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transition metal-free C(sp³)-H selenation of β-ketosulfones

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1. General Experimental

All reagents were purchased at the highest commercial quality and used without further purification. Reactions were monitored by thin layer chromatography (TLC) using ultra violet light (UV) as the visualizing agent. Flash column chromatography was performed on silica gel (particle size 200–300 mesh) and eluted with petroleum ether/ethyl acetate. Nuclear magnetic resonance spectra (NMR) were recorded on Bruker AV-600 instruments and were calibrated using residual undeuterated solvent as an internal reference (¹H NMR: CHCl₃ 7.26 ppm, ¹³C NMR: CHCl₃ 77.16 ppm). High resolution mass spectra (HRMS) were recorded on a Q-TOF mass spectrometer (Agilent G6545B, Germany). The following abbreviations were used to indicate multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, dt = doublet of triplets, m = multiplet).

2. General procedure for preparation of β-ketosulfones compounds



Sodium sulfinates (RSO₂Na, 6.0 mmol) was added to a solution of substituted 2bromoacetophenones (5.0 mmol) in a cosolvent of DMSO and THF (20 mL, v/v = 1:1) at rt. The reaction mixture was stirred at 2 h. The reaction mixture was partitioned with CH₂Cl₂ (3 × 30 mL) and water (30 mL). The combined organic layers were washed with brine, dried, filtered, and evaporated to afford crude β -ketosulfones under reduced pressure. Crude β -ketosulfones were recrystallized from EtOAc in nearly quantitative yields. β -Ketosulfones are known compounds and the analytical data are consistent with those in previous literature ¹.

3. General procedure for preparation of Diselenides compounds



To a Schlenk tube were added arylboronic acid (0.4 mmol), selenium (1.2 mmol), AgNO₃ (0.04 mmol), and DMSO (2.0 mL). The mixture was stirred in a heating mantle preheated to 120 °C for 2 h. After cooled to room temperature, the reaction mixture was

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diluted with H_2O (10 mL), and extracted with ethyl acetate (3×10 mL). The combined organic phase was washed with water and brine (30 mL), dried over anhydrous Na_2SO_4 , and then evaporated under reduced pressure. The residue was purified by column chromatography to give the desired diselen ².

4. Optimization of reaction conditions

Table 4.1. Optimization of reaction conditions

	0 00 	PhSeSePh Cs ₂ CO ₃ DMSO 2a		O O O O O O O O O O O O O O O O O O O
Entry	Oxidant	Temperature	Time (h)	Yield
1	air	rt	4	42%
2	air	rt	6	63%
3	air	rt	8	85%
4	air	rt	10	91%
5	air	rt	12	93%
6	air	rt	14	94%
7	air	rt	24	93%
8	air	40 °C	6	85%
9	air	60 °C	2	80%
10	air	80 °C	1	78%

Reaction conditions: **1a** (0.2 mmol scale, 1 equiv), **2a** (0.8 equiv) Cs_2CO_3 (1.2 equiv), and DMSO (2 mL).

5 General procedure for the synthesis of product



A mixture of β -ketosulfones 1 (0.20 mmol), diselenides 2 (0.16 mmol), and Cs₂CO₃ (0.45 mmol) in DMSO (2.0 mL) was placed in a test tube (25 mL) equipped with a magnetic stirring bar. The reaction mixture was stirred at room temperature for 12 h. After the reaction was completed, the mixture addition of satd aq NaCl (5 mL). Further stirring was followed by extraction with ethyl acetate (3×15 mL). The organic layer was dried with anhydrous MgSO₄, concentrated in vacuo and purified by flash silica gel chromatography using petroleum ether/ethyl acetate (8:1) to give the desired prod.

6. Gram-scale experiment



A mixture of β -ketosulfones **1a** (6 mmol), diselenides **2a** (4.8 mmol), and Cs₂CO₃ (7.2 mmol) in DMSO (30.0 mL) was placed in a Schlenk tube (100 mL) equipped with a magnetic stirring bar. The reaction mixture was stirred at room temperature for 24 h. After the reaction was completed, the mixture addition of satd aq NaCl (50 mL). Further stirring was followed by extraction with ethyl acetate (3×30 mL). The organic layer was dried with anhydrous MgSO₄, concentrated in vacuo and purified by flash silica gel chromatography using petroleum ether/ethyl acetate (8:1) to afford **3a** (2.34 g, 91 %) as white solid.

7. Control experiment



^a Standard conditions: **1a** (0.2 mmol), **2a** (0.16 mmol), Cs_2CO_3 (0.24 mmol), DMSO (2 mL), at room temperature, air, and 12 h. Isolated yields. ND = not detected.

8. X-Ray crystallographic data of products 3a

J 1	1		,		
Bond precision:	C-C = 0.0242 A		Wavelength=0.71073		
Cell:	a=10.005(4)	b=11.035(4)	c=18.194(7)		
	alpha=90	beta=101.800(7)	gamma=90		
Temperature: 296 K					
	Calculated		Reported		
Volume	1966.3(13)		1966.2(13)		
Space group	P 21/n		P 1 21/n 1		
Hall group	-P 2yn		-P 2yn		
Moiety formula	C21 H18 O	3 S Se	C21 H18 O3 S Se		
Sum formula	C21 H18 O	3 S Se	C21 H18 O3 S Se		
Mr	429.37		429.37		
Dx,g cm-3	1.450		1.451		
Ζ	4		4		
Mu (mm-1)	2.033		2.033		
F000	872.0		872.0		
F000'	872.46				
h,k,lmax	13,14,23		13,14,23		
Nref	4710		4544		
Tmin,Tmax			0.569,0.746		
Tmin'					
Correction method= # Reported T Limits: Tmin=0.569 Tmax=0.746 AbsCorr = MULTI-SCAN					
Data completeness= 0.965		Theta(max)= 27.	Theta(max)= 27.928		
R(reflections)= 0.1494(3182)	wR2(reflections)= 0.4049(4544)			
S = 1.141		Npar= 237			

 Table 8.1. Crystal parameters of compound 3a (CCDC: 2288402)

The crystal of product **3a** was obtained by slow evaporation in ethanol. The single crystal X-ray analysis determined the structure of product **3a** (Figure 7.1) as expected.



