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Stereoselective preparation of 7-*exo*-amino-7-*endo*-substituted bicyclo[4.1.0]heptanes

Morshed Alam Chowdhury,[a] Hisanori Senboku,[a] Masao Tokuda[a,*] and Toshiro Chiba[b]

[a]*Laboratory of Organic Synthesis, Division of Molecular Chemistry, Graduate School of Engineering, Hokkaido University, Sapporo 060-8628, Japan*

[b]*Department of Applied Chemistry, Kitami Institute of Technology, Kitami 090-8507, Japan*

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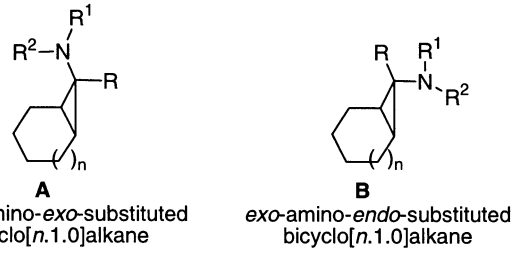
Abstract—Bicyclo[4.1.0]heptan-7-one *N*,*O*-hemiacetals undergo substitution reaction with Wittig reagents ($Ph_3P{=}CHR^3$) to give stereoselectively the corresponding 7-*exo*-amino-7-*endo*-substituted bicyclo[4.1.0]heptanes in good yields.

1. Introduction

Cyclopropanone *N*,*O*-hemiacetals are versatile precursors for various organic syntheses.[1] It is known that the hydroxy groups of *N*,*O*-hemiacetals are readily displaced by various nucleophiles such as alcohols, amines, thiols, hydrogen cyanide[2] and hydrazoic acid.[3] Indole, *N*-methylpyrrole, silyl enol ethers or active methylene compounds in the presence of titanium tetrachloride,[4] Grignard reagents[4] and C–H acidic compounds[5–8] also work as nucleophiles to give the corresponding displacement products of the hydroxy groups from *N*,*O*-hemiacetals. In the case of ring-fused cyclopropanone *N*,*O*-acetals, *endo*-amino-*exo*-substituted bicyclo[*n*.1.0]alkanes **A** are generally formed as the predominant products, because the displacement involves the cyclopropaniminium cation as an intermediate, which is attacked by nucleophiles from a less-hindered *exo*-position (Scheme 1).[9,10] On the other hand, there have been no reports on stereoselective preparation of the corresponding *exo*-amino-*endo*-substituted bicyclo[*n*.1.0]alkanes **B** (Scheme 1).



Scheme 1.

In this paper, we report a convenient method for preparing 7-*exo*-amino-7-*endo*-substituted bicyclo[4.1.0]heptanes by the reaction of bicyclo[4.1.0]heptan-7-one *N*,*O*-hemiacetals, which are readily prepared by our procedure,[11,12] with monosubstituted Wittig reagents.

2. Results and discussion

The reaction of bicyclo[4.1.0]heptan-7-one *N*,*O*-hemiacetals **1a–e** with one equivalent of triphenylphosphorane in the presence of a catalytic amount of benzoic acid[13] in toluene under reflux conditions predominantly gave the corresponding 7-*exo*-amino-7-*endo*-substituted bicyclo-[4.1.0]heptanes **2–5** in 52–87% yields (Scheme 2). In most cases, a small amount of ring-fused alkylidenecyclopropanes **6** was detected as a by-product.[14] The results are summarized in Table 1.

The reaction of bicyclo[4.1.0]heptan-7-one *N*,*O*-hemiacetal **1a** with a Wittig reagent ($R^3{=}COOMe$) was monitored by GLC analysis, and the results are shown in Fig. 1. A peak of ring-fused alkylidenecyclopropane **6** ($R^3{=}COOMe$) appeared as a major product in the initial stage of the reaction, and its yield reached about 75% within a few minutes after the start of the reaction. However, we failed to obtain **6** in a reasonable yield, and **2a** was obtained as a final and major product. These results indicate that, in the present reaction, **6** was formed initially and then the liberated amine immediately attacked the β-carbon of α,β-unsaturated ester **6** to give **2a** predominantly. It has already been

Keywords: bicyclo[4.1.0]heptane; bicyclo[4.1.0]heptan-7-one *N*,*O*-hemiacetal; Wittig reagent; substitution; 7-*endo*-amino-7-*exo*-substituted bicyclo[4.1.0]heptane; 7-*exo*-amino-7-*endo*-substituted bicyclo[4.1.0]-heptane.

