Supplementary Information for

Photoelectrochemical Nickel-Catalyzed Carboacylation/

Silanoylation of Alkenes with Unactivated C/Si-H Bonds

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1. General Experimental Details

All new compounds were fully characterized. Compounds were visualized by exposure to UV-light. All reactions and manipulations involving air- or moisture-sensitive compounds were performed using standard Schlenk techniques or in a glovebox. Toluene was purified using Pure Solv MD-5 solvent purification system, from Innovative Technology, Inc., by passing the solvent through two activated alumina columns after purging with argon. ¹H, ¹³C, ³¹P and ¹⁹F NMR spectra were recorded on a Bruker AVANCE III 400 MHz or 500 MHz spectrometer. Chemical shifts (δ values) were reported in ppm with CDCl₃ (7.26 and 77.16 ppm for ¹H and ¹³C respectively). Mass spectra were conducted at Agilent 6540 Ultra-High-Definition (UHD) Accurate-Mass Quadrupole Time-of-Flight (Q-TOF) liquid chromatography/mass spectrometry (LC/MS) system and Thermo Scientific TRACE 1300 ISQ LT gas chromatography/mass spectrometry (GC/MS) system. Unless otherwise noted, materials obtained from commercial suppliers were used without further purification.



2. Optimization of the Reaction Condition

	I	L4		L5		L6	
Entry	1/2/3	[Ni]	L	Current	Electrodes	Solvent	Yield
	(equiv)	(10 mol%)	(10 mol%)	(X mA)	(+)-(-)	(mL)	(%) ^[b]
1	10/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-RVC(-)	MeCN/Acetone	71
						(2/1, 3 mL)	
2	10/3/1	NiBr ₂ •DME	L2	4.0	GF(+)-RVC(-)	MeCN/Acetone	26
						(2/1, 3 mL)	
3	10/3/1	NiBr ₂ •DME	L3	4.0	GF(+)-RVC(-)	MeCN/Acetone	15
						(2/1, 3 mL)	
4	10/3/1	NiBr ₂ •DME	L4	4.0	GF(+)-RVC(-)	MeCN/Acetone	55
						(2/1, 3 mL)	
5	10/3/1	NiBr ₂ •DME	L5	4.0	GF(+)-RVC(-)	MeCN/Acetone	22
						(2/1, 3 mL)	
6	10/3/1	NiBr ₂ •DME	L6	4.0	GF(+)-RVC(-)	MeCN/Acetone	0
						(2/1, 3 mL)	
7	10/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-RVC(-)	MeCN	42
						(3 mL)	
8	10/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-RVC(-)	Acetone	17
						(3 mL)	
9	10/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-RVC(-)	MeCN/Acetone	55
						(1/2, 3 mL)	

10	10/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-GF(-)	MeCN/Acetone	64
						(1/1, 3 mL)	
11	10/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-RVC(-)	DMF	0
						(3 mL)	
12	10/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-RVC(-)	DMA	0
						(3 mL)	
13	10/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-RVC(-)	DMSO	0
						(3 mL)	
14	10/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-RVC(-)	HFIP	0
						(3 mL)	
15	10/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-RVC(-)	TFE	0
						(3 mL)	
16	5/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-RVC(-)	MeCN/Acetone	41
						(2/1, 3 mL)	
17	5/1/1	NiBr ₂ •DME	L1	4.0	GF(+)-RVC(-)	MeCN/Acetone	33
						(2/1, 3 mL)	
18	10/3/1	NiBr ₂ •DME	L1	6.0	GF(+)-RVC(-)	MeCN/Acetone	57
						(2/1, 3 mL)	
19	10/3/1	NiBr ₂ •DME	L1	2.0	GF(+)-RVC(-)	MeCN/Acetone	66
						(2/1, 3 mL)	
20	10/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-GF (-)	MeCN/Acetone	60
						(2/1, 3 mL)	
21	10/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-	MeCN/Acetone	15
					Ni foam (-)	(2/1, 3 mL)	
22	10/3/1	NiBr ₂ •DME	L1	4.0	RVC(+)-	MeCN/Acetone	36
					RVC(-)	(2/1, 3 mL)	
23	10/3/1	NiCl ₂ •DME	L1	4.0	Fe(+)-RVC(-)	MeCN/Acetone	<5
						(2/1, 3 mL)	
24	10/3/1	NiBr ₂	L1	4.0	GF(+)-RVC(-)	MeCN/Acetone	52
						(2/1, 3 mL)	
25	10/3/1	NiCl ₂ ·DME	L1	4.0	GF(+)-RVC(-)	MeCN/Acetone	62
						(2/1, 3 mL)	
26	10/3/1	Ni(acac)2	L1	4.0	GF(+)-RVC(-)	MeCN/Acetone	23
						(2/1, 3 mL)	
27	10/3/1	Ni(OAc) ₂	L1	4.0	GF(+)-RVC(-)	MeCN/Acetone	17
						(2/1, 3 mL)	
28	10/3/1	NiBr ₂ •DME	L1	0	GF(+)-RVC(-)	MeCN/Acetone	0

						(2/1, 3 mL)	
29	10/3/1	-	L1	4.0	GF(+)-RVC(-)	MeCN/Acetone	0
						(2/1, 3 mL)	
30	10/3/1	NiBr ₂ •DME	-	4.0	GF(+)-RVC(-)	MeCN/Acetone	0
						(2/1, 3 mL)	
31 ^[d]	10/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-RVC(-)	MeCN/Acetone	0
						(2/1, 3 mL)	
32 ^[e]	10/3/1	NiBr ₂ •DME	L1	4.0	GF(+)-RVC(-)	MeCN/Acetone	42
						(2/1, 3 mL)	

[a] Reaction conditions: **1a** (2.0 mmol), **2a** (0.6 mmol), **3a** (0.2 mmol), NiBr₂•DME (10 mol%), FeCl₃•6H₂O (10 mol%), dtbbpy (10 mol%), LiCl (3.0 equiv.), anhydrous MeCN/acetone (3.0 mL, 2/1), 4 mA, 24 h, 390 nm, 10 °C, nitrogen, graphite felt (GF) as an anode, reticulated vitreous carbon (RVC) as a cathode, undivided cell. [b] GC yields using dodecane as an internal standard. [c] isolated yields. [d] No blue LEDs. [e] At 30 °C.

3. General Procedures



A dry 10 mL vial equipped with a Teflon-coated magnetic stir bar was charged with acyl chlorides (0.30 mmol, 1.0 equiv.), alkanes (3.0 mmol, 10 equiv.) or silanes (1.5 mmol, 5 equiv.), alkenes (0.90 mmol, 3.0 equiv.), LiCl (37.8 mg, 3.0 equiv.) and FeCl₃·6H₂O (8.1 mg, 10 mol%) were dissolved in acetone (1.0 mL). Afterward, a precatalyst solution (it was prepared by a mix of NiBr₂·DME (6.6 mg, 10 mol%), 4,4'-ditert-butyl-2,2'-bipyridine (8.1 mg, 10 mol%) in anhydrous MeCN (2.0 mL) under N₂ atmosphere, and was stirred for 10 minutes.) was added via syringe. Then, it was capped with a Teflon lid equipped with Graphite Felt ($20 \times 10 \times 15$ mm) as the anode and reticulated vitreous carbon ($20 \times 10 \times 1$ mm) as the cathode. The reaction mixture was stirred under N₂ and electrolyzed at a constant current of 4 mA under irradiation by a 10-W 390-nm LED lamp with a cooling fan to keep the reaction temperature at 10 °C for 24 h. After the reaction was completed, the mixture was transferred to a 50 mL round bottom flask, electrodes were washed with ethyl acetate. Then H₂O (20 mL) was added and the mixture was extracted with EtOAc (20 mL) for three times. The combined organic layer was washed with H₂O (20 mL) and brine (20 mL). The organic layer was dried with anhydrous Na₂SO₄, then concentrated under vacuum. The product was purified by flash column chromatography on silica gel using hexane/EtOAc as eluent.

4. Characterization Data of Products

Methyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (4)



This compound was prepared according to General procedure, 61.3 mg, 71 % yield as a colorless oil. ¹H NMR (400 MHz, **Chloroform-***d*) δ 7.89 (d, J = 8.3 Hz, 2H), 7.28 (d, J = 7.9 Hz, 2H), 4.44 (dd, J = 7.9, 6.4 Hz, 1H), 3.68 (s, 3H), 2.42 (s, 3H), 1.87 – 1.66 (m, 6H), 1.19 – 1.13 (m, 2H), 0.95 – 0.80 (m, 5H). ¹³C NMR (101 MHz, Chloroform-d) δ 195.0, 170.9, 144.5, 133.6, 129.5, 128.8, 52.4, 51.5, 36.5, 35.8, 33.3, 33.0, 29.7, 26.4, 26.1, 21.7. HRMS m/z (ESI): calcd for $C_{18}H_{25}O_3$ (M + H)⁺ 289.1798, found 289.1780.

