New reaction of glycidols with oxalyl chloride and phosgene — an approach to cyclic esters

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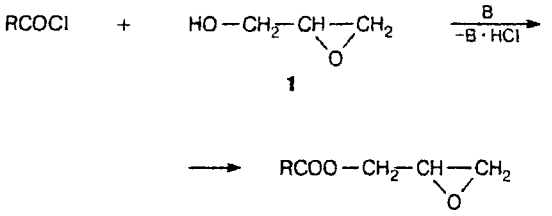
2,3-Epoxy alcohols (glycidols) react with carboxylic acid dichlorides to form cyclic esters of 3-chloro-1,2-diols. The reaction proceeds with complete retention of the configuration of the C(2) atom of the initial glycidol and with predominant (but not complete) inversion of the configuration of the C(3) atom in the final heterocycle.

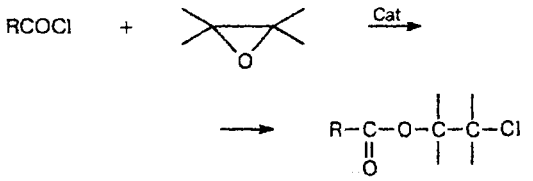
Key words: epoxyalcohols, oxalyl chloride, phosgene, cyclic esters, stereochemistry.

Glycidols and related 2,3-epoxy alcohols are widely used in organic synthesis.[1] These compounds assumed much greater importance when Sharpless asymmetric epoxidation[2] received wide acceptance. This procedure allows one to reliably prepare scalemic (*i.e.*, enantiomerically enriched or enantiomerically pure[3]) glycidols with a specified configuration.

Carboxylic acid chlorides can react with glycidols at the alcoholic functional group to form esters.[4]



At the same time, these reagents can add to the epoxide ring, which is accompanied by opening of the latter to give esters of β-chloro-substituted alcohols.[5,6]



In the case of monosubstituted epoxides, the chlorine atom is located predominantly at the terminal carbon atom. In the case of disubstituted oxiranes, mixtures of regioisomers are formed.[6]

Reactions of oxalyl chloride (2) with glycidols have not been studied. Although these reagents, formally, are mixed in the course of Swern oxidation of glycidols[7–9] with the use of $(COCl)_2$ as an activating agent for DMSO, free oxalyl chloride is absent in the reaction mixture at the instant a hydroxyl component (glycidol) is added.[10]

We demonstrated that the reaction of oxalyl chloride with a twofold excess of glycidol in the presence of two equivalents of triethylamine afforded diglycidyl oxalate (3), which has been prepared previously in a similar yield by transesterification of diethyl oxalate with glycidol catalyzed by thallium acetate.[11] When the reaction was performed with the use of racemic glycidol **1** (in our study as well as in the study reported previously[11]), diglycidyl oxalate was obtained as a mixture of D,L- and *meso* diastereomers. In the case of geminal superposition of two glycidyl residues, as in the case of diglycidylalkoxymethanes, this stereoisomerism is manifested[12] as the complication of the ^1H and ^{13}C NMR spectra. On the contrary, no complications are observed in the routine spectra of diglycidyl oxalate, and the proton resonance spectra of compound **3** closely resemble those of glycidyl acetate.

When the reagent ratio was changed (**1** : **2** = 1 : 1, in the absence of a base), glycidyl-containing compounds were absent among the products detected in the reaction mixture by ^1H NMR spectroscopy. Apparently, side reactions, which were accompanied by the epoxide ring opening, proceeded under these conditions.