* Corresponding author. Tel.: +81-11-706-6599; fax: +81-11-706-6598; e-mail: tokuda@org-mc.eng.hokudai.ac.jp

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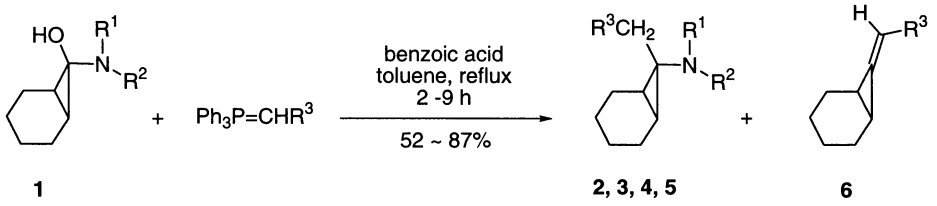


1

2, 3, 4, 5

6

1-5	R^1	R^2
a	Me	Me
b	Et	Et
c	$-(CH_2)_4-$	
d	$-(CH_2)_5-$	
e	$-(CH_2)_2-O-(CH_2)_2-$	

2, R^3 = COOMe
3, R^3 = COMe
4, R^3 = COPh
5, R^3 = CN

Scheme 2.

Table 1. Reactions of bicyclo[4.1.0]heptan-7-one *N*,*O*-hemiacetals **1** with Wittig reagents

N,*O*-Hemiacetal **1**	$Ph_3P=CHR^3$	Time (h)	Product	Yield (%)[a]
1a	R^3=COOMe	6	**2a**	52
1b		9	**2b**	53
1c		2	**2c**	73
1d		2	**2d**	76
1e		6	**2e**	76
1c	R^3=COOMe	3	**3c**	68
	R^3=COPh	1[b]	**4c**	66
1e	R^3=COMe	2	**3e**	74
	R^3=COPh	2	**4e**	87
	R^3=CN	8	**5e**	63

A mixture of bicyclo[4.1.0]heptan-7-one *N*,*O*-hemiacetal **1** (5 mmol), Wittig reagent (6 mmol), and benzoic acid (0.02 g) in toluene (25 mL) was heated under reflux conditions for 2–9 h.

[a] Isolated yields based on bicyclo[4.1.0]heptan-7-one *N*,*O*-hemiacetal **1** employed.

[b] Refluxed in benzene for 1 h.

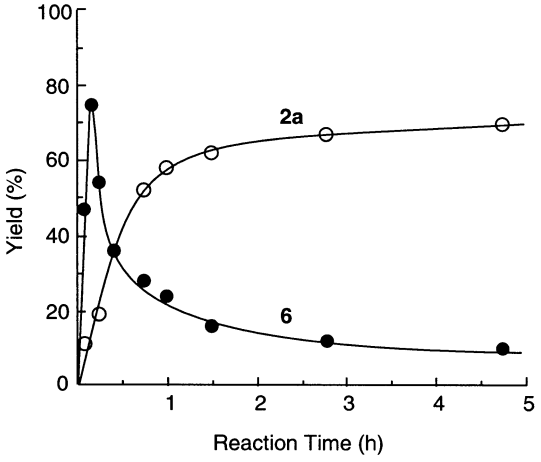
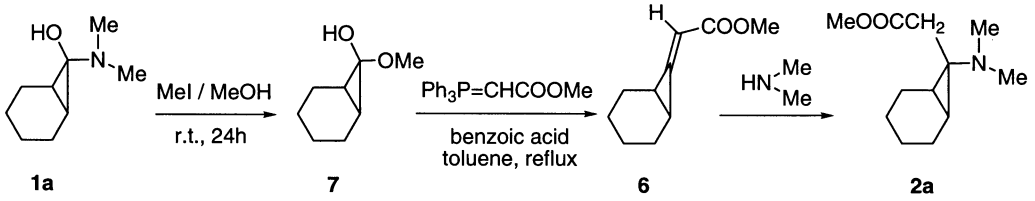