Methyl 2-(cyclohexylmethyl)-3-oxo-3-(o-tolyl)propanoate (5)

Me This compound was prepared according to General procedure, 0 53.6 mg, 62 % yield as a colorless oil. ¹H NMR (400 MHz, COOMe **Chloroform-***d*) δ 7.72 – 7.63 (m, 1H), 7.40 (td, J = 7.5, 1.4 Hz, 1H), 7.31 – 7.27 (m, 2H), 4.36 (dd, J = 8.3, 6.2 Hz, 1H), 3.70 (s, 3H), 2.50 (s, 3H), 1.94 -1.62 (m, 9H), 1.17 - 1.12 (m, 2H), 0.96 - 0.86 (m, 2H). ¹³C NMR (101 MHz, **Chloroform-***d*) δ 199.1, 170.8, 138.9, 137.2, 132.0, 131.6, 128.3, 125.7, 54.2, 52.3, 36.3, 35.9, 33.3, 32.9, 26.4, 26.1, 26.1, 21.0. HRMS m/z (ESI): calcd for C₁₈H₂₅O₃ (M + H)⁺ 289.1798, found 289.1799.

Methyl -2-(cyclohexylmethyl)-3-oxo-3-(m-tolyl)propanoate (6)



This compound was prepared according to General procedure, 63.9 mg, 74 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-d) δ 7.83 – 7.71 (m, 2H), 7.43 – 7.32 (m, 2H), 4.44 (dd, J = 7.9, 6.4 Hz, 1H), 3.68 (s, 3H), 2.42 (s, 3H), 1.98 – 1.77 (m, 3H), 1.71 – 1.60 (m, 4H), 1.28 – 1.17 (m, 4H), 0.98 – 0.87 (m,

2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 195.5, 170.8, 138.7, 136.2, 134.3, 129.1, 128.6, 125.8, 52.4, 51.6, 36.5, 35.9, 33.3, 33.0, 26.4, 26.2, 26.1, 21.4. HRMS m/z (ESI): calcd for C₁₈H₂₅O₃ (M + H)⁺ 289.1798, found 289.1798.

Methyl 2-(cyclohexylmethyl)-3-oxo-3-phenylpropanoate (7)



This compound was prepared according to General procedure, 56.7 mg, 69 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.99 (dd, *J* = 8.4, 1.4 Hz, 2H), 7.58 (d, *J* = 7.6

Hz, 1H), 7.48 (dd, J = 8.3, 7.1 Hz, 2H), 4.47 (dd, J = 7.9, 6.3 Hz, 1H), 3.69 (s, 3H), 1.95 – 1.60 (m, 7H), 1.31 – 1.10 (m, 4H), 0.96 – 0.93 (m, 2H). ¹³**C NMR (101 MHz, Chloroform-***d*) δ 195.3, 170.7, 136.0, 128.7, 128.5, 52.4, 51.5, 36.4, 35.7, 33.2, 32.9, 26.3, 26.0. HRMS m/z (ESI): calcd for C₁₇H₂₃O₃ (M + H)⁺ 275.1642, found 275.1643.

Methyl 2-(cyclohexylmethyl)-3-(naphthalen-2-yl)-3-oxopropanoate (8)



This compound was prepared according to General procedure, 58.3 mg, 60 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ8.57 – 8.52 (m, 1H), 8.07 – 8.05 (m, 2H), 7.95 – 7.90 (m, 2H), 7.67 – 7.58 (m, 2H), 4.65 (dd, *J* = 7.8, 6.5 Hz, 1H), 3.72 (s, 3H), 2.04 – 1.95 (m, 2H), 1.90 – 1.84 (m, 1H),

1.75 - 1.64 (m, 4H), 1.35 - 1.18 (m, 4H), 1.02 - 0.94 (m, 2H). ¹³**C NMR (101 MHz, Chloroform-***d*) δ 195.3, 170.8, 135.8, 133.5, 132.5, 130.5, 129.8, 128.8, 128.7, 127.8, 126.9, 124.2, 52.5, 51.6, 36.6, 35.9, 33.3, 33.1, 26.4, 26.1. HRMS m/z (ESI): calcd for $C_{21}H_{25}O_3$ (M + H)⁺ 325.1798, found 325.1800.

Methyl 3-(4-(tert-butyl)phenyl)-2-(cyclohexylmethyl)-3-oxopropanoate (9)



This compound was prepared according to General procedure, 65.3 mg, 66 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.93 (d, *J* = 8.6 Hz, 2H), 7.49 (d, *J* = 8.6 Hz, 2H), 4.44 (dd, *J* = 7.9, 6.4 Hz, 1H), 3.69 (s, 3H), 1.98 – 1.78

(m, 3H), 1.72 - 1.70 (m, 4H), 1.34 (s, 9H), 1.22 - 1.12 (m, 4H), 0.97 - 0.89 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 194.9, 170.9, 157.3, 133.5, 128.6, 125.7, 52.4, 51.5, 36.5, 35.9, 35.2, 33.3, 33.0, 31.0, 26.4, 26.1. HRMS m/z (ESI): calcd for C₂₁H₃₁O₃ (M + H)⁺ 331.2268, found 331.2270.

Methyl 2-(cyclohexylmethyl)-3-(4-methoxyphenyl)-3-oxopropanoate (10)



This compound was prepared according to General procedure, 65.7 mg, 72 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-d) δ 7.97 (d, J = 8.7 Hz, 2H), 6.94 (d, J = 8.7 Hz, 2H), 4.41 (t, J = 7.2 Hz, 1H), 3.87 (s, 3H),

3.67 (s, 3H), 1.95 - 1.78 (m, 3H), 1.66 (d, J = 12.6 Hz, 4H), 1.26 - 1.18 (m, 4H), 0.95 - 0.85 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 193.7, 170.5, 131.4, 131.3, 116.0, 115.8, 52.5, 51.6, 36.4, 35.8, 33.3, 33.0, 26.4, 26.1. HRMS m/z (ESI): calcd for C₁₈H₂₅O₄ (M + H)⁺ 305.1747, found 305.1747.

Methyl 2-(cyclohexylmethyl)-3-(3-methoxyphenyl)-3-oxopropanoate (11)



This compound was prepared according to General procedure, 49.2 mg, 72 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.58 – 7.57 (m, 1H), 7.52 – 7.51 (m, 1H), 7.41 – 7.37 (m, 1H), 7.15 – 7.12 (m, 1H), 4.45 – 4.42 (m, 1H), 3.86 (s, 3H), 3.69 (s, 3H), 1.74 – 1.61 (m, 5H), 1.16 – 1.09 (m, 2H), 0.97

- 0.83 (m, 6H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 195.2, 160.0, 137.4, 129.8, 121.2, 120.2, 112.7, 55.5, 52.5, 51.7, 36.5, 35.8, 33.3, 33.0, 29.7, 26.4, 26.1. HRMS m/z (ESI): calcd for C₁₈H₂₅O₄ (M + H)⁺ 305.1747, found 305.1747.

Methyl 2-(cyclohexylmethyl)-3-oxo-3-(4-(trifluoromethyl)phenyl)propanoate (12)



This compound was prepared according to General procedure, 52.3 mg, 51 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.09 – 7.96 (m, 2H), 7.80 – 7.55 (m, 2H), 4.36 (dd, J = 7.7, 6.5 Hz, 1H), 3.62 (s, 3H), 1.95 – 1.55 (m,

8H), 1.14 - 1.02 (m, 3H), 0.90 - 0.81 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 194.4, 170.3, 138.8, 128.9, 125.9 (d, J = 4.1 Hz), 52.7, 51.9, 36.2, 35.8, 33.2, 33.0, 26.3, 26.1. ¹⁹F NMR (376 MHz, Chloroform-*d*) δ -63.21. HRMS m/z (ESI): calcd for C₁₈H₂₂F₃O₃ (M + H)⁺ 343.1516, found 343.1519.

Methyl 4-(2-(cyclohexylmethyl)-3-methoxy-3-oxopropanoyl)benzoate (13)



This compound was prepared according to General procedure, 44.8 mg, 45 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.14 (d, J = 8.4 Hz, 2H), 8.02 (d, J = 8.5 Hz, 2H), 4.44 (dd, J = 7.8, 6.5 Hz,

1H), 3.95 (s, 3H), 3.68 (s, 3H), 1.94 – 1.84 (m, 2H), 1.80 – 1.76 (m, 4H), 1.22 – 1.11 (m, 3H), 0.97 – 0.80 (m, 4H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 194.9, 170.4, 139.4, 134.2, 130.0, 128.5, 52.53, 52.48, 52.0, 36.3, 35.8, 33.3, 33.0, 26.3, 26.1. MS (EI) *m*/*z* (relative intensity): 234 [M]+ (100). HRMS m/*z* (ESI): calcd for C₁₉H₂₅O₅ (M + H)⁺ 333.1697, found 333.1698.

