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Indium(III) chloride-catalyzed Mukaiyama–Michael addition: synthesis of 2,6-*anti*-tetrahydropyrans

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ABSTRACT

In the presence of a catalytic amount of indium(III) chloride ($InCl_3$), silyl enol ethers react with dihydropyranones at ambient temperature via Mukaiyama–Michael addition to afford the corresponding 2,6-anti-tetrahydropyran adducts in moderate to good yields. This simple and mild method proceeds under neat conditions and involves simple aqueous work-up. Indium(III) chloride can be recovered and reused without significant decrease in reactivity.

1. Introduction

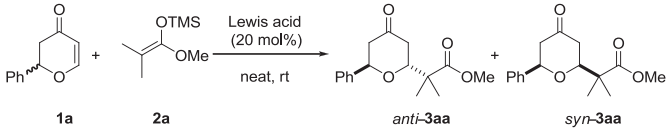
The ubiquitous presence of tetrahydropyran scaffolds in biologically active natural products warrants relentless ongoing efforts towards the development of new methods for the construction of functionalized tetrahydropyran rings.[1] In 2005, Clarke reviewed on various efficient methodologies for the asymmetric synthesis of tetrahydropyran rings, which include hetero Diels–Alder cyclization, Maitland–Japp multi-component reaction, Prins cyclization and intramolecular oxy-Michael reactions.[2] While most approaches can achieve satisfactory diastereoselectivities for the preparation of 2,6-*syn*-tetrahydropyrans, reported access to prepare the complementary 2,6-*anti*-tetrahydropyrans remained scarce.[3] In light of this, our group has recently vested interest in exploring new methodologies to construct 2,6-*anti*-tetrahydropyrans.[4]

Previously, we discovered that commercially available indium(III) chloride ($InCl_3$), a mild but versatile Lewis acid, is capable of catalyzing the Mukaiyama–Michael reaction between silyl enol ethers and α,β-unsaturated carbonyl compounds.[5] In the same report, we found that diastereoselectivity in the resultant adducts was inclined towards the *anti* configuration. This finding was gratifying and henceforth prompted us to apply similar methodology to synthesize tetrahydropyran rings, preferably resulting in 2,6-*anti* configuration.

2. Results and discussion

We embarked on our investigation with reactions of pyranone **1a** and silyl enol ether **2a** in the presence of a catalytic amount of Lewis acid (20 mol %) in neat conditions under N_2 atmosphere at room temperature. Table 1 lists representative data for the optimization of reaction conditions.

Table 1
Optimization of reaction conditions[a]



Entry	Lewis Acid	Yield (%)[b] (*anti*:*syn*)
1	None	0
2	$InCl_3$	83 (>99:1)
3	$InCl_3$[c]	82 (>99:1)
4	$InBr_3$	12 (>99:1)
5	$In(OTf)_3$	Trace
6	$ZnCl_2$	23 (>99:1)

[a] Reactions were carried out on the scale of 0.2 mmol of **1a** (racemic mixture) and **2a** in neat conditions under N_2 atmosphere at room temperature for 1 h.
[b] Isolated yields.
[c] Yield of second run. $InCl_3$ was recycled: after the removal of water under reduced pressure and subsequently oven-dried at 150 °C for 2 h.

As shown in Table 1 (entry 1), there was no desired product formed when the reaction was performed in the absence of any Lewis acid. This finding implied that the Lewis acid plays a crucial

