1**, 5,5-dimethyl-2,2-diphenyl-1,3-dioxane, in carbon disulfide solution is 9.6 kcal/mol [2].

It should also be noted that no intermediate minimum (1,4-*twist* conformer) typical of conformational equilibria of unsubstituted and 2-, 4-, 5-, 2,5-, and 4,4-substituted 1,3-dioxanes [11] was localized on the PES of compound **1**.

## EXPERIMENTAL

The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a Bruker Avance 400 spectrometer (Germany) at 400.13 and 100.62 MHz, respectively, using  $\text{CDCl}_3$ ,  $\text{CD}_2\text{Cl}_2$ ,  $\text{DMSO}-d_6$ , and  $\text{C}_6\text{D}_6$  as solvents; the chemical shifts were measured relative to residual proton and carbon signals of the solvent. All solvents for NMR experiments had a purity of 99.5–99.8% (Sigma–Aldrich). The X-ray diffraction data for compound **1** were obtained at 293(2) K on an XCalibur Eos automated four-circle diffractometer (Mo  $K_\alpha$  radiation,  $\lambda$  0.71073 Å; graphite monochromator;  $\omega$ -scanning,  $2\theta_{\text{max}} = 62^\circ$ ). The data were acquired and processed using CrysAlis<sup>Pro</sup> version 1.171.36.20 (Oxford Diffraction). The structure was solved by the direct method and was refined by the full-matrix least-squares method in anisotropic approximation for non-hydrogen atoms. Hydrogen atoms were localized by the difference Fourier synthesis and were refined in isotropic approximation. The calculations were performed using SHELX97 [16]. Triclinic crystal system, space group  $P\bar{1}$ ; unit cell parameters:  $a = 9.8063(5)$  Å,  $b = 11.4309(4)$  Å,  $c = 23.4002(15)$  Å;  $\alpha = 90.864(4)$ ,  $\beta = 91.837(4)$ ,  $\gamma = 102.439(4)^\circ$ ;  $V = 2559.5(2)$  Å<sup>3</sup>;  $Z = 6$ ;  $\rho_{\text{calc}} = 1.659$  mg/mm<sup>3</sup>;  $\mu = 4.755$  mm<sup>−1</sup>;  $F(000) = 1272.0$ ; scan range  $4.54 < \theta < 58.52^\circ$ ;  $-10 \leq h \leq 12$ ,  $-13 \leq k \leq 15$ ,  $-27 \leq l \leq 29$ ; number of independent reflections 11 759 ( $R_{\text{int}} = 0.0289$ ); goodness of fit  $S = 0.976$ ; final divergence factors:  $R_1 = 0.0477$ ,  $wR_2 = 0.0788$  [reflections with  $I_{hkl} > 2\sigma(I)$ ];  $R_1 = 0.1170$ ,  $wR_2 = 0.0969$  (all independent reflections); maximum and minimum residual electron density peaks  $\Delta\rho_{\text{min/max}} 0.66/-0.74$  e/Å<sup>3</sup>.

The geometric parameters of the *chair* conformer of **1** were preliminarily optimized by the AM1 method (HyperChem 8.0) [17] and then by the PBE/3 $\zeta$  method, as well as in the RI-MP2/ $\lambda/2$  approximation (PRIRODA [12]) for the isolated molecule. The ring inversion was simulated by scanning the CCCO torsion angle in the

range from  $-60$  to  $25^\circ$  (Fig. 2). The potential barriers were determined by the transition state search algorithm implemented in PRIRODA. Stationary points on the PES were identified as transition states by the presence of one imaginary frequency in the corresponding Hessian matrix, and as minima, by the absence of imaginary frequencies therein. Model clusters were built up by successively placing solvent molecules in the vicinity of molecule **1** using HyperChem, and the resulting structures were then optimized in the PBE/3 $\zeta$  approximation.

**5,5-Bis(bromomethyl)-2,2-diphenyl-1,3-dioxane (1).** An equimolar mixture of 2,2-bis(bromomethyl)-propane-1,3-diol (0.02 mol, 5.24 g; Sigma–Aldrich) and benzaldehyde (3.64 g) in benzene (50 mL) containing *p*-toluenesulfonic acid (0.1 g) was heated under reflux in a flask equipped with a Dean–Stark trap until water no longer separated. The mixture was cooled and washed with 5% aqueous sodium hydrogen carbonate (10 mL) and water (2×10 mL), the solvent was distilled off, and the residue was recrystallized twice from 95% ethanol. Yield 0.85 g (10%), mp 78–79°C.