Figure 1. Time-course of the reaction of bicyclo[4.1.0]heptan-7-one *N*,*O*-hemiacetal **1a** (5 mmol) with Wittig reagent (R^3=COOMe, 6 mmol) in the presence of benzoic acid (0.02 g) in refluxing toluene.

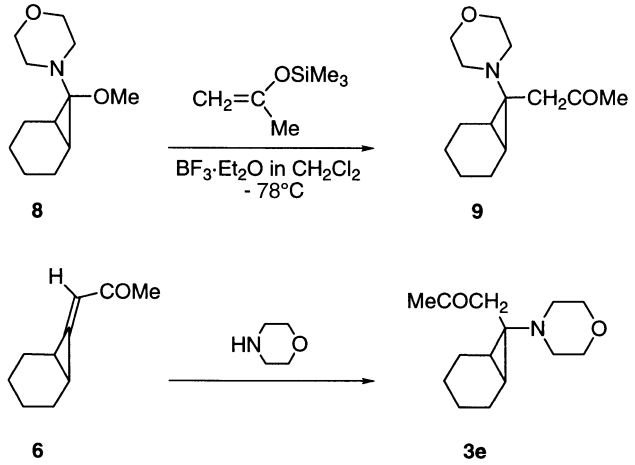
reported that the C=C double bond of methylenecyclopropane derivatives carrying an electron-withdrawing group is reactive as a Michael acceptor.[15] Actually, a reaction of the alkylidene derivative **6**, independently prepared from hemiacetal **7** and Wittig reagent (R^3=COOMe), with dimethyl amine readily afforded **2a** (Scheme 3).

The stereochemical configuration of the products was established by comparison of their spectral data with those of authentic samples of 7-*endo*- and 7-*exo*-morpholinobicyclo[4.1.0]heptane derivatives which were prepared independently. The *endo*-amino isomer **9** was obtained by the reaction of bicyclo[4.1.0]heptan-7-one *N*,*O*-acetal **8** with trimethylsiloxypropene in the presence of BF_3 etherate, using our previously reported method[11] (Scheme 4). On the other hand, the *exo*-amino isomer **3e** was prepared by an addition of morpholine to ring-fused alkylidenecyclopropane **6** (R^3=COMe), which was prepared by Wittig reaction of **2**-oxopropylidenetriphenylphosphorane with 7-methoxybicyclo[4.1.0]heptan-7-ol[13a,b] (Scheme 4). The spectral data of product **3e** obtained in the present reaction were identical with those of the authentic *exo*-amino isomer **3e**, and the ^{1}H NMR spectra of methylene protons of the morpholino ring displayed an AA′XX′ pattern, supporting the *exo*-morpholino configuration.[16,17]

Proposed reaction pathways of the present reaction are shown in Scheme 5. The formation of ring-fused alkylidenecyclopropanes **6** proceeds probably via iminium cation **10**, which is formed from bicyclo[4.1.0]heptan-7-one *N*,*O*-hemiacetals **1** under mild acidic conditions. Iminium cation **10** is sufficiently stable in situ to be trapped by various nucleophiles.[6,10] The reaction of **10** with a Wittig reagent gives 1:1 adducts **11**, and then an elimination of an iminium salt of triphenylphosphine **12** gives **6**. The iminium salt of triphenylphosphine **12** is converted into triphenylphosphine oxide **13** and amine **14** by reaction with water, which is produced initially during the process of formation of iminium cation **10**. The liberated amine **14** immediately attacks the β-carbon of α,β-unsaturated derivative **6** from a sterically less-hindered *exo*-position[10] to give **2**–**5** predominantly, because the C=C double bond of methylenecyclopropane derivatives **6** carrying an electron-withdrawing group is reactive as a Michael acceptor.[15]



Scheme 3.