Methyl 2-(cyclohexylmethyl)-3-(4-fluorophenyl)-3-oxopropanoate (14)



This compound was prepared according to General procedure, 61.3 mg, 70 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.04 (dd, *J* = 8.7, 5.4 Hz, 2H), 7.17 (t, *J* = 8.6 Hz, 2H), 4.42 (dd, *J* = 7.8, 6.5 Hz, 1H), 3.70 (s, 3H), 2.03 – 1.78

(m, 3H), 1.72 - 1.62 (m, 4H), 1.29 - 1.21 (m, 4H), 1.00 - 0.89 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 193.7, 170.5, 166.0 (d, J = 255.8 Hz), 132.6 (d, J = 3.0 Hz), 131.3 (d, J = 9.5 Hz), 115.9 (d, J = 22.0 Hz), 52.5, 51.6, 36.4, 35.8, 33.3, 33.0, 26.3, 26.1. HRMS m/z (ESI): calcd for C₁₇H₂₂O₃F (M + H)⁺ 293.1547, found 293.1547.

Methyl 3-(4-chlorophenyl)-2-(cyclohexylmethyl)-3-oxopropanoate (15)



This compound was prepared according to General procedure, 53.6 mg, 58 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-d) δ 8.07 – 7.98 (m, 2H), 7.15 (t, *J* = 8.6 Hz, 2H), 4.43 – 4.37 (m, 1H), 3.68 (s, 3H), 1.99 – 1.69 (m, 8H), 1.21 –

1.11 (m, 3H), 0.96 - 0.88 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 193.7, 170.5, 131.4, 131.3, 116.0, 115.8, 52.5, 51.6, 36.4, 35.8, 33.3, 33.0, 26.4, 26.1. HRMS m/z (ESI): calcd for C₁₇H₂₂O₃Cl (M + H)⁺ 309.1252, found 309.1253.

Methyl 3-(4-(chloromethyl)phenyl)-2-(cyclohexylmethyl)-3-oxopropanoate (16)



This compound was prepared according to General procedure, 30.9 mg, 32 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.98 (d, *J* = 8.4 Hz, 2H), 7.50 (d, *J* = 8.4 Hz, 2H), 4.62 (s, 2H), 4.43 (dd, *J* = 7.8, 6.5 Hz,

1H), 3.68 (s, 3H), 1.96 – 1.79 (m, 3H), 1.71 – 1.63 (m, 4H), 1.29 – 1.23 (m, 4H), 0.97 – 0.87 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 194.7, 170.6, 142.8, 135.9, 129.1, 128.9, 52.5, 51.7, 45.2, 36.4, 35.8, 33.3, 33.0, 26.4, 26.1. HRMS m/z (ESI): calcd for C₁₈H₂₄O₃F (M + H)⁺ 323.1408, found 323.1409.

Methyl 2-(cyclohexylmethyl)-3-(furan-2-yl)-3-oxopropanoate (17)



This compound was prepared according to General procedure, 43.6 mg, 55 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-d) $\delta 8.15$ (dd, J = 2.8, 1.3 Hz, 1H), 7.57 (dd, J = 5.1,

1.3 Hz, 1H), 7.33 (dd, J = 5.1, 2.9 Hz, 1H), 4.28 – 4.22 (m, 1H),

3.69 (s, 3H), 1.97 - 1.84 (m, 2H), 1.72 - 1.67 (m, 5H), 1.23 - 1.13 (m, 4H), 0.97 - 0.88 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 189.1, 170.6, 141.3, 133.1, 127.3, 126.6, 53.5, 52.5, 36.3, 35.8, 33.3, 33.0, 26.37, 26.07. HRMS m/z (ESI): calcd for C₁₅H₂₁O₄ (M + H)⁺ 265.1434, found 265.1438.

Methyl 3-(benzofuran-5-yl)-2-(cyclohexylmethyl)-3-oxopropanoate (18)



This compound was prepared according to General procedure, 65.9 mg, 70 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-d) δ 8.31 (d, J = 1.9 Hz, 1H), 8.00 (dd, J = 8.7, 1.9 Hz, 1H), 7.71 (d, J = 2.2 Hz, 1H), 7.57 (dt, J = 8.8, 0.8 Hz, 1H),

6.88 (dd, J = 2.2, 1.0 Hz, 1H), 4.55 (dd, J = 7.9, 6.5 Hz, 1H), 3.69 (s, 3H), 2.03 – 1.89 (m, 2H), 1.86 – 1.59 (m, 6H), 1.30 – 1.16 (m, 3H), 0.99 – 0.92 (m, 2H). ¹³**C NMR (101 MHz, Chloroform-***d*) δ 194.7, 170.8, 157.6, 146.5, 131.5, 127.6, 125.2, 122.9, 111.7, 107.3, 52.4, 51.5, 36.5, 35.7, 33.2, 32.9, 26.3, 26.0. HRMS m/z (ESI): calcd for C₁₉H₂₃O₄ (M + H)⁺ 315.1591, found 315.1597.

Ethyl 2-(3-methylbenzoyl)-4-oxooctanoate (19)



This compound was prepared according to General procedure, 62.4 mg, 63 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.48 (d, *J* = 1.1 Hz, 1H), 7.97 (d, *J* = 1.4 Hz, 2H), 7.55 (d, *J* = 5.4 Hz, 1H), 7.47 (d, *J* = 5.5 Hz, 1H), 4.56 (t,

J = 7.2 Hz, 1H), 3.69 (s, 3H), 2.02 – 1.90 (m, 2H), 1.86 – 1.79 (m, 1H), 1.76 – 1.63 (m, 4H), 1.35– 1.31 (m, 1H), 1.22 – 1.09 (m, 3H), 0.99 – 0.92 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 195.2, 170.9, 144.7, 139.5, 132.7, 128.1, 124.8, 124.7, 123.7, 122.9, 52.6, 51.7, 36.6, 35.9, 33.3, 33.1, 26.4, 26.1. HRMS m/z (ESI): calcd for C₁₉H₂₃O₃S (M + H)⁺ 331.1362, found 331.1366.

Methyl 2-(cyclopentylmethyl)-3-oxo-3-(p-tolyl)propanoate (20)



This compound was prepared according to General procedure, 59.1 mg, 72 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.90 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.3 Hz, 2H), 4.41 – 4.32 (m, 1H), 3.68 (s, 3H), 2.42 (s, 3H), 2.10 –

2.01 (m, 2H), 1.77 – 1.45 (m, 7H), 1.15 – 1.12 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 195.0, 170.8, 144.5, 133.7, 130.7, 129.5, 128.8, 53.2, 52.4, 38.3, 35.3,

32.6 (d, J = 4.4 Hz), 25.0, 21.7. HRMS m/z (ESI): calcd for $C_{17}H_{23}O_3$ (M + H)⁺ 275.1642, found 275.1642.

Methyl 2-(bicyclo[2.2.1]heptan-2-ylmethyl)-3-oxo-3-phenylpropanoate (21)



This compound was prepared according to General procedure, 53.2 mg, 62 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.95 – 7.87 (m, 2H), 7.31 – 7.29 (m, 2H), 4.39 – 4.31 (m, 1H), 3.69 (d, *J* = 4.5 Hz, 3H),

2.44 (s, 3H), 2.23 – 2.21 (m, 1H), 2.07 – 1.97 (m, 2H), 1.84 – 1.71 (m, 1H), 1.51 – 1.32 (m, 5H), 1.12 – 1.05 (m, 4H). ¹³**C NMR** (**101 MHz**, **Chloroform-***d*) δ 195.1, 195.0, 170.84, 170.80, 144.5, 129.6, 129.5, 128.77, 128.75, 52.4, 52.3, 52.2, 41.2, 41.1, 40.13, 40.05, 38.0, 37.9, 36.6, 35.9, 35.3, 35.3, 29.9, 28.6, 28.6, 21.7. HRMS m/z (ESI): calcd for C₁₈H₂₃O₃ (M + H)⁺ 287.1642, found 287.1642.