When reagents **1** and **2** were mixed upon cooling in the presence of a base (**1** : **2** : Et_3N = 1 : 1 : 1), the ^1H NMR spectrum of the reaction mixture in benzene, which was measured after separation of a precipitate of $Et_3N \cdot HCl$, had multiplets at δ 2.50—2.90 and 2.95—3.40 and two doublets at δ 3.67 (J = 12 and 6 Hz) and 4.03 (J = 12 and 3 Hz). Their structures being characteristic of esters of glycidol allows one to assign these

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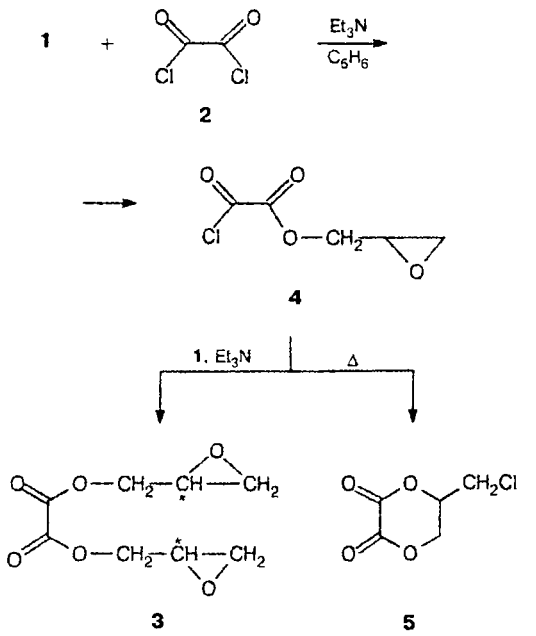
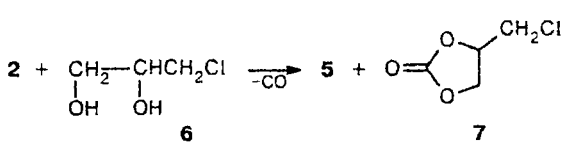
signals to glycidyl chlorooxalate (**4**). Brief heating of the reaction mixture afforded 5-chloromethyl-1,4-dioxane-2,3-dione (**5**) in nearly quantitative yield.

Scheme 1



The formation of 5-substituted 1,4-dioxane-2,3-diones in the reaction of compound **2** with vicinal diols has been studied previously.[13] It was demonstrated that in all cases, the closure of the six-membered ring was accompanied by expulsion of CO to give mixtures of the expected dioxanediones and cyclic carbonates as the final reaction products. Attempts to isolate products in individual form were complicated by decomposition both in the course of distillation and chromatography on silica gel.

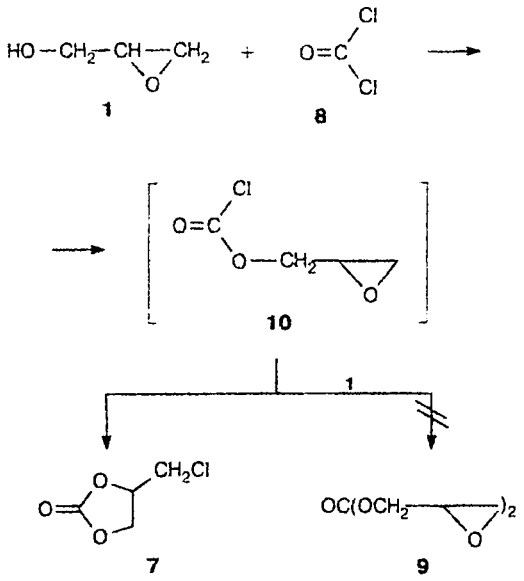
We performed the reaction of compound **2** with 3-chloropropane-1,2-diol (**6**) under conditions proposed previously.[13] After separation of a precipitate of $Et_3N \cdot HCl$, the reaction mixture was analyzed by ^1H and ^{13}C NMR spectroscopy. We detected dioxanedione **5** (about 60—70%), cyclic carbonate of diol (5—10%), and insignificant amounts of oligomeric oxalates of diol **6** among the reaction products.



Therefore, from the preparative standpoint, the reaction of oxalyl chloride with glycidol proceeds more selectively than that with diol and gives a product of higher purity.