Displacement ellipsoids are drawn at 50% probability level.

Figure 8.1. The crystal structure of compound 3a (CCDC: 2288402).



3a CCDC: 2288402

9. Analytical data for 3a-3v, 4a-4q, 5a

1-phenyl-2-(phenylselanyl)-2-tosylethan-1-one (3a)



White solid, (80 mg, 93%), Mp: 95-97 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.79 (d, *J* = 7.22 Hz, 2H), 7.58 (t, *J* = 7.6 Hz, 3H), 7.42 (t, *J* = 6.6 Hz, 3H), 7.37 (t, *J* = 7.22 Hz, 3H), 7.32 (d, *J* = 7.8 Hz, 2H), 7.28 (t, *J* = 7.8 Hz, 2H), 5.78 (s, 1H),

2.44 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 189.7, 145.6, 135.8, 135.2, 134.2, 134.1, 130.7, 129.7, 129.6, 129.4, 129.1, 128.9, 127.9, 68.4, 21.8. HRMS (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₁H₁₉O₃SSe: 431.0215; found: 431.0216.

2-(phenylselanyl)-1-(p-tolyl)-2-tosylethan-1-one (3b)



White solid, (75.8 mg, 85%), Mp: 119-120 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.89 (d, *J* = 7.8 Hz, 2H), 7.70 (d, *J* = 8.4 Hz, 2H), 7.60 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.37 (t, *J* = 7.8 Hz, 1H), 7.31 (d, *J* = 7.8 Hz, 2H), 7.28 (t, *J* = 7.8 Hz, 2H), 7.22 (d, *J* = 8.4 Hz,

2H), 5.76 (s, 1H), 2.43 (s, 3H), 2.40 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 189.3, 145.5, 145.5, 135.8, 134.1, 132.7, 130.7, 129.7, 129.6, 129.4, 129.3, 128.1, 68.5, 21.9, 21.9. HRMS (+ESI-TOF): m/z [M + H]⁺ calcd for C₂₂H₂₁O₃SSe: 445.0371; found: 445.0360.

1-(4-methoxyphenyl)-2-(phenylselanyl)-2-tosylethan-1-one (3c)



White solid, (82.7 mg, 90%), Mp: 81-83 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.88 (d, *J* = 7.8 Hz, 2H), 7.80 (d, *J* = 9.0 Hz, 2H), 7.60 (dd, *J* = 7.8, 1.2 Hz, 2H), 7.37 (t, *J* = 7.2 Hz, 1H), 7.31 (d, *J* = 7.8 Hz, 2H), 7.28 (t, *J* = 7.8 Hz, 2H), 6.89 (d, *J* =

9.0 Hz, 2H), 5.74 (s, 1H), 3.87 (s, 3H), 2.44 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 188.2, 164.5, 145.5, 135.7, 134.0, 131.7, 130.7, 129.6, 129.5, 129.4, 128.3, 128.1, 114.2, 68.7, 55.7, 21.9. HRMS (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₂H₂₁O₄SSe: 461.0320; found: 461.0317.

1-(4-ethoxyphenyl)-2-(phenylselanyl)-2-tosylethan-1-one (3d)



2H), 7.60 (dd, J = 8.4, 1.2 Hz, 2H), 7.36 (t, J = 7.2 Hz, 1H), 7.30 (d, J = 7.8 Hz, 2H), 7.27 (d, J = 7.2 Hz, 2H), 6.86 (d, J = 8.4 Hz, 2H), 5.74 (s, 1H), 4.09 (q, J = 7.2 Hz, 2H), 2.43 (s, 3H), 1.43 (t, J = 7.8 Hz, 3H). ¹³**C NMR** (151 MHz, CDCl₃) δ 188.1, 164.0, 145.4, 135.7, 134.0, 131.7, 130.9, 129.6, 129.5, 129.4, 128.3, 127.9, 114.6, 68.6, 64.1, 21.8, 14.7. **HRMS** (+ESI-TOF): m/z [M + H]⁺ calcd for C₂₃H₂₃O₄SSe: 475.0477; found: 475.0476.

1-(4-fluorophenyl)-2-(phenylselanyl)-2-tosylethan-1-one (3e)



White solid, (84 mg, 94%), Mp: 83-85 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.89 (d, *J* = 8.4 Hz, 2H), 7.84 (dd, *J* = 8.4, 4.8 Hz, 2H), 7.57 (d, *J* = 7.2 Hz, 2H), 7.37 (t, *J* = 7.8 Hz, 1H), 7.33 (d, *J* = 8.4 Hz, 2H), 7.28 (t, *J* = 7.8 Hz, 2H), 7.09 (t, *J* = 8.4 Hz, 2H), 7.28 (t, *J* = 7.8 Hz, 2H), 7.09 (t, *J* = 8.4 Hz, 2H), 7.28 (t, *J* = 7.8 Hz, 2H), 7.09 (t, *J* = 8.4 Hz, 2H), 7.28 (t, *J* = 7.8 Hz, 2H), 7.09 (t, *J* = 8.4 Hz, 2H), 7.09 (t, J = 8.4 Hz, 2H), 7.09 (t, J = 8.4 Hz, 14), 8.4 Hz, 14), 8.4 Hz, 14 (t, 14), 8.4 Hz, 14), 8.4 Hz, 14 (t, 14), 14 (t,

2H), 5.72 (s, 1H), 2.44 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 188.3, 167.3 (d, $J_{C-F}=$ 255.8 Hz), 145.7, 135.8, 133.9, 132.0 (d, $J_{C-F}=$ 9.5 Hz), 131.6 (d, $J_{C-F}=$ 2.6 Hz), 130.7, 129.8, 129.7, 129.5, 127.9, 116.2 (d, $J_{C-F}=$ 21.8 Hz), 68.6, 21.9. HRMS (+ESI-TOF): m/z [M + H]⁺ calcd for C₂₁H₁₈FO₃SSe: 449.0120; found: 449.0112.

