Research Paper



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Ferric chloride-catalyzed deoxygenative chlorination of carbonyl compounds: A comparison of chlorodimethylsilane and dichloromethylsilane system

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Abstract

Deoxygenative chlorination of carbonyl compounds using the HMe₂SiCl/FeCl₃/EtOAc and HMeSiCl₂/FeCl₃/EtOAc systems has been systemically investigated. The HMe₂SiCl-FeCl₃ system showed the advantages of good substrate applicability, mild reaction conditions, simple operation, low cost, and easy availability of raw materials. Also, it provided a simple and efficient synthesis route for carbonyl deoxychlorination via a one-pot method. Using the HMeSiCl₂/FeCl₃/EtOAc system, the β -methylchalcone derivative could be obtained in good yields in addition to obtaining the chlorinated compound. Finally, two plausible reaction routes were proposed to describe the formation of the chlorinated compound and the β -methylchalcone derivative.

Keywords

carbonyl compound, chlorodimethylsilane, deoxygenative chlorination, dichloromethylsilane, ferric chloride

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Introduction

Reduction of carbonyl compounds to hydroxyl compounds via catalytic hydrosilylation is an essential synthetic method that has been widely used in academia and industry.^{1–4} Since Ojima et al.⁵ first discovered this reaction in 1972, an enormous amount of work has been published. The published works have mainly focused on searching for meaningful catalysts or activators that are cheap, easy-to-handle, tolerant, and chemo- and/or regioselective.^{6–12} However, several research groups have focused on the direct conversion of hydroxyl groups to chlorides during hydrosilylation reactions.^{13–18} For example, Onishi et al.¹⁹ reported that the effective $In(OH)_3$ -catalyzed deoxygenative halogenation of carbonyl compounds using chlorodimethylsilane (HMe₂SiCl)

bearing both hydrogen and chlorine moieties instead of triethylsilane (HSiEt₃) and chlorotrimethylsilane (Me₃SiCl) as separate hydrogen and chlorine sources, respectively. Obviously, such direct conversion of carbonyl compounds

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	~	ou +HMe _s SiCl	yst Cl		
		solven	it, rt. T	1 ₃	
	1		2		
Entry	Silane (equiv.)	Catalyst (equiv.)	Solvent	Time (h)	Yield ^b (%)
1	HMe ₂ SiCl (1.5)	_	EtOAc	24.0	0
2	HMe ₂ SiCI (1.5)	Fe(II) acetate (0.05)	EtOAc	4.0	93
3	HMe ₂ SiCI (1.5)	Fe(ClO ₄) ₃ ·3H ₂ O(0.05)	EtOAc	7.0	95
4	HMe ₂ SiCI (1.5)	FeCl ₂ (0.05)	EtOAc	10.0	96
5	HMe ₂ SiCl (1.5)	In(OH) ₃ (0.05)	EtOAc	10.0	93
6	HMe ₂ SiCl (1.5)	Fe(acac) ₃ (0.05)	EtOAc	12.0	97
7	HMe ₂ SiCl (1.5)	AICI ₃ (0.05)	EtOAc	24.0	88
8	HMe ₂ SiCl (1.5)	FeCl ₃ (0.05)	EtOAc	2.0	97
9	HMe ₂ SiCl (1.8)	FeCl ₃ (0.05)	EtOAc	1.0	97
10	HMe ₂ SiCl (1.2)	FeCl ₃ (0.05)	EtOAc	6.0	93
11	HMe ₂ SiCl (1.0)	FeCl ₃ (0.05)	EtOAc	24.0	73
12	HMe ₂ SiCl (1.5)	FeCl3 (0.05)	I,4-Dioxane	7.0	66
13	HMe ₂ SiCl (1.5)	FeCl ₃ (0.05)	DME	24.0	44
14	HMe ₂ SiCl (1.5)	FeCl ₃ (0.05)	MeCN	24.0	Trace
15	HMe ₂ SiCl (1.5)	FeCl ₃ (0.05)	Et ₂ O	24.0	Trace
16	HMe ₂ SiCl (1.5)	FeCl ₃ (0.05)	Hexane	24.0	0
17	HMe ₂ SiCl (1.5)	FeCl ₃ (0.05)	DMF	24.0	0
18	HMe ₂ SiCl (1.5)	FeCl ₃ (0.05)	DCM	24.0	0
19	HMe ₂ SiCl (1.5)	FeCl ₃ (0.05)	CHCl ₃	24.0	0

Table I. Optimized reaction conditions.^a

DME: 1,2-dimethoxyethane; DMF: *N*,*N*-dimethylformamide; DCM: dichloromethane.

^aAll reactions were carried out with I (10.0 mmol) in solvent (20 mL) at room temperature.

^bYields were determined by ¹H NMR.

The bold values in Table I are used to highlight that this is the optimal reaction condition.

into chlorides under mild conditions shows significant superiority to the conventional multistep approach involving separate reduction and chlorination under harsh conditions. Subsequently, Li et al.²⁰ realized a similar reaction using dichloromethylsilane (HMeSiCl₂) with FeCl₃ as the catalyst. In addition, the deoxygenative chlorination of aromatic carbonyl compounds has also been explored. Savela et al.²¹ reported that using Fe(III) oxo acetate as a catalyst, hydrosilylation of benzylic carbonyl compounds followed by subsequent chlorination could be easily achieved with HSiEt₃ (as a hydrogen source) and Me₃SiCl (as a chlorine source). Savela and Leino²² also found that if less Me₃SiCl was used (e.g. 8 mol%), symmetrical and nonsymmetrical ethers were obtained.