We failed to obtain the product of the double replacement of the chlorine atoms in the phosgene molecule (**8**) by the glycidyl residues, *viz.*, diglycidyl carbonate (**9**), by passing gaseous phosgene through an excess of glycidol in a mixture with triethylamine, by adding a solution of phosgene in dichloromethane dropwise to an analogous mixture, or by mixing the reagents in the reverse order. In all cases as well as when liquid phosgene was mixed with glycidol **1** in dichloromethane in the absence of a base, 1,3-dioxolane **7** was the only product isolated.

Scheme 2



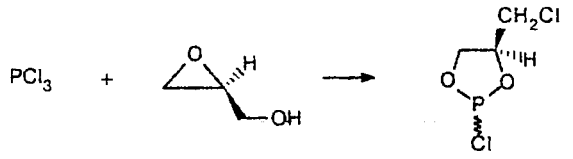
It is known that in the absence of a catalyst[14] or in the presence of guanidinium salts,[6] phosgene adds only one epoxide molecule and the reaction is terminated at the stage of formation of the corresponding chloroformate. To prepare bisadducts of phosgene or to involve chloroformates, as such, in the reaction of epoxide opening, it is necessary to use other catalysts[15] or to perform the reaction under substantially more drastic conditions (120 °C, 8 h).[6] Under the conditions used in this study (−70 to 0 °C), the reaction of phosgene with glycidol would be expected to be terminated at the stage of formation of glycidyl chloroformate (**10**). However, we did not detect the latter compound. Moreover, the high yield of cyclization product **7** in the presence of an excess of free alcohol **1** suggests that the intramolecular addition of the OC(O)Cl fragment to the oxirane ring of intermediate **10** is more favorable than the replacement of the C—Cl bond in the OC(O)Cl group by the oxy function.

The formation of the only five-membered carbonate **7** (racemic in the case of the use of racemic glycidol) in the reaction of glycidol with phosgene is indicative of

the high regioselectivity of the epoxide ring opening in the reaction conditions under consideration. To reveal its stereochemical features, we studied the reaction of phosgene with scalemic 3-substituted glycidol, namely, with (2*S*,3*S*)-2,3-epoxy-3-phenylpropan-1-ol (**11**). This alcohol was chosen for two reasons. First, it can be prepared from readily available cinnamyl alcohol by Sharpless epoxidation.[16] Second, alcohol **11** can be obtained in 100% enantiomeric purity by simple recrystallization.[17]

It is of great importance that in the reaction of phosgene with compound **11**, the epoxide ring opening can take two pathways. The attack of activated chlorine on the C(3) atom of the epoxide ring should afford five-membered carbonates **12** containing the exocyclic chlorobenzyl fragment, while the attack on the C(2) atom should give six-membered cyclic carbonates, *viz.*, 5-chloro-4-phenyl-1,3-dioxan-2-ones (**13**). The ^{13}C NMR spectrum of the distilled reaction mixture, which was obtained by the reaction of compound **8** with **11**, has signals of two major products in a ratio of 7 : 1. The chemical shifts of the related carbon atoms in these compounds differ by no more than 0.3 ppm, which allows one to unambiguously assign a common chemical structure to these compounds. The structures differ in details of spatial organization. Treatment of the reaction mixture with ether made it possible to isolate more than 65% of the major product in the crystalline state. X-ray diffraction study of this product confirmed that the adduct is a five-membered carbonate. The absolute configuration of the molecule was determined by studying anomalous scattering by the crystal, and the major isomer was identified as (4*S*)-4-((1*R*)-1-chloromethyl-1-phenyl)-1,3-dioxolan-2-one (**12a**). The molecular structure of **12a** and the atomic numbering scheme are shown in Fig. 1.

The (*S*) configuration of the C(4) atom in the carbonate ring suggests the retention of the configuration of the C(2) atom in the initial molecule **11**. Previously,[8] we have reported the retention of the configuration of this atom in the similar reaction of (*S*)-glycidol with PCl_3.