1-(4-bromophenyl)-2-(phenylselanyl)-2-tosylethan-1-one (3f)



White solid, (96 mg, 94%), Mp: 93-94 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.88 (d, *J* = 8.4 Hz, 2H), 7.65 (d, *J* = 8.4 Hz, 2H), 7.56 (t, *J* = 5.4 Hz, 4H), 7.38 (t, *J* = 7.8 Hz, 1H), 7.32 (d, *J* = 7.8 Hz, 2H), 7.28 (t, *J* = 7.8 Hz, 2H), 5.69 (s, 1H), 2.44 (s, 3H). ¹³C

NMR (151 MHz, CDCl₃) δ 188.8, 145.7, 135.8, 133.9, 133.9, 132.3, 130.7, 130.5, 129.9, 129.7, 129.7, 129.5, 127.8, 68.4, 21.9. **HRMS** (+ESI-TOF): m/z [M + H]⁺ calcd for C₂₁H₁₈BrO₃SSe: 508.9319; found: 508.9309.

1-(4-iodophenyl)-2-(phenylselanyl)-2-tosylethan-1-one (3g)



White solid, (105.1 mg, 95%), Mp: 73-75 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.88 (d, *J* =7.8 Hz, 2H), 7.78 (d, *J* = 8.4 Hz, 2H), 7.57 (dd, *J* = 7.8, 1.2Hz, 2H), 7.49 (d, *J* = 9.0 Hz, 2H), 7.38 (t, *J* = 7.8 Hz, 1H), 7.32 (d, *J* = 7.8 Hz, 2H), 7.28 (t, *J* = 7.8 Hz, 2H),

5.68 (s, 1H), 2.44 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 189.1, 145.7, 138.3, 135.8,

134.4, 133.9, 130.7, 130.3, 129.9, 129.7, 129.5, 127.8, 102.7, 68.4, 21.9. **HRMS** (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₁H₁₈IO₃SSe: 556.9181; found: 556.9172.

1-(3,4-dichlorophenyl)-2-(phenylselanyl)-2-tosylethan-1-one (3h)



Yellow solid, (89.7 mg, 90%), Mp: 115-116 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.89 (d, *J* = 8.4 Hz, 2H), 7.80 (d, *J* = 2.4 Hz, 1H), 7.62 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.57 (d, *J* = 6.6 Hz, 2H), 7.50 (d, *J* = 8.4 Hz, 1H), 7.40 (t, *J* = 7.2 Hz, 1H), 7.34 (d, *J* = 8.4 Hz,

2H), 7.30 (t, J = 7.2 Hz, 2H), 5.62 (s, 1H), 2.45 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 187.6, 145.9, 139.0, 135.9, 134.7, 133.9, 133.7, 131.0, 130.9, 130.7, 130.1, 129.8, 129.6, 128.0, 127.6, 68.5, 21.9, **HRMS** (+ESI-TOF): m/z [M + H]⁺ calcd for $C_{21}H_{17}Cl_2O_3SSe: 498.9435$; found: 498.9426.

4-(2-(phenylselanyl)-2-tosylacetyl)benzonitrile(3i)



White solid, (89.3 mg, 98%), Mp: 111-112 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.89 (t, *J* = 8.4 Hz, 4H), 7.71 (d, *J* = 8.4 Hz, 2H), 7.52 (d, *J* = 7.2 Hz, 2H), 7.38 (t, *J* = 7.8 Hz, 1H), 7.34 (d, *J* = 7.8 Hz, 2H), 7.28 (t, *J* = 7.8 Hz, 2H), 5.70 (s, 1H), 2.45 (s,

3H). ¹³C NMR (151 MHz, CDCl₃) δ 188.5, 146.0, 138.2, 135.8, 133.8, 132.7, 130.6, 130.1, 129.8, 129.6, 129.4, 127.4, 117.7, 117.3, 68.5, 21.9. HRMS (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₂H₁₈NO₃SSe: 456.0167; found: 456.0168.

1-(4-nitrophenyl)-2-(phenylselanyl)-2-tosylethan-1-one (3j)



White solid, (86.0 mg, 91%), Mp: 118-120 °C; ¹H NMR (600 MHz, CDCl₃) δ 8.25 (d, *J* = 8.4 Hz, 2H), 7.95 (d, *J* = 8.4 Hz, 2H), 7.91 (d, *J* = 7.8 Hz, 2H), 7.53 (d, *J* = 7.2 Hz, 2H), 7.39 (t, *J* = 7.8Hz, 1H), 7.35 (d, *J* = 7.8 Hz, 2H), 7.28 (t, *J* = 7.2Hz, 2H), 7.28 (t, *J* = 7.2Hz, 2H), 7.28 (t, *J* = 7.2Hz), 7.28 (t, J = 7.2Hz), 7.28 (t, J

2H), 5.72 (s, 1H), 2.46 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 188.3, 150.8, 146.0, 139.7, 135.8, 133.8, 130.7, 130.2, 130.1, 129.8, 129.7, 127.4, 124.0, 68.7, 21.90. **HRMS** (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₁H₁₈NO₅SSe: 476.0065; found: 476.0063.

