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Tetrahedron: *Asymmetry*

Preparation of chiral cyclopropanecarboxylic acids and 3-oxabicyclo[3.1.0]hexane-2-ones from levoglucosenone

Alexander V. Samet,* Dmitriy N. Lutov, Leonid D. Konyushkin, Yuri A. Strelenko and Victor V. Semenov

N. D. Zelinsky Institute of Organic Chemistry RAS, Leninsky Pr., 47 Moscow 119991, Russia

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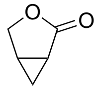
Abstract—Levoglucosenone (a chiral α,β-unsaturated ketone derivative of cellulose) serves as a starting compound in 2 and 3 step syntheses of chiral cyclopropanecarboxylic acids and 3-oxabicyclo[3.1.0]hexane-2-ones, respectively.

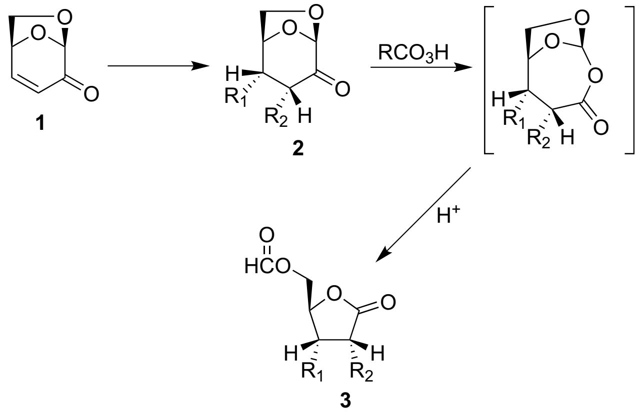
1. Introduction

Chiral molecules with a cyclopropane motif are of major interest as biologically active natural and synthetic products[1,2] and as intermediates in organic synthesis.[3] In particular, chiral 3-oxabicyclo[3.1.0]hexane-2-ones **A** can serve as valuable starting compounds in the preparation of chiral cyclopropanecarboxylic acid derivatives,[4–6] chiral butanolides,[7] and nucleoside analogues.[8] Compounds **A** are, in turn, prepared either by intramolecular cyclization of allyl diazoacetates in the presence of chiral catalysts[9–11] or from various chiral precursors.[12–16]



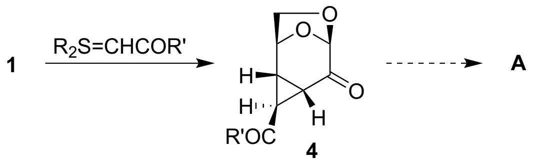
A

Levoglucosenone **1** [(1*S*,5*R*)-6,8-dioxabicyclo[3.2.1]oct-2-ene-4-one]—an unsaturated ketone prepared by acid-catalyzed pyrolysis of cellulose—is known to undergo stereoselective Michael addition of nucleophiles and cycloaddition to the C=C bond on the side opposite to the 1,6-anhydro bridge.[17–19] With peracids, these addition products **2** (as well as compound **1** itself) undergo an unusual transformation to butanolides **3** (Scheme 1) with retention of configuration of the asymmetric centers[20–23] (evidently, this is an interesting case of a Baeyer–Villiger rearrangement with subsequent acid-catalyzed recyclization, although the mechanism involved was never investigated in detail).



Scheme 1.

By cyclopropanation of levoglucosenone **1** with sulfur ylides, we have recently prepared chiral cyclopropanes **4**,[24] (Scheme 2), which may be able to undergo a similar conversion to the target chiral 3-oxabicyclo[3.1.0]hexane-2-ones.



Scheme 2.