## ACKNOWLEDGMENTS

Structural studies were carried out at the Agidel joint center (Institute of Petrochemistry and Catalysis, Russian Academy of Sciences).

## FUNDING

This study was performed under financial support by the Ministry of Science and Higher Education of the Russian Federation (project no. 16.1969.2017/4.6).

## CONFLICT OF INTEREST

The authors declare the absence of conflict of interest.

## REFERENCES

1. Rakhmankulov, D.L., Karakhanov, R.A., Zlotskii, S.S., Kantor, E.A., Imashev, U.B., and Syrkin, A.M., *Itogi Nauki Tekh., Ser.: Tekhnol. Org. Veshchestv*, 1979, no. 5, p. 6.
2. *Internal Rotation in Molecules*, Orville-Thomas, W.J., Ed., London: Wiley, 1974.
3. Kuznetsov, V.V., *Chem. Heterocycl. Compd.*, 2006, vol. 42, p. 559.  
<https://doi.org/10.1007/s10593-006-0127-x>
4. Kuznetsov, V.V., *Russ. Chem. Bull., Int. Ed.*, 2005, vol. 54, p. 1543.  
<https://doi.org/10.1007/s11172-006-0001-0>



5. Kurmaeva, E.S., Chalova, O.B., Chistoedova, G.I., Lapuka, L.F., Kiladze, T.K., Kantor, E.A., and Rakhmankulov, D.L., *Zh. Org. Khim.*, 1985, vol. 21, p. 131.
6. Bochkor, S.A., Lapuka, L.F., Kurmaeva, E.S., Chalova, O.B., Zlotskii, S.S., and Rakhmankulov, D.L., *Chem. Heterocycl. Compd.*, 1987, vol. 23, p. 500.  
<https://doi.org/10.1007/BF00476374>
7. Khazhiev, Sh.Yu., Khusainov, M.A., and Kantor, E.A., *Russ. J. Gen. Chem.*, 2011, vol. 81, p. 153.  
<https://doi.org/10.1134/S1070363211010282>
8. Khazhiev, Sh.Yu., Khusainov, M.A., Khalikov, R.A., Tyumkina, T.V., Meshcheryakova, E.S., Khalilov, L.M., and Kuznetsov, V.V., *Russ. J. Org. Chem.*, 2018, vol. 54, p. 1076.  
<https://doi.org/10.1134/S1070428018070175>
9. Khazhiev, Sh.Yu., Khusainov, M.A., Khalikov, R.A., Tyumkina, T.V., Meshcheryakova, E.S., Khalilov, L.M., and Kuznetsov, V.V., *Russ. J. Gen. Chem.*, 2018, vol. 88, p. 397.  
<https://doi.org/10.1134/S1070363218030040>
10. Khazhiev, Sh.Yu., Khusainov, M.A., Khalikov, R.A., Tyumkina, T.V., Meshcheryakova, E.S., Khalilov, L.M., and Kuznetsov, V.V., *Russ. J. Gen. Chem.*, 2019, vol. 89, p. 199.  
<https://doi.org/10.1134/S0044460X19020057>
11. Kuznetsov, V.V., *Russ. J. Org. Chem.*, 2014, vol. 50, p. 1227.  
<https://doi.org/10.1134/S1070428014090012>
12. Laikov, D.N. and Ustynyuk, Yu.A., *Russ. Chem. Bull., Int. Ed.*, 2005, vol. 54, p. 820.  
<https://doi.org/10.1007/s11172-005-0329-x>
13. Khazhiev, Sh.Yu., Khusainov, M.A., and Kantor, E.A., *Russ. J. Org. Chem.*, 2011, vol. 47, p. 450.  
<https://doi.org/10.1134/S1070428011030225>
14. Raskildina, G.Z., Spirikhin, L.V., Zlotskij, S.S., and Kuznetsov, V.V., *Russ. J. Org. Chem.*, 2019, vol. 55, p. 502.  
<https://doi.org/10.1134/S107042801904014>
15. Kuznetsov, V.V., *J. Struct. Chem.*, 2018, vol. 59, p. 1374.  
<https://doi.org/10.26902/JSC20180617>
16. Sheldrick, G.M., *Acta Crystallogr., Sect. A*, 2008, vol. 64, p. 112.  
<https://doi.org/10.1107/S0108767307043930>
17. HyperChem 8.0. <http://www.hyper.com>