# **Supporting Information**

## Aromatic Cation Activation of Alcohols: Conversion to Alkyl Chlorides using Dichlorodiphenylcyclopropenes

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**General Information.** All reactions were performed using oven-dried glassware under an atmosphere of dry argon. Non-aqueous reagents were transferred by syringe under argon. Organic solutions were concentrated using a Buchi rotary evaporator. Tetrahydrofuran, toluene, and methylene chloride (CH<sub>2</sub>Cl<sub>2</sub>) were dried using a J.C. Meyer solvent purification system. 1,2-Dichloroethane (DCE) and acetonitrile (CH<sub>3</sub>CN) were freshly distilled over CaH<sub>2</sub> under argon. Acetone and dimethyl sulfoxide were used in their deuterated form as packaged in ampules. All other commercial reagents were used as provided. Flash column chromatography was performed employing 32-63  $\mu$ m silica gel (Dynamic Adsorbents Inc). Thin-layer chromatography (TLC) was performed on silica gel 60 F<sub>254</sub> plates (EMD).

<sup>1</sup>H and <sup>13</sup>C NMR were recorded in CDCl<sub>3</sub> on Bruker DRX-300 and DRX-400 spectrometers as noted. Data for <sup>1</sup>H NMR are reported as follows: chemical shift ( $\delta$  ppm), multiplicity (s = singlet, brs = broad singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant (Hz), integration, and assignment. Data for <sup>13</sup>C NMR are reported in terms of chemical shift. Gas chromatography was performed on a Varian 3900 gas chromatograph equipped with a Varian 25m CP-Chirasil-Dex CB capillary column using the following conditions: 220 °C injector temp, 0.7 mL/min flow rate (see below for oven temperatures).

### Synthesis of Cyclopropenes:



**1,2-Diphenylcyclopropenone:** Following the method of Breslow,<sup>1</sup> 1,3-diphenyl acetone (17.5 g, 83.2 mmol) was added to a 500-mL round-bottomed flask, followed by glacial acetic acid (62 mL). A dropping funnel containing bromine (27.5 g, 172.1 mmol) in glacial acetic acid (125 mL) was fitted to the flask. The solution was added over a period of 15 min at 23 °C. After addition was complete, the mixture was stirred for an additional 15 min. The mixture was then poured into water (250 mL). Solid Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> was added to the mixture until the initial yellow color disappeared and the mixture was allowed to stand for 1 h. The light yellow solid was filtered and air-dried. The yellow solid was recrystallized from petroleum ether (with a few drops of benzene), and dried under vacuum to afford the intermediate di-bromide as a white solid (24.2 g, 65.8 mmol, 79% yield).

To a 500-mL round-bottomed flask containing  $CH_2Cl_2$  (55 mL), was added triethylamine (24.0 mL, 172 mmol) at 23 °C. The flask was fitted with a dropping funnel containing the intermediate di-bromide (24.0 g, 65.2 mmol) in  $CH_2Cl_2$  (110 mL). This solution was added over 1 h. After addition was complete, the solution was stirred for an additional 30 min. The red mixture was then washed with 3 N HCl (3 x 40 mL). The organic layer was transferred to a 500-mL Erlenmeyer flask and cooled to 0 °C in an ice bath. To this stirring solution was slowly added a cold solution of sulfuric acid (12.5 mL) in water (6 mL). Upon addition, a pink precipitate formed, which was collected on a fritted funnel and washed with  $CH_2Cl_2$ . The solid was returned to the flask and diluted with  $CH_2Cl_2$  (60 mL) and water (125 mL). After neutralization by addition of  $Na_2CO_3$  (1.1 g) in small portions, the layers were separated and the aqueous layer was extracted with  $CH_2Cl_2$  (3 x 60 mL). The combined organics were washed with brine (100 mL), dried (MgSO<sub>4</sub>) and concentrated under vacuum to afford a pink solid. The crude pink solid was purified by silica gel chromatography (50%-100% EtOAc:hexanes) to provide the title compound as a white solid (8.1 g, 39.3 mmol, 60% yield). <sup>1</sup>H NMR (400 MHz,

<sup>&</sup>lt;sup>1</sup> Breslow, R; Posner, J. Org. Syntheses. 1967, 47, 62.

CDCl<sub>3</sub>) δ 7.97-7.94 (m, 4H, Ar**H**), 7.57-7.55 (m, 6H, Ar**H**). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 155.7, 148.3, 132.6, 131.4, 129.3, 124.0.



