# **Supporting Information for**

# **Efficient Preparation of Unsymmetrical Alkyl-Aryl Tellurides by Nickel-catalyzed Reductive Coupling Strategy**

Fei Wang<sup>a</sup>, Ying Chen<sup>a</sup>, Lutz Ackermann<sup>b,c,\*</sup>, Shun-Yi Wang<sup>a,\*</sup>

<sup>a</sup>Key Laboratory of Organic Synthesis of Jiangsu Province, College of Chemistry, Chemical Engineering and Materials Science, Soochow University, 199 Ren-Ai Road, Suzhou, Jiangsu 215123 (China). Correspondence and requests for materials should be addressed to S.Y.W. (<u>shunyi@suda.edu.cn</u>).

<sup>b</sup>Institut für Organische und Biomolekulare Chemie Georg-August-Universität Göttingen Tammannstraße 2, 37077 Göttingen (Germany). E-mail: <u>Lutz.Ackermann@chemie.uni-goettingen.de</u>.

<sup>c</sup>Prof. Dr. L. Ackermann Wöhler Research Institute for Sustainable Chemistry Georg-August-Universität Göttingen Tammannstraße 2, 37077 Göttingen (Germany)

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# I. General Information

Unless otherwise noted, all commercially available compounds were used as provided without further purification. Solvents for chromatography were analytical grade and used without further purification. Anhydrous DMF, was purchased from Beijing InnoChem Science & Technology Co., Ltd. Analytical thin-layer chromatography (TLC) was performed on silica gel, visualized by irradiation with UV light. For column chromatography, 300-400 mesh silica gel was used. <sup>1</sup>H-NMR and <sup>13</sup>C-NMR were recorded on a BRUKER 400 MHz spectrometer in CDCl<sub>3</sub>. Chemical shifts ( $\delta$ ) were reported referenced to an internal tetramethylsilane standard or the CDCl<sub>3</sub> residual peak ( $\delta$  7.26) for <sup>1</sup>H NMR. Chemical shifts of <sup>13</sup>C NMR are reported relative to CDCl<sub>3</sub> ( $\delta$  77.16). Data are reported in the following order: chemical shift ( $\delta$ ) in ppm; multiplicities are indicated s (singlet), bs (broad singlet), d (doublet), t (triplet), m (multiplet); coupling constants (J) are in Hertz (Hz). For the NMR data of the products, these peaks multplets: 2.91 - 2.75 (m, 2H), 2.21 - 2.10 (m, 2H), it is because of Te-Hcoupling. IR spectra were recorded on a BRUKER VERTEX 70 spectrophotometer and are reported in terms of frequency of absorption (cm<sup>-1</sup>). HRMS spectra were obtained by using BRUKER micrOTOF-Q III instrument with ESI source. The starting materials were isolated by SepaBean machine Flash Chromatography, which purchased from Santai Technologies Inc.

# **II. Synthesis of Substrates**

## General procedure for the synthesis of symmetrical aryl-aryl tellurides

$$(2a-2k, 2m, 2n)$$
.<sup>1</sup>

$$R + Te \xrightarrow{KOH, DMSO, N_2} R + Te \xrightarrow{Te} R$$

A sealed tube containing a magnetic stirring bar was charged with aryl iodide (10.0 mmol), Te<sup>0</sup> (10.0 mmol), KOH (20.0 mmol) and DMSO (15 mL) under nitrogen. The reaction mixture was heated in an oil bath at 110 °C and stirred at this temperature for 24 h. The progress of the reaction was monitored by TLC via syringe. After the reaction was complete, the reaction mixture was allowed to cool, and treated with CH<sub>2</sub>Cl<sub>2</sub> and H<sub>2</sub>O. The organic layer was washed with sat. NH<sub>4</sub>Cl and brine solution, dried with Na<sub>2</sub>SO<sub>4</sub> and concentrated under vacuum. The residue was purified by column chromatography on silica gel with an eluent consisting of hexanes and ethyl acetate to affording the corresponding diaryl tellurides in good yields.

## General Procedure for the synthesis of aryl telluride (20)<sup>2</sup>.



A solution of **1a'** (2.4 g, 8.5 mmol) was dissolved in  $CH_2Cl_2$  (35 mL). To the solution was added *m*-CPBA (75%, 2.6 g, 12.75 mmol) in 30 mL  $CH_2Cl_2$ , followed by triflic acid (2.3 ml, 25.5 mmol) with stirring, during which a sticky black gum formed on the flask-wall. After stirred for 1 h, the solvent was decanted. Ether (20 mL) was added to the residue and a well-dispersed white solid precipitated, and stirred for 10 min. The solid was filtered and washed with ether (3×15 mL), affording **1a** as a white solid

A 25 mL Schlenk tube was charged with cyclc dirayliodonium salts **1a** (1.5 mmol), tellurium (287 mg, 2.25 mmol), 2-picoline (5 mL) and DMSO (5 mL) under a nitrogen atmosphere. The reaction mixture was stirred at 120 °C for 12 h. After cooled to room temperature, the reaction mixture was diluted with H<sub>2</sub>O (15 mL). The mixture extracted with dichloromethane ( $3 \times 15$  mL). The combined organic phase was washed with water and brine (20 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered, and then evaporated under reduced pressure. The residue was purified by column chromatography on silica gel to give the desired product **20**.

#### General Procedure for the synthesis of phenyl(p-tolyl)tellane (2p).

General Procedure for the synthesis of 1,2-diphenylditellane  $(2a)^3$ . To a stirred solution of bromobenzene 2a' (8.5 mmol) in dry tetrahydrofuran (THF) (40 mL) under N<sub>2</sub> at  $-78^{\circ}$ C was added *tert*-butyllithium (7.5 mL, 1.7 M; 12.75 mmol). After 1h, the cooling bath was removed, and freshly crushed finely ground elemental tellurium (8.5 mmol) was added while a brisk stream of nitrogen was passed through the open system. After 1 h, when only trace amounts of tellurium remained, the dark solution was quenched with a saturated solution of NH<sub>4</sub>Cl. After extraction with diethyl ether, separation, drying of the organic phase with Na<sub>2</sub>SO<sub>4</sub>, and evaporation, the pure product was obtained by flash chromatography with an appropriate mixture of hexane/ethyl acetate.