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* Corresponding author. Tel.: +7 495 7161418; e-mail addresses: sametav@yahoo.com; sametav@server.ioc.ac.ru

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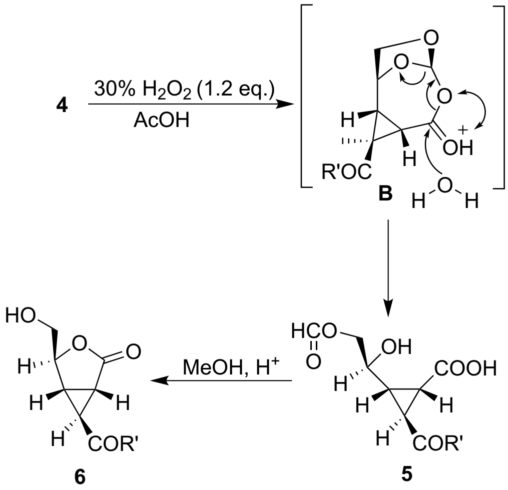
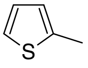
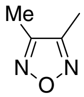
2. Results and discussion

We have found that annulated cyclopropanes **4** can be converted by 30% aq H_2O_2 in AcOH to cyclopropanecarboxylic acids **5**; heating of the latter in MeOH/HCl produces 4-hydroxymethyl-3-oxabicyclo[3.1.0]hexane-2-ones **6** (Scheme 3, Table 1).



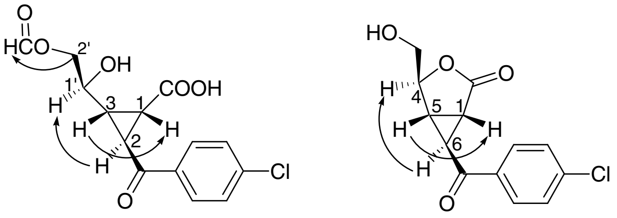
Scheme 3.

Table 1. Preparation of compounds **5a–d**, **6a–g**

Starting compound	R′	Product **5** (yield)	Product **6** (yield)
4a	Ph	**5a** (79)	**6a** (81)
4b	4-ClC$_6$H$_4$	**5b** (83)	**6b** (84)
4c	4-BrC$_6$H$_4$	**5c** (82)	**6c** (79)
4d		**5d** (79)	**6d** (80)
4e		[a]	**6e** (79[b])
4f	1-Adamantyl	[a]	**6f** (63[b])
4g	OEt	[a]	**6g**[c] (67[b])

[a] Not isolated in pure form.
[b] Yield in two steps from **4**.
[c] For **6g**, R′ = OMe due to transesterification.

It should be noted that there is no need to use hazardous peracetic acid or a large excess of H_2O_2 (cf. lit.[20–23]). The transformation of **4e–g** to **5e–g** is accompanied by partial hydrolysis of the formyl group in the products, so the corresponding crude **5** was converted to **6** without isolation. At the same time, oxidation of **4a–d** (R′ = Ar or 2-thienyl) affords pure acids **5a–d** in high yields. Most likely, the acids **5** result from the acid-catalyzed hydrolysis of the intermediate lactones **B** (Scheme 3) Interestingly, the rearrangement selectively affects a cyclic keto group in molecules **4a–f**, while the acyl substituents (COR′) in the cyclopropane ring are left intact. This is, probably, due to the remarkably high reactivity of the cyclic keto group adjacent to an acetal fragment (thus, it is known to readily add water and MeOH yielding *gem*-diols and ketals, respectively).[17,24,25]



5b **6b**

Figure 1. Key cross-peaks in 2D ^{1}H NOESY spectra of **5b** and **6b**.

The structures of compounds **5** and **6** are confirmed by NOE spectra of **5b** and **6b** (Fig. 1). The 2D ^{1}H NOESY spectrum of **6b** features a strong cross-peak between the signals of H-1 (δ 2.65) and H-5 (δ 2.71) protons[†] and weak cross-peaks between these two signals and the signal of the H-6 proton (δ 3.42), thus indicating that the H-6 proton is *trans*- to the protons H-1 and H-5. This is consistent with the coupling constants values $J_{1,6} = 2.4–2.6$ Hz, $J_{5,6} = 3.2–3.4$ Hz, $J_{1,5} = 6.1–6.2$ Hz in ^{1}H NMR spectra of **6a–g**, considering that *cis*-constants in cyclopropanes are larger than *trans*-. The configurations of the C-6 (and C-4) centers are further confirmed by a strong cross-peak between the signals of H-6 and H-4 (δ 4.63) protons (any other configuration of these centers would increase the distance between the H-6 and H-4 protons and thus render such a cross-peak impossible, cf. NOE experiments with similar structures[15,26]).