Consequently, the second diastereomeric reaction product **12b** should differ only in the configuration of the exocyclic C(6) atom and has the structure (4*S*)-4-((1*S*)-1-chloromethyl-1-phenyl)-1,3-dioxolan-2-one.

Analysis of the ^{13}C NMR spectra of mother liquors enriched with minor components after separation of the major amount of **12a** revealed very weak signals, which we assigned to structural isomer **13** (at δC: 65.0

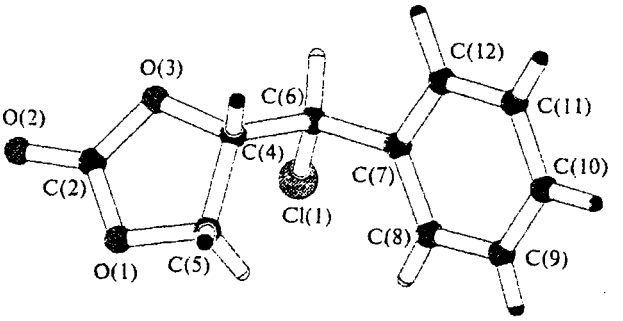
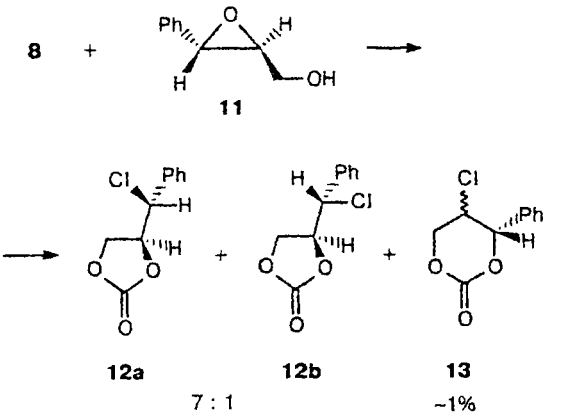


Fig. 1. Three-dimensional structure of molecule **12a**.

(CH_2—O); 72.3 (CH—Cl); 79.2 (CH—O); 155.5 (C=O); signals of the phenyl fragment: 126.2 (*m*-CH), 129.1 (*o*-CH), and 137.9 (*i*-CH); the signal of *p*-CH overlaps with the signals of the major products), along with signals of dioxolanones **12**. The total amount of product **13** was less than 1%, which, on the one hand, does not allow one to discuss its stereoisomerism and, on the other hand, indicates that the reaction under consideration is also highly regioselective in the case of disubstituted glycidols.

Scheme 3



It should be noted that the regioselective addition of hydrogen halides to disubstituted oxiranes is not a simple task. In particular, the regioselective addition of the chloride anion to the C(3) atom of epoxycinnamyl alcohol (glycidol **11** and its racemic modification) can be attained only with the use of complex lithium and/or titanium chlorides[19] or under the action of Et_2AlCl.[20] In the latter study, it was mentioned that 3-chloro-3-phenylpropane-1,2-diol was formed from compound **11** as a mixture of the *erythro* and *threo* isomers in a ratio of approximately 7 : 3. In the case under consideration, a somewhat higher degree of stereoselectivity of the addition of the chloride anion to the oxirane ring can be achieved with the use of inexpensive and readily

available phosgene. An additional advantage is the fact that the pure *erythro* isomer of chloroglycol can be readily isolated from the reaction mixture as crystalline carbonate **12a**.

Experimental

The IR spectra were recorded on a UR-20 spectrometer in Nujol mulls for solid samples or in thin films for liquids. The NMR spectra were obtained on Varian T-60 (^1H, 60 MHz) and Bruker MSL-400 (^{13}C, 100.6 MHz) spectrometers with Me_4Si as the internal standard. The optical rotation was measured on a Polamat A polarimeter. The solvents were dried according to standard procedures.