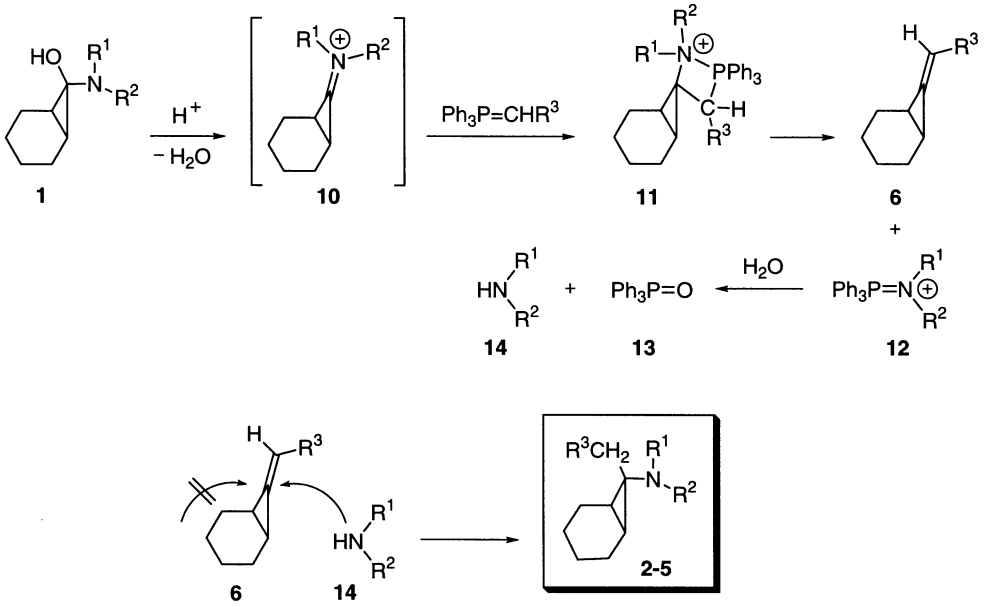


Scheme 4.

as a solvent. Chemical shifts are given in ppm downfield (δ) from TMS as an internal standard. IR spectra were obtained from neat films unless otherwise noted. MS spectra were obtained at an ionization potential of 70 eV.

3.2. General procedure for preparation of 7-*exo*-amino-7-*endo*-substituted bicyclo[4.1.0]heptanes (2–5)

A mixture of bicyclo[4.1.0]heptan-7-one *N*,*O*-hemiacetal **1** (5 mmol), triphenylphosphorane (6 mmol), and a catalytic amount of benzoic acid (0.02 g) in 25 mL of toluene was stirred under reflux conditions for 2–9 h. The mixture was evaporated in vacuo, and the residue was extracted with pentane (20 mL×5). The combined pentane extracts were concentrated and column-chromatographed on alumina (ether–hexane).



Scheme 5.

3. Experimental

3.1. General

Wittig reagents were prepared as described in the literature.[18–20] Bicyclo[4.1.0]heptan-7-one *N*,*O*-hemiacetals **1a–e** were prepared using our previously reported method.[11,12] Unless otherwise noted, ^{1}H and ^{13}C NMR spectra were recorded at 270 and 67.5 MHz, respectively, using $CDCl_3$

3.2.1. Methyl 2-(7-*exo*-dimethylaminobicyclo[4.1.0]hept-7-yl)acetate (2a). IR ν 1740 cm^{-1}; ^{1}H NMR δ 0.95–1.02 (m, 2H), 1.02–1.35 (m, 4H), 1.45–1.58 (m, 2H), 1.84–2.00 (m, 2H), 2.28 (s, 6H), 2.48 (s, 2H), 3.68 (s, 3H); ^{13}C NMR δ 19.30 (CH_2), 21.55 (CH_2), 22.57 (CH), 27.80 (CH_2), 40.38 (CH_3), 48.05 (C), 51.48 (OCH_3), 173.84 (CO); MS *m*/*z* (relative intensity) 211 (M^+, 48), 196 (82), 156 (95), 138 (100), 114 (88). Anal. Calcd for $C_{12}H_{21}NO_2$: C, 68.21; H, 10.02; N, 6.63. Found: C, 68.19; H, 10.12; N, 6.49.