1-(4-(methylsulfonyl)phenyl)-2-(phenylselanyl)-2-tosylethan-1-one (3k)



White solid, (89.3 mg, 88%), Mp: 149-150 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.98 (q, *J* = 8.4Hz, 4H), 7.90 (d, *J* = 8.4 Hz, 2H), 7.53 (d, *J* = 7.2 Hz, 2H), 7.38 (t, *J* = 7.8 Hz, 1H), 7.34 (d, *J* = 8.4 Hz, 2H), 7.28 (t, *J* = 7.8 Hz, 2H), 5.73 (s, 1H), 3.07 (s,

3H), 2.45 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 188.6, 146.0, 145.0, 139.2, 135.7, 133.8, 130.6, 130.1, 129.9, 129.8, 129.6, 128.0, 127.5, 68.6, 44.4, 21.9. HRMS (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₂H₂₁O₅S₂Se: 508.9990; found: 508.9982.

1-(phenylselanyl)-1-tosylpropan-2-one (3l)



White solid, (70.9 mg, 96%), Mp: 92-93 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.83 (d, *J* = 8.4 Hz, 2H), 7.48 (d, *J* = 7.2 Hz, 2H), 7.35 – 7.33 (m, 3H), 7.27 – 7.24 (m, 3H), 4.77 (s, 1H), 2.46 (s, 3H), 2.45 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 196.7, 145.8, 135.2, 134.1, 129.8,

129.8, 129.7, 129.6, 127.5, 73.6, 29.0, 21.9. **HRMS** (+ESI-TOF): m/z [M + H]⁺ calcd for C₁₆H₁₇O₃SSe: 369.0058; found: 369.0053.

1-phenyl-2-(phenylselanyl)-2-(phenylsulfonyl)ethan-1-one (3m)



White solid, (81.8 mg, 98%), Mp: 156-157 °C; ¹H NMR (600 MHz, CDCl₃) δ 8.03 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.77 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.63 (t, *J* = 7.2 Hz, 1H), 7.58 – 7.51 (m, 5H), 7.40 (t, *J* = 8.4 Hz, 2H), 7.36 (t, *J* = 7.8 Hz, 1H), 7.26 (t, *J* = 7.8Hz, 2H), 5.79 (s, 1H).

¹³**C NMR** (151 MHz, CDCl₃) δ 189.5, 137.1, 135.9, 135.1, 134.4, 134.3, 130.7, 129.8, 129.6, 129.1, 128.9, 128.8, 127.8, 68.2. **HRMS** (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₀H₁₇O₃SSe: 417.0058; found: 417.0057.

2-((4-(tert-butyl)phenyl)sulfonyl)-1-phenyl-2-(phenylselanyl)ethan-1-one(3n)



White solid, (91.5 mg, 94%), Mp: 120-121 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.97 (d, *J* = 8.4 Hz, 2H), 7.80 (d, *J* = 7.8 Hz, 2H), 7.59 (t, *J* = 7.2 Hz, 1H), 7.54 (dd, *J* = 13.2, 8.4 Hz, 4H), 7.43 (t, *J* = 7.8 Hz, 2H), 7.38 (t, *J* = 7.8 Hz, 1H), 7.27 (t, *J* = 7.2 Hz, 2H),

5.80 (s, 1H), 1.36 (s, 9H). ¹³C NMR (151 MHz, CDCl₃) δ 189.7, 158.4, 135.8, 135.3, 134.2, 134.1, 130.0, 129.7, 129.7, 129.0, 128.9, 127.9, 125.8, 68.4, 35.43, 31.2. HRMS (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₄H₂₅O₃SSe: 473.0684; found: 473.0684.

2-(naphthalen-2-ylsulfonyl)-1-phenyl-2-(phenylselanyl)ethan-1-one(3o)



White solid, (92.4 mg, 96%), Mp: 125-126 °C; ¹H NMR (600 MHz, CDCl₃) δ 8.61 (s, 1H), 7.99 (d, J = 9.0 Hz, 2H), 7.93 (d, J = 9.0 Hz, 1H), 7.90 (d, J = 8.4 Hz, 1H), 7.79 (d, J = 7.2 Hz, 2H), 7.67 (t, J = 7.8 Hz, 1H), 7.61 (t, J = 8.4 Hz, 1H), 7.55 (t, J = 7.2

Hz, 3H), 7.39 (t, J = 7.8 Hz, 2H), 7.33 (t, J = 7.2 Hz, 1H), 7.22 (t, J = 7.8 Hz, 2H), 5.89 (s, 1H). ¹³C NMR (151 MHz, CDCl₃) δ 189.5, 135.8, 135.7, 135.1, 134.4, 134.2, 132.9, 131.9, 129.8, 129.7, 129.6, 129.5, 129.0, 128.9, 128.8, 128.0, 127.8, 127.6, 124.9, 68.5. HRMS (+ESI-TOF): m/z [M + H]⁺ calcd for C₂₉H₁₉O₃SSe: 467.0215; found: 467.0214.

2-((4-chlorophenyl)sulfonyl)-1-phenyl-2-(phenylselanyl)ethan-1-one (3p)



White solid, (85.0 mg, 94%), Mp: 108-110 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.98 (d, J = 8.4 Hz, 2H), 7.80 (dd, J = 8.4, 1.2 Hz, 2H), 7.59 (dd, J = 16.2, 7.8 Hz, 3H), 7.51 (d, J = 8.4 Hz, 2H), 7.44 (t, J = 8.4 Hz, 2H), 7.39 (t, J = 7.8 Hz, 1H), 7.30 (t

2H), 5.82 (s, 1H). ¹³C NMR (151 MHz, CDCl₃) δ 189.5, 141.3, 135.8, 135.4, 135.0, 134.5, 132.3, 130.0, 129.7, 129.0, 127.6, 68.1. HRMS (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₀H₁₆ClO₃SSe: 450.9668; found:450.9666.