General Procedure for the synthesis of phenyl(*p*-tolyl)tellane  $(2p)^4$ . To a 5 mL Schlenk tube equipped with a small magnetic stirring bar were added the 1,2-diphenylditellane 2a (1 mmol), the appropriate *p*-tolylboronic acid 3a (2 mmol), AgNO<sub>3</sub> (10 mol %) and 1,4-dioxane (5 mL). The resulting mixture was stirred at 100 °C for 6 h. After that, the reaction mixture was cooled to room temperature, and was quenched using water (5 mL). The mixture was then extracted using ethyl acetate (10 mL) and washed with water (3 x 10 mL). The combined organic layers were dried over anhydrous MgSO<sub>4</sub> and concentrated under vacuum to yield the crude product, which

was purified by flash chromatography on silica gel using hexane or a mixture of hexane/ethyl acetate as eluent.

Ph Br	+ Ph <sup>Te</sup> _Ph	Ni(PPh <sub>3</sub> ) <sub>2</sub> Cl <sub>2</sub> L1 (10 mol %) Mn (1.5 equiv)	Ph Ph Té Ph
1b	2a	DMF, 40 °C, 12 h	3b
entry	Devation from standard co	nditions 3b(%)	$\mathbb{R}^2$ $\mathbb{R}^2$
1	none	70	
2	NiCl <sub>2</sub> instead of Ni(PPh 3)2	Cl <sub>2</sub> 68	
3	NiI <sub>2</sub> NiI <sub>2</sub> NiI <sub>2</sub> NiI <sub>2</sub>	2 63	R' R'
4	NiBr <sub>2</sub> instead of Ni(PPh	Cl <sub>2</sub> 48	$R^1 = Me, R_2 = Ph, R_3 = H, L1$ $R^1 = Me, R_2 = Ph, R_3 = H, L2$
5	Ni(acac) <sub>2</sub> instead of Ni(PPh	3)2Cl2 65	$R^{1} = H, R_{2} = Me, R_{3} = Me, L3$
6	L2 instead of L1	27	R <sup>2</sup> R <sup>2</sup>
7	L3 instead of L1	52	$\searrow$ $\checkmark$
8	L4 instead of L1	42	
9	L5 instead of L1	55	
10	L6 instead of L1	35	$R^{1} = Me, R_{2} = H, L4$
11	L7 instead of L1	42	$R_1^1 = H, R_2 = t_B u, L5$
12	L8 instead of L1	34	$R_1^1 = H, R_2^2 = OMe, L6$
13	DMA instead of DMF	53	
14	MeCN, DMSO instead of D	MF n. r	
15	without Mn, Zn, (Bpin) <sub>2</sub> /K <sub>3</sub>	PO4 instead of trace	
16	Mn (1.0 equiv) instead of M	In (1.5 equiv) 84	
17	24h instead of 12h	98 <sup>c</sup>	
18	without Ligand	55	

# **III.** Optimization of the reaction conditions<sup>*a,b*</sup>

<sup>a</sup>Reaction conditions: **1b** (0.2 mmol, 1.0 equiv.), **2a** (0.34 mmol, 1.7 equiv.), Ni(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (5.0 mol %), **L1** (10 mol %); Mn (0.3 mmol, 1.5 equiv.), DMF (1 mL); N<sub>2</sub> atmosphere; 40 °C; 12 h. <sup>b</sup>Yields were determined by GC with biphenyl as the internal standard. <sup>c</sup>Isolated yield.

We tested the reaction of 1-bromo-3-phenylpropane **1b** with diphenyltellane **2a**, Ni(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (5 mol %), Mn (1.5 equiv), ligand (10 mol %) in DMF at 40 °C for 12 hours. Gratifyingly, the reaction proceeded selectively to give the target product phenyl(3-phenylpropyl)tellane (**3b**). With this promising result in hand, we tried to further optimize the reaction conditions. As briefly illustrated in Table 1, after systematic testing with **L1** as the ligand, Ni(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> was chosen as catalyst because of highest yield (Table 1, entries 1-5). Next, we conducted the ligand optimization with a series of bipyridine and phenanthroline ligands. Thus, **L1** provided best result (Table 1, entries 6-12). Then, we probed the reactions in several different solvents, such as DMF, DMA, DMSO, and MeCN. It was found that DMF was crucial for the successful transformation of substrate **1b** (Table 1, entries 13 and 14). When Zn, (Bpin)<sub>2</sub>/K<sub>3</sub>PO<sub>4</sub> was used instead of Mn, the target product **3b** could not be obtained (Table 1, entry 15). Satisfactorily, when 1.0 equiv of Mn was applied to the reaction, the target product **3b** could be obtained in 84% GC-yield (Table 1, entry 16). The reaction time was extended to 24 hours, and the target product could be obtained in excellent yield (Table 1, entry 17). Finally, the target product could be obtained in 55% yield without adding ligand (Table 1, entry 18).

# **IV. General Procedure and Product Characterization**

# 1. General Procedure A

A representative procedure synthesis of phenyl(3-phenylpropyl)tellane (3b) is shown below.



In glovebox, an oven-dried screw-capped 8 mL vial equipped with a magnetic stir bar was charged with (3-bromopropyl)benzene **1b** (99.0 mg, 0.5 mmol) and diphenyltellane **2a** (241.3 mg 0.85 mmol), Ni(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (17.5 mg, 5.0 mol %), Ligand (10 mol %), Mn (1.0 equiv), DMF (1.0 mL) was added via syringe. The reaction mixture was stirred for 24 h at 40 °C. After 24h, the crude reaction mixture was diluted with ethyl acetate (20 mL) and washed with water (20 mL × 3). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated. The residue was purified by flash chromatography to afford pure product **3b** (98% yield).

#### General procedure synthesis of phenyl(3-phenylpropyl)selane (41) is shown below.



In glovebox, an oven-dried screw-capped 8 mL vial equipped with a magnetic stir bar was charged with (3-bromopropyl)benzene **1b** (0.2 mmol) and diphenylselane **2l** (0.34 mmol), Ni(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (5.0 mol %), Ligand (10 mol %), Mn (1.0 equiv), DMF (1.0 mL) was added via syringe. The reaction mixture was stirred for 24 h at 20 °C. After 24h, the crude reaction mixture was diluted with ethyl acetate (20 mL) and washed with water (20 mL × 3). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated. The residue was purified by flash chromatography to afford pure product **4l** (68% yield).