**3,3-Dichloro-1,2-diphenylcyclopropene**:<sup>2</sup> Following the method of Perkins, diphenylcyclopropenone (4.0 g, 19.4 mmol) was added to a 100-mL round-bottomed flask fitted with a reflux condenser. To this, was added neat thionyl chloride (40 mL, 550 mmol) and solution was heated to 50 °C for 2 h. After 2 h, the reaction was cooled to 23 °C and concentrated under vacuum to yield a light yellow solid. The solid was recrystallized from hexanes to afford a white solid (4.4 g, 16.9 mmol, 87% yield). <sup>1</sup>H NMR (400 MHz, CD<sub>3</sub>CN)  $\delta$  8.18-8.16 (m, 4H, Ar**H**), 7.77-7.73 (m, 2H, Ar**H**), 7.71-7.67 (m, 4H, Ar**H**). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  131.3, 130.2, 129.3, 125.8, 123.9.

#### Synthesis of Substrates:



(Z)-4-Hydroxybut-2-enyl acetate:<sup>3</sup> To a stirring solution of NaH (60% in mineral oil, 1.36 g, 34 mmol) in THF (50 mL) was slowly added (Z)-2-butene-1,4-diol (9.6 mL, 102 mmol) at 23 °C. After stirring for an additional 12 h, Ac<sub>2</sub>O (3.2 mL, 34 mmol) was added and the solution was stirred for 2 h at 23 °C. After 2 h, the solution was poured into ice and the aqueous layer that developed was extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 x 40 mL). The combined organic were dried (Na<sub>2</sub>SO<sub>4</sub>) and concentrated under vacumm. The crude residue was purified by silica gel chromatography (50% EtOAc:hexanes) to provide the title compound as a clear oil (2.9 g, 22.4 mmol, 66% yield overall). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  5.79-5.72 (m, 1H, AcOCH<sub>2</sub>CH=CH), 5.57-5.50 (m, 1H, AcOCH<sub>2</sub>CH=CH), 4.58 (d, J = 6.9 Hz, 2H, AcOCH<sub>2</sub>), 4.15 (d, J = 6.6 Hz, 2H, CH<sub>2</sub>Cl), 1.99 (s, 3H, CH<sub>3</sub>). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.1, 133.3, 125.1, 60.1, 58.0, 20.7.

<sup>&</sup>lt;sup>2</sup> Perkins, W.C.; Wadsworth, D.H. Synthesis. 1972, 205.

<sup>&</sup>lt;sup>3</sup> Genet, J.P.; Thorimbert, S; Mallart, S; Kardos, N. Synthesis. 1993, 321.

#### **Synthesis of Chlorides**

**General Procedure:** To a stirring solution of alcohol in 0.75-1.3 mL of freshly distilled  $CH_2Cl_2$  (or  $CH_3CN$ ) was added the dichlorocyclopropene. The mixture was stirred at 23 °C (or 80 °C) for 3 to 65 min, depending on the alcohol. When the reaction was complete (monitored by TLC), the reaction mixture was eluted through a short silica gel plug eluting with 10% EtOAc:hexanes. When necessary, the crude chloride was purified by silica gel chromatography.



(Chloromethyl)benzene (Table 1, entry 1):<sup>4</sup> Prepared according to the general procedure from benzyl alcohol (21.6 mg, 0.20 mmol) and dichlorocyclopropene (57.5 mg, 0.22 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (1.3 mL) at 23 °C to yield a pale yellow oil (20.4 mg, 0.16 mmol, 81% yield). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.42-7.33 (m, 5H, ArH), 4.60 (s, 2H, CH<sub>2</sub>Cl). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  137.5, 128.7, 128.6, 128.4, 46.2.



**Cinnamyl chloride (Table 1, entry 1)**:<sup>5</sup> Prepared according to the general procedure from cinnamyl alcohol (31.0 mg, 0.23 mmol) and dichlorocyclopropene (66.1 mg, 0.25 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (1.5 mL) at 23 °C to yield a pale yellow oil (32.4 mg, 0.21 mmol, 92% yield). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.44-7.30 (m, 5H, Ar**H**), 6.68 (d, J = 15.6, 1H, C**H**=CHCH<sub>2</sub>Cl), 6.35 (dt, J = 15.6, 7.1 Hz, 1H, CH=C**H**CH<sub>2</sub>Cl), 4.27 (d, J = 7.1, 2H, C**H**<sub>2</sub>Cl). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  135.9, 134.1, 128.6, 128.2, 126.7, 124.9, 45.4.