Methyl 2-((7-oxabicyclo[2.2.1]heptan-2-yl)methyl)-3-oxo-3-phenylpropanoate (22)



This compound was prepared according to General procedure, 43.2 mg, 50 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.93 – 7.86 (m, 2H), 7.31 – 7.28 (m, 2H), 4.57 (q, *J* = 5.1 Hz, 1H), 4.38 – 4.23 (m, 2H),

3.70 (d, J = 4.4 Hz, 3H), 2.44 (s, 3H), 2.17 (dd, J = 14.0, 7.4 Hz, 1H), 1.91 – 1.85 (m, 1H), 1.77 – 1.59 (m, 7H), 1.40 – 1.36 (m, 1.6 Hz, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 194.6, 194.5, 170.53, 170.51, 144.7, 144.6, 133.7, 133.6, 129.6, 128.8, 128.7, 80.2, 80.0, 76.54, 76.53, 52.5, 51.9, 51.7, 41.4, 41.3, 37.9, 37.9, 34.5, 29.7, 29.6, 29.6, 29.5, 21.7. HRMS m/z (ESI): calcd for C₁₈H₂₃O₄ (M + H)⁺ 303.1591, found 303.1595.

Methyl -3-oxo-3-phenyl-2-((tetrahydrofuran-2-yl)methyl)propanoate (23)



This compound was prepared according to General procedure, 44.0 mg, 56 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-d) $\delta 8.08 - 7.97$ (m, 2H), 7.61 - 7.54 (m, 1H), 7.50 -7.43 (m, 2H), 4.70 - 4.63 (m, 1H), 3.98 - 3.74 (m, 2H), 3.73 -

3.62 (m, 4H), 2.35 – 2.31 (m, 1H), 2.15 – 1.81 (m, 4H), 1.55 – 1.49 (m, 1H). ¹³**C NMR** (**101 MHz, Chloroform-***d***)** δ 195.5, 195.4, 170.5, 170.4, 136.6, 133.5, 133.4, 128.8, 128.8, 128.7, 76.8, 76.4, 67.6, 67.6, 52.5, 52.4, 51.3, 50.8, 35.2, 35.0, 31.5, 31.5, 29.7, 25.7, 25.6. HRMS m/z (ESI): calcd for C₁₅H₁₉O₄ (M + H)⁺ 263.1278, found 263.1279.

Methyl -2-((1,4-dioxan-2-yl)methyl)-3-oxo-3-phenylpropanoate (24)



This compound was prepared according to General procedure, 26.6 mg, 32 % yield as colorless oil. ¹H NMR (400 MHz,

Chloroform-*d*) δ 8.05 – 8.01 (m, 2H), 7.64 – 7.55 (m, 1H), 7.52

-7.47 (m, 2H), 4.72 - 4.68 (m, 1H), 3.82 - 3.52 (m, 9H), 3.32 - 3.24 (m, 1H), 2.24 - 2.14 (m, 1H), 2.06 - 1.91 (m, 1H). ¹³**C NMR (101 MHz, Chloroform-***d***)** δ 195.3, 170.4, 133.7, 133.6, 128.9, 128.8, 73.3, 72.6, 71.1, 71.0, 66.8, 66.6, 66.4, 66.4, 52.6, 52.6, 49.6, 48.9, 30.8, 30.7, 29.7, 29.3. HRMS m/z (ESI): calcd for C₁₅H₁₉O₅ (M + H)⁺ 279.1227, found 279.1230.

Methyl 3-oxo-2-((3-oxocyclopentyl)methyl)-3-phenylpropanoate (25)



This compound was prepared according to General procedure, 35.4 mg, 41 % yield as colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.90 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 8.2 Hz,

2H), 4.44 - 4.32 (m, 1H), 3.69 (s, 3H), 2.43 (s, 3H), 2.39 - 2.06 (m, 7H), 1.87 - 1.75 (m, 1H), 1.58 - 1.52 (m, 1H). ¹³**C NMR (101 MHz, Chloroform-***d***)** δ 218.4, 194.1, 144.9, 144.9, 129.6, 128.7, 52.6, 52.3, 52.1, 44.9, 44.8, 38.5, 35.3, 35.3, 34.61, 34.58, 29.5, 29.5, 21.7. HRMS m/z (ESI): calcd for C₁₆H₁₉O₄ (M + H)⁺ 275.1278, found 275.1279.

Ethyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (26)



This compound was prepared according to General procedure, 63.4 mg, 70 % yield as colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 6.5 Hz, 2H), 7.27 (d, *J* = 7.4 Hz, 2H), 4.44 – 4.37 (m, 1H), 4.14 (q, *J* = 7.1 Hz, 2H), 2.41 (s, 3H),

2.01 - 1.80 (m, 3H), 1.72 - 1.60 (m, 4H), 1.33 - 1.24 (m, 2H), 1.22-1.16 (m, 5H), 0.96 - 0.85 (m, 2H).¹³**C NMR (101 MHz, Chloroform-***d***)** δ 195.0, 170.3, 144.3, 133.6, 129.4, 128.7, 61.2, 51.6, 36.3, 35.8, 33.3, 32.9, 26.3, 26.1, 26.0, 21.6, 14.0. HRMS m/z (ESI): calcd for C₁₉H₂₇O₃ (M + H)⁺ 303.1955, found 303.1955.

Tert-butyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (27)



This compound was prepared according to General procedure, 72.2 mg, 73 % yield as colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.81 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 7.9 Hz, 2H), 4.19 (dd, *J* = 7.7, 6.6 Hz, 1H), 2.34 (s, 3H), 1.82 – 1.55

(m, 8H), 1.37 (d, J = 2.3 Hz, 1H), 1.28 (s, 9H), 1.12 – 1.06 (m, 2H), 0.89 – 0.80 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 195.3, 169.6, 144.0, 134.0, 129.3, 128.7, 81.5, 52.9, 36.1, 35.8, 33.4, 33.1, 29.7, 27.8, 26.4, 26.2, 21.6. HRMS m/z (ESI): calcd for C₂₁H₃₁O₃ (M + H)⁺ 331.2268, found 331.2268.

Isobutyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (28)



This compound was prepared according to General procedure, 67.3 mg, 68 % yield as colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.90 (d, *J* = 8.3 Hz, 2H), 7.27 (d, *J* = 8.0 Hz, 2H), 4.42 (dd, *J* = 7.8, 6.5 Hz, 1H), 3.86 (dd, *J* = 6.6, 2.8 Hz,

2H), 2.41 (s, 3H), 1.97 – 1.79 (m, 4H), 1.72 – 1.63 (m, 4H), 1.20 – 1.11 (m, 3H), 0.98 – 0.88 (m, 3H), 0.82 (dd, J = 6.7, 1.1 Hz, 6H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 194.9, 170.5, 144.3, 133.8, 129.4, 128.7, 71.3, 51.7, 36.3, 35.8, 33.3, 33.0, 29.7, 27.6, 26.4, 26.1, 21.7, 18.9. MS (EI) m/z (relative intensity): 234 [M]+ (100). HRMS m/z (ESI): calcd for C₂₁H₃₁O₃ (M + H)⁺ 331.2268, found 331.2269.

Cyclohexyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (29)



This compound was prepared according to General procedure, 66.2 mg, 62 % yield as colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 8.3 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 4.78 (dt, *J* = 8.6, 4.4 Hz, 1H), 4.36 (dd, *J* = 8.0, 6.4 Hz,

1H), 2.41 (s, 3H), 1.95 - 1.80 (m, 3H), 1.76 - 1.62 (m, 7H), 1.36 - 1.32 (m, 3H), 1.23 - 1.08 (m, 5H), 0.99 - 0.85 (m, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 195.0, 169.9, 144.2, 133.9, 129.3, 128.7, 73.3, 52.1, 36.2, 35.8, 33.4, 33.0, 31.3, 31.1, 29.7, 26.4, 26.2, 26.1, 25.3, 23.4, 21.7. HRMS m/z (ESI): calcd for C₂₃H₃₃O₃ (M + H)⁺ 357.2424, found 357.2425.

Benzyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (30)



This compound was prepared according to General procedure, 65.5 mg, 60 % yield as colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.86 (d, *J* = 8.1 Hz, 2H), 7.32 – 7.20 (m, 7H), 5.12 (s, 2H), 4.45 (dd, *J* = 8.2, 6.2 Hz, 1H), 2.40 (s, 3H), 1.99

- 1.77 (m, 3H), 1.70 - 1.59 (m, 4H), 1.24 - 1.08 (m, 4H), 0.86 - 0.96 (m, 2H). ¹³C **NMR (101 MHz, Chloroform-***d***)** δ 194.8, 170.3, 144.4, 135.6, 133.6, 129.5, 128.8, 128.5, 128.2, 128.1, 66.9, 51.7, 36.4, 35.8, 33.4, 32.9, 26.4, 26.1, 26.1, 21.7. HRMS m/z (ESI): calcd for C₂₄H₂₉O₃ (M + H)⁺ 365.2111, found 365.2120.

Phenyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (31)



This compound was prepared according to General procedure, 56.7 mg, 54 % yield as colorless oil. ¹H NMR (400 MHz, Chloroform-d) δ 7.96 (d, J = 7.9 Hz, 2H), 7.36 – 7.29 (m,

4H), 7.20 (t, *J* = 7.4 Hz, 1H), 6.99 (d, *J* = 7.9 Hz, 2H), 4.63 (dd, *J* = 8.6, 5.7 Hz, 1H), 2.44 (s, 3H), 2.07 (td, *J* = 8.3, 4.2 Hz, 1H), 1.96 – 1.87 (m, 2H), 1.82 – 1.60 (m, 5H), 1.45 – 1.38 (m, 1H), 1.22 – 1.14 (m, 2H), 1.05 – 0.97 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 218.4, 194.1, 144.9, 144.9, 129.6, 128.7, 52.6, 52.3, 52.1, 44.9, 44.8, 38.5, 35.3, 35.3, 34.61, 34.58, 29.5, 29.5, 21.7. HRMS m/z (ESI): calcd for C₂₃H₂₇O₃ (M + H)⁺ 351.1955, found 351.1956.

Allyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (32)



This compound was prepared according to General procedure, 41.4 mg, 44 % yield as colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 8.2 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 5.88 – 5.75 (m, 1H), 5.27 – 5.14 (m, 2H),

4.59 (dt, J = 5.6, 1.5 Hz, 2H), 4.45 (dd, J = 8.0, 6.4 Hz, 1H), 2.42 (s, 3H), 1.94 – 1.58 (m, 9H), 1.14 (dt, J = 15.6, 5.9 Hz, 2H), 0.95 – 0.91 (m, 2H). ¹³**C NMR (101 MHz, Chloroform-***d*) δ 194.93, 170.1, 144.4, 133.6, 131.7, 129.5, 128.8, 118.4, 65.8, 51.6, 36.5, 35.8, 33.3, 33.0, 26.4, 26.1, 26.1, 21.7. HRMS m/z (ESI): calcd for C₂₀H₂₇O₃ (M + H)⁺ 315.1955, found 315.1957.

2-Chloroethyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (33)



This compound was prepared according to General procedure, 46.4 mg, 46 % yield as colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 8.2 Hz, 2H), 7.28 (d, *J* = 9.5 Hz, 2H), 4.47 (dd, *J* = 8.4, 6.0 Hz, 1H), 4.35 (t,

J = 5.7 Hz, 2H), 3.61 (td, J = 5.7, 3.1 Hz, 2H), 2.42 (s, 3H), 1.96 – 1.92 (m, 1H), 1.88 – 1.79 (m, 2H), 1.75 – 1.62 (m, 4H), 1.31 – 1.14 (m, 4H), 0.98 – 0.88 (m, 2H). ¹³C **NMR (101 MHz, Chloroform-***d*) δ 194.7, 170.0, 144.5, 133.4, 129.5, 128.7, 64.5, 51.4, 41.2, 36.4, 35.8, 33.3, 32.8, 26.3, 26.1, 26.0, 21.7. HRMS m/z (ESI): calcd for C₁₉H₂₆O₃Cl (M + H)⁺ 337.1565, found 337.1565.

2,2,2-trifluoroethyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (34)



This compound was prepared according to General procedure, 72.6 mg, 68 % yield as colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.86 (d, *J* = 8.3 Hz, 2H), 7.29

(d, J = 8.0 Hz, 2H), 4.58 – 4.38 (m, 3H), 2.43 (s, 3H), 1.97 – 1.94 (m, 1H), 1.87 – 1.80 (m, 2H), 1.73 – 1.61 (m, 4H), 1.33 – 1.13 (m, 4H), 0.96 – 0.91 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 194.1, 168.9, 144.8, 133.1, 129.6, 128.8, 124.1, 121.4, 60.7 (q, J = 36.9 Hz), 51.2, 36.4, 35.88, 33.4, 32.8, 26.3, 26.1 (d, J = 4.6 Hz), 21.7. HRMS m/z (ESI): calcd for C₁₉H₂₄O₃F₃ (M + H)⁺ 357.1672, found 357.1680.

2-methoxyethyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (35)



This compound was prepared according to General procedure, 57.7 mg, 58 % yield as colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.95 – 7.84 (m, 2H), 7.28 – 7.26 (m, 2H), 4.50 – 4.40 (m, 1H), 4.32 – 4.19 (m, 2H),

3.53 – 3.50 (m, 2H), 3.29 (d, J = 1.9 Hz, 3H), 2.42 (d, J = 1.9 Hz, 3H), 1.95 – 1.63 (m, 7H), 1.31 – 1.11 (m, 4H), 0.99 – 0.84 (m, 2H). ¹³**C NMR** (**101 MHz, Chloroform-***d*) δ 194.9, 170.4, 144.4, 133.6, 129.4, 128.8, 70.3, 64.2, 58.9, 51.6, 36.4, 35.9, 33.4, 33.0, 26.4, 26.1, 26.1, 21.7. HRMS m/z (ESI): calcd for C₂₀H₂₉O₄ (M + H)⁺ 333.2060, found 333.2062.

Diethyl (3-cyclohexyl-1-oxo-1-(p-tolyl)propan-2-yl)phosphonate (36)



This compound was prepared according to General procedure, 50.5 mg, 46 % yield as colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ ¹H NMR (400 MHz, Chloroform-*d*) δ 7.93 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 6.6 Hz, 2H), 4.25 – 4.00 (m,

5H), 2.44 (s, 3H), 1.86 – 1.64 (m, 8H), 1.28 (d, J = 3.2 Hz, 3H), 1.20 (d, J = 7.1 Hz, 3H), 1.11 (t, J = 10.7, 3H), 0.92 – 0.86 (m, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 199.6, 195.6, 144.1, 135.2, 129.2, 128.8, 62.7, 62.5, 45.2, 43.9, 36.4, 36.2, 33.7, 32.5, 26.3, 26.1, 26.0, 21.6, 16.4. HRMS m/z (ESI): calcd for C₂₀H₃₂O₄P (M + H)⁺ 367.2033, found 367.2036.

(1S,2R,5S)-2-isopropyl-5-methylcyclohexyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl) propanoate (37)



This compound was prepared according to General procedure, 66.7 mg, 54 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.88 (dd, *J* = 8.2, 3.7 Hz, 2H), 7.25 (d, J = 7.8 Hz, 2H), 4.66 – 4.60 (m, 1H), 4.37 – 4.33 (m, 1H), 2.41 (s, 3H), 1.97 – 1.82 (m,

4H), 1.74 - 1.63 (m, 5H), 1.45 - 1.09 (m, 7H), 1.03 - 0.72 (m, 12H), 0.63 - 0.54 (m, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 133.9, 129.3, 128.7, 128.7, 75.3, 75.2, 52.5, 52.3, 46.8, 46.8, 40.4, 40.3, 36.2, 36.1, 35.8, 35.7, 34.2, 33.5, 32.93, 32.90, 31.3, 26.4, 26.2, 26.1, 26.1, 25.8, 25.7, 23.0, 22.9, 22.0, 21.7, 20.8, 20.7, 15.8, 15.7. HRMS m/z (ESI): calcd for C₂₇H₄₁O₃ (M + H)⁺ 413.3050, found 413.3055.

Methyl 3-oxo-3-(p-tolyl)-2-((triphenylsilyl)methyl)propanoate (38)



This compound was prepared according to General procedure, 94.6 mg, 68 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.64 – 7.52 (m, 8H), 7.39–7.31 (m, 9H), 7.13 (d, *J* = 7.9 Hz, 2H), 4.48– 4.44 (m, 1H), 3.32 (s, 3H), 2.37 (s,

3H), 2.25 – 2.19 (m, 1H), 2.09 – 2.03 (m, 1H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 195.0, 170.8, 144.2, 135.8, 133.8, 133.1, 129.7, 129.2, 128.8, 128.0, 52.3, 49.4, 21.7, 12.9. HRMS m/z (ESI): calcd for C₃₀H₂₉O₃Si (M + H)⁺ 465.1880, found 465.1882.

Methyl 3-oxo-3-(m-tolyl)-2-((triphenylsilyl)methyl)propanoate (39)



This compound was prepared according to General procedure, 98.7 mg, 71 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-d) δ 7.66 – 7.48 (m, 8H), 7.47 – 7.32 (m, 10H), 7.26 – 7.24 (m, 1H), 4.51 (dd, J = 8.5, 5.8 Hz, 1H), 3.38 (s, 3H), 2.35 (s, 3H), 2.25 (dd, J = 15.2, 8.5 Hz, 1H), 2.11 (dd, J = 15.2, 5.8 Hz, 1H). ¹³C NMR (101

MHz, Chloroform-*d*) δ 195.8, 170.8, 135.8, 135.6, 134.1, 133.7, 129.7, 129.2, 128.4,

128.0, 125.9, 52.3, 49.5, 21.3, 12.9. HRMS m/z (ESI): calcd for C₃₀H₂₉O₃Si (M + H)⁺ 465.1880, found 465.1884.