Di(2,3-epoxypropyl) oxalate (3). A solution of oxalyl chloride (**2**) (2.35 g, 18.5 mmol) in CH_2Cl_2 (5 mL) was added dropwise to a solution of glycidol (**1**) (2.75 g, 37.0 mmol) and Et_3N (3.75 g, 37.0 mmol) in dry CH_2Cl_2 (10 mL) cooled to −45 °C. Then the temperature of the reaction mixture was increased to −20 °C and the precipitate of $Et_3N \cdot HCl$ that formed was filtered off. Highly volatile components and the solvent were evaporated *in vacuo*. The solid residue was purified by recrystallization from $CHCl_3$. Compound **3** was obtained in a yield of 4.8 g (65%), m.p. 104—105 °C (*cf.* Ref. 11: 104 °C). ^1H NMR ($CHCl_3$), δ: 2.58—2.93 (m, 2 H, CH_2 of oxirane); 3.07—3.40 (m, 1 H, CH of oxirane); 4.07 (dd, 1 H, CH_aH_b, J = 12 and 6.2 Hz); 4.50 (dd, 1 H, CH_aH_b, J = 12 and 2.8 Hz). ^{13}C NMR ($CHCl_3$), δ: 44.56 (t, CH_2 of oxirane, J = 177.1 Hz); 48.41 (d, CH of oxirane, J = 179.1 Hz); 67.14 (t, OCH_2, J = 149.9 Hz); 156.90 (s, C=O). IR, ν/cm^{-1}: 750, 960, 1260 (oxirane ring); 3040 (CH_2 of oxirane); 1740 (C=O).

5-Chloromethyl-1,4-dioxane-2,3-dione (5). A mixture of compound **1** (1.28 g, 17.4 mmol) and Et_3N (1.8 g, 17.5 mmol) in benzene (8 mL) was added dropwise to a solution of compound **2** (2.21 g, 17.4 mmol) in dry benzene (10 mL) cooled in an ice bath under an inert atmosphere. Then the temperature of the reaction mixture was increased to −20 °C and the precipitate of $Et_3N \cdot HCl$ that formed was filtered off under an inert atmosphere. The filtrate was refluxed for 1.5 h and highly volatile components were evaporated *in vacuo*. Glassy compound **5** was obtained in a yield of 2.65 g (93%). ^1H NMR ($(CD_3)_2CO$), δ: 4.00 (br.d, 2 H, J = 5.4 Hz, CH_2Cl); 4.63—4.77 (m, 2 H, OCH_2); 5.53—5.73 (m, 1 H, OCH). ^{13}C NMR ($(CH_3)_2CO$), δ: 42.35 (t, CH_2Cl, J = 147.5 Hz); 64.80 (t, OCH_2, J = 154.0 Hz); 73.83 (d, OCH, J = 143.4 Hz); 156.68 (s, C=O); 158.13 (s, C=O). IR, ν/cm^{-1}: 1760 (C=O).

4-Chloromethyl-1,3-dioxolan-2-one (7). A mixture of compound **1** (5.9 g, 79.5 mmol) and Et_3N (8.1 g, 80 mmol) in CH_2Cl_2 (5 mL) was added dropwise with cooling to −78 °C to a solution of phosgene (7.9 g, 80 mmol) in dry CH_2Cl_2 (25 mL). Then the temperature of the reaction mixture was slowly increased to −20 °C. The precipitate of $Et_3N \cdot HCl$ that formed was filtered off, the solvent was evaporated, and the reaction product was purified by vacuum distillation. Compound **7** was obtained in a yield of 6.76 g (65%). The analogous reaction in the absence of Et_3N afforded compound **7** in a yield of 7.57 g (73%). B.p. 153—155 °C (23 Torr) (*cf.* Ref. 21: 175 °C (25 Torr)), n_D^{20} 1.4682 (*cf.* Ref. 21: 1.4688). ^1H NMR ($CHCl_3$), δ: 3.57 (br.d, J = 4.6 Hz, 2 H, CH_2Cl); 4.07—4.53 (m, 2 H, OCH_2); 4.63—4.97 (m, 1 H, OCH). ^{13}C NMR ($CHCl_3$), δ: 44.67 (t, CH_2Cl, J = 153.0 Hz); 67.04 (t, OCH_2, J = 159.3 Hz); 74.86 (d, OCH, J = 158.0 Hz) 154.68 (s, C=O). IR, ν/cm^{-1}: 1800 (C=O).