<sup>&</sup>lt;sup>4</sup> Altamura, M; Perrotta, E. J. Org. Chem. 1993, 58, 272.

<sup>&</sup>lt;sup>5</sup> Goren, Z; Heeg, M.J.; Mobashery, S. J. Org. Chem.. 1991, 56, 7186.



**Geranyl chloride (Table 1, entry 1)**:<sup>6</sup> Prepared according to the general procedure from geraniol (30.9 mg, 0.20 mmol) and dichlorocyclopropene (57.5 mg, 0.22 mmol) in  $CH_2Cl_2$  (1.3 mL) at 23 °C to yield a clear oil (32.7 mg, 0.19 mmol, 95% yield).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  5.45 (t, J = 8.0 Hz, 1H, CHCH<sub>2</sub>Cl), 5.09-5.06 (m, 1H, (CH<sub>3</sub>)<sub>2</sub>C=CH), 4.09 (d, J = 8.0 Hz, 2H, CHCH<sub>2</sub>Cl), 2.10-2.06 (m, 4H, CH<sub>2</sub>CH<sub>2</sub>), 1.73 (s, 3H, CH<sub>3</sub>), 1.69 (s, 3H, CH<sub>3</sub>), 1.60 (s, 3H, CH<sub>3</sub>). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  142.7, 131.9, 123.6, 120.3, 41.1, 39.4, 26.2, 25.6, 17.7, 16.1.



(Z)-4-Chlorobut-2-enyl acetate (Table 1, entry 1):<sup>7</sup> Prepared according to the general procedure from (Z)-4-hydroxybut-2-enyl acetate (26.0 mg, 0.20 mmol) and dichlorocyclopropene (57.5 mg, 0.22 mmol) in  $CH_2Cl_2$  (1.3 mL) at 23 °C to yield a clear oil (24.9 mg, 0.17 mmol, 84% yield).

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  5.89-5.83 (m, 1H, AcOCH<sub>2</sub>CH=CH), 5.80-5.69 (m, 1H, AcOCH<sub>2</sub>CH=CH), 4.66 (d, J = 6.7 Hz, 2H, AcOCH<sub>2</sub>), 4.13 (d, J = 7.6 Hz, 2H, CH<sub>2</sub>Cl), 2.07 (s, 3H, CH<sub>3</sub>). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  170.6, 129.7, 127.9, 59.3, 38.6, 20.8.



**1-Chlorooct-2-yne (Table 1, entry 1**):<sup>8</sup> Prepared according to the general procedure from 2octyn-1-ol (25.2 mg, 0.20 mmol) and dichlorocyclopropene (57.5 mg, 0.22 mmol) in  $CH_2Cl_2$  (1.3 mL) at 23 °C to yield a clear oil (26.8 mg, 0.21 mmol, 92% yield).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  4.14 (t, J = 2.2 Hz, 2H, CH<sub>2</sub>Cl), 2.24-2.20 (m, 2H, CH<sub>2</sub>CH), 1.53-1.49 (m, 2H, CH<sub>2</sub>CH<sub>2</sub>CH), 1.38-1.31 (m, 4H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH), 0.91-0.89 (t, J = 7.1 Hz, 3H CH<sub>3</sub>CH<sub>2</sub>). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  87.8, 74.9, 31.3, 31.0, 28.0, 22.1, 18.8, 13.9.

<sup>&</sup>lt;sup>6</sup> Nowotny, S; Tucker, C.E.; Jubert, C; Knochel, P. J. Org. Chem.. 1995, 60, 2762.

<sup>&</sup>lt;sup>7</sup> Backvall, J-E.; Nystrom, J-E.; Nordberg, R.E. J. Am. Chem. Soc. **1985**, 107, 3676.

<sup>&</sup>lt;sup>8</sup> Condon-Gueugnot, S; Linstrumelle, G. Tetrahedron. 2000, 56, 1851.



**3-Chlorocyclohex-1-ene (Table 1, entry 1)**:<sup>9</sup> Prepared according to the general procedure from 2-cyclohexen-1-ol (9.8 mg, 0.10 mmol) and dichlorocyclopropene (26.1 mg, 0.1 mmol) in d-CH<sub>3</sub>CN (0.75 mL) at 80 °C. Yield calculated using benzyl ether (5  $\mu$ L, 0.0263 mmol) as a NMR standard (88% yield).