Methyl 3-(4-fluorophenyl)-3-oxo-2-((triphenylsilyl)methyl)propanoate (40)



This compound was prepared according to General procedure, 91.2 mg, 65 % yield as a colorless oil. ¹H NMR (400 MHz, **Chloroform-***d*) δ 7.74 – 7.65 (m, 2H), 7.55 – 7.53 (m, 6H), 7.41 - 7.31 (m, 9H), 7.01 - 6.97 (m, 2H), 4.45 - 4.42 (m, 1H), 3.37 (s, 3H), 2.26 - 2.07 (m, 2H). ¹³C NMR (101 MHz, Chloroform-d) δ 193.8, 170.7,

165.8 (d, J = 255.5 Hz), 135.8, 133.7, 132.1(d, J = 3.2 Hz), 131.3 (d, J = 9.3 Hz), 129.8, 128.0, 115.6 (d, J = 21.9 Hz), 52.4, 49.5, 12.8. ¹⁹F NMR (376 MHz, Chloroform-d) δ -104.7. HRMS m/z (ESI): calcd for C₂₉H₂₆FO₃Si (M + H)⁺ 469.1630, found 469.1631.

Methyl 3-(4-chlorophenyl)-3-oxo-2-((triphenylsilyl)methyl)propanoate (41)



This compound was prepared according to General procedure, 90.2 mg, 62 % yield as a colorless oil. ¹H NMR (400 MHz, **Chloroform-d**) δ 7.62 – 7.49 (m, 8H), 7.41 – 7.28 (m, 11H), 4.42 (dd, *J* = 7.7, 6.5 Hz, 1H), 3.38 (s, 3H), 2.25 – 2.08 (m, 2H).

¹³C NMR (101 MHz, Chloroform-d) δ 194.2, 170.6, 139.8, 135.8, 134.0, 133.6, 130.0, 129.8, 128.8, 128.0, 52.4, 49.5, 12.7. HRMS m/z (ESI): calcd for C₂₉H₂₆ClO₃Si (M + H)⁺ 485.1334, found 485.1337.

Isobutyl 3-oxo-3-(p-tolyl)-2-((triphenylsilyl)methyl)propanoate (42)



This compound was prepared according to General procedure, 76.1 mg, 69 % yield as a colorless oil. ¹H NMR (400 MHz, **Chloroform-***d*) δ 7.75 – 7.53 (m, 8H), 7.50 – 7.29 (m, 9H), 7.16 (d, J = 8.0 Hz, 2H), 4.49 (dd, J = 8.0, 6.1 Hz, 1H), 3.55

(ddd, J = 39.2, 10.6, 6.7 Hz, 2H), 2.40 (s, 3H), 2.30 - 2.11 (m, 2H), 1.70 (dt, J = 13.4, 1.20)6.7 Hz, 1H), 0.76 (dd, J = 6.7, 1.8 Hz, 6H). ¹³C NMR (101 MHz, Chloroform-d) δ 195.0, 170.6, 144.0, 135.9, 133.9, 133.3, 129.7, 129.1, 128.8, 127.9, 71.4, 49.5, 27.4, 21.7, 18.9, 12.5. HRMS m/z (ESI): calcd for $C_{33}H_{35}O_3Si (M + H)^+$ 507.2350, found 507.2361.

Cyclohexyl 3-oxo-3-(p-tolyl)-2-((triphenylsilyl)methyl)propanoate (43)



(dd, J = 7.7, 6.2 Hz, 1H), 2.40 (s, 3H), 2.28 – 2.10 (m, 2H), 1.59 – 1.09 (m, 11H). ¹³C **NMR (101 MHz, Chloroform-***d***)** δ 170.0, 135.9, 134.0, 129.6, 129.0, 128.8, 127.9, 73.6, 49.9, 30.92, 30.89, 25.3, 23.3, 21.7, 12.3. HRMS m/z (ESI): calcd for C₃₆H₃₃O₃Si (M + H)⁺ 533.2506, found 533.2515.

Benzyl 3-oxo-3-(p-tolyl)-2-((triphenylsilyl)methyl)propanoate (44)



This compound was prepared according to General procedure, 76.1 mg, 47 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.62 – 7.50 (m, 8H), 7.41 – 7.28 (m, 11H), 4.42 (dd, J = 7.7, 6.5 Hz, 1H), 3.38 (s, 3H), 2.23 – 2.07 (m,

2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 194.9, 170.3, 144.1, 135.9, 133.8, 129.7, 129.1, 128.8, 128.4, 128.1, 128.0, 127.95, 66.9, 49.6, 21.6, 12.6. HRMS m/z (ESI): calcd for C₃₆H₃₃O₃Si (M + H)⁺ 541.2193, found 541.2208.

3-oxo-3-(p-tolyl)-2-((triphenylsilyl)methyl)propanoate (45)



This compound was prepared according to General procedure, 79.2 mg, 52 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.61 – 7.52 (m, 8H), 7.39 – 7.32 (m, 9H), 7.12 (d, *J* = 8.0 Hz, 2H), 4.48 – 4.45

(m, 1H), 3.96 - 3.81 (m, 2H), 3.35 - 3.27 (m, 2H), 3.21 (s, 3H), 2.36 (s, 3H), 2.21 (dd,

J = 15.2, 8.6 Hz, 1H), 2.08 (dd, J = 15.2, 5.6 Hz, 1H). ¹³C NMR (101 MHz, **Chloroform-***d*) δ 194.9, 170.5, 144.1, 135.9, 133.8, 133.1, 129.7, 129.1, 128.9, 128.0, 70.0, 64.1, 58.8, 49.5, 21.6, 12.7. HRMS m/z (ESI): calcd for C₃₂H₃₃O₄Si (M + H)⁺ 509.2143, found 509.2150.

Methyl 2-((methyldiphenylsilyl)methyl)-3-oxo-3-(p-tolyl)propanoate (46)

This compound was prepared according to General procedure, 86.8 mg, 72 % yield as a colorless oil. ¹H NMR (400 MHz, Chloroform-d) δ 7.67 – 7.61 (m, 2H), 7.53 – 7.49 (m, 4H), 7.39 – 7.31 (m, 6H), 7.16 (d, J = 8.0 Hz, 2H), 4.31 (dd, J = 8.5,

6.0 Hz, 1H), 3.49 (s, 3H), 2.38 (s, 3H), 1.89 (dd, J = 15.0, 8.6 Hz, 1H), 1.73 (dd, J = 15.0, 6.0 Hz, 1H), 0.55 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 195.3, 171.2, 144.3, 135.9, 135.7, 134.6, 133.1, 129.5, 129.49, 129.3, 128.8, 128.0, 127.9, 52.4, 49.3, 21.6, 14.1, -4.1. HRMS m/z (ESI): calcd for C₂₅H₂₇O₃Si (M + H)⁺ 403.1724, found 403.1728.

5. Mechanistic Investigations



A dry 10 mL vial equipped with a Teflon-coated magnetic stir bar was charged with acyl chlorides (0.30 mmol, 1.0 equiv.), alkanes (3.0 mmol, 10 equiv.), alkenes (0.90 mmol, 3.0 equiv.), and 2,2,6,6-Tetramethylpiperidinooxy (0.9 mmol, 3.0 equiv.), LiCl (37.8 mg, 3.0 equiv.) and FeCl₃· $6H_2O$ (8.1 mg, 10 mol%) were dissolved in acetone (1.0 mL). Afterward, a pre-catalyst solution (it was prepared by a mix of NiBr₂· DME (6.6 mg, 10 mol%), 4,4'-di-tert-butyl-2,2'-bipyridine (8.1 mg, 10 mol%) in anhydrous MeCN (2.0 mL) under N₂ atmosphere, and was stirred for 10 minutes.) was added via

syringe. Then, it was capped with a Teflon lid equipped with Graphite Felt $(20 \times 10 \times 15 \text{ mm})$ as the anode and reticulated vitreous carbon $(20 \times 10 \times 1 \text{ mm})$ as the cathode. The reaction mixture was stirred under N₂ and electrolyzed at a constant current of 4 mA under irradiation by a 10-W 390-nm LED lamp with a cooling fan. After being stirred at 10 °C for 24 h, the reaction mixture was analyzed by GCMS.