3.2.2. Methyl 2-(7-*exo*-diethylaminobicyclo[4.1.0]hept-7-yl)acetate (2b). IR ν 1740 cm^{-1}; ^1H NMR δ 0.99 (t, J=7.3 Hz, 6H), 0.95–1.05 (m, 2H), 1.05–1.36 (m, 4H), 1.46–1.60 (m, 2H), 1.81–1.99 (m, 2H), 2.46 (s, 2H), 2.59 (q, J=7.3 Hz, 4H), 3.66 (s, 3H); ^{13}C NMR δ 15.15 (CH_3), 18.98 (CH_2), 21.66 (CH_2), 21.93 (CH), 30.01 (CH_2), 45.95 (CH_2), 47.03 (C), 51.36 (OCH_3), 174.09 (CO); MS *m/z* (relative intensity) 239 (M^+, 45), 224 (48), 210 (100), 180 (55). Anal. Calcd for $C_{14}H_{25}NO_2$: C, 70.25; H, 10.53; N, 5.85. Found: C, 70.48; H, 10.68; N, 5.65.

3.2.3. Methyl 2-(7-*exo*-pyrrolidinobicyclo[4.1.0]hept-7-yl)acetate (2c). Bp 108°C (13 mmHg); IR ν 1740 cm^{-1}; ^1H NMR (90 MHz) δ 0.91–1.10 (m, 2H), 1.10–1.46 (m, 4H), 1.46–1.76 (m, 6H), 1.76–2.18 (m, 2H), 2.50 (s, 2H), 2.51–2.71 (m, 4H), 3.66 (s, 3H); ^{13}C NMR (22.4 MHz) δ 19.46 (CH_2), 20.73 (CH), 21.87 (CH_2), 23.78 (CH_2), 29.65 (CH_2), 43.21 (C), 47.48 (CH_2), 51.35 (OCH_3), 173.80 (CO); MS *m/z* (relative intensity) 237 (M^+, 43), 208 (26), 182 (54), 178 (100), 164 (52). Anal. Calcd for $C_{14}H_{23}NO_2$: C, 70.85; H, 9.77; N, 5.90. Found: C, 70.80; H, 9.77; N, 5.86.

3.2.4. Methyl 2-(7-*exo*-piperidinobicyclo[4.1.0]hept-7-yl)acetate (2d). IR ν 1740 cm^{-1}; ^1H NMR δ 0.91–1.01 (m, 2H), 1.01–1.57 (m, 12H), 1.81–1.99 (m, 2H), 2.47 (s, 2H), 2.45–2.55 (m, 4H), 3.67 (s, 3H); ^{13}C NMR δ 19.37 (CH_2), 21.64 (CH_2), 22.18 (CH), 24.67 (CH_2), 26.78 (CH_2), 28.72 (CH_2), 48.36 (C), 49.76 (CH_2), 51.38 (OCH_3), 173.94 (CO); MS *m/z* (relative intensity) 251 (M^+, 25), 194 (39), 192 (100), 178 (34). Anal. Calcd for $C_{15}H_{25}NO_2$: C, 71.67; H, 10.02; N, 5.57. Found: C, 71.61; H, 10.05; N, 5.80.