<sup>1</sup>H NMR (400 MHz, CD<sub>3</sub>CN) δ 5.90-5.85 (m, 1H, CH=CHCHCl), 5.80-5.77 (m, 1H, CH=CHCHCl), 4.70-4.66 (m, 1H, CH=CHCHCl), 2.06-1.95 (m, 4H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH=CH), 1.83-1.78 (m, 1H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH=CH), 1.66-1.62 (m, 1H, CH<sub>2</sub>CH<sub>2</sub>CH=CH).



**Methyl 2-chloro-2-phenylacetate (Table 1, entry 1)**:<sup>10</sup> Prepared according to the general procedure from (*S*)-methyl mandelate (25.0 mg, 0.15 mmol) and dichlorocyclopropene (58.8 mg, 0.23 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (1.0 mL) at 23 °C to yield a clear oil (25.8 mg, 0.14 mmol, 93% yield). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.51-7.49 (m, 2H, Ar**H**), 7.40-7.7.37 (m, 3H, Ar**H**), 5.37 (s, 1H, C**H**Cl), 3.78 (s, 3H, C**H**<sub>3</sub>). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  168.8, 135.7, 129.3, 128.8, 127.9, 58.9, 53.3.



(2-Chloroethyl)benzene (Table 1, entry 1):<sup>11</sup> Prepared according to the general procedure from 1-phenylethanol (24.4 mg, 0.20 mmol) and dichlorocyclopropene (57.5 mg, 0.22 mmol) in  $CH_2Cl_2$  (1.3 mL) at 23 °C to yield a pale yellow oil (25.1 mg, 0.18 mmol, 89% yield). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.38-7.24 (m, 5H, ArH), 3.75 (t, J = 7.4 Hz, 2H, CH<sub>2</sub>CH<sub>2</sub>Cl),

 $3.10 \text{ (t, J} = 7.4 \text{ Hz, 2H, CH}_2\text{CH}_2\text{Cl}). \ ^{13}\text{C NMR} (75 \text{ MHz, CDCl}_3) \delta 138.1, 128.8, 128.6, 126.9, 44.9, 39.2.$ 

<sup>&</sup>lt;sup>9</sup> Bentley, T.W.; Christl, M; Kemmer, R; Llewellyn, G; Oakley, J.E. J. Chem. Soc., Perkin Trans. 2. **1994**, 2531.

<sup>&</sup>lt;sup>10</sup> Yus, M; Herrera, R.P; Guijarro, A. *Chem. Eur. J.* **2002**, 8, 2574

<sup>&</sup>lt;sup>11</sup> Drabowicz, J; Luczak, J; Mikolajczyk, M. J. Org. Chem. **1998**, 63, 9565.



(2-Chloropropyl)benzene (Table 1, entry 1):<sup>12</sup> Prepared according to the general procedure from 1-phenyl-2-propanol (24.3 mg, 0.18 mmol) and dichlorocyclopropene (70.0 mg, 0.27 mmol) in CH<sub>3</sub>CN (1.0 mL) at 80 °C to yield a pale yellow oil (26.4 mg, 0.17 mmol, 95% yield). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.38-7.24 (m, 5H, ArH), 4.26 (q, J = 6.7 Hz, 1H, CHCl), 3.13 (dd, J = 7.0, 13.9 Hz, 1H, CH<sub>2</sub>CHCl), 3.00 (dd, J = 7.0, 13.9 Hz, 1H, CH<sub>2</sub>CHCl), 1.55 (d, J = 6.5 Hz, 3H, CH<sub>3</sub>). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  138.0, 129.3, 128.4, 126.8, 58.5, 46.7, 24.6.



**2-Chlorooctane (Table 1, entry 1)**:<sup>13</sup> Prepared according to the general procedure from 2-octanol (26.0 mg, 0.2 mmol) and dichlorocyclopropene (78.0 mg, 0.3 mmol) in d-CH<sub>3</sub>CN (1.0 mL) at 80 °C. Yield calculated using benzyl ether (10  $\mu$ L, 0.0526 mmol) as a NMR standard (93% yield).

<sup>1</sup>H NMR (400 MHz, CD<sub>3</sub>CN)  $\delta$  4.06 (sextet, J = 6.6 Hz, 1H, CHCl), 1.69-1.64 (m, 2H, CH<sub>2</sub>CHCl), 1.46 (d, J = 6.6 Hz, 3H, CHClCH<sub>3</sub>), 1.40-1.24 (m, 8H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>), 0.89 (t, J = 7.0 Hz, 3H, CH<sub>2</sub>CH<sub>3</sub>). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  58.9, 40.4, 31.7, 28.8, 26.6, 25.3, 22.6, 14.0.