Recently, we reported the K₂CO₃-activated hydrosilylation of aldehydes and ketones with inexpensive polymethylhydrosiloxane as a reductive reagent.²³ In this work, we expanded our strategy to the deoxygenative halogenation of carbonyls using silicon reagents and inexpensive catalysts. Herein, we present FeCl₃-catalyzed deoxygenative chlorination of carbonyl compounds using HMe₂SiCl or HMeSiCl₂ as the hydride and chloride sources, respectively, in an efficient and simple manner.

Results and discussion

HMe₂SiCl as hydride and chloride sources

First, acetophenone was used as a standard substrate, and the catalytic activity of various catalysts was studied (Table 1).

Ethyl acetate (EtOAc) was used as the solvent, and the deoxygenative chlorination could not be conducted without catalyst (Table 1, entry 1). However, high conversion was obtained when the reaction was carried out in the presence of Lewis acids (Table 1, entries 2-8). Common Lewis acids, such as Fe(C₂H₃O₂)₂, Fe(ClO₄)₃·3H₂O, FeCl₂, In(OH)₃, Fe(acac)₃, AlCl₃, and FeCl₃, all exhibited excellent catalytic activity. When FeCl₃ was used, 97% yield of chloroethylbenzene was obtained within 2h (Table 1, entry 8). Because FeCl₃ is one of the least expensive inorganic catalysts, other catalysts were not investigated in subsequent studies. To further optimize the reaction, the molar ratios of HMe₂SiCl to acetophenone varied (Table 1, entries 9-11). The results indicate that yields of the corresponding chlorides decreased with a decrease in the amount of HMe₂SiCl (Table 1, entries 10 and 11). When diminishing the equivalent of HMe₂SiCl to 1.0, the yield of the chloroethylbenzene was only 73% after 24h (Table 1, entry 11). Notably, the 1.8 equiv. of HMe₂SiCl could not lead to the yield improvement, while the reaction time could be reduced to 1 h (Table 1, entry 9).

The influences of solvents on deoxygenative chlorination were also investigated (Table 1, entries 12–19). When 1,4-dioxane was used as the solvent, the desired compounds were only obtained in 66% yield (Table 1, entry 12), while 1,2-dimethoxyethane (DME) led to an even lower yield of 44% (Table 1, entry 13). For other solvents, such as acetonitrile (MeCN), ether (Et₂O), hexane, and *N*,*N*dimethylformamide (DMF), the starting substrate was fully recovered or only a trace amount of the desired compound was obtained (Table 1, entries 14–17); this indicates that no

	⁺ HMe ₂ SiCl R ₂	FeCl₃ (0.05 eq.) EtOAc, rt.	$R_1 R_2$
Entry	Substrate	Product	Yield⁵ (%)
I	3a 0		97
2	3b	4b	96
3	3c °	4c C	88
4	3d F	4d F	91
5	3e	4e Cl	96
6	3f CI		93
7	3g ci		95
8	3h Br	4h Br	100
9	3i F ₃ C	4i F ₃ c	92
10	3j °5 °5	4j ~ 5	79
11	3k 0 ₂ N	4k 0 ₂ N	81
12	31 °2N 0	41 °2N CI	88
13	3m NC	4m NC	91
14	3n - J	4n •	95
15	Зо ноос	40 ноос	81
16	3p CI	4p ^c cl cl	100
17	3q 0 "	4q ci ci	100
18	3r Sr	4r 4r	93
			(Continued)

Tal	ble	2.	Substrate scope	of	different	carbonyl	compounds. ^a
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Table 2. (Continued)

Entry Substrate Product Yield^b (%) 19 30 100 20 4t (3t 89 3u 21 0 22 0 0 23 24 94 25 83 26 54 3aa 27 4aa 95 3ab 79 4ab 28 29 3ac 4ac 5 40^d 30 0 31 3ad 4ad

^aReaction condition: **3** (10.0 mmol), HMe₂SiCl (15.0 mmol), and FeCl₃ (0.05 mmol) in EtOAc (20 mL) at room temperature. ^bThe values below the structures refer to NMR yields.

^c30.0 mmol of HMe₂SiCl was used.

 $^{\rm d}Reaction$ carried out at 77 °C, 2-chloroheptane (**4ac**) was obtained in 40% yield.

deoxygenative chlorination occurred. When dichloromethane (DCM) and chloroform were used, some side products were generated, and the desired compound was not detected (Table 1, entries 18 and 19). Although it was reported that the carbonyl substrates gave good yield of the chloro compounds in chloroform using HMe₂SiCl with In(OH)₃ as catalyst,¹⁹ using chloroform as solvent in our experiment, we could only recover most of the starting material. Thus, EtOAc is the preferred solvent for the deoxygenative chlorination reaction, and this is probably because the good solubility of FeCl₃ in EtOAc and the involving of a solventcoordinated hexavalent silicate which acts as an active hydride species.

After the reaction conditions were optimized, various ketones were tested as substrates for the deoxygenative chlorination reaction (Table 2). Unsubstituted aromatic ketones gave corresponding chlorides in high yields (Table 2, entries 1–3). While the aromatic ketones that had a deactivating substituent (Table 2, entries 4–17) and those that had a weak-activating substituent (Table 2, entries 18-20) were converted to chlorides in good-to-excellent yields (79%-100%); the substituents susceptible to reduction such as MeSO₂- (Table 2, entry 10), NO₂-(Table 2, entries 11 and 12), NC- (Table 2, entry 13), CH₃OOC- (Table 2, entry 14), and HOOC- (Table 2, entry 15) were not affected. For the acetophenones that had the same substituent at different positions, the yields were similar (Table 2, entries 4 and 5; entries 6 and 7; entries 11 and 12). However, each aromatic ketone that had an activating substituent (Table 2, entries 21-23) failed to give the desired chloride, and the Clemmensen-type reduction product was obtained. These results agree well with Onishi's report, in which chloroform was used as the solvent.¹⁹ For aromatic ketones that had an activating substituent, the reaction conditions were further optimized to obtain the desired chloride.