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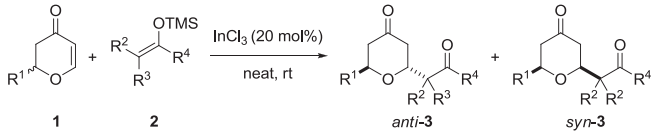
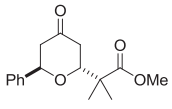
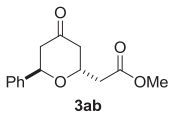
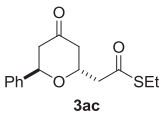
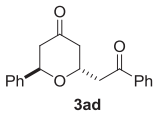
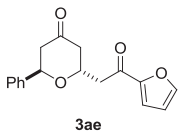
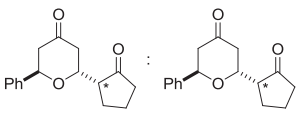
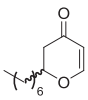
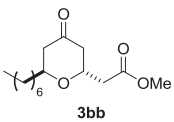
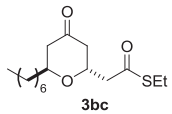
role in the reaction. Among the Lewis acids screened, indium(III) chloride ($InCl_3$) provided the desired product in good yield (83%) (Table 1, entry 2). It is noteworthy that the yield was reproducible even with recycled $InCl_3$ (Table 1, entry 3). Although other Lewis acids were less efficient, it is gratifying that all Lewis acids screened gave high diastereoselectivities, except for $In(OTf)_3$ where the low yield excluded the possibility of analyzing the diastereoselectivity. It is also worth mentioning that only 1,4-addition adducts (Mukaiyama–Michael products) were obtained, with no 1,2-addition adducts (Mukaiyama-aldol products). By utilizing $InCl_3$ as our choice of Lewis acid, the scope of the present Mukaiyama–Michael reaction was explored (Table 2). Silyl enol ethers with heteroatoms, such as **2a**, **2c** and **2e** (Table 2, entries 1, 3 and 5) generally gave better yields compared to silyl enol ethers, such as **2d** (Table 2, entry 4). Sluggish transformations were observed with silyl enol ether **2b**, affording only 48% and 63% yields with pyranones **1a** and **1b**, respectively, after 20 h (Table 2, entries 2 and 7). Pyranone with an aliphatic substituent **1b** gave a higher yield than that with an aromatic substituent **1a** (Table 2, compare entries 2 and 7 or entries 3 and 8). When prochiral silyl enol ether **2f** was used, a combined yield of 60% was obtained with no diastereoselectivity observed (*anti*:*syn*=1:1) (Table 2, entry 6). From the results shown in Tables 1 and 2, we propose that $InCl_3$ activates the conjugated system of the substituted pyranone **1** towards nucleophilic attack by the silyl enol ether **2**. The activated substituted pyranone **1** would preferentially assume a chair conformation in the transition state, which is of a lower energy than the alternative twist boat conformation (Scheme 1). Hence, giving rise to 2,6-*anti*-tetrahydropyran adducts selectively.

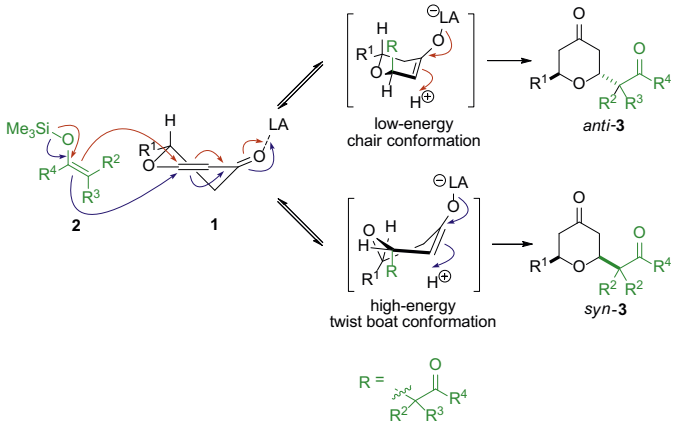
Table 2
$InCl_3$-catalyzed Mukaiyama–Michael reaction[a]



1 **2** *anti*-**3** *syn*-**3**

Entry	Pyranone **1**	Silyl enol ether **2**	Michael adduct **3**/yield[b]
1	 **1a**	 **2a**	 **3aa** (83%; *anti* only)
2	**1a**	 **2b**	 **3ab** (48%; *anti* only)
3	**1a**	 **2c**	 **3ac** (78%; *anti* only)
4	**1a**	 **2d**	 **3ad** (28%; *anti* only)
5	**1a**	 **2e**	 **3ae** (83%; *anti* only)
6	**1a**	 **2f**	 *anti*-**3af** *syn*-**3af** (60%; *anti*:*syn* = 1:1)
7	 **1b**	**2b**	 **3bb** (63%; *anti* only)
8	**1b**	**2c**	 **3bc** (84%; *anti* only)

[a] Reactions were carried out on the scale of 0.2 mmol of **1** (racemic mixture) and **2** in neat conditions under N_2 atmosphere at room temperature for 3–20 h.
[b] Isolated yields.



Scheme 1. Proposed mechanism for *anti*-selective Mukaiyama–Michael reaction.

3. Conclusion

In summary, we have developed a simple methodology to access 2,6-*anti*-tetrahydropyran rings via $InCl_3$-catalyzed Mukaiyama–Michael addition. The mild and solvent-free conditions employed would allow application of substrates that are acid-sensitive. $InCl_3$ can be recycled without losing its reactivity to give reproducible results. Efforts to apply this methodology in the syntheses of natural products are ongoing in our group.