(2-Chloro-2-methylpropyl)benzene (Table 1, entry 1): Prepared according to the general procedure from 2-methyl-1-phenyl-2-propanol (22.5 mg, 0.15 mmol) and dichlorocyclopropene

<sup>&</sup>lt;sup>12</sup> Yasuda, M; Yamasaki, S; Onishi, Y; Baba, A. J. Am. Chem. Soc. 2004, 126, 7186.

<sup>&</sup>lt;sup>13</sup> Haughton, L; Williams, J.M.J. Synthesis. 2001, 943.

(43.1 mg, 0.17 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (1.0 mL) at 23 °C. Yield calculated using benzyl ether (10  $\mu$ L, 0.0526 mmol) as a NMR standard (45% yield of the chloride<sup>14</sup>, 33% of the styrene<sup>15</sup>).

*Chloride* - <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.31-7.21 (m, 5H, Ar**H**), 3.08 (s, 2H, C**H**<sub>2</sub>), 1.58 (s, 6H, C**H**<sub>3</sub>).

*Styrene* - <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.31-7.21 (m, 5H, Ar**H**), 6.25 (bs, 1H, C**H**), 1.90 (d, J = 1.2 Hz. 3H, C**H**<sub>3</sub>), 1.86 (d, J = 1.2 Hz, 3H, C**H**<sub>3</sub>).



**Gram-scale preparation of (R)-(1-chloroethyl)benzene**:<sup>16</sup> Prepared from (*S*)-methylbenzyl alcohol (1.00 g, 8.19 mmol, 99% ee) and dichlorocyclopropene (2.25 g, 6.82 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (40 mL) at 23 °C. After 10 min, the reaction solution was concentrated *in vacuo* to produce an off-white, solid mixture. This crude mixture was diluted with hexanes and the resultant suspension was decanted/filtered through a short silica plug, leaving an off-white solid behind. The solid mixture was triturated twice more and the filtered solution was concentrated *in vacuo* to yield the desired chloride as a clear oil (1.04 g, 7.4 mmol, 90% yield, 93% ee). The off-white solid remaining after trituration was purified by means of a short silica plug (50% EtOAc:hexanes  $\rightarrow$  100% EtOAc) to yield the cyclopropenone as a white solid (1.60 g, 7.8 mmol, 90% recovery from dichlorocyclopropene – NMR of recovered cyclopropenone provided below). Enantiomeric excess determined by chiral GC chromatography.<sup>17</sup> Injection of 1 uL (1:100 split) of a 0.5 mg/mL sample on a Varian CP-Chiralsil-Dex CB column, retention time = 13.6 min (*S* isomer) and 13.8 min (*R* isomer) using the following method: 60 °C, hold for two min; 5 °C/min to 95 °C, hold for 20 min.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.46-7.30 (m, 5H, Ar**H**), 5.12 (q, J = 6.8 Hz, 1H, C**H**Cl), 1.88 (d, J = 6.9 Hz, 3H, C**H**<sub>3</sub>). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  142.8, 128.6, 128.2, 126.4, 58.7, 26.5.

<sup>&</sup>lt;sup>14</sup> Shelvin, P.B.; Hansem, H.J. J. Org. Chem. 1977, 42, 3011.

<sup>&</sup>lt;sup>15</sup> Song, C; Ma, Y; Chai, Q; Ma, C; Jiang, W; Andrus, M.B. *Tetrahedron.* **2005**, 61, 7438.

<sup>&</sup>lt;sup>16</sup> Yadav, V.K.; Babu, K.G. *Eur. J. Org. Chem.* **2005**, 452.

<sup>&</sup>lt;sup>17</sup> Tanaka, K; Ajiki, K. Org. Lett. 2005. 7, 1537.



**NMR experiment with 1-phenylethanol**: The reaction was set up according to the general procedure with 1-phenylethanol (23.0 mg, 0.18 mmol) and dichlorocyclopropene (51.7 mg, 0.20 mmol) except that  $CD_3CN$  was used in place of  $CH_2Cl_2$  as the solvent (to take advantage of slower reaction profile). The reaction was transferred from the vial to an NMR tube and followed by NMR over the course of the reaction. See below for overlaid NMR spectra.





























