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# **Supporting Information**

# Photoinduced Synthesis and Biological Investigation of Aryl Trifluoromethyl Tellurides

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#### **1.** General information

All reactions were carried out under a nitrogen atmosphere. Unless otherwise specified, NMR spectra were recorded in CDCl<sub>3</sub>, CD<sub>3</sub>CN, or acetone-d<sub>6</sub> on a 500 MHz (for <sup>1</sup>H), 471 MHz (for <sup>19</sup>F), and 126 MHz (for <sup>13</sup>C) spectrometer. All chemical shifts were reported in ppm relative to TMS (0 ppm for <sup>1</sup>H NMR) and PhOCF<sub>3</sub> (-58.0 ppm for <sup>19</sup>F NMR) as an internal or external standard. The coupling constants were reported in Hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd =doublet of doublets. The HPLC experiments were carried out on a Wufeng LC-100 II instrument (column: SHIMEN Superb II, C18, 5  $\mu$ m, 4.6  $\times$  250 mm), and the yields of product were determined by using the corresponding pure compound as an external standard. Melting points were measured and uncorrected. HRMS experiments were performed on a TOF-Q ESI or APCI instrument. Thianthrene 5-oxide (TTO),<sup>1</sup> dibenzo[b,d]thiophene 5-oxide (DBTO),<sup>2</sup> and the corresponding arylsulfonium salts<sup>3-6</sup> were prepared according to the literature. Reagent [Me<sub>4</sub>N][TeCF<sub>3</sub>] was synthesized according to the literature.<sup>7</sup> The dry solvents were purchased from commercial source and used without further purification.

#### The equipment and light source for the reactions



The reaction tube for 0.1 mmol scales (left), green LEDs (5 W  $\times$  8, 505-520 nm) (middle). The reaction tube(s) were irradiated with the green LEDs (right). In each case, the distance between the light source and the reaction vessel was about 2.5 cm.

# 2. Screening of optimal reaction conditions for the trifluoromethyltellurolation of arylsulfonium salt (1a) with [Me4N][TeCF3] (2).

Table S1. Photoinduced trifluoromethyltellurolation of 1a with 2 in DMF under

different light irradiation.<sup>a</sup>

NC 1a OTf 0.1 mmol	+ $[Me_4N][TeCF_3]$ S 2 $N_2$ , 12 h $NC$	3a TeCF <sub>3</sub>
Entry	Light source	Yield ( <b>3a</b> , %)
1	365-370 nm LED (5 W)	72
2	380-385 nm LEDs (5 W $\times$ 8)	65
3	395-400 nm LED (5 W)	67
4	blue LEDs (450-460 nm, 5 $W \times 8$ )	73
5 <sup>b</sup>	blue LEDs (450-460 nm, 5 $W \times 8$ )	80
6	460-465 nm LED (5 W)	76
7	green LEDs (505-520 nm, 5 W $\times$ 8)	89
8	natural light	trace

<sup>a</sup> Reaction conditions: **1a** (0.1 mmol), **2** (0.16 mmol), DMF (0.5 mL), light source, 30-32 °C, N<sub>2</sub>, and 12 h. The yield was determined by HPLC using pure **3a** as an external standard (water / methanol = 20 / 80 (v/v), flow rate = 0.7 mL/min,  $\lambda_m = 253$  nm, t<sub>R</sub> = 7.55 min). <sup>b</sup> The reaction was run at 0 °C.

**Table S2.** Photoinduced trifluoromethyltellurolation of **1a** with **2** in different solventsunder green LEDs (5 W) irradiation.<sup>a</sup>

NC 1a OTf S 0.1 mmol	+ [Me <sub>4</sub> N][TeCF <sub>3</sub> ] green LEDs (5 W) <b>2</b> solvent, 30-32 °C 1.6 equiv N <sub>2</sub> , 12 h	NC 3a TeCF3
Entry	Solvent	Yield ( <b>3a</b> , %)
1	DMF	89
2	dimethyl carbonate	80
3	MeCN	49
4	DMAc	83
5	acetone	trace
6	DMSO	87
7	THF	80

8	DME	81
9	1,4-dioxane	86
10	ethyl acetate	66
11	DG	72
12	toluene	55
13	DCE	13
14	<i>n</i> -hexane	4

<sup>a</sup> Reaction conditions: **1a** (0.1 mmol), **2** (1.6 equiv), solvent (0.5 mL), green LEDs (5 W), 30-32 °C, N<sub>2</sub>, and 12 h. The yield was determined by HPLC using pure **3a** as an external standard (water / methanol = 20 / 80 (v/v), flow rate = 0.7 mL/min,  $\lambda_m = 253$  nm, t<sub>R</sub> = 7.55 min).