4. Experimental section

Analytical thin layer chromatography (TLC) was performed using Merck 60 F_{254} precoated silica gel plate (0.2 mm thickness). Subsequent to elution, plates were visualized using UV radiation (254 nm) with a Spectroline Model ENF-24061/F_{254} nm. Further visualization was possible by staining with an acidic solution of ceric molybdate or an ethanolic solution of ninhydrin. Flash-column chromatography was performed using Merck silica gel 60 with freshly distilled solvents. ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance DPX 300 and Bruker AMX 400 spectrophotometers using TMS as an internal standard. Chemical shifts for ^1H NMR spectra are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-*d* (δ 7.26, s). Multiplicities were given as: s (singlet); d (doublet); dd (doublets of doublet); ddd (doublets of

doublets of doublet); t (triplet); q (quartet); or m (multiplets). The number of protons (*n*) for a given resonance is indicated by *n*H. Coupling constants are reported as a *J* value in hertz (Hz). Carbon nuclear magnetic resonance spectra (^{13}C NMR) are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-*d* (δ 77.0, t). HRMS spectra were obtained using Finnigan MAT95XP GC/HRMS (Thermo Electron Corporation). IR spectra were recorded on a Bio-Rad FTS 165 FTIR spectrometer.

4.1. Preparation of pyranones 1 and silyl enol ethers 2

All racemic pyranones **1** were prepared from Lewis acid-catalyzed hetero Diels–Alder reaction of the corresponding aldehydes with Danishefsky diene.[6]

Silyl enol ethers **2** (with the exception of **2c**) were prepared according to the literature.[7]

*4.1.1. (1-(Ethylthio)vinyloxy)trimethylsilane (**2c**)*[8]. A stirred solution of ethyl thioacetate (5.3 mL, 50 mmol, 1 equiv) in dichloromethane (50 mL) was added to triethylamine (8.4 mL, 60 mmol, 1.2 equiv). The mixture was cooled down to 0 °C. Trimethylsilyl trifluoromethanesulfonate (9.0 mL, 50 mmol, 1 equiv) was added dropwise and the reaction was stirred at 0 °C for 2 h. Dichloromethane was removed in vacuo and the two layers were separated. The upper layer was collected and was sufficiently pure for subsequent reactions. (1-(*ethylthio*)*vinyloxy*)*trimethylsilane* **2c** was afforded as yellow oil (7.40 g, 84% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.42 (d, *J*=1.6 Hz, 1H), 4.35 (d, *J*=2.0 Hz, 1H), 2.69 (q, *J*=7.2 Hz, 2H), 1.28 (t, *J*=7.6 Hz, 3H), 0.26 (s, 9H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.4, 93.0, 25.6, 14.3, −0.1 ppm.

4.2. Representative experimental procedure for $InCl_3$-catalyzed Mukaiyama–Michael addition

A mixture of pyranone (0.2 mmol, 1 equiv) and indium trichloride (8.8 mg, 0.04 mmol, 0.2 equiv) was stirred neat at room temperature for 15 min. Silyl enol ether (0.4 mmol, 2 equiv) was added and the mixture stirred for 1 h. A mixture of THF: 1 M hydrochloric acid (1:1) (3 mL) was added and the reaction mixture was stirred for 30 min. The mixture was extracted with ethyl acetate (3×15 mL). The combined organic extracts were washed with water (20 mL) and brine (20 mL). The organic layer was dried over anhydrous magnesium sulfate, filtered and concentrated in vacuo. The residual crude product was purified by flash column chromatography.

*4.2.1. Methyl 2-methyl-2-(4-oxo-6-phenyltertrahydro-2H-pyran-2-yl) propanoate (**3aa**).* Oil (83%). R_f=0.55 (ethyl acetate/hexane=1:2); FTIR (NaCl, neat) ν_{max}: 1722, 1643, 1267, 698 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37–7.28 (m, 5H), 5.45, (dd, *J*=7.2, 2.4 Hz, 1H), 3.74 (dd, *J*=11.6, 2.4 Hz, 1H), 3.60 (s, 3H), 2.98 (d, *J*=15.2 Hz, 1H), 2.89 (dd, *J*=15.2, 7.2 Hz, 1H), 2.52 (dd, *J*=14.8, 11.6 Hz, 1H), 2.30 (d, *J*=14.4 Hz, 1H), 1.20 (s, 3H), 1.09 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 207.4, 176.1, 139.2, 128.6, 128.3, 127.9, 74.6, 74.5, 52.0, 46.4, 43.7, 42.6, 21.3 ppm; HRMS (EI) *m*/*z* calcd for $C_{16}H_{20}O_4$ [M^+]= 276.1362; found 276.1351.