At room temperature, the equivalent of the HMe₂SiCl was increased to 2 or 3, and no target chloride was obtained, whereas the yield of the reduction product (alkyl benzene) increased. With subsequent heating to 77 °C, the target chloride was still not obtained. Polycyclic aromatic substrates (Table 2, entries 24 and 25) also effectively produced the desired chloride. Benzophenone (Table 2, entry 26) produced chloride in 54% yield along with diphenylmethane in 43% yield. When aliphatic ketones are used as the substrates, the yields of the products were medium to high (Table 2, entries 27-29); sometimes higher temperature was necessary to obtain a satisfactory yield (Table 2, entry 30). In this context, aromatic heterocyclic ketones, such as thiophene-2-ketone (Table 2, entry 31), could not be converted to the corresponding chloride, although a trace amount of the Clemmensen-type reduction product was obtained.

HMeSiCl₂ as hydride and chloride sources

Sheng et al.24 reported that FeCl3-catalysted deoxygenative chlorination of acetophenone can also be performed using HMeSiCl₂ as hydride and chloride sources with DME as the solvent. Following their report, we used p-chlorobenzophone as the substrate in our experiment. Surprisingly, a β methylchalcone derivative was obtained as the major product in addition to the desired chloride. Also, there existed a trace amount of 4-chlorostyrene. Subsequently, EtOAc was used as the solvent to further study this reaction and the results are summarized in Table 3. Aromatic ketones that had a deactivating substituent (Table 3, entries 1 and 2) afforded β methylchalcone derivatives along with the chlorinated compound. Aromatic ketones that had a weak-activating substituent (Table 3, entries 3 and 4) resulted in only β methylchalcone derivatives in 82% and 72% yields, respectively, and no chloride was obtained. However, aromatic ketones that had an activating substituent, such as methoxy and hydroxy (Table 3, entries 5 and 6), failed to give β methylchalcone derivatives and chlorides. These results agree with results reported by Elmorsy et al.,25 who treated tetrachlorosilane with aromatic ketones; the substituents that increased the negativity of the acetyl group decreased the yield of β methylchalcone derivatives.

We also found that no allyl chloride could be detected, implying that β -methylchalcone derivative could not be deoxychlorinated in the HMeSiCl₂/FeCl₃/EtOAc system. Aliphatic ketones (Table 3, entries 7–9) failed to give the desired products at room temperature, and almost all of the starting arenes were recovered. These results imply that HMeSiCl₂ probably reacted in a very different role than HMe₂SiCl in the deoxygenative chlorination reaction.

Mechanism proposal

On the basis of the above results, two plausible reaction routes were proposed to explain the formation of the chlorinated compound and β -methylchalcone derivative (Scheme 1). In 2002, Onishi et al.¹⁹ reported that deoxyhalogenation of carbonyl compounds with HMe2SiCl was catalyzed by In(OH)₃ and gave the proposed mechanism. We believe that the HMe₂SiCl/FeCl₂ and HMeSiCl₂/FeCl₂ systems in our experiment each gave the chlorinated compound in a similar way to that proposed by Onishi (Scheme 1, path A). However, when HMeSiCl₂ is employed as both the hydride and chloride sources, alcoholysis of HMeSiCl₂ should be considered. Alcoholysis of organochlorosilane and alcohols is an important chemical characteristic of the Si-Cl bond.²⁶ HMeSiCl₂ is more electrophilic than HMe₂SiCl because of one more chlorine atom bonded to silicon. When HMeSiCl, is used, the enol form of ketones (nucleophile) can easily attack dichloromethylsilane to give enol silyl ether (Scheme 1, path B). First, alcoholysis of the enol form of ketones occurs and affords an enol silyl ether. Then, nucleophilic attack of enol silyl ether on another ketone under the activation of FeCl₃ produces an intermediate chelate via loss of dichloromethylsilane. Finally, the chelate collapses to produce the final β methylchalcone derivative via loss of FeCl₂ and H₂O.

Conclusion

In summary, we have achieved deoxygenative halogenation of carbonyl compounds using the HMe₂SiCl/FeCl₂/ EtOAc system. This method is eco-friendly and shows good tolerance. We also performed deoxygenative halogenation using the HMeSiCl₂/FeCl₃/EtOAc system, and β -methylchalcone derivative was obtained in high yields in addition to obtaining the target chlorinated compound. The differences between the HMe₂SiCl/FeCl₃/EtOAc and HMeSiCl₂/FeCl₂/EtOAc systems were investigated and the corresponding mechanisms were proposed. We propose that the chlorinated compound was given in a similar way to the mechanism proposed by Onishi for both the systems, while a possible route, involving the alcoholysis of enol form of ketone followed by nucleophilic attack on another ketone, was proposed for the formation of the β -methylchalcone derivative in the HMeSiCl₂/FeCl₃/EtOAc system.