(2*S*,3*S*)-2,3-Epoxy-3-phenylpropan-1-ol (11) was prepared from cinnamyl alcohol according to a procedure reported previously.[16] M.p. 51 °C, $[\alpha]_D^{25}$ −50.5° (*c* 1.5, $CHCl_3$) (*cf.* Ref. 17: m.p. 50—51 °C, $[\alpha]_D^{20}$ −50.4° (*c* 2.4, $CHCl_3$)).

Reaction of 11 with phosgene. A solution of epoxyalcohol **11** (1.5 g, 10 mmol) in CH_2Cl_2 (5 mL) was added dropwise to a solution of phosgene (3.7 g, 37.5 mmol) in dry CH_2Cl_2 (10 mL) under an inert atmosphere at −70 °C. The reaction mixture was stirred at −70 °C for 5 h and then the temperature was gradually increased to −20 °C. After evaporation of highly volatile components *in vacuo*, the resulting product was purified by distillation and a mixture of isomeric 4-(α-chlorobenzyl)-1,3-dioxolan-2-ones (**12**) was isolated as a viscous oil in a yield of 0.7 g (33%), b.p. 120 °C (0.01 Torr). ^1H NMR ($CDCl_3$), δ: 4.37—4.86 (m, 2 H); 5.07—5.45 (m, 2 H); 7.20—7.43 (m, 5 H, Ph). ^{13}C NMR (CH_2Cl_2), δ: 62.22 (d, CHClPh, J = 153.5 Hz), 66.88 (t, OCH_2, **12b**, J = 155.6 Hz), 67.18 (t, OCH_2, **12a**, J = 152.1 Hz), 78.20 (d, OCH, **12a**, J = 158.7 Hz), 78.52 (d, OCH, **12b**, J = 159.3 Hz), 127.82 (d, *o*-CH, **12a**, J = 157.8 Hz), 128.15 (d, *o*-CH, **12b**, J = 165.2 Hz), 129.32 (d, *m*-CH, **12a**, J = 162.2 Hz), 129.36 (d, *m*-CH, **12b**, J = 158.4 Hz), 129.82 (d, *p*-CH, **12a**, J = 167.8 Hz), 129.88 (d, *p*-CH, **12b**, J = 167.2 Hz), 135.3 (s, *i*-CH, **12a**), 135.49 (s, *i*-CH, **12b**), 154.08 (s, C=O, **12a**), 154.14 (s, C=O, **12b**). Crystallization of **12** from Et_2O afforded (4*S*)-4-((1*R*)-1-chloromethyl-1-phenyl)-1,3-dioxolan-2-one (**12a**) in a yield of 0.47 g (22%): m.p. 81 °C, $[\alpha]_D^{20}$ −57.2° (*c* 0.9, CH_2Cl_2). ^{13}C NMR (CH_2Cl_2), δ: 62.14 (d, CHClPh, J = 153.5 Hz); 67.12 (t, OCH_2, J = 152.1 Hz); 78.11 (d, OCH, J = 158.7 Hz); 127.8 (d, *o*-CH, J = 157.8 Hz); 129.3 (d, *m*-CH, J =

Table 1. Atomic coordinates in the structure of **12a**, equivalent temperature factors of the nonhydrogen atoms $B = 4/3 \cdot \sum_{i=1}^{3}\sum_{j=1}^{3}(a_i \cdot a_j)B(i,j)$ (Å^2), and isotropic temperature factors of the hydrogen atoms B_{iso} (Å^2)