#### General procedure synthesis of phenyl(3-phenylpropyl)selane (9) is shown below.<sup>5</sup>



An oven-dried screw-capped 8 mL vial equipped with a magnetic stir bar was charged with phenyl(3-phenylpropyl)tellane **3b** (0.2 mmol) and potassium salt **8** (0.6 mmol), Pd(PPh<sub>3</sub>)<sub>4</sub> (10 mol %), Ag<sub>2</sub>O (2.0 equiv), Et<sub>3</sub>N (3.0 equiv) and MeOH was added. The reaction mixture was stirred for 2 h at 60 °C. After 2h, the crude reaction mixture was diluted with ethyl acetate (20 mL) and washed with water (20 mL × 3). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated. The residue was purified by flash chromatography to afford pure product **9** (78% yield).

### 2. General Procedure B

#### The procedure scale-up synthesis of 3b is shown below.

In glovebox, An oven-dried screw-capped 50-mL vial equipped with a magnetic stir bar was charged with (3-bromopropyl)benzene **1b** (5 mmol) and diphenyltellane **2a** 

(8.5 mmol), Ni(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (5.0 mol %), Ligand (10 mol %), Mn (1.0 equiv), DMF was added via syringe. The reaction mixture was stirred for 36 h at 40 °C. After 36h, the crude reaction mixture was diluted with ethyl acetate (20 mL) and washed with water (20 mL  $\times$  3). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated. The residue was purified by flash chromatography to afford pure product (60% yield).



## V. Mechanism experiment

## VI. The investigation of other free radical precursors



We investigated the activity of other alkyl radical precursors. It was found that under standard conditions, when alkyl chloride and alkyl OTs were applied to the reaction, the target product 3b could be obtained in moderate yield. When alkyl iodide was applied to the reaction, the target product could only be obtained in 32% yield. At the same time, to our delight, the reaction of bromobenzene **11** with **2d** could give the target product **12** in 12% yield at 80 °C, which proves that our strategy has potential application value in the construction of unsymmetrical arylaryl tellurides.

## **VII. Product Characterization**



#### phenethyl(phenyl)tellane (3a)

**Yield**: 68% (105.3mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 3023, 1573, 1493, 1473, 1432, 1152, 1017, 726, 690. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.74 – 7.67 (m, 2H), 7.31 – 7.23 (m, 3H), 7.23 – 7.12 (m, 5H), 3.16 – 3.06 (m, 4H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  142.8, 138.5, 129.3, 128.6, 128.2, 127.7, 126.4, 111.8, 38.3, 9.2. <sup>125</sup>Te (126 MHz, CDCl<sub>3</sub>)  $\delta$  490.6. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>14</sub>H<sub>14</sub>Te: 312.0158, found 312.0157.



#### phenyl(3-phenylpropyl)tellane (3b)

**Yield**: 98% (160.0mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2925, 1573, 1473, 1432, 1017, 726, 690. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.72 – 7.64 (m, 2H), 7.25 (m, *J* = 7.2, 6.4, 1.1 Hz, 3H), 7.17 (m, *J* = 6.9, 1.4 Hz, 3H), 7.14 – 7.10 (m, 2H), 2.93 – 2.82 (m, 2H), 2.68 (t, *J* = 7.5 Hz, 2H), 2.10 (p, *J* = 7.5 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  141.3, 138.4, 129.3, 128.6, 128.5, 127.6, 126.0, 111.7, 38.0, 33.4, 8.0. <sup>125</sup>Te NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  470.5. HRMS (M<sup>+</sup>) m/z calcd for C<sub>15</sub>H<sub>16</sub>Te: 326.0314, found 326.0306.



### (2-phenoxyethyl)(phenyl)tellane (3c)

**Yield**: 49% (79.5mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 1728, 1431, 1293, 1242, 1125, 1007, 755, 730, 692. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.82 – 7.74 (m, 2H), 7.32 – 7.19 (m, 5H), 6.93 (m, *J* = 7.3, 1.1 Hz, 1H), 6.86 – 6.79 (m, 2H), 4.35 – 4.26 (m, 2H), 3.25 – 3.12 (m, 2H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  158.4, 138.8, 129.6, 129.4, 128.1, 121.1, 114.8, 111.1, 69.5, 7.0. <sup>125</sup>**Te NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  443.77. **HRMS** (M<sup>+</sup>) m/z calcd for C<sub>14</sub>H<sub>14</sub>OTe: 328.0107, found 328.0113.



## phenyl(4-phenylbutyl)tellane (3d)

**Yield**: 71% (120.2mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 1709, 1590, 1490, 1358, 1220, 819, 734, 690. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.71 – 7.65 (m, 2H), 7.24 (m, *J* = 7.7 Hz, 3H), 7.18 – 7.09 (m, 5H), 2.88 (t, *J* = 7.4 Hz, 2H), 2.58 (t, *J* = 7.6 Hz, 2H), 1.86 – 1.76 (m, 2H), 1.73 – 1.63 (m, 2H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  142.2, 138.4, 129.2, 128.5, 128.4, 127.6, 125.8, 111.8, 35.2, 33.7, 31.4, 8.5. <sup>125</sup>**Te NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  471.3. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>16</sub>H<sub>18</sub>Te: 340.0471, found 340.0478.



#### phenyl(5-phenylpentyl)tellane (3e)

**Yield**: 85% (150.1mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2924, 2852, 1573, 1432, 1018, 728, 691. <sup>1</sup>H **NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.72 – 7.64 (m, 2H), 7.23 (dt, *J* = 7.3, 5.9 Hz, 3H), 7.18 – 7.08 (m, 5H), 2.84 (t, *J* = 7.6 Hz, 2H), 2.58 – 2.50 (m, 2H), 1.79 (p, *J* = 7.5 Hz, 2H), 1.58 (p, *J* = 7.5 Hz, 2H), 1.43 – 1.34 (m, 2H). <sup>13</sup>C **NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  142.5, 138.3, 129.1, 128.4, 128.3, 127.5, 125.7, 111.9, 35.8, 31.7, 31.6, 30.8, 8.6. <sup>125</sup>Te **NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  472.5. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>17</sub>H<sub>20</sub>Te: 354.0627, found 354.0631.



#### (4-methoxyphenethyl)(phenyl)tellane (3f)

**Yield**: 98% (173.8mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2930, 1712, 1609, 1573, 1509, 1433, 1299, 1242, 1175, 1033, 814, 729, 690. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.87 – 7.74 (m, 2H), 7.39 – 7.33 (m, 1H), 7.31 – 7.25 (m, 2H), 7.20 – 7.13 (m, 2H), 6.94 – 6.87 (m, 2H), 3.85 (s, 3H), 3.25 – 3.10 (m, 4H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  158.2, 138.4, 134.9, 129.3, 129.2, 127.6, 114.0, 112.0, 55.3, 37.4, 9.9. <sup>125</sup>Te NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  484.7. HRMS (CI) m/z (M<sup>+</sup>) calcd for C<sub>15</sub>H<sub>16</sub>OTe: 342.0263, found 342.0263.