Experimental

General information

Chemicals were purchased from commercial sources and used without further purification unless otherwise stated. Deuterochloroform (CDCl₃) and HMe₂SiCl were purchased

able 3. Reaction under the fit rester, rees, Etore system	Table 3	 Reaction 	under the	HMeSiCl	/FeCl ₃	/EtOAc s	ystem. ^a
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	R ₁	+ HMeSiCl ₂ $\frac{\text{FeCl}_3 (f)}{\text{EtOAc}}$	0.05 eq.) rt. R	R_2 + R_1 + R_1 + R_1	
Entry	Substrate	Chlorinated product	Yield ^b (%)	Aldol condensation product	Yield ^b (%)
I			28		70
2	O ₂ N	4k 0 ₂ N	60	5k 02N	37
3	° °	4s CI	0	5s 0 0	82
4	Ph O	4t Ph	0	St Ph	72
5		4u O CI	0	5u o o	0
6	НОО	4• но СІ	0	5 чно он	0
7		4aa Cl	0	5aa O	0
8	\bigcirc	4ab	0	5ab	0
9	0 C ₅ H ₁₁	CI 4ac C ₅ H ₁₁	0	5ac C ₅ H ₁₁	0



from J&K Scientific Ltd. All of the other chemicals were purchased from Beijing InnoChem Science & Technology Co., Ltd., Acros, and Alfa Aesar. Column chromatography was performed using 100–200 mesh silica gel, which was purchased from Qingdao Haiyang Chemical Co., Ltd. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker 400 MHz spectrometer (1H: 400 MHz; 13C: 101 MHz).

General procedure for the deoxygenative chlorination of carbonyl compounds with HMe₂SiCl

A 50mL single-neck round-bottom flask was loaded with carbonyl compound (10.0 mmol), FeCl_3 (0.0811 g, 0.5 mmol), and EtOAc (20 mL). The reaction mixture was stirred at room temperature for 1 min, and then HMe₂SiCl (1.4193 g,

15.0 mmol) was added. Subsequently, the reaction flask was equipped with a 90° glass joint with a balloon to protect the mixture from moisture. The reaction mixture was stirred vigorously at room temperature until the reaction was completed (as detected by thin-layer chromatography (TLC)). The solution was washed with saturated NaHCO₃ solution $(3 \times 10 \text{ mL})$ to remove FeCl₃. The organic layer was dried over anhydrous Na₂SO₄. The solvent was then removed under vacuum, and the mixture was purified via column chromatography to afford the product.

General procedure for the deoxygenative chlorination of carbonyl compounds with HMeSiCl₂

A 50 mL single-neck round-bottom flask was loaded with carbonyl compound (10.0 mmol), FeCl_3 (0.0811 g, 0.5 mmol), and EtOAc (20 mL). The reaction mixture was



Scheme 1. Possible routes for the formation of chlorinated compound and β -methylchalcone derivative.

stirred at room temperature for 1 min, and then HMeSiCl₂ (1.7255 g, 15.0 mmol) was added. Subsequently, the reaction flask was equipped with a 90° glass joint with a balloon to protect the mixture from moisture. The reaction mixture was stirred vigorously at room temperature until the reaction was completed (as detected by TLC). The solution was washed with saturated NaHCO₃ solution $(3 \times 10 \text{ mL})$ to remove FeCl₃. The organic layer was dried over anhydrous Na₂SO₄. The solvent was then removed under vacuum, and the mixture was purified via column chromatography to afford the product.

Characterization of the products 4a-5t

(1-Chloroethyl)benzene (4a):¹⁹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 200:1) in 97% yield. Colorless liquid; 1H NMR (400 MHz, CDCl₃): δ 7.34 (d, J=7.1 Hz, 2H), 7.27 (t, J=7.3 Hz, 2H), 7.21 (t, J=7.1 Hz, 1H), 5.01 (q, J=6.8 Hz, 1H), 1.77 (d, J=6.8 Hz, 3H);13C NMR (101 MHz, CDCl₃): δ 142.8, 128.7, 128.3, 126.5, 58.8, 26.5. High-resolution mass spectrometry (HRMS) (electrospray ionization (ESI)) m/z calcd for [C₈H₁₀Cl]⁺ (M + H)⁺: 141.0466; found: 141.0467.

(1-Chloropropyl)benzene (**4b**):²⁷ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 96% yield. Pale yellow liquid; ¹H NMR (400 MHz, CDCl₃) δ 7.40–7.27 (m, 5H), 4.78 (dd, *J*=7.6, 6.8 Hz, 1H), 2.20–2.01 (m, 2H), 0.99 (t, *J*=7.3 Hz, 3H);¹³C NMR (101 MHz, CDCl₃): δ 141.8, 128.6, 128.2, 127.0, 65.5, 33.2, 11.8. HRMS (ESI) m/z calcd for [C₉H₁₂Cl]⁺ (M + H)⁺: 155.0622; found: 155.0618.

(Chloro(cyclohexyl)methyl)benzene (**4c**):²¹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 88% yield. Colorless liquid; ¹H NMR

 $\begin{array}{l} (400\,{\rm MHz},{\rm CDCl}_3)\,\delta\,7.38{-}7.28\,({\rm m},\,{\rm 5H}),\,4.63\,({\rm d},J{=}8.4\,{\rm Hz},\\ 1{\rm H}),\,2.22{-}2.20\,({\rm m},\,1{\rm H}),\,1.95{-}1.87\,({\rm m},\,1{\rm H}),\,1.86{-}1.78\,({\rm m},\\ 1{\rm H}),\,1.70{-}1.67\,({\rm m},\,2{\rm H}),\,1.48{-}1.45\,({\rm m},\,1{\rm H}),\,1.33{-}1.23\,({\rm m},\\ 1{\rm H}),\,1.22{-}1.03\,({\rm m},\,3{\rm H}),\,0.97{-}0.88\,({\rm m},\,1{\rm H});\,^{13}{\rm C}\,\,{\rm NMR}\\ (101\,{\rm MHz},\,{\rm CDCl}_3){\rm :}\,\,\delta\,\,141.0,\,128.4,\,128.0,\,127.6,\,69.9,\\ 45.7,\,30.5,\,30.3,\,26.2,\,26.0,\,25.9.\,\,{\rm HRMS}\,({\rm ESI})\,\,{\rm m/z}\,\,{\rm calcd}\\ {\rm for}\,\,[{\rm C}_{13}{\rm H}_{18}{\rm Cl}]^+\,({\rm M}+{\rm H})^+{\rm :}\,209.1092;\,{\rm found:}\,209.1089. \end{array}$