2-((4-nitrophenyl)sulfonyl)-1-phenyl-2-(phenylselanyl)ethan-1-one (3q)



White solid, (87.1 mg, 94%), Mp: 154-155 °C; ¹H NMR (600 MHz, CDCl₃) δ 8.37 (d, *J* = 8.4 Hz, 2H), 8.27 (d, *J* = 8.4 Hz, 2H), 7.80 (d, *J* = 7.2 Hz, 2H), 7.61 (d, *J* = 7.8 Hz, 3H), 7.45 (t, *J* = 7.2 Hz, 2H), 7.41 (t, *J* = 7.2 Hz, 1H), 7.32 (t, *J* = 7.8 Hz, 2H), 5.89

(s, 1H). ¹³C NMR (151 MHz, CDCl₃) δ 189.3, 151.2, 142.6, 135.9, 134.8, 134.7, 132.4, 130.3, 129.8, 129.1, 129.0, 127.3, 123.7, 67.9. **HRMS** (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₁H₁₉O₅SSe: 477.0144; found: 477.0173.

1-phenyl-2-(phenylselanyl)-2-(pyridin-3-ylsulfonyl)ethan-1-one(3r)



Yellow solid, (78.6 mg, 94%), Mp: 140-141 °C; ¹H NMR (600 MHz, CDCl₃) δ 9.26 (d, J = 1.8 Hz, 1H), 8.86 (dd, J = 4.8, 1.8 Hz, 1H), 8.36 – 8.34 (m, 1H), 7.81 (d, J = 7.2 Hz, 2H), 7.61 (t, J = 7.8 Hz,

1H), 7.59 (dd, J = 8.4, 1.2 Hz, 2H), 7.50 (dd, J = 7.8, 4.8 Hz, 1H), 7.45 (t, J = 7.8 Hz, 2H), 7.40 (t, J = 7.2 Hz, 1H), 7.30 (t, J = 7.8 Hz, 2H), 5.86 (s, 1H).¹³C NMR (151 MHz, CDCl₃) δ 189.5, 154.6, 151.5, 139.0, 135.9, 134.8, 134.6, 133.6, 130.1, 129.8, 129.1, 129.0, 127.4, 123.4, 67.9. HRMS (+ESI-TOF): m/z [M + H]⁺ calcd for C₁₉H₁₆O₃SSe: 418.0011; found: 418.0001.

1-phenyl-2-(phenylselanyl)-2-(thiophen-2-ylsulfonyl)ethan-1-one (3s)



White solid, (83.1 mg, 98%), Mp: 160-161 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.85 – 7.76 (m, 4H), 7.59 (d, J = 7.8 Hz, 3H), 7.43 (t, J = 7.8 Hz, 2H), 7.39 (t, J = 7.2 Hz, 1H), 7.29 (t, J = 7.2 Hz, 2H), 7.14 (t, J = 4.2 Hz, 2H), 5.87 (s, 1H). ¹³C NMR (151 MHz, CDCl₃) δ 189.4,

137.5, 136.0, 135.8, 135.0, 134.4, 130.0, 129.6, 129.0, 127.7, 127.6, 68.6. **HRMS** (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₁₈H₁₅O₃SSe: 422.9622; found: 422.9612.

2-(methylsulfonyl)-1-phenyl-2-(phenylselanyl)ethan-1-one (3t)

White solid, (69.8 mg, 98%), Mp: 126-127 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.88 (dd, J = 8.4, 1.2 Hz, 2H), 7.73 (dd, J = 8.4, 1.2 Hz, 2H), 7.64 (t, J = 7.8 Hz, 1H), 7.48 (t, J = 7.8 Hz, 2H), 7.43 (t, J = 7.8 Hz, 1H), 7.35 (t, J = 7.8 Hz, 2H), 5.60 (s, 1H), 3.35 (s, 3H). ¹³C NMR (151 MHz,

CDCl₃) δ 190.6, 136.0, 134.8, 134.7, 130.2, 129.8, 129.1, 127.4, 65.6, 38.8. **HRMS** (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₁₅H₁₅O₃SSe: 354.9902; found: 354.9894.

2-(cyclopropylsulfonyl)-1-phenyl-2-(phenylselanyl)ethan-1-one(3u)



1.50 –1.45 (m, 1H), 1.30 –1.26 (m, 1H), 1.18 – 1.14 (m, 1H), 1.07–1.2 (m, 1H). ¹³C **NMR** (151 MHz, CDCl₃) δ 190.2, 135.9, 135.0, 134.4, 129.9, 129.7, 129.1, 129.0, 127.8, 66.3, 29.0, 6.9, 5.1. **HRMS** (+ESI-TOF): m/z [M + H]⁺ calcd for C₁₇H₁₇O₃SSe: 381.0058; found: 381.0058.

2-(benzylsulfonyl)-1-phenyl-2-(phenylselanyl)ethan-1-one (3v)



White solid, (82.6 mg, 96%), Mp: 108-109 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.72 (d, *J* = 7.2 Hz, 2H), 7.66 (d, *J* = 7.2 Hz, 2H), 7.61 (t, *J* = 7.8 Hz, 1H), 7.44 – 7.38 (m, 5H), 7.35 (t, *J* = 7.8 Hz, 1H), 7.32 – 7.29 (m, 4H), 5.54 (s, 1H), 4.83 (d, *J* = 13.2 Hz, 1H),

4.67 (d, J = 13.2 Hz, 1H). ¹³C NMR (151 MHz, CDCl₃) δ 190.2, 136.3, 134.8, 134.5, 131.5, 130.1, 129.7, 129.2, 129.1, 129.0, 128.9, 127.1, 126.8, 64.5, 57.6. HRMS (+ESI-TOF): m/z [M + H]⁺ calcd for C₂₁H₁₉O₃SSe: 431.0215; found: 431.0216.

1-phenyl-2-(p-tolylselanyl)-2-tosylethan-1-one (4a)



White solid, (79.1 mg, 89%), Mp: 145-147 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.79 (d, *J* = 7.2 Hz, 2H), 7.57 (t, *J* = 7.2 Hz, 1H), 7.45 (d, *J* = 7.8 Hz, 2H), 7.41 (t, *J* = 7.8 Hz, 2H), 7.32 (d, *J* = 7.8 Hz, 2H), 7.08 (d, *J* = 7.8 Hz, 2H) 5.73 (s,

1H), 2.43 (s, 3H), 2.39 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 189.7, 145.5. 140.2, 136.1, 135.2, 134.2, 134.1, 130.7, 130.4, 129.4, 129.0, 128.9, 124.3, 68.4, 21.8, 21.34. HRMS (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₂H₂₁O₃SSe: 445.0371; found: 445.0373.