## 2-(3-(phenyltellanyl)propyl)isoindoline-1,3-dione (3g)

**Yield**: 56% (110.2mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 1771, 1697, 1432, 1395, 1359, 1189, 1061, 972, 867, 716, 688. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.81 (m, *J* = 5.4, 3.1 Hz, 2H), 7.75 – 7.64 (m, 4H), 7.29 – 7.22 (m, 1H), 7.21 – 7.10 (m, 2H), 3.75 (t, *J* = 6.9 Hz, 2H), 2.91 – 2.75 (m, 2H), 2.21 – 2.10 (m, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  168.3, 138.7, 134.0, 132.0, 129.2, 127.8, 123.2, 111.5, 40.0, 30.8, 4.5. <sup>125</sup>Te NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  492.41. **HRMS** (M<sup>+</sup>) m/z calcd for C<sub>17</sub>H<sub>15</sub>NO<sub>2</sub>Te: 395.0165, found 395.0162.



**2-(4-(phenyltellanyl)butyl)benzo[d]oxazole (3h) Yield**: 91% (173.5mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2329, 1612, 1571, 1455, 1243,

1145, 940, 750, 729, 689. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.72 – 7.61 (m, 3H), 7.47 – 7.40 (m, 1H), 7.29 – 7.19 (m, 3H), 7.16 – 7.09 (m, 2H), 2.89 (td, *J* = 7.4, 2.3 Hz, 4H), 2.00 – 1.93 (m, 2H), 1.93 – 1.86 (m, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  166.6, 150.7, 141.3, 138.4, 129.1, 127.5, 124.4, 124.0, 119.5, 111.5, 110.2, 31.1, 28.8, 27.8, 7.6. <sup>125</sup>Te NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  477.20. HRMS (CI) m/z (M<sup>+</sup>) calcd for C<sub>17</sub>H<sub>17</sub>NOTe: 381.0372, found 381.0375.

## H0\_\_\_\_\_Te\_\_ph

#### 3-(phenyltellanyl)propan-1-ol (3i)

**Yield**: 56% (74.9mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2925, 1726, 1573, 1473, 1432, 1292, 1017, 727. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.76 – 7.69 (m, 2H), 7.29 – 7.25 (m, 1H), 7.24 – 7.15 (m, 2H), 3.67 (t, J = 6.1 Hz, 2H), 2.95 (t, J = 7.4 Hz, 2H), 2.03 (tt, J = 7.4, 6.2 Hz, 2H), 1.81 (s, 1H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  138.4, 129.3, 127.7, 111.8, 63.9, 34.3, 4.3. <sup>125</sup>**Te NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  471.7. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>9</sub>H<sub>12</sub>OTe: 265.9950, found 265.9942.



#### 2-(2-(phenyltellanyl)ethyl)-1,3-dioxolane (3j)

**Yield**: 78% (120.1 mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2880, 1711, 1574, 1433, 1359, 1220, 1123, 1017, 841, 731, 692. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.74 – 7.67 (m, 2H), 7.25 (m, *J* = 8.2, 6.4, 1.4 Hz, 1H), 7.18 (m, *J* = 6.6, 1.7 Hz, 2H), 4.91 (t, *J* = 4.3 Hz, 1H), 3.97 – 3.92 (m, 2H), 3.87 – 3.79 (m, 2H), 3.00 – 2.86 (m, 2H), 2.19 (td, *J* = 7.7, 4.3 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  138.2, 129.2, 127.6, 112.4, 104.7, 65.0, 35.8, 0.2. <sup>125</sup>Te NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  498.7. HRMS (CI) m/z (M<sup>+</sup>) calcd for C<sub>11</sub>H<sub>14</sub>O<sub>2</sub>Te: 308.0056, found 308.0057.

#### (2-methoxyethyl)(phenyl)tellane (3k)

**Yield**: 64% (85.3 mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2923, 1574, 1473, 1433, 1101, 1018, 942, 728. <sup>1</sup>H **NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.77 – 7.69 (m, 2H), 7.29 – 7.24 (m, 1H), 7.19 (m, J = 8.3, 6.6, 1.3 Hz, 2H), 3.71 (dd, J = 7.8, 6.9 Hz, 2H), 3.33 (s, 3H), 3.05 (dd, J = 7.8, 6.9 Hz, 2H). <sup>13</sup>C **NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  138.5, 129.3, 127.8, 111.4, 73.7, 58.3, 7.9. <sup>125</sup>Te **NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  437.2. **HRMS** (M<sup>+</sup>) m/z calcd for C<sub>9</sub>H<sub>12</sub>OTe: 265.9950, found 265.994.



#### tert-butyldimethyl(2-(phenyltellanyl)ethoxy)silane (31)

**Yield**: 50% (88.8 mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2927, 1575, 1472, 1433, 1253, 1067, 833, 774, 728, 690. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.74 – 7.64 (m, 2H), 7.25 – 7.19 (m, 1H), 7.14 (m, *J* = 8.1, 6.6 Hz, 2H), 3.97 – 3.83 (m, 2H), 3.07 – 2.96 (m, 2H), 0.84 (s, 9H), 0.00 (s, 6H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  138.4, 129.2, 127.6, 111.6, 64.7, 26.0, 18.4, 12.3, -5.1. <sup>125</sup>**Te NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  422.3. **HRMS** (M<sup>+</sup>) m/z calcd for C<sub>14</sub>H<sub>24</sub>ONSiTe: 366.0659, found 366.0655.



#### ethyl 6-(phenyltellanyl)hexanoate (3m)

**Yield**: 84% (146.8mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2929, 1730, 1574, 1433, 1371, 1177, 1115, 1018, 729, 691. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.74 – 7.66 (m, 2H), 7.29 – 7.22 (m, 1H), 7.17 (m, J = 8.2, 6.7 Hz, 2H), 4.16 – 4.04 (m, 2H), 2.87 (t, J = 7.5 Hz, 2H), 2.25 (t, J = 7.5 Hz, 2H), 1.79 (p, J = 7.5 Hz, 2H), 1.65 – 1.57 (m, 2H), 1.39 (tt, J = 9.7, 6.2 Hz, 2H), 1.26 – 1.20 (m, 3H). <sup>13</sup>C **NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  173.5, 138.3, 129.1, 127.5, 111.7, 60.1, 34.1, 31.4, 31.3, 24.2, 14.2, 8.2. <sup>125</sup>Te **NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  473.5. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>14</sub>H<sub>20</sub>O<sub>2</sub>Te: 350.0526, found 350.0527.