A dry 10 mL vial equipped with a Teflon-coated magnetic stir bar was charged with benzoyl chloride (0.30 mmol, 1.0 equiv.), cyclohexane (3.0 mmol, 10 equiv.), methyl acrylate (0.90 mmol, 3.0 equiv.), TBAClO₄ (846.9 mg, 3.0 equiv.) and FeCl₃· $6H_2O$ (8.1 mg, 10 mol%) were dissolved in acetone (1.0 mL). Afterward, a pre-catalyst solution (it was prepared by a mix of NiBr₂· DME (6.6 mg, 10 mol%), 4,4'-di-tert-butyl-2,2'-bipyridine (8.1 mg, 10 mol%) in anhydrous MeCN (2.0 mL) under N₂ atmosphere, and was stirred for 10 minutes.) was added via syringe. Then, it was capped with a Teflon lid equipped with Graphite Felt ($20 \times 10 \times 15$ mm) as the anode and reticulated vitreous carbon ($20 \times 10 \times 1$ mm) as the cathode. The reaction mixture was stirred under N₂ and electrolyzed at a constant current of 4 mA under irradiation by a 10-W 390-nm LED lamp for 24 h. After the reaction is completed, the reaction mixture was analyzed by GCMS. We have no found the corresponding compound **7**.



A dry 10 mL vial equipped with a Teflon-coated magnetic stir bar was charged with benzoyl chloride (0.30 mmol, 1.0 equiv.), chlorocyclohexane (3.0 mmol, 10 equiv.), methyl acrylate (0.90 mmol, 3.0 equiv.), LiCl (37.8 mg, 3.0 equiv.) and FeCl₃·6H₂O (8.1 mg, 10 mol%) were dissolved in acetone (1.0 mL). Afterward, a pre-catalyst solution (it was prepared by a mix of NiBr₂·DME (6.6 mg, 10 mol%), 4,4'-di-tert-butyl-2,2'-bipyridine (8.1 mg, 10 mol%) in anhydrous MeCN (2.0 mL) under N₂ atmosphere, and was stirred for 10 minutes.) was added via syringe. Then, it was capped with a Teflon lid equipped with Graphite Felt ($20 \times 10 \times 15$ mm) as the anode and reticulated vitreous carbon ($20 \times 10 \times 1$ mm) as the cathode. The reaction mixture was stirred under N₂ and electrolyzed at a constant current of 4 mA under irradiation by a 10-W 390-nm LED lamp for 24 h. After the reaction is completed, the reaction mixture was analyzed by GCMS. We have no found the corresponding compound **7**.



completed, the reaction mixture was analyzed by GCMS.



A dry 10 mL vial equipped with a Teflon-coated magnetic stir bar was charged with cyclohexane (3.0 mmol, 10 equiv.), methyl acrylate (0.90 mmol, 3.0 equiv.), LiCl (37.8 mg, 3.0 equiv.) and FeCl₃· $6H_2O$ (8.1 mg, 10 mol%) were dissolved in acetone (1.0 mL). Afterward, complex **47** (0.3 mmol, 1.0 equiv.) in anhydrous MeCN (2.0 mL) under N₂ atmosphere, and was stirred for 10 minutes.) was added via syringe. Then, it was capped with a Teflon lid equipped with Graphite Felt ($20 \times 10 \times 15$ mm) as the anode and reticulated vitreous carbon ($20 \times 10 \times 1$ mm) as the cathode. The reaction mixture was stirred under N₂ and electrolyzed at a constant current of 4 mA under irradiation by a 10-W 390-nm LED lamp with a cooling fan to keep the reaction temperature at 10 °C for 24 h. After the reaction was completed, the reaction mixture was analyzed by GCMS.



A dry 10 mL vial equipped with a Teflon-coated magnetic stir bar was charged with benzoyl chloride (0.30 mmol, 1.0 equiv.), cyclohexane (3.0 mmol, 10 equiv.), methyl acrylate (0.90 mmol, 3.0 equiv.), LiCl (37.8 mg, 3.0 equiv.) and FeCl₃·6H₂O (8.1 mg, 10 mol%) were dissolved in acetone (1.0 mL). Afterward, complex **47** (0.03 mmol. 10 mol%) in anhydrous MeCN (2.0 mL) under N₂ atmosphere, and was stirred for 10 minutes.) was added via syringe. Then, it was capped with a Teflon lid equipped with Graphite Felt ($20 \times 10 \times 15$ mm) as the anode and reticulated vitreous carbon ($20 \times 10 \times 1$ mm) as the cathode. The reaction mixture was stirred under N₂ and electrolyzed at a constant current of 4 mA with a cooling fan to keep the reaction temperature at 10 °C under irradiation by a 10-W 390-nm LED lamp for 24 h. After the reaction was completed, the reaction mixture was analyzed by gas chromatography to obtain the yield of **7** using dodecane as an internal standard.



A dry 10 mL vial equipped with a Teflon-coated magnetic stir bar was charged with benzoyl chloride (0.30 mmol, 1.0 equiv.), **1a** or **[D]-1a** (3.0 mmol, 10 equiv.), methyl acrylate (0.90 mmol, 3.0 equiv.), LiCl (37.8 mg, 3.0 equiv.) and FeCl₃·6H₂O (8.1 mg, 10 mol%) were dissolved in acetone (1.0 mL). Afterward, a pre-catalyst solution (it was prepared by a mix of NiBr₂·DME (6.6 mg, 10 mol%), 4,4'-di-tert-butyl-2,2'-bipyridine (8.1 mg, 10 mol%) in anhydrous MeCN (2.0 mL) under N₂ atmosphere, and was stirred for 10 minutes.) was added via syringe. Then, it was capped with a Teflon lid equipped with Graphite Felt ($20 \times 10 \times 15$ mm) as the anode and reticulated vitreous carbon ($20 \times 10 \times 1$ mm) as the cathode. The reaction mixture was stirred under N₂ and electrolyzed at a constant current of 4 mA with a cooling fan to keep the reaction temperature at 10 °C under irradiation by a 10-W 390-nm LED lamp for the indicated time (five parallel runs). After the reaction was completed, the reaction mixture was analyzed by gas chromatography to obtain the yield of **7** using dodecane as an internal standard. KIE value was measured averaging the slopes obtained for each run, and kH/kD = 1.15 was obtained.



6. Cyclic Voltammetry (CV) Measurements

All measurements were performed under anhydrous conditions in argon-filled glovebox. All supporting electrolytes were dried under dynamic vacuum (less than 0.1 mbar) over 24 h at 100 °C and stored inside the glovebox. The cell for the analysis was equipped with a glass vial (working volume is 10 mL) and Teflon cap, equipped with O-ring for tight sealing. Glassy carbon was used as working electrodes (circle, d = 3 mm), and an Ag/AgCl as the reference electrode. Each sample was calibrated against the Fc/Fc⁺ reference. All measurements were conducted in 0.1 M solutions of nBu₄NPF₆ in MeCN/acetone (2/1). All analyte concentration was 10 mM. The scan rate was typically 50 mV/s.





7. NMR Spectra

Methyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (4)

7.7.7.90 7.7.7.88 7.7.7.27 7.7.28 7.7.27 7.7.28 7.7.29 7.7





Methyl 2-(cyclohexylmethyl)-3-oxo-3-(o-tolyl)propanoate (5)





Methyl -2-(cyclohexylmethyl)-3-oxo-3-(m-tolyl)propanoate (6)





Methyl 2-(cyclohexylmethyl)-3-oxo-3-phenylpropanoate (7)

88.00 88.00 88.00 88.00 88.00 77.557





Methyl 2-(cyclohexylmethyl)-3-(naphthalen-2-yl)-3-oxopropanoate (8)





Methyl 3-(4-(tert-butyl)phenyl)-2-(cyclohexylmethyl)-3-oxopropanoate (9)







Methyl 2-(cyclohexylmethyl)-3-(4-methoxyphenyl)-3-oxopropanoate (10)





Methyl 2-(cyclohexylmethyl)-3-(3-methoxyphenyl)-3-oxopropanoate (11)

 $\begin{array}{c} 7.57\\ 7.55\\$





Methyl 2-(cyclohexylmethyl)-3-oxo-3-(4-(trifluoromethyl)phenyl)propanoate (12)

8.803 8.903 8.903








20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1 (ppm)



Methyl 4-(2-(cyclohexylmethyl)-3-methoxy-3-oxopropanoyl)benzoate (13)



Methyl 2-(cyclohexylmethyl)-3-(4-fluorophenyl)-3-oxopropanoate (14)

8.806 8.805 8.805 8.802 8.802 8.802 8.802 8.804 8.9049



210 200 190 110 100 f1 (ppm) 180 170 160 150 140 130 120 80 70 60 50 40 30 10 -10 90 20 0



Methyl 3-(4-chlorophenyl)-2-(cyclohexylmethyl)-3-oxopropanoate (15)

210 200 110 100 fl (ppm)

Methyl 3-(4-(chloromethyl)phenyl)-2-(cyclohexylmethyl)-3-oxopropanoate (16)

7.751 7.749 4.445 4.445 3.58 3.57.99 7.97



Methyl 2-(cyclohexylmethyl)-3-(furan-2-yl)-3-oxopropanoate (17)