Similarly, in the 2D ^{1}H NOESY spectrum of **5b**, strong cross-peaks between the signals of H-1 (δ 2.29) and H-3 (δ 1.94) protons and between the signals of H-2 (δ 3.30) and H-1′ (δ 3.93) protons, together with weak cross-peaks H-1–H-2, H-2–H-3 and the coupling constant values $J_{1,2} = 4.5–4.6$ Hz, $J_{2,3} = 5.7–5.8$ Hz, $J_{1,3} = 9.4–9.5$ Hz in ^{1}H NMR spectra of **5a–d**, indicate that the stereochemistry of **5** is the same as that of **6**. In addition, the formyl proton (δ 8.20) shows a cross-peak with CH$_2$-protons (δ 4.12 and 4.18) rather than with H-1′ proton, thus supporting the proposed 2-formyloxy-1-hydroxyethyl side chain structure against a 1-formyloxy-2-hydroxyethyl structure, which initially seemed possible as well.

These data are in agreement with the previously reported retention of the configuration of the stereocenters in the process of the rearrangement (Scheme 1).[20–23]

3. Conclusion

Thus, levoglucosenone proved to be a suitable chiral template for the convenient and straightforward synthesis of optically active substituted cyclopropanecarboxylic acids and 3-oxabicyclo[3.1.0]hexane-2-ones.

[†] The assignments of H-1 and H-5 protons are also based on the 2D ^{1}H NOESY spectrum of **6b**. The signal at δ 2.71, in contrast to the one at δ 2.65, shows cross-peaks with the signals of H-4 (δ 4.63) and CH$_2$-protons (δ 3.62), thus indicating the former to be the H-5 proton, and the latter to be H-1.

4. Experimental

4.1. General

NMR spectra were recorded on a Bruker-DRX500 spectrometer (500.13 MHz for ^{1}H NMR and 2D ^{1}H NOESY, 125.75 MHz for ^{13}C). Optical rotations were measured on a Jasco-340 polarimeter. Compounds **4** were prepared by a known procedure.[24] Levoglucosenone **1** was supplied by Chemical Block Ltd (www.chemblock.com).

4.2. Synthetic procedures

4.2.1. Preparation of 5a–d (general procedure). To a solution of 30% aq H_2O_2 (0.27 g, 2.4 mmol) in glacial AcOH (2 ml), **4a**–**d** (2 mmol) was added in portions with stirring. The resulting suspension was gently heated at 30–40 °C until a clear solution was formed and left for 5 h at rt. The resulting precipitate of **5a**–**d** was filtered off, washed thoroughly with water, and dried.

4.2.1.1. (1*S*,2*S*,3*S*)-2-Benzoyl-3-((*S*)-2-formyloxy-1-hydroxyethyl)cyclopropanecarboxylic acid 5a. Mp 186–189 °C (EtOH) (decomp.). $[\alpha]_D^{20} = +112.4$ (*c* 1.0, DMSO). ^{1}H NMR (DMSO-d_6): 1.95 (td, *J* = 9.5, 5.7 Hz, 1H, H-3); 2.27 (dd, *J* = 9.3, 4.6 Hz, 1H, H-1); 3.29 (t, *J* = 5.2 Hz, 1H, H-2); 3.95 (m, 1H, H-1′); 4.16 (m, 2H, CH_2); 5.25 (br s, 1H, OH); 7.57 (t, *J* = 8.4 Hz, 2H); 7.68 (t, *J* = 8.4 Hz, 1H); 8.03 (d, *J* = 8.3 Hz, 2H); 8.20 (s, 1H, HCOO); 12.61 (br s, 1H, COOH). Anal. Calcd for $C_{14}H_{14}O_6$: C, 60.43; H, 5.07. Found: C, 60.05; H, 5.32.