1-(1-Chloroethyl)-4-fluorobenzene (**4d**):²¹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 91% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.43–7.35 (m, 2H), 7.06–7.00 (m, 2H), 5.08 (q, *J*=6.8 Hz, 1H), 1.83 (d, *J*=6.8 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ 162.4 (d, *J*_{C-F}=248 Hz), 138.7 (d, *J*_{C-F}=3 Hz), 128.3 (d, *J*_{C-F}=8 Hz), 115.5 (d, *J*_{C-F}=22 Hz), 58.0, 26.6. ¹⁹F NMR (565 MHz, CDCl₃) δ –113.68 (s, 1F). HRMS (ESI) m/z calcd for $[C_8H_9ClF]^+$ (M+H)⁺: 159.0371; found: 159.0375.

l-(*l*-Chloroethyl)-3-fluorobenzene (4e):²⁸ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 96% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.24 (td, *J*=8.0, 5.9 Hz, 1H), 7.08 (ddd, *J*=9.8, 8.0, 4.9 Hz, 2H), 6.95–6.89 (m, 1H), 4.98 (q, *J*=6.8 Hz, 1H), 1.76 (d, *J*=6.8 Hz, 3H);¹³C NMR (101 MHz, CDCl₃): δ 130.2 (d, *J*_{C-F}=8 Hz), 122.2 (d, *J*_{C-F}=2 Hz), 115.2 (d, *J*_{C-F}=21 Hz), 113.6 (d, *J*_{C-F}=23 Hz), 57.7, 26.5. ¹⁹F NMR (565 MHz, CDCl₃) δ -112.46 (s, 1F). HRMS (ESI) m/z calcd for $[C_8H_9CIF]^+$ (M + H)⁺: 159.0371; found: 159.0368.

1-Chloro-2-(1-chloroethyl)benzene (**4f**):²¹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 93% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.65 (dd, *J*=7.8, 1.5 Hz, 1H), 7.37–7.29 (m, 2H), 7.26–7.20 (m, 1H), 5.58 (q, *J*=6.8 Hz, 1H), 1.83 (d, *J*=6.8 Hz, 3H);¹³C NMR (101 MHz, CDCl₃): δ

140.0, 132.4, 129.6, 129.3, 127.8, 127.4, 54.5, 25.7. HRMS (ESI) m/z calcd for $[C_8H_9Cl_2]^+$ (M + H)⁺: 175.0076; found: 175.0074.

1-Chloro-4-(1-chloroethyl)benzene (4g):²¹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 95% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.38–7.29 (m, 4H), 5.05 (q, *J*=6.8 Hz, 1H), 1.82 (d, *J*=6.8 Hz, 3H);¹³C NMR (101 MHz, CDCl₃): δ 141.3, 134.0, 128.8, 127.9, 57.8, 26.5. HRMS (ESI) m/z calcd for [C₈H₉Cl₂]⁺ (M + H)⁺: 175.0076; found: 175.0070.

1-Bromo-4-(1-chloroethyl)benzene (**4h**):²¹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 100% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.39 (d, *J*=8.4 Hz, 2H), 7.21 (d, *J*=8.3 Hz, 2H), 4.95 (q, *J*=6.8 Hz, 1H), 1.73 (d, *J*=6.8 Hz, 3H);¹³C NMR (101 MHz, CDCl₃): δ 141.9, 131.8, 128.3, 122.1, 57.8, 26.5. HRMS (ESI) m/z calcd for [C₈H₉BrCl]⁺ (M + H)⁺: 218.9571; found: 218.9576.

1-(1-Chloroethyl)-4-(trifluoromethyl)benzene (**4i**):²¹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 92% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.54 (d, *J*=8.2 Hz, 2H), 7.45 (d, *J*=8.3 Hz, 2H), 5.02 (q, *J*=6.8 Hz, 1H), 1.77 (d, *J*=6.8 Hz, 3H);¹³C NMR (151 MHz, CDCl₃) δ 146.7, 130.5 (q, *J*_{C-F}=33 Hz), 127.0, 125.7 (q, *J*_{C-F}=4 Hz), 126.7–121.3 (q, *J*_{C-F}=272 Hz), 57.5, 26.4.¹⁹ F NMR (565 MHz, CDCl₃) δ -62.66 (s, 3F). HRMS (ESI) m/z calcd for $[C_8H_9ClF_3]^+$ (M + H)⁺: 209.0339; found: 209.0341.

1-(1-Chloroethyl)-4-(methylsulfonyl)benzene (4j): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 30:1) in 79% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.95 (d, J=8.3 Hz, 2H), 7.63 (d, J=8.4 Hz, 2H), 5.13 (q, J=6.8 Hz, 1H), 3.07 (s, 3H), 1.87 (d, J=6.8 Hz, 3H);¹³C NMR (101 MHz, CDCl₃): δ 148.7, 140.2, 128.0, 127.7, 57.0, 44.5, 26.4. HRMS (ESI) m/z calcd for [C₉H₁₂ClO₂S]⁺ (M + H)⁺: 219.0241; found: 219.0244.