Methyl 3-(benzofuran-5-yl)-2-(cyclohexylmethyl)-3-oxopropanoate (18)

8.31 8.31 8.31 8.31 8.31 8.31 8.31 8.45 7.75 8.55 8.56 8.86 8.87 8.75 8.87 7.75 8.87 7.75 8.87 7.75 8.87 7.75 8.81 8.81 8.81 8.81 8.81 8.81 8.81 8.81 8.81 8.81 8.82 8.82 8.82 8.83 8.84 8.84 8.84 8.84 8.84 8.84 8.84 8.84 8.84 8.84 8.85</l



30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 F1 (ppm)

Ethyl 2-(3-methylbenzoyl)-4-oxooctanoate (19)



Methyl 2-(cyclopentylmethyl)-3-oxo-3-(p-tolyl)propanoate (20)

$\begin{array}{c} 7.91\\ 7.791\\ 7.728\\ 7.728\\ 3.368\\ 3.368\\ 3.368\\ 3.368\\ 3.368\\ 1.77\\ 1.79\\ 1.79\\ 1.79\\ 1.79\\ 1.76\\ 1.7$



110 100 f1 (ppm) -1

Methyl 2-(bicyclo[2.2.1]heptan-2-ylmethyl)-3-oxo-3-phenylpropanoate (21)

$\begin{array}{c} 7,7,9\\ 7,$



Methyl 2-((7-oxabicyclo[2.2.1]heptan-2-yl)methyl)-3-oxo-3-phenylpropanoate (22)



110 100 f1 (ppm)

Methyl -3-oxo-3-phenyl-2-((tetrahydrofuran-2-yl)methyl)propanoate (23)

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210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 F1 (ppm)

Methyl -2-((1,4-dioxan-2-yl)methyl)-3-oxo-3-phenylpropanoate (24)

$\begin{array}{c} 88.82 \\ 88.05 \\ 88.04 \\ 88.04 \\ 88.04 \\ 88.01 \\ 88.01 \\ 88.01 \\ 88.01 \\ 88.01 \\ 88.01 \\ 88.01 \\ 88.01 \\ 88.01 \\ 88.01 \\ 77.51 \\ 77.51 \\ 77.55 \\$



f1 (ppm)

Methyl 3-oxo-2-((3-oxocyclopentyl)methyl)-3-phenylpropanoate (25)



250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -: f1 (ppm)

Ethyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (26)



220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -: f1 (ppm)

Tert-butyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (27)

$\begin{array}{c} 7.7_{-7.82}\\ 7.7_{-7.81}\\ 7.7_{-7.18}$



220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1 f1 (ppm)

Isobutyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (28)

$\begin{array}{c} 7,7,91\\ 7,7,26\\ 7,7,26\\ 7,7,26\\ 7,7,26\\ 7,7,26\\ 7,7,26\\ 7,7,26\\ 7,7,26\\ 7,7,26\\ 7,7,26\\ 7,7,26\\ 7,7,26\\ 7,7,26\\ 7$



Cyclohexyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (29)



Benzyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (30)

$\begin{array}{c} 7.3.7\\ 7.7.28\\ 7.7.28\\ 7.7.28\\ 7.7.28\\ 7.7.28\\ 7.7.28\\ 7.7.28\\ 7.7.28\\ 7.7.28\\ 7.7.28\\ 7.7.28\\ 7.7.28\\ 7.7.28\\ 7.7.28\\ 7.7.28\\ 7.7.28\\ 1.7.28\\ 1.1.28\\ 1.1.96\\ 1.1.96\\ 1.1.96\\ 1.1.96\\ 1.1.96\\ 1.1.88\\$



230 220 210 200 120 110 100 f1 (ppm) -10 150 140

Phenyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (31)

$\begin{array}{c} 7,7,7\\ 7,7,7\\ 7,7,3\\ 7,7,3\\ 7,7,2\\ 7,$



Allyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (32)

77.90 77.90 77.88 77.88 5.5.21 5.5.5.21 5.5.



2-Chloroethyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (33)

77.90 77.90 77.88 77.88 77.84 44.47 74.44 44.47 77.88 44.43 77.28 44.43 53.60 53.50 1.195 44.35 53.50 53.50 1.195 44.35 53.50 1.195 1.115 1.155



230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 f1 (ppm)

2,2,2-trifluoroethyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (34)



220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 r1 (ppm)

2-methoxyethyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (35)



240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -: f1 (ppm)

Diethyl (3-cyclohexyl-1-oxo-1-(p-tolyl)propan-2-yl)phosphonate (36)

$\begin{array}{c} 7.7_{94}\\ 7.7_{94}\\ 7.7_{22}\\ 7.7_{22}\\ 7.7_{22}\\ 7.7_{22}\\ 7.7_{22}\\ 7.7_{22}\\ 7.7_{22}\\ 7.7_{22}\\ 7.7_{22}\\ 7.7_{22}\\ 7.7_{22}\\ 7.7_{22}\\ 7.7_{22}\\ 7.7_{23}\\ 7.7_{24}\\$



110 100 f1 (ppm) 210 200 -10

(1S,2R,5S)-2-isopropyl-5-methylcyclohexyl 2-(cyclohexylmethyl)-3-oxo-3-(p-tolyl)propanoate (37)

 $\begin{array}{c} 7.79\\ 7.88\\ 7.78\\ 7.78\\ 7.78\\ 7.78\\ 7.78\\ 7.78\\ 7.78\\ 7.78\\ 7.78\\ 7.78\\ 7.78\\ 7.78\\ 7.78\\ 7.72\\ 8.8\\ 1.19\\ 8.8\\ 1.19\\ 8.8\\ 1.18\\ 8.8\\ 1.18\\ 8.8\\ 1.18\\ 8.8\\ 1.18\\ 8.8\\ 1.18\\ 8.8\\ 1.18\\ 8.8\\ 1.18\\ 8.8\\ 1.18\\ 8.8\\ 1.18\\ 1.16\\ 1.12\\ 0.09\\ 1.12\\ 0.09\\ 1.12\\ 0.09\\ 1.12\\ 0.09\\ 1.12\\ 0.09\\ 1.12\\ 0.09\\ 1.12\\ 0.09\\ 1.12\\ 0.09\\ 1.12\\ 0.09\\ 1.12\\ 0.09\\ 1.12\\ 0.09\\ 1.12\\ 0.09\\ 1.12\\ 0.09\\ 1.12\\ 0.00\\ 0.09\\ 0.00\\ 0.05\\ 0.00\\ 0.05\\ 0.00\\ 0.05\\ 0.00\\ 0.05\\ 0.00\\ 0.05\\ 0.00\\ 0.05\\ 0.00\\ 0.05\\ 0.00\\ 0.05\\ 0.00\\ 0.05\\ 0.00$





Methyl 3-oxo-3-(p-tolyl)-2-((triphenylsilyl)methyl)propanoate (38)

230 220 210 200 190 180 170 160 150 140 150 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 F1 (ppm)

Methyl 3-oxo-3-(p-tolyl)-2-((triphenylsilyl)methyl)propanoate (40)



Methyl 3-(4-fluorophenyl)-3-oxo-2-((triphenylsilyl)methyl)propanoate (39)

- 3.37 2.24 2.22 2.22 2.18 2.14 2.14 2.12 2.12 2.10





Methyl 3-(4-chlorophenyl)-3-oxo-2-((triphenylsilyl)methyl)propanoate (40)





Isobutyl 3-oxo-3-(p-tolyl)-2-((triphenylsilyl)methyl)propanoate (42)





Cyclohexyl 3-oxo-3-(p-tolyl)-2-((triphenylsilyl)methyl)propanoate (43)

77/54 77/52 77/52 77/52 77/57







Benzyl 3-oxo-3-(p-tolyl)-2-((triphenylsilyl)methyl)propanoate (44)

7.50 7.55 7.55 7.55 7.53 7.53 7.53 7.53 7.53	4.43 4.42 4.42 4.40	3.38	2.23 2.21 2.17 2.13 2.13 2.13 2.13 2.09 2.09
		1.1	





3-oxo-3-(p-tolyl)-2-((triphenylsilyl)methyl)propanoate (45)

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Methyl 2-((methyldiphenylsilyl)methyl)-3-oxo-3-(p-tolyl)propanoate (46)

7,7,65 7,69 7,63 7,53 7,53 7,53 7,53 7,55 7,55 7,55 7,5	4.32 4.31 4.30 4.29	3.49	2.38 1.92 1.87 1.76 1.74 1.74 1.72	0.55
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-195.3 - 195.3 - 171.2 - 171.2 - 171.2 - 171.2 - 171.2 - 171.2 - 135.7 - 135.1 - 135.1 - 135.1 - 135.1 - 121.2 - 121.6 - 121.2 - 21.6 - -4.1



230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 11 (ppm)