2-(naphthalen-2-ylselanyl)-1-phenyl-2-tosylethan-1-one (4b)



Colorless oil, (79.3 mg, 83%); ¹H NMR (600 MHz, CDCl₃) δ 7.99 (s, 1H), 7.93 (d, *J* = 8.4 Hz, 2H), 7.82 (d, *J* = 7.2 Hz, 1H), 7.80 (d, *J* = 7.2 Hz, 2H), 7.74 (d, *J* = 9 Hz, 1H), 7.71 (d, *J* = 9.0 Hz, 1H), 7.62 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.57 (t, *J* = 7.2 Hz, 1H), 7.54-7.50

(m, 2H), 7.40 (t, J = 7.2Hz, 2H), 7.31 (d, J = 8.4 Hz, 2H), 5.86 (s, 1H), 2.42 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 189.7, 145.6, 135.8, 135.2, 134.2, 134.1, 133.7, 133.4, 131.8, 130.7, 129.5, 129.2, 129.1, 128.0, 127.9, 127.4, 126.9, 125.2, 68.4, 21.8. HRMS (+ESI-TOF): m/z [M + H]⁺ calcd for C₂₅H₂₁O₃SSe: 481.0371; found: 481.0367.

2-(benzo[d][1,3]dioxol-5-ylselanyl)-1-phenyl-2-tosylethan-1-one (4c)



White solid, (75.9 mg, 80%), Mp: 120-121 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.90 (d, J = 7.8 Hz, 2H), 7.80 (d, J = 7.8 Hz, 2H), 7.58 (t, J = 7.2 Hz, 1H), 7.43 (t, J = 7.8 Hz, 2H), 7.32 (d, J = 8.4 Hz, 2H), 7.03 – 7.00 (m, 2H), 6.68 (d, J = 7.8 Hz, 2H), 5.98 (s, 2H),

5.71 (s, 1H), 2.44 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 189.6, 149.5, 148.3, 145.6,

135.3, 134.2, 134.1, 131.1, 130.7, 129.5, 129.0, 128.9, 118.9, 116.5, 109.2, 101.7, 68.6, 21.8. **HRMS** (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₂H₁₉O₅SSe: 475.0113; found: 475.0113.

2-([1,1'-biphenyl]-4-ylselanyl)-1-phenyl-2-tosylethan-1-one (4d)



White solid, (94.1 mg, 93%), Mp: 141-142 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.92 (d, J = 8.4 Hz, 2H), 7.83 (d, J = 8.4 Hz, 2H), 7.66 (d, J = 7.2 Hz, 2H), 7.60 – 7.57 (m, 3H), 7.50 (d, J = 8.4 Hz, 2H), 7.47 – 7.42 (m, 4H), 7.39 (t, J = 8.4 Hz, 1H), 7.33 (d, J = 7.8 Hz, 2H), 5.82 (s, 1H), 2.44 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ

189.7, 145.6, 142.7, 140.0, 136.3, 135.2, 134.3, 134.1, 130.7, 129.5, 129.1, 129.0, 128.9, 128.2, 128.1, 127.2, 126.8, 68.5, 21.9. **HRMS** (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₇H₂₃O₃SSe: 507.0528; found: 507.0526.

2-(mesitylselanyl)-1-phenyl-2-tosylethan-1-one (4e)



White solid, (57.2 mg, 61%), Mp: 125-126 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.83 (d, J = 7.8 Hz, 2H), 7.73 (d, J = 7.2 Hz, 2H), 7.55 (t, J = 7.2 Hz, 1H), 7.36 (t, J = 7.8 Hz, 2H), 7.29 (d, J = 8.4 Hz, 2H), 6.89 (s, 2H), 5.50 (s, 1H), 2.42 (s, 3H), 2.38 (s, 6H),

2.26 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 191.2, 145.5, 144.0, 140.4, 135.5, 134.2, 134.1, 130.3, 129.5, 129.2, 129.1, 128.8, 127.1, 68.0, 24.5, 21.8, 21,2. HRMS (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₄H₂₅O₃SSe: 473.0684; found: 473.0685.

2-((4-fluorophenyl)selanyl)-1-phenyl-2-tosylethan-1-one (4f)



White solid, (109.8 mg, 93%), Mp: 121-122 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.88 (d, J = 7.8 Hz, 2H), 7.78 (dd, J = 8.4, 1.2 Hz, 2H), 7.60 – 7.55 (m, 3H), 7.42 (t, J = 8.4 Hz, 2H), 7.32 (d, J = 7.8 Hz, 2H), 6.96 (t, J = 8.4 Hz, 2H), 5.75 (s, 1H), 2.43 (s, 3H). ¹³C

NMR (151 MHz, CDCl₃) δ 189.4, 164.8 (d, J_{C-F} = 249.6 Hz), 145.6, 138.7 (d, J_{C-F} = 8.52 Hz), 135.2, 134.3, 134.0, 130.6, 129.5, 129.0, 128.9, 122.3 (d, J_{C-F} = 3.3 Hz), 116.9 (d, J_{C-F} = 21.6 Hz), 68.2, 21.9. **HRMS** (+ESI-TOF): m/z [M + H]⁺ calcd for C₂₁H₁₈FO₃SSe: 449.0120; found: 449.0111.