Atom	*x*	*y*	*z*	*B*/Å^2
Cl(1)	0.3584(1)	0.82066(7)	0.94285(3)	6.64(1)
O(1)	0.0334(3)	0.6351(1)	0.80415(8)	5.08(3)
O(2)	0.3627(3)	0.5304(2)	0.7626(1)	6.66(4)
O(3)	0.3643(2)	0.7562(1)	0.78510(8)	4.77(3)
C(2)	0.2627(4)	0.6331(2)	0.7829(1)	4.69(5)
C(4)	0.1930(3)	0.8574(2)	0.8099(1)	3.79(4)
C(5)	−0.0335(3)	0.7733(2)	0.8228(1)	4.77(4)
C(6)	0.2960(3)	0.9364(2)	0.8716(1)	3.74(4)
C(7)	0.1315(3)	1.0540(2)	0.8921(1)	3.71(4)
C(8)	−0.0657(4)	1.0386(2)	0.9370(1)	5.07(5)
C(9)	−0.2224(4)	1.1480(3)	0.9477(1)	6.58(6)
C(10)	−0.1869(5)	1.2723(3)	0.9155(1)	6.60(6)
C(11)	0.0086(5)	1.2891(2)	0.8728(1)	6.05(6)
C(12)	0.1686(4)	1.1811(2)	0.8608(1)	4.57(4)
H(4)	0.167(3)	0.915(2)	0.7704(9)	4.4(4)*
H(6)	0.453(3)	0.976(2)	0.8572(8)	3.4(4)*
H(8)	−0.089(3)	0.958(2)	0.963(1)	4.4(4)*
H(9)	−0.341(4)	1.130(2)	0.974(1)	6.1(5)*
H(10)	−0.292(5)	1.346(2)	0.925(1)	8.4(7)*
H(11)	0.036(6)	1.368(2)	0.851(1)	8.6(7)*
H(12)	0.316(3)	1.196(2)	0.825(1)	5.0(4)*
H(51)	−0.083(3)	0.774(2)	0.869(1)	5.1(5)*
H(52)	−0.155(3)	0.794(2)	0.796(1)	6.0(5)*

* Atoms were refined isotropically.

166.8 Hz); 129.7 (d, *p*-CH, J = 167.8 Hz); 135.4 (s, *i*-C); 154.02 (s, C=O). IR, ν/cm^{-1}: 1780 (C=O).

X-ray diffraction analysis of (4*S*)-4-((1*R*)-1-chloromethyl-1-phenyl)-1,3-dioxolan-2-one (12a). Crystals of **12a** belong to the orthorhombic system. At 20 °C, a = 5.525(2), b = 9.669(3), c = 19.015(5) Å, V = 1015.8(5) Å^3, Z = 4, d_{calc} = 1.39 g cm^{-3}, space group $P2_12_12_1$. The unit cell parameters and intensities of 3192 reflections (reflections in two independent regions were recorded, of which 2022 reflections were with $I \geq 3\sigma$) were measured on an automated four-circle Enraf-Nonius CAD-4 diffractometer (λ(Mo-Kα), graphite monochromator, ω/2θ scanning technique, $\theta \leq 26°$). Intensities of three checking reflections showed no decrease in the course of X-ray data collection. Because of the low value of the coefficient (μMo = 3.50 cm^{-1}), absorption was ignored. The structure was solved by the direct method using the SIR program[22] and refined first isotropically and then anisotropically. All hydrogen atoms were revealed from the difference electron density synthesis and refined isotropically. With the aim of establishing the absolute structure and, consequently, the absolute configuration of the molecule, the "direct" and inverted structures were refined taking into account anomalous scattering of all nonhydrogen atoms. The values of the R factors for the "direct" structure were as follows: R = 0.04067, R_w = 0.04880 based on 1951 reflections for 163 refinable parameters. The values of the R factors for the inverted structure were as follows: R = 0.04185, R_w = 0.05063. Consequently, according to the Hamilton test,[23] the "direct" structure corresponds to the absolute structure with a probability of 95%. The final values of the R factors were as follows: R = 0.04067, R_w = 0.04880 based on 1951 reflections. All calculations were carried out on an Alpha Station 200 computer using the MolEn program package.[24] The atomic coordinates are given in Table 1. The atomic numbering scheme and the geometry of molecule **12a** are shown in Fig. 1.