4.2.1.2. (1*S*,2*S*,3*S*)-2-(4-Chlorobenzoyl)-3-((*S*)-2-formyloxy-1-hydroxyethyl)cyclopropanecarboxylic acid 5b. Mp 188–190 °C (EtOH) (decomp.). $[\alpha]_D^{20} = +98.0$ (*c* 1.0, DMSO). ^{1}H NMR (DMSO-d_6): 1.94 (m, 1H, H-3); 2.29 (dd, *J* = 9.3, 4.6 Hz, 1H, H-1); 3.30 (t, *J* = 5.3 Hz, 1H, H-2); 3.93 (m, 1H, H-1′); 4.12 (dd, *J* = 11.0, 4.3 Hz, 1H, CH_2); 4.18 (dd, *J* = 11.0, 5.8 Hz, 1H, CH_2); 5.24 (br s, 1H, OH); 7.64 (d, *J* = 8.5 Hz, 2H); 8.07 (d, *J* = 8.5 Hz, 2H); 8.20 (s, 1H, HCOO); 12.63 (br s, 1H, COOH). ^{13}C NMR (DMSO-d_6): 27.90, 28.98, 33.77, 64.91, 67.28, 129.07, 130.12, 135.08, 138.65, 162.17, 170.81, 195.09. Anal. Calcd for $C_{14}H_{13}ClO_6$: C, 53.77; H, 4.19; Cl, 11.34. Found: C, 54.08; H, 3.96; Cl, 11.10.

4.2.1.3. (1*S*,2*S*,3*S*)-2-(4-Bromobenzoyl)-3-((*S*)-2-formyloxy-1-hydroxyethyl)cyclopropanecarboxylic acid 5c. Mp 190–193 °C (EtOH) (decomp.). $[\alpha]_D^{20} = +89.6$ (*c* 0.5, DMSO). ^{1}H NMR (DMSO-d_6): 1.94 (td, *J* = 9.5, 5.8 Hz, 1H, H-3); 2.29 (dd, *J* = 9.5, 4.6 Hz, 1H, H-1); 3.30 (t, *J* = 5.2 Hz, 1H, H-2); 3.93 (m, 1H, H-1′); 4.12 (dd, *J* = 11.1, 4.6 Hz, 1H, CH_2); 4.18 (dd, *J* = 11.1, 5.7 Hz, 1H, CH_2); 5.25 (br s, 1H, OH); 7.79 (d, *J* = 8.5 Hz, 2H); 7.97 (d, *J* = 8.5 Hz, 2H); 8.20 (s, 1H, HCOO); 12.63 (br s, 1H, COOH). Anal. Calcd for $C_{14}H_{13}BrO_6$: C, 47.08; H, 3.67; Br, 22.37. Found: C, 46.78; H, 3.60; Br, 22.60.

4.2.1.4. (1*S*,2*S*,3*S*)-2-((*S*)-2-Formyloxy-1-hydroxyethyl)-3-(2-thienoyl)cyclopropanecarboxylic acid 5d. Mp 192–195 °C (EtOH) (decomp.). $[\alpha]_D^{20} = +140.0$ (*c* 0.8, DMSO). ^{1}H NMR (DMSO-d_6): 1.91 (td, *J* = 9.5, 5.8 Hz, 1H, H-3); 2.26 (dd, *J* = 9.4, 4.5 Hz, 1H, H-1); 3.22 (t, *J* = 5.2 Hz, 1H, H-2); 3.93 (m, 1H, H-1′); 4.14 (m, 2H, CH_2); 5.27 (br s, 1H, OH); 7.29 (dd, *J* = 4.9, 3.8 Hz, 1H); 8.08 (dd, *J* = 4.9, 1.6 Hz, 1H); 8.19 (m, 1H, HCOO); 12.63 (br s, 1H, COOH). Anal. Calcd for $C_{12}H_{12}O_6S$: C, 50.70; H, 4.25; S, 11.28. Found: C, 50.61; H, 4.40; S, 10.93.

4.2.2. Preparation of 6a–d (general procedure). To a solution of **5a**–**d** (2 mmol) in MeOH (3 ml), concd HCl (0.1 ml) was added and the mixture was refluxed for 3 h. Then, the solvent was distilled off at atmospheric pressure, the residue was kept for 30 min at 80 °C and recrystallized from a suitable solvent.