3.2.5. Methyl 2-(7-*exo*-morpholinobicyclo[4.1.0]hept-7-yl)acetate (2e). Mp 41–43°C (pentane, −30°C); IR (KBr) ν 1744 cm^{-1}; ^1H NMR δ 0.95–1.05 (m, 2H), 1.05–1.34 (m, 4H), 1.41–1.55 (m, 2H), 1.82–1.99 (m, 2H), 2.51 (s, 2H), 2.58 (t, J=4.6 Hz, 4H), 3.55 (J=4.6 Hz, 4H), 3.69 (s, 3H); ^{13}C NMR δ 19.32 (CH_2), 21.58 (CH_2), 21.66 (CH), 28.81 (CH_2), 47.80 (C), 49.08 (CH_2), 51.57 (OCH_3), 67.76 (CH_2), 173.53 (CO); MS *m/z* (relative intensity) 253 (M^+, 54), 194 (100), 180 (73). Anal. Calcd for $C_{14}H_{23}NO_3$: C, 66.37; H, 9.15; N, 5.53. Found: C, 66.21; H, 8.97; N, 5.43.

3.2.6. 1-(7-*exo*-Pyrrolidinobicyclo[4.1.0]hept-7-yl)-2-propanone (3c). IR ν 1715 cm^{-1}; ^1H NMR (90 MHz) δ 0.85–1.38 (m, 6H), 1.38–1.69 (m, 6H), 1.69–2.15 (m, 2H), 2.21 (s, 3H), 2.38–2.65 (m, 4H), 2.57 (s, 2H); ^{13}C NMR (22.4 MHz) δ 17.92 (CH), 19.42 (CH_2), 21.75 (CH_2), 23.41 (CH_2), 29.56 (CH_3), 40.92 (CH_2), 43.78 (C), 47.77 (CH_2), 208.37 (CO); MS *m/z* (relative intensity) 221 (M^+, 8), 178 (100). Anal. Calcd for $C_{14}H_{23}NO$: C, 75.97; H, 10.47; N, 6.33. Found: C, 75.76; H, 10.54; N, 6.22.

3.2.7. 1-(7-*exo*-Morpholinobicyclo[4.1.0]hept-7-yl)-2-propanone (3e). Mp 59–61°C (hexane, −30°C); IR (KBr) ν 1713 cm^{-1}; ^1H NMR δ 0.97–1.15 (m, 4H), 1.15–1.45 (m, 4H), 1.80–2.00 (m, 2H), 2.23 (s, 3H), 2.48 (t, J=4.6 Hz, 4H), 2.61 (s, 2H), 3.55 (t, J=4.6 Hz, 4H); ^{13}C NMR δ 19.54 (CH_2), 20.38 (CH), 21.64 (CH_2), 30.42 (CH_3), 38.13 (CH_2), 47.51 (C), 49.44 (CH_2), 67.58 (CH_2), 207.49 (CO); MS *m/z* (relative intensity) 237 (M^+, 6), 194 (100). Anal. Calcd for $C_{14}H_{23}NO_2$: C, 70.85; H, 9.77; N, 5.90. Found: C, 71.00; H, 9.65; N, 5.88.

3.2.8. 2-(7-*exo*-Pyrrolidinobicyclo[4.1.0]hept-7-yl)acetophenone (4c). IR ν 1693 cm^{-1}; ^1H NMR δ 0.95–1.40 (m, 6H), 1.40–1.69 (m, 6H), 1.85–2.01 (m, 2H), 2.44–2.60 (m, 4H), 3.17 (s, 2H), 7.39–7.59 (m, 3H), 7.95–8.05 (m, 2H); ^{13}C NMR δ 19.75 (CH_2), 20.36 (CH), 21.71 (CH_2), 23.13 (CH_2), 32.28 (CH_2), 42.64 (C), 48.09 (CH_2), 127.71 (CH), 128.34 (CH), 132.45 (CH), 137.72 (C), 198.81 (CO); MS *m/z* (relative intensity) 212 (M^+−pyrrolidine, 100), 183 (80). Anal. Calcd for $C_{19}H_{25}NO$: C, 80.52; H, 8.89; N, 4.94. Found: C, 80.43; H, 9.10; N, 4.83.