1-(1-Chloroethyl)-4-nitrobenzene (**4k**):²⁹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 60:1) in 81% yield. Yellow liquid; ¹H NMR (400 MHz, CDCl₃): δ 8.14 (d, J=8.8 Hz, 2H), 7.52 (d, J=8.7 Hz, 2H), 5.06 (q, J=6.8 Hz, 1H), 1.79 (d, J=6.8 Hz, 3H);¹³C NMR (101 MHz, CDCl₃): δ 149.7, 147.6, 127.5, 123.9, 56.9, 26.4. HRMS (ESI) m/z calcd for [C₈H₉ClNO₂]⁺ (M + H)⁺: 186.0316; found: 186.0310.

1-(1-Chloroethyl)-3-nitrobenzene (**4**): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 120:1) in 88% yield. Yellow liquid; ¹H NMR (400 MHz, CDCl₃): δ 8.31 (s, 1H), 8.18 (d, J=8.2Hz, 1H), 7.78 (d, J=7.7Hz, 1H), 7.57 (t, J=8.0Hz, 1H), 5.17 (q, J=6.8Hz, 1H), 1.90 (d, J=6.8Hz, 3H);¹³C NMR (101 MHz, CDCl₃): δ 148.4, 144.8, 132.8, 129.8, 123.2, 121.6, 56.9, 26.4. HRMS (ESI) m/z calcd for [C₈H₉ClNO₂]⁺ (M+H)⁺: 186.0316; found: 186.0314.

4-(1-Chloroethyl)benzonitrile (**4m**):¹⁹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 150:1) in 91% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 6.44 (d, J=8.2 Hz, 2H), 6.31 (d, J=8.3 Hz, 2H), 3.86 (q, J=6.8 Hz, 1H), 0.62 (d, J=6.8 Hz, 3H);¹³C NMR (101 MHz, CDCl₃): δ 147.7, 132.5, 127.3, 118.4, 112.0, 57.2, 26.3. HRMS (ESI) m/z calcd for $[C_9H_9CIN]^+$ (M + H)⁺: 166.0418; found: 166.0410.

Methyl 4-(1-chloroethyl)benzoate (**4n**):¹⁹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 100:1) in 95% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 8.03 (dd, *J*=8.4, 1.9 Hz, 2H), 7.52– 7.46 (m, 2H), 5.15–5.06 (m, 1H), 3.91 (d, *J*=2.3 Hz, 3H), 1.84 (dd, *J*=6.8, 2.6 Hz, 3H);¹³C NMR (101 MHz, CDCl₃): δ 166.6, 147.6, 130.0, 126.5, 57.8, 52.2, 26.4. HRMS (ESI) m/z calcd for [C₁₀H₁₂ClO₂]⁺ (M + H)⁺: 199.0520; found: 199.0524.

4-(1-Chloroethyl)benzoic acid (40):¹⁹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 10:1) in 81% yield. White solid; m.p. 201–203 °C. ¹H NMR (400 MHz, dimethyl sulfoxide (DMSO)): δ 13.03 (s, 1H), 7.95 (d, J=8.3 Hz, 2H), 7.60 (d, J=8.3 Hz, 2H), 5.42 (q, J=6.8 Hz, 1H), 1.79 (d, J=6.8 Hz, 3H); ¹³C NMR (101 MHz, DMSO): δ 167.4, 147.7, 131.1, 130.1, 127.3, 58.6, 26.3. HRMS (ESI) m/z calcd for [C₉H₁₀ClO₂]⁺ (M + H)⁺: 185.0364; found: 185.0362.

1,3-Bis(1-chloroethyl)benzene (**4p**): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 150:1) in 100% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.33 (d, *J*=36.3 Hz, 4H), 5.02 (q, *J*=6.8 Hz, 2H), 1.78 (d, *J*=6.8 Hz, 6H);¹³C NMR (101 MHz, CDCl₃): δ 143.3, 129.0, 126.4, 124.8, 58.5, 26.6. HRMS (ESI) m/z calcd for [C₁₀H₁₃Cl₂]⁺ (M+H)⁺: 203.0389; found: 203.0390.

1-Chloro-3-(1-chloropropyl)benzene (**4q**): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 100% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃) δ 7.30 (s, 1H), 7.22–7.15 (m, 3H), 4.65 (dd, *J*=7.8, 6.5 Hz, 1H), 2.10–1.91 (m, 2H), 0.92 (t, *J*=7.3 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ 143.7, 134.4, 129.9, 128.4, 127.2, 125.2, 64.4, 33.2, 11.6. HRMS (ESI) m/z calcd for [C₉H₁₁Cl₂]⁺ (M+H)⁺: 189.0232; found: 189.0233.

1-(1-Chloroethyl)-2-methylbenzene (**4r**):³⁰ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 93% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.53 (d, *J*=7.6Hz, 1H), 7.23 (dd, *J*=15.3, 8.5Hz, 2H), 7.16 (d, *J*=6.9Hz, 1H), 5.35 (q, *J*=6.8Hz, 1H), 2.41 (s, 3H), 1.86 (d, *J*=6.8Hz, 3H);¹³C NMR (101 MHz, CDCl₃): δ 140.5, 135.3, 130.6, 128.2, 126.6, 125.7, 55.0, 25.2, 19.0. HRMS (ESI) m/z calcd for [C₉H₁₂Cl]⁺ (M + H)⁺: 155.0622; found: 155.0628.