*4.2.2. Methyl 2-(4-oxo-6-phenyltetrahydro-2H-pyran-2-yl)acetate (**3ab**).* Oil (48%). R_f=0.37 (ethyl acetate/hexane=1:2); FTIR (NaCl, neat) ν_{max}: 1721, 1643, 1258, 700 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.41–7.28 (m, 5H), 5.31 (t, *J*=5.4 Hz, 1H), 4.40–4.31 (m, 1H), 3.68 (s, 3H), 2.92 (ddd, *J*=14.7, 5.4, 1.2 Hz, 1H), 2.83 (ddd, *J*=14.7, 5.4, 1.5 Hz, 1H), 2.70 (dd, *J*=15.0, 7.8 Hz, 1H), 2.59 (ddd, *J*=14.7, 4.5, 1.5 Hz, 1H), 2.51 (dd, *J*=15.3, 5.7 Hz, 1H), 2.44 (ddd, *J*=15.0, 8.4, 1.2 Hz, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ 206.2, 170.6, 139.4, 128.8, 128.4, 127.2, 74.3, 68.7, 52.0, 46.6, 45.5, 40.1 ppm; HRMS (EI) *m*/*z* calcd for $C_{14}H_{16}O_4$ [M^+]=248.1049; found 248.1049.

*4.2.3. S-Ethyl 2-((2R,6R)-4-oxo-6-phenyltetrahydro-2H-pyran-2-yl) ethanethioate (**3ac**).* Oil (78%). R_f=0.61 (ethyl acetate/hexane=1:1); FTIR (NaCl, neat) ν_{max}: 1722, 1690, 1456, 1232, 756 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.36–7.29 (m, 5H), 5.29 (t, *J*=5.4 Hz, 1H), 4.46–4.37 (m, 1H), 2.98–2.85 (m, 4H), 2.81 (dd, *J*=15.0, 5.7 Hz, 1H), 2.68 (dd, *J*=15.0, 5.4 Hz, 1H), 2.58 (dd, *J*=14.7, 4.2 Hz, 1H), 2.42 (dd, *J*=14.7, 7.8 Hz, 1H), 1.25 (t, *J*=4.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ 205.8, 195.6, 139.2, 128.6, 127.3, 126.9, 74.1, 68.9, 48.7, 46.3, 45.5, 23.5, 14.6 ppm; HRMS (EI) *m*/*z* calcd for $C_{15}H_{18}O_3S$ [M^+]= 278.0977; found 278.0970.

*4.2.4. 2-(2-Oxo-2-phenylwthyl)-6-phenyldihydro-2H-pyran-4(3H)-one (**3ad**).* Oil (28%). R_f=0.50 (ethyl acetate/hexane=1:2); FTIR (NaCl, neat) ν_{max}: 1715, 1643, 690, 523 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J*=7.2 Hz, 2H), 7.59 (t, *J*=7.3 Hz, 1H), 7.46 (t, *J*=7.8 Hz, 2H), 7.36–7.31 (m, 5H), 5.29 (t, *J*=5.6 Hz, 1H), 4.64–4.57 (m, 1H), 3.40 (dd, *J*=16.1, 6.5 Hz, 1H), 3.15 (dd, *J*=16.1, 6.4 Hz, 1H), 2.93 (ddd, *J*=14.6, 5.8, 1.1 Hz, 1H), 2.84 (ddd, *J*=14.6, 5.6, 0.9 Hz, 1H), 2.71 (dd, *J*=13.7, 4.1 Hz, 1H), 2.49 (dd, *J*=14.8, 8.2 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 206.6, 197.0, 139.6, 137.0, 133.6, 128.9, 128.8, 128.3, 127.0 (2C), 74.6, 68.8, 47.0, 45.9, 43.9 ppm; HRMS (EI) *m*/*z* calcd for $C_{19}H_{18}O_3$ [M^+]=294.1256; found 294.1291.