2-((4-bromophenyl)selanyl)-1-phenyl-2-tosylethan-1-one (4g)

White solid, (88.0 mg, 87%), Mp: 138-139 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.87 (d, J = 8.4 Hz, 2H), 7.79 (d, J = 7.2 Hz, 2H), 7.59 (t, J = 7.2 Hz, 2H), 7.42 (dt, J = 25.2, 8.4 Hz, 6H), 7.31 (d, J = 8.4 Hz, 2H), 5.77 (s, 1H), 2.43 (s, 3H). ¹³C NMR (151 MHz,

CDCl₃) δ 189.4, 145.7, 137.6, 135.1, 134.4, 133.9, 132.7, 130.6, 129.5, 129.1, 129.0, 126.4, 124.8, 68.2, 21.9. **HRMS** (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₁H₁₈BrO₃SSe: 508.9320; found: 508.9318.

2-((4-iodophenyl)selanyl)-1-phenyl-2-tosylethan-1-one (4h)



White solid, (94.5 mg, 85%), Mp: 152-154 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.86 (d, *J* = 7.8 Hz, 2H), 7.79 (dd, *J* = 8.4, 0.6 Hz, 2H), 7.59 (t, *J* = 7.8 Hz, 3H), 7.43 (dd, *J* = 8.1, 7.8 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 4H), 5.77 (s, 1H), 2.43 (s, 3H). ¹³C NMR (151 MHz,

CDCl₃) δ 189.4, 145.7, 138.7, 137.5, 135.1, 134.4, 133.9, 130.6, 129.5, 129.1, 129.0, 127.4, 96.6, 68.2, 21.87. **HRMS** (+ESI-TOF): m/z [M + H]⁺ calcd for C₂₁H₁₈IO₃SSe: 556.9181; found: 556.9179.

2-((4-chlorophenyl)selanyl)-1-phenyl-2-tosylethan-1-one (4i)



White solid, (81.8 mg, 88%), Mp: 119-121 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.87 (d, *J* = 8.4 Hz, 2H), 7.79 (d, *J* = 7.8 Hz, 2H), 7.59 (t, *J* = 7.2 Hz, 1H), 7.52 (d, *J* = 8.4 Hz, 2H), 7.43 (t, *J* = 7.8 Hz, 2H), 7.31 (d, *J* = 7.8 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 5.76 (s,

1H), 2.43 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) 189.4, 145.7, 137.4, 136.5, 135.1, 134.4, 133.9, 130.6, 129.8, 129.5, 129.0, 128.9, 125.7, 68.2, 21.9. HRMS (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₁H₁₈ClO₃SSe: 462.9833; found: 462.9829.

2-((3-chlorophenyl)selanyl)-1-phenyl-2-tosylethan-1-one (4j)



White solid, (80.3 mg, 87%), Mp: 103-104 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.89 (d, J = 7.8 Hz, 2H), 7.82 (d, J = 7.8 Hz, 2H), 7.60 (t, J = 7.2 Hz, 1H), 7.45 (t, J = 7.2 Hz, 3H), 7.37 – 7.32 (m, 4H), 7.20 (t, J = 7.8 Hz, 1H), 5.80 (s, 1H), 2.45 (s, 3H). ¹³C NMR

(151 MHz, CDCl₃) δ 189.4, 145.9, 135.3, 135.1, 134.9, 134.4, 133.8, 133.7, 130.7,

130.6, 130.0, 129.6, 129.1, 129.0, 128.9, 68.3, 21.9. **HRMS** (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₁H₁₈ClO₃SSe: 464.9825; found: 464.9823.

2-((2-chlorophenyl)selanyl)-1-phenyl-2-tosylethan-1-one (4k)



White solid, (78.6 mg, 85%), Mp: 100-102°C; ¹H NMR (600 MHz, CDCl₃) δ 7.91 (d, *J* = 7.8 Hz, 2H), 7.81 (d, *J* = 8.4Hz, 3H), 7.60 (t, *J* = 7.2 Hz, 1H), 7.45 (t, *J* = 8.4 Hz, 2H), 7.40 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.31 – 7.26 (m, 3H), 7.22 (td, *J* = 9.0, 1.8 Hz, 1H),

6.06 (s, 1H), 2.43 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 190.2, 145.8, 137.7, 136.6, 135.3, 134.4, 133.4,130.7, 130.6, 130.0, 129.5, 129.4, 128.9, 128.5, 127.9, 67.7, 21.9. **HRMS** (+ESI-TOF): m/z [M + H]⁺ calcd for C₂₁H₁₈ClO₃SSe: 464.9825; found: 464.9824.

1-phenyl-2-tosyl-2-((4-(trifluoromethyl)phenyl)selanyl)ethan-1-one (41)



White solid, (82.6 mg, 83%), Mp: 123-124 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.85 (d, *J* = 8.4 Hz, 2H), 7.81 (dd, *J* = 7.8, 0.6 Hz, 2H), 7.74 (d, *J* = 7.8 Hz, 2H), 7.60 (t, *J* = 7.2 Hz, 1H), 7.52 (d, *J* = 7.8 Hz, 2H), 7.44 (t, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 7.8 Hz, 2H), 5.86

(s, 1H), 2.43 (s, 3H). ¹³**C NMR** (151 MHz, CDCl₃) δ 189.4, 145.9, 135.6, 135.0, 134.5, 133.6, 132.4, 131.7 (q, J_{C-F} = 32.6 Hz), 130.6, 129.6, 129.1, 129.0, 126.3 (q, J_{C-F} = 4.0 Hz), 124.8 (q, J_{C-F} = 270.9 Hz), 68.2, 21.8. HRMS (+ESI-TOF): m/z [M + H]⁺ calcd for C₂₂H₁₈F₃O₃SSe: 499.0089; found: 499.0090.

4-((2-oxo-2-phenyl-1-tosylethyl)selanyl)benzonitrile (4m)



for C₂₂H₁₈NO₃SSe: 456.0167; found: 456.0164.