#### 5-(phenyltellanyl)pentanenitrile (3n)

**Yield**: 84% (121.7mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2929, 2245, 1710, 1573, 1474, 1432, 1359, 1219, 1017, 731. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.75 – 7.67 (m, 2H), 7.31 – 7.24 (m, 1H), 7.24 – 7.16 (m, 2H), 2.86 (t, *J* = 7.4 Hz, 2H), 2.29 (t, *J* = 7.1 Hz, 2H), 1.95 – 1.86 (m, 2H), 1.76 – 1.70 (m, 2H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  138.6, 129.3, 127.8, 119.4, 111.2, 30.6, 27.3, 16.5, 6.8. <sup>125</sup>**Te NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  479.6. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>11</sub>H<sub>13</sub>NTe: 289.0110, found 289.0109.



### diethyl (3-(phenyltellanyl)propyl)phosphonate (30)

**Yield**: 48% (88.8mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2979, 1730, 1434, 1256, 1229, 1052, 1019, 953, 731, 691. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.74 – 7.70 (m, 2H), 7.31 – 7.25 (m, 1H), 7.19 (dd, J = 8.2, 6.6 Hz, 2H), 4.05 (ddq, J = 14.4, 7.2, 3.4 Hz, 4H), 2.97 – 2.91 (m, 2H), 2.12 – 2.00 (m, 2H), 1.90 – 1.80 (m, 2H), 1.28 (t, J = 7.1 Hz, 6H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  138.6, 129.2, 127.8, 111.2, 61.5 (J = 6.52), 52.6, 27.9 (J = 139.29), 25.92, 24.8 (J = 4.68), 16.5 (J = 6.22), 9.0 (J = 18.06). <sup>125</sup>**Te NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  470.5. **HRMS** (M<sup>+</sup>) m/z calcd for C<sub>13</sub>H<sub>21</sub>PO<sub>3</sub>Te: 386.0291, found 386.0294.



#### 4-(tert-butyldisulfanyl)butanenitrile (3p)

**Yield**: 11% (21.4mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2928, 1738, 1712, 1498, 1434, 1248, 1160, 1018, 733, 692. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.80 – 7.73 (m, 2H), 7.32 – 7.27 (m, 1H), 7.20 (dd, J = 8.1, 6.7 Hz, 2H), 5.33 (d, J = 8.2 Hz, 1H), 4.72 (d, J = 7.6 Hz, 1H), 3.53 (s, 3H), 3.29 (t, J = 6.5 Hz, 2H), 1.42 (s, 9H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.8, 139.3, 129.4, 128.2, 111.0, 80.2, 53.8, 52.5, 28.4, 11.8. <sup>125</sup>Te NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  422.6. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>15</sub>H<sub>21</sub>NO<sub>4</sub>Te: 409.0533, found 409.0530.



#### (8S,9R,13R,14R)-13-methyl-3-(3-(phenyltellanyl)propoxy)-

**6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (3q) Yield**: 68% (172.2mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2329, 1728, 1570, 1468, 1247, 1157, 1055, 875, 733, 693. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.75 – 7.66 (m, 2H), 7.28 – 7.21 (m, 1H), 7.19 – 7.13 (m, 3H), 6.67 (m, *J* = 8.5, 2.8 Hz, 1H), 6.59 (d, *J* = 2.7 Hz, 1H), 3.96 (t, *J* = 6.0 Hz, 2H), 3.02 (t, *J* = 7.3 Hz, 2H), 2.90 – 2.81 (m, 2H), 2.48 (dd, *J* = 18.8, 8.6 Hz, 1H), 2.37 (dq, *J* = 11.9, 3.3 Hz, 1H), 2.22 (tq, *J* = 10.0, 4.8, 3.8 Hz, 3H), 2.16 – 1.92 (m, 4H), 1.64 – 1.38 (m, 6H), 0.89 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  220.8, 156.8, 138.3, 137.7, 132.1, 129.2, 127.6, 126.3, 114.6, 112.2, 111.8, 68.6, 50.4, 48.0, 44.0, 38.4, 35.9, 31.6, 31.3, 29.7, 26.6, 25.9, 21.6, 13.9, 4.4. <sup>125</sup>Te NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  473.2. HRMS (M<sup>+</sup>) m/z calcd for C<sub>27</sub>H<sub>32</sub>O<sub>2</sub>Te: 518.1465, found 518.1464.



#### 1-benzyl-2-(2-phenoxyethyl)disulfane (4a)

**Yield**: 68% (132.2mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2928, 1599, 1584, 1493, 1453, 1238, 1171, 1013, 752, 690. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.84 – 7.73 (m, 2H), 7.36 – 7.30 (m, 1H), 7.21 (t, *J* = 7.5 Hz, 2H), 3.80 (d, *J* = 13.4 Hz, 2H), 3.49 (tt, *J* = 10.6, 3.9 Hz, 1H), 2.93 (ddd, *J* = 13.5, 10.3, 3.0 Hz, 2H), 2.03 (dq, *J* = 12.5, 3.9 Hz, 2H), 1.78 (dtd, *J* = 14.1, 10.4, 4.1 Hz, 2H), 1.45 – 1.39 (m, 9H). <sup>13</sup>C **NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  154.8, 140.5, 129.2, 128.2, 110.7, 79.5, 49.5, 35.0, 28.5, 23.3. <sup>125</sup>Te **NMR** (126 MHz,



#### (3-phenylpropyl)(o-tolyl)tellane (4a)

**Yield**: 31% (52.3mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2924, 1583, 1495, 1451, 1377, 1272, 1023, 739. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.56 (m, *J* = 7.6, 1.2 Hz, 1H), 7.26 (m, *J* = 8.1, 6.7 Hz, 2H), 7.22 – 7.10 (m, 5H), 6.97 (m, *J* = 7.4, 1.8 Hz, 1H), 2.85 (t, *J* = 7.5 Hz, 2H), 2.70 (t, *J* = 7.4 Hz, 2H), 2.41 (s, 3H), 2.10 (p, *J* = 7.5 Hz, 2H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  142.5, 141.3, 136.9, 129.2, 128.7, 128.5, 127.7, 126.6, 126.1, 116.4, 38.1, 33.2, 26.6, 7.0. <sup>125</sup>**Te NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  353.9. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>16</sub>H<sub>18</sub>Te: 340.0471, found 340.0469.