*4.2.5. Methyl 2-methyl-2-(4-oxo-6-phenyltertrahydro-2H-pyran-2-yl) propanoate (**3ae**).* Oil (83%). R_f=0.33 (ethyl acetate/hexane=1:2); FTIR (NaCl, neat) ν_{max}: 3063, 1643, 1265, 737 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (s, 1H), 7.36–7.27 (m, 5H), 7.21 (d, *J*=3.6 Hz, 1H), 6.55–6.53 (m, 1H), 5.30 (t, *J*=5.6 Hz, 1H), 4.62–4.56 (m, 1H), 3.25 (dd, *J*=15.4, 7.5 Hz, 1H), 2.98 (dd, *J*=15.4, 5.8 Hz, 1H), 2.92 (dd, *J*=14.8, 5.6 Hz, 1H), 2.84 (dd, *J*=14.8, 5.6 Hz, 1H), 2.66 (dd, *J*=14.8, 4.3 Hz, 1H), 2.48 (dd, *J*=14.7, 8.2 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 206.5, 185.8, 146.8, 142.8, 139.5, 128.8, 128.3, 127.0, 117.8, 112.7, 74.4, 68.6, 46.9, 45.8, 43.8 ppm; HRMS (EI) *m*/*z* calcd for $C_{17}H_{16}O_4$ [M^+]=284.1049; found 284.2482.

*4.2.6. 2-(2-Oxocyclopentyl)-6-phenyldihydro-2-H-pyran-4(3H)-one (**3af**).* Oil (60%). R_f=0.67 (ethyl acetate/hexane=1:8); FTIR (NaCl, neat) ν_{max}: 1730, 1450, 1265, 737 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) (for two diastereomers): δ 7.40–7.29 (m, 5H), 5.40 (dd, *J*=6.3, 3.8 Hz, 0.5H), 5.28 (t, *J*=5.4 Hz, 0.5H), 4.12 (dt, *J*=9.5, 4.3 Hz, 0.5H), 3.90 (dt, *J*=10.8, 3.2 Hz, 0.5H), 3.12 (dd, *J*=15.0, 10.8 Hz, 0.5H), 2.98–2.80 (m, 2H), 2.60 (ddd, *J*=15.0, 9.6, 0.8 Hz, 0.5H), 2.49 (ddd, *J*=15.0, 3.6, 1.4 Hz, 0.5H), 2.36–2.03 (m, 5.5H), 1.96–1.79 (m, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) (for two diastereomers): δ 218.9, 217.9, 207.2, 206.9, 139.4, 128.9, 128.8, 128.3, 127.6, 127.2, 74.7, 74.4, 71.2, 70.1, 52.6, 51.1, 45.6, 45.1, 44.7, 44.2, 39.8, 39.2, 26.9, 24.8, 21.0, 20.8 ppm; HRMS (EI) *m*/*z* calcd for $C_{16}H_{18}O_3$ [M^+]=258.1256; found 258.1238.

*4.2.7. Methyl 2-(6-heptyl-4-oxotetrahydro-2H-pyran-2-yl)acetate (**3bb**).* Oil (63%). R_f=0.30 (ethyl acetate/hexane=1:4); FTIR (NaCl, neat) ν_{max}: 1732, 1436, 1265, 704 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.54–4.51 (m, 1H), 4.14–4.08 (m, 1H), 3.70 (s, 3H), 2.68–2.46 (m, 6H), 1.27–1.23 (m, 12H), 0.88 (t, *J*=6.3 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ 206.6, 170.6, 72.9, 68.5, 51.9, 46.7, 46.4, 39.9, 33.9, 31.8, 29.3, 29.2, 25.2, 22.6, 14.1 ppm; HRMS (EI) *m*/*z* calcd for $C_{15}H_{27}O_4$ [M+H]$^+$=271.1909; found 271.1909.

*4.2.8. S-Ethyl 2-(6-heptyl-4-oxotetrahydro-2H-pyran-2-yl)ethanethioate (**3bc**).* Oil (84%). R_f=0.41 (ethyl acetate/hexane=1:4); FTIR (NaCl, neat) ν_{max}: 1720, 1685, 1456, 1232, 756 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.62–4.53 (m, 1H), 4.16–4.08 (m, 1H), 2.94–2.84 (m, 3H), 2.68–2.52 (m, 3H), 2.35–2.26 (m, 2H), 1.29–1.24 (m, 15H), 0.88 (t, *J*=6.3 Hz, 3H) ppm; ^{13}C NMR (75 MHz,

$CDCl_3$): δ 206.5, 195.5, 72.9, 68.7, 48.7, 46.7, 46.4, 34.1, 31.8, 31.6, 29.3, 29.2, 25.2, 23.5, 22.6, 14.1 ppm; HRMS (EI) m/z calcd for $C_{16}H_{29}O_3S$ $[M+H]^+$=301.1837; found 301.1832.

Acknowledgements

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References and notes

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