References

1. R. M. Hanson, *Chem. Rev.*, 1991, **91**, 437.
2. (a) T. Katsuki and V. S. Martin, *Org. Reactions*, 1996, **48**, 1; (b) R. A. Johnson and K. B. Sharpless, in *Catalytic Asymmetric Synthesis*, Ed. I. Ojima, VCH, New York, 1993, 103.
3. B. M. Trost, *Acc. Chem. Res.*, 1996, **29**, 355.
4. C. M. Lok, J. P. Ward, and D. A. Van Dorp, *Chem. Phys. Lipids*, 1976, **16**, 115.
5. A. Rosowsky, in *Chemistry of Heterocyclic Compounds*, 1964, **19**, 1.
6. P. Gros, P. Le Perchec, and J. P. Senet, *J. Org. Chem.*, 1994, **59**, 4925.
7. M. E. Jung and D. C. D'Amico, *J. Am. Chem. Soc.*, 1995, **117**, 7379.
8. D. A. Evans and J. M. Williams, *Tetrahedron Lett.*, 1988, **29**, 5065.
9. G. A. Molander and D. C. Shubert, *J. Am. Chem. Soc.*, 1987, **109**, 576.
10. A. J. Mancuso and D. Swern, *Synthesis*, **1981**, 165.
11. H. Zondler, D. Trachsler, and F. Lohse, *Helv. Chim. Acta*, 1977, **60**, 1845.
12. A. A. Bredikhin and S. N. Lazarev, *Mendeleev Commun.*, 1998, 81.
13. I. Takehiko and I. Taisuke, *Tetrahedron*, 1993, **49**, 10511.
14. M. C. Malinovskii and N. M. Medyantseva, *Zh. Obshch. Khim.*, [*J. Gen. Chem. USSR*], 1953, **23**, 221 (in Russian).
15. J. I. Jones, *J. Chem. Soc.*, 1957, **6**, 2735.
16. Y. Gao, R. M. Hanson, J. M. Klunder, S. Y. Ko, H. Masamune, and K. B. Sharpless, *J. Am. Chem. Soc.*, 1987, **109**, 5765.
17. S. Takano, M. Yanase, and K. Ogasewara, *Heterocycles*, 1989, **29**, 249.
18. A. A. Bredikhin, S. N. Lazarev, Z. A. Bredikhina, and V. A. Al'fonsov, *Phosphorus, Sulfur, and Silicon*, 1997, **131**, 173.
19. B. Lon, Y. Zhang, G. Guo, and L. Dai, *Acta Chem. Sin.*, 1985, **6**, 554 [*Chem. Abstrs.*, 1991, **114**, 42013j].
20. F. Benedetti, F. Berti, and S. Norbedo, *Tetrahedron Lett.*, 1998, **39**, 7971.
21. J. Katzhendler, I. Ringler, and S. Sarel, *J. Chem. Soc., Perkin Trans. 2*, 1972, 2019.
22. A. Altomare, G. Cascarano, C. Giacovazzo, and D. Viterbo, *Acta Crystallogr. A*, 1991, **47**, 744.
23. W. C. Hamilton, *Acta Crystallogr*, 1965, **18**, 502.
24. L. H. Straver and A. J. Schierbeek, *MolEN. Structure Determination System*, Nonius B. V., 1994, No. 1, 2.

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