#### (3-phenylpropyl)(*m*-tolyl)tellane (4b)

**Yield**: 60% (102.0mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2920, 1586, 1494, 1452, 1209, 769, 746, 687. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.55 – 7.50 (m, 1H), 7.50 – 7.45 (m, 1H), 7.28 – 7.22 (m, 2H), 7.20 – 7.10 (m, 3H), 7.08 – 7.04 (m, 2H), 2.92 – 2.82 (m, 2H), 2.69 (t, *J* = 7.5 Hz, 2H), 2.29 (s, 3H), 2.15 – 2.06 (m, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  141.4, 139.1, 139.0, 135.4, 129.1, 128.7, 128.5, 128.5, 126.0, 111.6, 38.0, 33.5, 21.3, 7.9. <sup>125</sup>Te NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  465.8. HRMS (CI) m/z (M<sup>+</sup>) calcd for C<sub>16</sub>H<sub>18</sub>Te: 340.0471, found 340.0477.



#### (3-phenylpropyl)(p-tolyl)tellane (4c)

**Yield**: 58% (99.3mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2922, 1602, 1486, 1453, 1209, 1013, 798, 747, 698. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.53 (m, *J* = 8.0 Hz, 2H), 7.18 (m, *J* = 8.0, 6.7 Hz, 2H), 7.13 – 7.03 (m, 3H), 6.93 (m, *J* = 7.7 Hz, 2H), 2.77 (t, *J* = 7.5 Hz, 2H), 2.61 (t, *J* = 7.5 Hz, 2H), 2.26 (s, 3H), 2.01 (p, *J* = 7.5 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  141.4, 139.0, 137.8, 130.2, 128.7, 128.5, 126.0, 107.4, 38.0, 33.4, 21.3, 8.0. <sup>125</sup>Te NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  458.7. HRMS (ESI<sup>+</sup>, MeCN) m/z calcd for C<sub>16</sub>H<sub>18</sub>Te: 340.0471, found 340.0473.



#### (4-methoxyphenyl)(3-phenylpropyl)tellane (4d)

**Yield**: 74% (131.7mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2927, 1712, 1585, 1487, 1453, 1281, 1241, 1175, 1028, 820, 747, 698. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.70 – 7.62 (m, 2H), 7.27 – 7.22 (m, 2H), 7.20 – 7.09 (m, 3H), 6.78 – 6.71 (m, 2H), 3.78 (s, 3H), 2.84 – 2.78 (m, 2H), 2.70 – 2.62 (m, 2H), 2.06 (p, *J* = 7.5 Hz, 2H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  159.8, 141.4, 141.1, 128.7, 128.4, 126.0, 115.3, 100.5, 55.3, 37.9, 33.4, 8.3. <sup>125</sup>**Te NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  455.9. **HRMS** (M<sup>+</sup>) m/z calcd for C<sub>16</sub>H<sub>18</sub>OTe: 356.0420, found 356.0411.



#### (4-(*tert*-butyl)phenyl)(3-phenylpropyl)tellane (4e)

**Yield**: 81% (154.4mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2960, 1494, 1453, 1392, 1266, 1112, 1008, 818, 746, 697. <sup>1</sup>H **NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.66 – 7.59 (m, 2H), 7.26 – 7.18 (m, 4H), 7.17 – 7.08 (m, 3H), 2.84 (t, *J* = 7.5 Hz, 2H), 2.67 (dd, *J* = 8.2, 6.8 Hz, 2H), 2.09 (p, *J* = 7.4 Hz, 2H), 1.29 (s, 9H). <sup>13</sup>C **NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  150.9, 141.3, 138.5, 128.6, 128.4, 126.4, 126.0, 107.9, 37.9, 34.6, 33.4, 31.4, 7.8. <sup>125</sup>Te **NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  451.5. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>19</sub>H<sub>24</sub>Te: 382.0940, found 382.0938.



#### 4-((3-phenylpropyl)tellanyl)aniline (4f)

**Yield**: 90% (162.4mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 1708, 1590, 1490, 1358, 1220, 819, 700. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.58 – 7.49 (m, 2H), 7.29 – 7.20 (m, 2H), 7.17 – 7.09 (m, 3H), 6.49 – 6.41 (m, 2H), 3.62 (s, 2H), 2.80 – 2.69 (m, 2H), 2.66 – 2.60 (m, 2H), 2.07 – 1.98 (m, 2H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  146.5, 141.4, 141.2, 128.5, 128.3, 125.8, 116.1, 97.1, 37.8, 33.2, 8.2. <sup>125</sup>**Te NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  448.7. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>15</sub>H<sub>17</sub>NTe: 341.0423, found 341.0418.



#### (4-fluorophenyl)(3-phenylpropyl)tellane (4g)

**Yield**: 78% (134.4mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2926, 1712, 1580, 1483, 1359, 1219, 1160, 1012, 821, 747, 698. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.70 – 7.61 (m, 2H), 7.24 (m, *J* = 8.0, 6.6 Hz, 2H), 7.20 – 7.13 (m, 1H), 7.13 – 7.03 (m, 2H), 6.90 – 6.79 (m, 2H), 2.82 (t, *J* = 7.5 Hz, 2H), 2.66 (t, *J* = 7.4 Hz, 2H), 2.06 (p, *J* = 7.5 Hz, 2H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  163.0 (*J* = 246.2), 141.2, 141.0, 140.9, 128.6 (*J* = 13.8),

126.1, 116.6 (J = 20.4), 105.2 (J = 3.7), 37.9, 33.3, 8.5. <sup>125</sup>Te NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  476.0. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>15</sub>H<sub>15</sub>FTe: 344.0229, found 344.0217.



#### (4-chlorophenyl)(3-phenylpropyl)tellane (4h)

**Yield**: 76% (137.4mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2925, 1494, 1470, 1380, 1210, 1086, 1007, 805, 721, 696. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.60 – 7.52 (m, 2H), 7.24 (dd, J = 8.1, 6.7 Hz, 2H), 7.20 – 7.15 (m, 1H), 7.15 – 7.07 (m, 4H), 2.84 (t, J = 7.5 Hz, 2H), 2.66 (t, J = 7.4 Hz, 2H), 2.07 (p, J = 7.5 Hz, 2H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  141.1, 139.8, 134.2, 129.5, 128.6, 128.5, 126.1, 109.3, 37.9, 33.3, 8.4. <sup>125</sup>**Te NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  478.6. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>15</sub>H<sub>15</sub>ClTe: 359.9925, found 359.9915.