4.2.3. Preparation of 6e–g ('one-pot', general procedure). To a solution of 30% aq H_2O_2 (0.27 g, 2.4 mmol) in glacial AcOH (2 ml), **4e**–**g** (2 mmol) was added in portions with stirring. The resulting suspension was gently heated at 30–40 °C, until a clear solution was formed (ca. 20 min) and then left for 5 h at rt. The solvent was removed in vacuo, and the residue was dissolved in MeOH (3 ml) with the resulting solution treated as specified above for the preparation of **6a**–**d**.

4.2.3.1. (1*S*,4*S*,5*S*,6*S*)-6-Benzoyl-4-hydroxymethyl-3-oxabicyclo[3.1.0]hexane-2-one 6a. Mp 127–128 °C (MeOH). $[\alpha]_D^{20} = +112.8$ (*c* 1.0, DMSO). ^{1}H NMR (DMSO-d_6): 2.64 (dd, *J* = 6.1, 2.4 Hz, 1H, H-1); 2.70 (dd, *J* = 6.1, 3.3 Hz, 1H, H-5); 3.41 (t, *J* = 3.0 Hz, 1H, H-6); 3.62 (m, 2H, CH_2); 4.64 (t, *J* = 3.6 Hz, 1H, H-4); 5.14 (t, *J* = 5.7 Hz, 1H, OH); 7.57 (t, *J* = 7.6 Hz, 2H); 7.71 (t, *J* = 7.3 Hz, 1H); 8.13 (d, *J* = 7.9 Hz, 2H). ^{13}C NMR (DMSO-d_6): 26.83, 27.33, 29.96, 62.44, 81.03, 128.52, 128.91, 133.92, 136.18, 173.65, 194.99. Anal. Calcd for $C_{13}H_{12}O_4$: C, 67.23; H, 5.21. Found: C, 67.00; H, 5.33.

4.2.3.2. (1*S*,4*S*,5*S*,6*S*)-6-(4-Chlorobenzoyl)-4-hydroxymethyl-3-oxabicyclo[3.1.0]hexane-2-one 6b. Mp 184–185 °C (EtOH). $[\alpha]_D^{20} = +88.1$ (*c* 1.0, DMSO). ^{1}H NMR (DMSO-d_6): 2.65 (dd, *J* = 6.1, 2.5 Hz, 1H, H-1); 2.71 (dd, *J* = 6.1, 3.2 Hz, 1H, H-5); 3.42 (t, *J* = 3.0 Hz, 1H, H-6); 3.62 (m, 2H); 4.63 (t, *J* = 3.6 Hz, 1H, H-4); 5.12 (br s, 1H, OH); 7.64 (d, *J* = 8.5 Hz, 2H); 8.16 (d, *J* = 8.5 Hz, 2H). Anal. Calcd for $C_{13}H_{11}ClO_4$: C, 58.55; H, 4.16; Cl, 13.29. Found: C, 58.81; H, 3.99; Cl, 13.42.

4.2.3.3. (1*S*,4*S*,5*S*,6*S*)-6-(4-Bromobenzoyl)-4-hydroxymethyl-3-oxabicyclo[3.1.0]hexane-2-one 6c. Mp 195–197 °C (EtOH). $[\alpha]_D^{20} = +79.7$ (*c* 1.0, DMSO). ^{1}H NMR (DMSO-d_6): 2.63 (dd, *J* = 6.1, 2.5 Hz, 1H, H-1); 2.69 (dd, *J* = 6.2, 3.4 Hz, 1H, H-5); 3.41 (t, *J* = 3.0 Hz, 1H, H-6); 3.61 (m, 2H); 4.62 (t, *J* = 3.6 Hz, 1H, H-4); 5.13 (br s, 1H, OH); 7.78 (d, *J* = 8.5 Hz, 2H); 8.07 (d, *J* = 8.5 Hz, 2H). Anal. Calcd for $C_{13}H_{11}BrO_4$: C, 50.18; H, 3.56; Br, 25.68. Found: C, 49.89; H, 3.60; Br, 26.00.