3.2.9. 2-(7-*exo*-Morpholinobicyclo[4.1.0]hept-7-yl)acetophenone (4e). Mp 78–79°C (hexane); IR (KBr) ν 1690 cm^{-1}; ^1H NMR δ 0.95–1.34 (m, 6H), 1.41–1.58 (m, 2H), 1.85–2.19 (m, 2H), 2.54 (t, J=4.6 Hz, 4H), 3.18 (s, 2H), 3.52 (t, J=4.6 Hz, 4H), 7.41–7.61 (m, 3H), 7.95–8.05 (m, 2H); ^{13}C NMR δ 19.88 (CH_2), 21.37 (CH), 21.73 (CH_2), 31.95 (CH_2), 47.71 (C), 49.85 (CH_2), 67.64 (CH_2), 127.85 (CH), 128.57 (CH), 132.79 (CH), 137.61 (C), 198.71 (CO); MS *m/z* (relative intensity) 299 (M^+, 7), 282 (17), 194 (100). Anal. Calcd for $C_{19}H_{25}NO_2$: C, 76.22; H, 8.42; N, 4.68. Found: C, 76.18; H, 8.46; N, 4.64.

3.2.10. 2-(7-*exo*-Morpholinobicyclo[4.1.0]hept-7-yl)-acetonitrile (5e). Mp 91–92°C (hexane); IR (KBr) ν 2245 cm^{-1}; ^1H NMR δ 1.01–1.40 (m, 6H), 1.40–1.60 (m, 2H), 1.85–2.05 (m, 2H), 2.48 (s, 2H), 2.71 (t, J=4.6 Hz, 4H), 3.60 (t, J=4.6 Hz, 4H); ^{13}C NMR δ 11.39 (CH_2), 18.51 (CH_2), 21.37 (CH_2), 21.65 (CH), 46.88 (C), 48.95 (CH_2), 67.38 (CH_2), 119.64 (CN); MS *m/z* (relative intensity) 220 (M^+, 43), 180 (47), 165 (49), 140 (100). Anal. Calcd for $C_{13}H_{20}N_2O$: C, 70.87; H, 9.15; N, 12.72. Found: C, 71.11; H, 8.90; N, 12.44.

3.3. Preparation of 7-*endo*-amino-7-*exo*-substituted bicyclo[4.1.0]heptane 9

To a magnetically stirred solution of bicyclo[4.1.0]heptan-7-one *N,O*-acetal **8** (2.11 g, 10 mmol) and 2-trimetylsiloxypropene (1.95 g, 15 mmol) in CH_2Cl_2 (30 mL), $BF_3{\cdot}Et_2O$ (1.42 g, 10 mmol) was added via syringe over a period of 10 min at −78°C. The reaction mixture was stirred for 3 h at this temperature, allowed to warm to 0°C over a period of 2 h, and quenched with saturated NaCl aqueous solution. The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (10 mL×2). The combined organic layers were dried over Na_2SO_4, and evaporated. The residue was column-chromatographed on silica gel (ether–hexane, 2:1) to give **9** as an oil (1.32 g, 56% yield), which was solidified on standing. The analytical sample was obtained by recrystallization from hexane at −30°C.

3.3.1. 1-(7-*endo*-Morpholinobicyclo[4.1.0]hept-7-yl)-2-propanone (9). Mp 60–61°C (hexane, −30°C); IR (KBr) ν 1707 cm^{-1}; ^1H NMR δ 0.82–0.95 (m, 2H), 1.12–1.31 (m, 2H), 1.40–1.65 (m, 4H), 1.65–1.90 (m, 2H), 2.18 (s, 3H), 2.49 (s, 2H), 2.51–2.70 (m, 4H), 3.48–3.68 (m, 2H), 3.68–3.85 (m, 2H); ^{13}C NMR δ 19.25 (CH_2), 21.19 (CH), 22.01 (CH_2), 30.89 (CH_3), 44.94 (C), 45.95 (CH_2), 50.30 (CH_2), 67.39 (CH_2), 208.64 (CO); MS *m/z* (relative intensity) 237 (M^+, 19), 220 (31), 195 (53), 194 (100). Anal. Calcd for

$C_{14}H_{23}NO_2$: C, 70.85; H, 9.77; N, 5.90. Found: C, 70.93; H, 9.80; N, 5.87.

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