#### 3-((3-phenylpropyl)tellanyl)pyridine (4i)

**Yield**: 84% (137.2mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 1710, 1358, 1219, 1010, 791, 701, 615. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.87 (m, J = 1.9 Hz, 1H), 8.49 (m, J = 4.9, 1.6 Hz, 1H), 7.95 (m, J = 7.7, 1.9 Hz, 1H), 7.25 (m, J = 8.1, 6.7 Hz, 2H), 7.19 – 7.14 (m, 1H), 7.13 – 7.07 (m, 3H), 2.86 (t, J = 7.5 Hz, 2H), 2.67 (t, J = 7.4 Hz, 2H), 2.08 (p, J = 7.5 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  157.7, 148.4, 145.7, 140.8, 128.5, 128.4, 126.0, 124.4, 109.1, 37.7, 33.2, 8.2. <sup>125</sup>Te NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  435.6. HRMS (CI) m/z (M<sup>+</sup>) calcd for C<sub>14</sub>H<sub>15</sub>NTe: 327.0267, found 327.0262.



#### 2-((3-phenylpropyl)tellanyl)pyridine (4j)

**Yield**: 73% (119.9mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2923, 1565, 1552, 1447, 1409, 1273, 1094, 1076, 982, 746, 613. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.52 (m, *J* = 5.1, 1.8 Hz, 1H), 7.48 (m, *J* = 7.8 Hz, 1H), 7.41 – 7.29 (m, 3H), 7.27 – 7.18 (m, 3H), 7.07 – 7.00 (m, 1H), 3.18 (t, *J* = 7.4 Hz, 2H), 2.79 (t, *J* = 7.5 Hz, 2H), 2.30 (p, *J* = 7.5 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  150.8, 141.3, 141.1, 135.4, 131.6, 128.6, 128.4, 125.9, 121.1, 38.1, 33.5, 8.9. <sup>125</sup>Te NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  517.7. HRMS (CI) m/z (M<sup>+</sup>) calcd for C<sub>14</sub>H<sub>15</sub>NTe: 327.0267, found 327.0269.



#### **3-((3-phenylpropyl)tellanyl)thiophene (4k)**

**Yield**: 62% (103.3mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2924, 1601, 1494, 1452, 1193, 1150, 841, 766, 747, 696. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.45 (m, *J* = 2.8, 1.3 Hz, 1H), 7.29 – 7.21 (m, 3H), 7.21 – 7.14 (m, 2H), 7.14 – 7.08 (m, 2H), 2.77 (dd, *J* = 8.5, 6.4 Hz, 2H), 2.66 (t, *J* = 7.5 Hz, 2H), 2.10 – 2.00 (m, 2H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  141.3, 137.0, 133.5, 128.6, 128.5, 126.8, 126.0, 101.2, 37.8, 33.4, 8.4, 8.3. <sup>125</sup>**Te NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  333.9. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>13</sub>H<sub>14</sub>STe: 331.9878, found 331.9882.



#### phenyl(3-phenylpropyl)selane (41)

**Yield**: 68% (37.5mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2929, 1578, 1476, 1436, 1072, 1022, 732, 689. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.48 – 7.42 (m, 2H), 7.29 – 7.20 (m, 5H), 7.19 – 7.10 (m, 3H), 2.89 (t, J = 7.3 Hz, 2H), 2.72 (t, J = 7.5 Hz, 2H), 2.00 (p, J = 7.4 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  141.4, 132.6, 130.4, 129.1, 128.6, 128.5, 126.8, 126.0, 35.8, 31.7, 27.2. <sup>77</sup>Se NMR (76 MHz, CDCl<sub>3</sub>)  $\delta$  289.8. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>15</sub>H<sub>16</sub>Se: 276.0417, found 276.0419.



#### 2-(4-(phenethyltellanyl)butyl)benzo[d]oxazole (4r)

**Yield**: 75% (91.7mg). Yellow oil. **IR** (neat, v, cm<sup>-1</sup>): 2925, 1713, 1614, 1570, 1454, 1240, 1145, 743, 697. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.72 – 7.66 (m, 1H), 7.50 (m, *J* = 8.5, 3.2 Hz, 1H), 7.36 – 7.28 (m, 4H), 7.27 – 7.23 (m, 1H), 7.23 – 7.17 (m, 2H), 3.11 (t, *J* = 7.9 Hz, 2H), 3.01 – 2.86 (m, 4H), 2.65 (t, *J* = 7.5 Hz, 2H), 2.03 – 1.94 (m, 2H), 1.92 – 1.83 (m, 2H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  166.8, 150.8, 142.7, 141.4, 128.5, 128.1, 126.3, 124.5, 124.1, 119.6, 110.3, 38.8, 31.6, 29.0, 27.9, 3.5, 1.9. <sup>125</sup>**Te NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  261.1. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>19</sub>H<sub>21</sub>NOTe: 409.0685, found 409.0683.

#### (4-methoxyphenyl)(phenyl)tellane (12)

**Yield**: 12% (7.8 mg, 0.2 mmol). White solid. <sup>1</sup>**H** NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.70 – 7.62 (m, 2H), 7.52 – 7.47 (m, 2H), 7.11 (m, J = 14.5, 8.5, 6.1, 2.2 Hz, 3H), 6.76 – 6.70 (m, 2H), 3.73 (s, 3H). <sup>13</sup>**C** NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  160.0, 141.2, 136.4, 129.4, 127.3,

115.9, 115.5, 103.2, 55.2. **HRMS** (CI) m/z (M<sup>+</sup>) calcd for C<sub>13</sub>H<sub>12</sub>OTe: 313.9950, found 313.9948. The values of the NMR spectrum are accordance with reported literature data.<sup>6</sup>



#### 4-methoxy-1,1'-biphenyl (9)

**Yield**: 78% (28.7 mg). White solid. <sup>1</sup>**H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.53 (m, J = 8.7, 7.5, 1.6 Hz, 4H), 7.40 (t, J = 7.6 Hz, 2H), 7.32 – 7.25 (m, 1H), 7.02 – 6.91 (m, 2H), 3.83 (s, 3H). <sup>13</sup>**C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  159.3, 140.9, 133.9, 128.8, 128.3, 126.9, 126.8, 114.3, 55.5.