1-(1-Chloroethyl)-4-methylbenzene (4s):¹⁹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 200:1) in 100% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.29 (d, *J*=8.1 Hz, 2H), 7.13 (d, *J*=7.9 Hz, 2H), 5.05 (q, *J*=6.8 Hz, 1H), 2.32 (s, 3H), 1.81 (d, *J*=6.8 Hz, 3H);¹³C NMR (101 MHz, CDCl₃): δ 139.9, 138.1, 129.3, 126.5, 58.8, 26.5, 21.2. HRMS (ESI) m/z calcd for [C₉H₁₂Cl]⁺ (M + H)⁺: 155.0622; found: 155.0620.

4-(1-Chloroethyl)-1, 1'-biphenyl (**4t**):²¹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 89% yield. White solid; m.p. 52–54 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.50 (d, J=8.3 Hz, 4H), 7.44–7.33 (m, 4H), 7.28 (t, J=7.3 Hz, 1H), 5.07 (q, J=6.8 Hz, 1H), 1.82 (d, J=6.8 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ 141.8, 141.3, 140.6, 128.8, 127.5, 127.4, 127.2, 127.0, 58.6, 26.5. HRMS (ESI) m/z calcd for $[C_{14}H_{14}Cl]^+$ (M + H)⁺: 217.0779; found: 217.0779.

1-Ethyl-4-methoxybenzene (**4u**):³¹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 5:1) in 69% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃) δ 7.13 (d, *J*=8.6Hz, 2H), 6.85 (d, *J*=8.6Hz, 2H), 3.80 (s, 3H), 2.61 (q, *J*=7.6Hz, 2H), 1.23 (t, *J*=7.6Hz, 3H).¹³C NMR (101 MHz, CDCl₃) δ 157.6, 136.4, 128.7, 113.8, 55.3, 28.0, 15.9. HRMS (ESI) m/z calcd for [C₉H₁₃O]⁺ (M + H)⁺: 137.0961; found: 137.0959.

4-Ethylphenol (**4v**):³² Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 5:1) in 63% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃) δ 7.07 (d, *J*=8.5 Hz, 2H), 6.76 (d, *J*=6.5 Hz, 2H), 4.58 (s, 1H), 2.58 (q, *J*=7.6 Hz, 2H), 1.20 (t, *J*=7.6 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 153.3, 136.6, 128.9, 115.2, 28.0, 15.9. HRMS (ESI) m/z calcd for [C₈H₁₁O]⁺ (M + H)⁺: 123.0804; found: 123.0806.

N-(*4*-ethylphenyl)acetamide (**4w**): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 200:1) in 67% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃) δ 7.39 (d, *J*=8.4 Hz, 2H), 7.14 (d, *J*=8.4 Hz, 2H), 2.61 (q, *J*=7.6 Hz, 2H), 2.16 (s, 3H), 1.21 (t, *J*=7.6 Hz, 3H).¹³C NMR (101 MHz, CDCl₃) δ 168.2, 140.4, 135.5, 128.3, 120.1, 28.3, 24.6, 15.7. HRMS (ESI) m/z calcd for [C₁₀H₁₄NO]⁺ (M + H)⁺: 164.1070; found: 164.1070.

2-(1-Chloroethyl)naphthalene (**4x**):²¹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 94% yield. White solid; m.p. 67 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.90–7.83 (m, 4H), 7.60 (dd, J=8.5, 1.8 Hz, 1H), 7.56–7.48 (m, 2H), 5.30 (q, J=6.8 Hz, 1H), 1.97 (d, J=6.8 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃): δ 140.0, 133.1, 133.1, 128.6, 128.1, 127.7, 126.4, 126.4, 125.2, 124.5, 59.0, 26.4. HRMS (ESI) m/z calcd for [C₁₂H₁₂Cl]⁺ (M + H)⁺: 191.0622; found: 191.0620.

9-Chloro-9H-fluorene (**4y**):²¹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 100:1) in 83% yield. White solid; m.p. 92 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.61–7.54 (m, 4H), 7.32 (dt, *J*=7.5, 3.8 Hz, 2H), 7.26 (td, *J*=7.4, 1.2 Hz, 2H), 5.70 (s, 1H); ¹³C NMR (101 MHz, CDCl₃): δ 143.8, 140.0, 129.4, 128.0, 125.8, 120.1, 57.6. HRMS (ESI) m/z calcd for [C₁₃H₁₀Cl]⁺ (M + H)⁺: 201.0466; found: 201.0465.

(*Chloromethylene*)*dibenzene* (**4z**):³³ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 54% yield. Yellow color liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.41 (ddd, *J*=18.3, 17.8, 5.2 Hz, 10H), 6.20 (s, 1H); ¹³C NMR (101 MHz, CDCl₃): δ 141.1, 128.6, 128.1, 127.8, 64.3. HRMS (ESI) m/z calcd for [C₁₃H₁₂Cl]⁺ (M + H)⁺: 203.0622; found: 203.0623.

(3-Chlorobutyl)benzene (4aa):¹⁹ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 95% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃) δ 7.37 (t, *J*=7.6 Hz, 2H), 7.28 (d, *J*=6.1 Hz, 3H), 4.10–4.02 (m, 1H), 2.92–2.81 (m, 2H), 2.11–2.05 (m, 2H), 1.60 (d, *J*=6.6 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 141.1, 128.5, 128.5, 126.1, 57.9, 41.9, 32.9, 25.4. HRMS (ESI) m/z calcd for [C₁₀H₁₄Cl]⁺ (M+H)⁺: 169.0779; found: 169.0777.