4.2.3.4. (1*S*,4*S*,5*S*,6*S*)-4-Hydroxymethyl-6-(2-thienoyl)-3-oxabicyclo[3.1.0]hexane-2-one 6d. Mp 202–203 °C (MeOH). $[\alpha]_D^{20} = +112.4$ (*c* 1.0, DMSO). ^{1}H NMR (DMSO-d_6): 2.75 (dd, *J* = 6.2, 2.6 Hz, 1H, H-1); 2.89 (dd, *J* = 6.2, 3.3 Hz, 1H, H-5); 3.42 (t, *J* = 3.0 Hz, 1H, H-6); 3.63 (m, 2H); 4.60 (t, *J* = 3.6 Hz, 1H, H-4); 5.16 (t,

$J = 5.7$ Hz, 1H, OH); 7.32 (dd, $J = 4.9$, 3.9 Hz, 1H); 8.12 (d, $J = 4.9$ Hz, 1H); 8.37 (d, $J = 3.9$ Hz, 1H). Anal. Calcd for $C_{12}H_{12}O_6S$: C, 55.45; H, 4.23; S, 13.46. Found: C, 55.51; H, 4.35; S, 13.13.

4.2.3.5. (1*S*,4*S*,5*S*,6*S*)-4-Hydroxymethyl-6-(4-methylfurazane-3-yl)carbonyl-3-oxabicyclo[3.1.0]hexane-2-one 6e. Mp 125–126 °C (benzene). $[\alpha]_D^{24} = +97.2$ (*c* 1.0, DMSO). ^{1}H NMR (DMSO-d_6): 2.52 (s, 3H); 2.80 (dd, $J = 6.2$, 2.5 Hz, 1H, H-1); 2.87 (dd, $J = 6.2$, 3.3 Hz, 1H, H-5); 3.19 (t, $J = 2.9$ Hz, 1H, H-6); 3.63 (m, 2H, CH_2); 4.63 (t, $J = 3.5$ Hz, 1H, H-4); 5.19 (t, $J = 5.7$ Hz, 1H, OH). Anal. Calcd for $C_{10}H_{10}N_2O_5$: C, 50.42; H, 4.23; N, 11.76. Found: C, 50.65; H, 4.10; N, 12.11.

4.2.3.6. (1*S*,4*S*,5*S*,6*S*)-6-(1-Adamantoyl)-4-hydroxymethyl-3-oxabicyclo[3.1.0]hexane-2-one 6f. Mp 112–114 °C (MeOH). $[\alpha]_D^{20} = +84.1$ (*c* 1.0, DMSO). ^{1}H NMR (DMSO-d_6): 1.69 (m, 6H); 1.81 (m, 6H); 2.00 (m, 3H); 2.30 (dd, $J = 6.1$, 2.6 Hz, 1H, H-1); 2.40 (dd, $J = 6.1$, 3.3 Hz, 1H, H-5); 2.84 (t, $J = 3.0$ Hz, 1H, H-6); 3.58 (m, 2H); 4.50 (t, $J = 3.7$ Hz, 1H, H-4); 5.09 (br s, 1H, OH). Anal. Calcd for $C_{17}H_{22}O_4$: C, 70.32; H, 7.64. Found: C, 70.04; H, 7.79.

4.2.3.7. (1*S*,4*S*,5*S*,6*S*)-Methyl-4-hydroxymethyl-2-oxo-3-oxabicyclo[3.1.0]hexane-6-carboxylate 6g. Mp 115–116 °C (MeOH). $[\alpha]_D^{20} = +98.8$ (*c* 1.0, DMSO). ^{1}H NMR (DMSO-d_6): 2.18 (t, $J = 3.0$ Hz, 1H, H-6); 2.53 (dd, $J = 6.1$, 2.4 Hz, 1H, H-1); 2.63 (dd, $J = 6.1$, 3.3 Hz, 1H, H-5); 3.60 (m, 2H, CH_2); 3.69 (s, 3H); 4.50 (t, $J = 3.5$ Hz, 1H, H-4); 5.13 (br s, 1H, OH). ^{13}C NMR (DMSO-d_6): 23.22, 25.38, 27.25, 52.29, 62.30, 80.64, 169.90, 172.98. Anal. Calcd for $C_8H_{10}O_5$: C, 51.61; H, 5.41. Found: C, 51.85; H, 5.19.

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