## VIII. References

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# IX. Copies of <sup>1</sup>H NMR, <sup>13</sup>C NMR and <sup>125</sup>Te NMR Spectra

<sup>1</sup>H NMR Spectra of **3a** (400 MHz, CDCl<sub>3</sub>)

 $\begin{array}{c} 7.7\\ 7.72\\ 7.72\\ 7.72\\ 7.76\\ 7.76\\ 7.76\\ 7.72\\$ 



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)







1900 1700 1500 1300 1100 900 700 500 300 100 -100 -300 -500 -700 -900 -1100 -1300 -1500 -1700 -1900 fl (ppm)

<sup>1</sup>H NMR Spectra of **3c** (400 MHz, CDCl<sub>3</sub>)

7.77 7.72 7.727.72



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





# <sup>1</sup>H NMR Spectra of **3e** (400 MHz, CDCl<sub>3</sub>)

 $\begin{array}{c} 7.56\\ 7.67\\ 7.69\\ 7.67\\ 7.67\\ 7.67\\ 7.67\\ 7.67\\ 7.67\\ 7.67\\ 7.67\\ 7.67\\ 7.67\\ 7.67\\ 7.67\\ 7.72\\$ 





# <sup>1</sup>H NMR Spectra of **3f** (400 MHz, CDCl<sub>3</sub>)







# <sup>1</sup>H NMR Spectra of **3g** (400 MHz, CDCl<sub>3</sub>)

 $\begin{array}{c} 7.82\\ 7.82\\ 7.73\\ 7.79\\ 7.77\\ 7.76\\ 7.72\\$ 











<sup>1</sup>H NMR Spectra of **3i** (400 MHz, CDCl<sub>3</sub>)

 $\begin{array}{c} 7.73\\ 7.73\\ 7.71\\ 7.71\\ 7.72\\ 7.72\\ 7.72\\ 7.22\\$ 









<sup>1</sup>H NMR Spectra of **3k** (400 MHz, CDCl<sub>3</sub>)







<sup>1</sup>H NMR Spectra of **3m** (400 MHz, CDCl<sub>3</sub>)

 $\begin{array}{c} 7.71\\ 7.71\\ 7.70\\ 7.70\\ 7.70\\ 7.70\\ 7.70\\ 7.70\\ 7.72\\ 7.72\\ 7.72\\ 7.72\\ 7.72\\ 7.19\\ 7.10\\ 7.12\\ 7.12\\ 7.12\\ 7.12\\ 7.12\\ 7.12\\ 7.12\\ 7.12\\ 7.12\\ 7.12\\ 7.16\\ 1.17\\ 1.17\\ 1.17\\ 1.17\\ 1.17\\ 1.17\\ 1.17\\ 1.12\\$ 



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



# <sup>1</sup>H NMR Spectra of **3n** (400 MHz, CDCl<sub>3</sub>)







<sup>1</sup>H NMR Spectra of **30** (400 MHz, CDCl<sub>3</sub>)

 $\begin{array}{c} 7.73\\ 7.77\\ 7.77\\ 7.77\\ 7.77\\ 7.77\\ 7.77\\ 7.77\\ 7.77\\ 7.72\\$ 



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



<sup>1</sup>H NMR Spectra of **3p** (400 MHz, CDCl<sub>3</sub>)







<sup>125</sup>Te NMR Spectra of **3p** (400 MHz, CDCl<sub>3</sub>)



### 





\_422.59





00 1700 1500 1300 1100 900 700 500 300 100 -100 -300 -500 -700 -900 -1100 -1300 -1500 -1700 -190 f1 (ppm)

# <sup>1</sup>H NMR Spectra of **3r** (400 MHz, CDCl<sub>3</sub>)

# $\begin{array}{c} 7.3.\\ 7.7.\\$





<sup>1</sup>H NMR Spectra of **4a** (400 MHz, CDCl<sub>3</sub>)

 $\begin{array}{c} 7.55\\ 7.56\\$ 



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







# <sup>1</sup>H NMR Spectra of **4c** (400 MHz, CDCl<sub>3</sub>)

 $\begin{array}{c} 7.53\\ 7.52\\$ 



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)



# <sup>1</sup>H NMR Spectra of **4d** (400 MHz, CDCl<sub>3</sub>)

# $\begin{array}{c} 7.68\\ 7.67\\ 7.65\\ 7.65\\ 7.65\\ 7.65\\ 7.65\\ 7.65\\ 7.65\\ 7.75\\ 7.72\\$





<sup>1</sup>H NMR Spectra of **4e** (400 MHz, CDCl<sub>3</sub>)

 $\begin{array}{c} 7.64\\ 1.64\\$ 



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





200 1700 1500 1300 1100 900 700 500 300 100 -100 -300 -500 -700 -900 -1100 -1300 -1500 -1700 -190 f1 (ppm) <sup>1</sup>H NMR Spectra of 4g (400 MHz, CDCl<sub>3</sub>)

 $\begin{array}{c} 7.65\\ 7.65\\ 7.65\\ 7.65\\ 7.65\\ 7.75\\$ 



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)



200 1700 1500 1300 1100 900 700 500 300 100 -100 -300 -500 -700 -900 -1100 -1300 -1500 -1700 -19 f1 (ppm)

# <sup>1</sup>H NMR Spectra of **4h** (400 MHz, CDCl<sub>3</sub>)





# <sup>1</sup>H NMR Spectra of **4i** (400 MHz, CDCl<sub>3</sub>)

2.00 





<sup>1</sup>H NMR Spectra of 4j (400 MHz, CDCl<sub>3</sub>)











<sup>1</sup>H NMR Spectra of 4k (400 MHz, CDCl<sub>3</sub>)

 $\begin{array}{c} 7.46\\ 7.45\\ 7.26\\ 7.26\\ 7.27\\ 7.22\\$ 











# <sup>1</sup>H NMR Spectra of **4r** (400 MHz, CDCl<sub>3</sub>)

 $\begin{array}{c} 7.7.1\\ 7.7.6\\ 7.7.6\\ 7.7.6\\ 7.7.6\\ 7.7.5\\ 7.7.5\\ 7.7.5\\ 7.7.5\\ 7.7.2\\ 7.$ 



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

# <sup>125</sup>Te NMR Spectra of 4r (400 MHz, CDCl<sub>3</sub>)





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