Chlorocyclohexane (**4ab**):³⁴ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 79% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 4.08–3.95 (m, 1H), 2.08 (d, J=10.9 Hz, 2H), 1.81 (d, J=5.6 Hz, 2H), 1.74–1.62 (m, 2H), 1.55 (d, J=5.7 Hz, 1H), 1.43–1.28 (m, 3H); ¹³C NMR (101 MHz, CDCl₃): δ 60.3, 36.7, 25.2, 24.9. HRMS (ESI) m/z calcd for [C₆H₁₂Cl]⁺ (M + H)⁺: 119.0622; found: 119.0624.

2-Chloroheptane (4ac): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 200:1) in 40% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃) δ 4.12–3.96 (m, 1H), 1.70 (ddd, *J*=9.0, 8.4, 4.1 Hz, 2H), 1.51 (d, *J*=6.5 Hz, 4H), 1.42–1.22 (m, 5H), 0.90 (t, *J*=7.0 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 59.0, 40.4, 31.4, 26.4, 25.4, 22.6, 14.0. HRMS (ESI) m/z calcd for [C₇H₁₆Cl]⁺ (M + H)⁺: 135.0935; found: 135.0931.

Diphenylmethane (4ad):³⁵ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 300:1) in 43% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.16 (ddd, J=20.5, 14.0, 7.1 Hz, 10H), 3.91 (d, J=13.6 Hz, 2H); ¹³C NMR (101 MHz, CDCl₃): δ 141.1, 129.0, 128.5, 126.1, 42.0. HRMS (ESI) m/z calcd for [C₁₃H₁₃]⁺ (M + H)⁺: 169.1012; found: 169.1011.

1-(Heptan-2-yloxy)-2-methylheptane (**4ae**): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 200:1) in 52% yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃) δ 3.46–3.33 (m, 2H), 1.51–1.23 (m, 16H), 1.09 (d, *J*=6.1 Hz, 6H), 0.88 (t, *J*=6.9 Hz, 6H);¹³C NMR (101 MHz, CDCl₃) δ 73.0, 37.5, 32.0, 25.6, 22.7, 20.5, 14.1. HRMS (ESI) m/z calcd for [C₁₄H₃₁O]⁺ (M + H)⁺: 215.2369; found: 215.2371.

1,3-Bis(4-chlorophenyl)but-2-en-1-one (**5g**):³⁶ Isolated by silica gel column chromatography (petroleum ether/ ethyl acetate 150:1) in 70% yield. Yellow solid; m.p. 80 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.93 (d, J=8.6 Hz, 2H), 7.50 (d, J=8.7 Hz, 2H), 7.45 (d, J=10.9 Hz, 2H), 7.39 (d, J=8.7 Hz, 2H), 7.10 (s, 1H), 2.58 (s, 3H); ¹³C NMR (101 MHz, CDCl₃): δ 190.2, 154.3, 140.9, 139.0, 137.4, 135.3, 129.7, 128.9, 128.8, 127.8, 121.7, 18.8. HRMS (ESI) m/z calcd for [C₁₆H₁₃Cl₂O]⁺ (M + H)⁺: 291.0338; found: 291.0339.

1,3-Bis(4-nitrophenyl)but-2-en-1-one (**5k**):²⁵ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 20:1) in 37% yield. Yellow solid; m.p. 154 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.35 (dd, J=17.9, 8.8 Hz, 4H), 8.16 (d, J=8.8 Hz, 2H), 7.74 (d, J=8.8 Hz, 2H), 7.19 (s, 1H), 2.68 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 189.4, 154.9, 150.2, 148.6, 148.3, 143.4, 129.3, 127.5, 124.0, 124.0, 123.4, 19.2. HRMS (ESI) m/z calcd for [C₁₆H₁₃N₂O₅]⁺ (M + H)⁺: 313.0819; found: 313.0819.

1,3-Di-p-tolylbut-2-en-1-one (**5s**):²⁵ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 200:1) in 82% yield. Yellow solid; ¹H NMR (400 MHz, CDCl₃) δ 7.90 (d, *J*=8.2 Hz, 2H), 7.48 (d, *J*=8.2 Hz, 2H), 7.26 (d, *J*=8.7 Hz, 2H), 7.22 (d, *J*=8.0 Hz, 2H), 7.15 (s, 1H), 2.57 (s, 3H), 2.42 (s, 3H), 2.39 (s, 3H);¹³C NMR (101 MHz, CDCl₃) δ 191.6, 154.5, 143.2, 139.9, 139.3, 137.0, 129.3, 129.2, 128.4, 126.4, 121.5, 21.7, 21.3, 18.8. HRMS (ESI) m/z calcd for $[C_{18}H_{18}O]^+$ (M+H)⁺: 250.1358; found: 250.1355.

1,3-Di([1,1'-biphenyl]-4-yl)but-2-en-1-one (**5t**):²⁵ Isolated by silica gel column chromatography (petroleum ether/ethyl acetate 200:1) in 72% yield. Yellow solid; ¹H NMR (400 MHz, CDCl₃) δ 8.12 (d, J=8.4Hz, 2H), 7.74 (d, J=8.5 Hz, 2H), 7.70 (d, J=2.5 Hz, 4H), 7.68 (d, J=3.8 Hz, 4H), 7.50 (t, J=6.6 Hz, 4H), 7.43 (t, J=6.7 Hz, 2H), 7.31 (s, 1H), 2.69 (s, 3H);¹³C NMR (101 MHz, CDCl₃) δ 191.3, 154.5, 145.3, 142.1, 141.6, 140.3, 140.1, 138.2, 128.9, 128.2, 127.7, 127.3, 127.1, 127.0, 121.9, 18.8. HRMS (ESI) m/z calcd for (C₂₈H₂₃O)⁺ (M + H)⁺: 375.1743; found: 375.1743.

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Supplemental material

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