2-((3-nitrophenyl)selanyl)-1-phenyl-2-tosylethan-1-one (4n)



White solid, (87.4 mg, 89%), Mp: 147-149 °C; ¹H NMR (600 MHz, CDCl₃) δ 8.26 (t, *J* = 1.8 Hz, 1H), 8.20 (d, *J* = 8.4 Hz, 1H), 7.98 (d, *J* = 7.2 Hz, 1H), 7.84 (dd, *J* = 16.2, 8.4 Hz, 4H), 7.62 (t, *J* = 7.8 Hz, 1H), 7.49 – 7.44 (m, 3H), 7.32 (d, *J* = 7.8 Hz, 2H), 5.90

(s, 1H), 2.43 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 189.1, 148.2, 146.1, 142.0, 134.9, 134.7, 133.5, 130.6, 130.4, 130.3, 130.0, 129.6, 129.1, 128.8, 124.6, 68.1, 21.9. HRMS (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₁H₁₈NO₅SSe: 476.0065; found: 476.0058.

2-(methylselanyl)-1-phenyl-2-tosylethan-1-one (40)

White solid, (55.3 mg, 75%), Mp: 118-119 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.91 – 7.89 (m, 4H), 7.59 (t, *J* = 7.8 Hz, 1H), 7.45 (t, *J* = 7.8 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 5.63 (s, 1H), 2.43 (s,

3H), 2.16 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 188.8, 145.5, 135.1, 134.2, 134.1, 130.5, 129.5, 130.0, 128.8, 61.8, 21.9, 7.2. HRMS (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₁₆H₁₇O₃SSe: 369.0058; found: 369.0061.

2-(benzylselanyl)-1-phenyl-2-tosylethan-1-one (4p)



White solid, (69.0 mg, 78%), Mp: 127-128 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.48 (t, *J* = 7.2 Hz, 1H), 7.39 -7.36 (m, 7.1 Hz, 4H), 7.34 -7.30 (m, 5H), 7.25 (t, *J* = 7.8 Hz, 2H), 5.47 (s, 1H), 4.40 (d, *J* = 11.4 Hz, 1H), 3.96 (d, *J* = 11.4 Hz, 2H), 5.47 (s, 1H), 4.40 (d, *J* = 11.4 Hz, 1H), 3.96 (d, *J* = 11.4 Hz, 2H), 5.47 (s, 1H), 4.40 (d, *J* = 11.4 Hz, 1H), 3.96 (d, *J* = 11.4 Hz, 2H), 5.47 (s, 1H), 4.40 (d, *J* = 11.4 Hz, 1H), 3.96 (d, *J* = 11.4 Hz, 2H), 5.47 (s, 1H), 4.40 (d, *J* = 11.4 Hz, 1H), 3.96 (d, *J* = 11.4 Hz, 2H), 5.47 (s, 1H), 4.40 (d, *J* = 11.4 Hz, 1H), 3.96 (d, *J* = 11.4 Hz, 2H), 5.47 (s, 1H), 4.40 (d, *J* = 11.4 Hz, 1H), 3.96 (d, *J* = 11.4 Hz, 2H), 5.47 (s, 1H), 4.40 (d, *J* = 11.4 Hz, 1H), 3.96 (d, *J* = 11.4 Hz, 2H), 5.47 (s, 1H), 4.40 (d, *J* = 11.4 Hz, 1H), 3.96 (d, *J* = 11.4 Hz, 1H), 5.47 (s, 1H),

1H), 2.42 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 189.64, 145.5, 136.8, 134.8, 134.1, 134.0, 130.8, 130.0, 129.3, 129.1, 128.7, 128.6, 127.8, 61.8, 30.9, 21.9. **HRMS** (+ESI-TOF): *m/z* [M + H]⁺ calcd for C₂₂H₂₁O₃SSe: 445.0371; found: 445.0369.

1-phenyl-2-(phenylthio)-2-tosylethan-1-one (4q)³



White solid, (72.6 mg, 95%), m.p.: 85-87 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.88 (t, J = 8.4 Hz, 4H), 7.61 (t, J = 7.2 Hz, 1H), 7.53 (d, J = 6.6 Hz, 2H), 7.46 (t, J = 7.8 Hz, 2H), 7.36 – 7.30 (m, 5H), 5.80 (s, 1H), 2.45 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 189.6, 145.8,

135.3, 134.4, 133.6, 133.3, 132.4, 130.9, 129.6, 126.5, 129.4, 129.3, 129.0, 77.4, 77.2, 77.0, 75.7, 21.9.

3-(3,5-di-tert-butyl-4-hydroxyphenyl)-1-phenyl-2-tosylpropan 1-one (5a) White solid, Mp: 166-167 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.74

(d, J = 8.4 Hz, 2H), 7.68 (dd, J = 8.4, 0.6 Hz, 2H), 7.48 (t, J = 7.2 Hz, 1H), 7.35 – 7.32 (m, 4H), 6.78 (s, 2H), 5.28 (dd, J = 12.0, 3.0

Hz, 1H), 4.98 (s, 1H), 3.43 (dd, J = 13.2, 3.0 Hz, 1H), 3.24 (dd, J = 13.2, 12.0 Hz, 1H), 2.45 (s, 3H), 1.25 (s, 18H). ¹³**C NMR** (151 MHz, CDCl₃) δ 193.5, 152.8, 145.5, 137.6, 136.3, 133.9, 133.7, 130.1, 129.7, 128.8, 128.6, 126.3, 125.4, 71.7, 34.8, 34.3, 30.2, 21.9. **HRMS** (+ESI-TOF): m/z [M + H]⁺ calcd for C₃₀H₃₆NaO₄S: 515.2227; found: 515.2224.

10. References

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11. Spectra Data





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100 90 f1 (ppm)











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¹H NMR of 3k





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





¹H NMR of 3n



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¹H NMR of **3p**



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1 H NMR of **3**q



¹³C NMR of **3**q

















¹³C NMR of **3u**















¹H NMR of 4c









100 90 f1 (ppm) 1 H NMR of **4**f



















f1 (ppm)



附 录



附 录