**Table S3**. Photoinduced trifluoromethyltellurolation of **1a** with **2** in DMF at different concentrations under green LEDs (5 W) irradiation.<sup>a</sup>

NC 1a 0.1 mmol	+ [Me <sub>4</sub> N][TeCF <sub>3</sub> ] $\xrightarrow{\text{green LEDs (5 W)}}$ S 2 DMF, 30-32 °C 1.6 equiv N <sub>2</sub> , 12 h	NC 3a TeCF <sub>3</sub>
Entry	Concentration (M) / DMF (x mL)	Yield ( <b>3a</b> , %)
1	0.2 (0.5 mL)	89
2	0.1 (1 mL)	88
3	0.05 (2 mL)	74
4	0.025 (4 mL)	70
5	0.0125 (8 mL)	68
6	0.00625 (16 mL)	61

<sup>a</sup> Reaction conditions: **1a** (0.1 mmol), **2** (1.6 equiv), DMF (x mL), green LEDs (5 W), 30-32 °C, N<sub>2</sub>, and 12 h. The yield was determined by HPLC using pure **3a** as an external standard (water / methanol = 20 / 80 (v/v), flow rate = 0.7 mL/min,  $\lambda_m = 253$ nm, t<sub>R</sub> = 7.55 min).

**Table S4**. Photoinduced trifluoromethyltellurolation of **1a** with different equivalents of **2** in DMF under green LEDs (5 W) irradiation.<sup>a</sup>

NC 1a OTf S +	[Me <sub>4</sub> N][TeCF <sub>3</sub> ] green LEDs (5 W) 2 N2, 12 h x equiv N2, 12 h	NC 3a TeCF <sub>3</sub>
Entry	<b>2</b> (x equiv)	Yield ( <b>3a</b> , %)
1	1	62
2	1.2	79
3	1.4	87
4	1.5	89
5	1.6	89
6	1.8	94

<sup>a</sup> Reaction conditions: **1a** (0.1 mmol), **2** (x equiv), DMF (0.5 mL), green LEDs (5 W), 30-32 °C, N<sub>2</sub>, and 12 h. The yield was determined by HPLC using pure **3a** as an external standard (water / methanol = 20 / 80 (v/v), flow rate = 0.7 mL/min,  $\lambda_m = 253$ nm, t<sub>R</sub> = 7.55 min).

**Table S5**. Photoinduced trifluoromethyltellurolation of **1a** with **2** in DMF at different times under green LEDs (5 W) irradiation.<sup>a</sup>

NC 1a OTf S +	[Me <sub>4</sub> N][TeCF <sub>3</sub> ] green LEDs (5 W) <b>2</b> DMF, 30-32 °C 1.4 equiv N <sub>2</sub> , time	NC 3a TeCF 3
Entry	Time (h)	Yield ( <b>3a</b> , %)
1	0	0
2	2	45
3	4	56
4	8	60
5	12	82

<sup>a</sup> Reaction conditions: **1a** (0.1 mmol), **2** (1.4 equiv), DMF (0.5 mL), green LEDs (5 W), 30-32 °C, N<sub>2</sub>, and 0-12 h. The yield was determined by <sup>19</sup>F NMR analysis of the reaction mixtures using PhOCF<sub>3</sub> as an internal standard.

**Table S6**. Photoinduced trifluoromethyltellurolation of different type of arylsulfonium salts with **2** in DMF under green LEDs (5 W) irradiation.<sup>a</sup>



<sup>a</sup> Reaction conditions: arylsulfonium salt (0.1 mmol), **2** (0.16 mmol), DMF (0.5 mL), green LEDs (5 W), 30-32 °C, N<sub>2</sub>, and 12 h. The yield was determined by HPLC using pure **3a** as an external standard (water / methanol = 20 / 80 (v/v), flow rate = 0.7 mL/min,  $\lambda_m = 253$  nm,  $t_R = 7.55$  min). <sup>b</sup> Besides **3a**, phenyl(trifluoromethyl)tellane (**3a'**, 19% yield) was also observed according to the <sup>19</sup>F NMR analysis of the reaction mixture using PhOCF<sub>3</sub> as an internal reference (30.0 mg, 0.185 mmol).

# 3. General procedures for the synthesis of arylsulfonium salts.<sup>3-6</sup>



**Procedure A**: Under a N<sub>2</sub> atmosphere, a 50 mL round-bottom flask was charged with thianthrene 5-oxides, phenoxathiine 10-oxide, or dibenzo[*b*,*d*]thiophene 5-oxides (1.1 mmol), arene (1.0 mmol), and MeCN (4 mL) with stirring, and cooled to -40 °C. Then, trifluoroacetic anhydride (TFAA, 0.42 mL, 3.0 mmol) and trifluoromethanesulfonic acid (TfOH, 0.26 mL, 3.0 mmol) were added. The mixture was reacted at -40 °C for 1 hour, warmed to room temperature overnight, diluted with DCM (10 mL), and neutralized with a saturated aqueous NaHCO<sub>3</sub> solution (10 mL). The organic layer was washed with an aqueous NaOTf solution (3 × 20 mL, 5% (w/w)), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated to dryness under reduced pressure. The residue was crystallized from a mixture of DCM and *tert*-butyl methyl ether (1:10, v/v) to

give the corresponding arylsulfonium triflate (1a-b, 1e, 1g-j, 1l, 1n-o, 4a, 5a, 5c-d, 7a, 8a, 9a, and 10a).



**Procedure B**: Under a N<sub>2</sub> atmosphere, a 50 mL round-bottomed flask was charged with thianthrene 5-oxide or dibenzo[b,d]thiophene 5-oxide (1.1 mmol), arene (1.0 mmol), and DCM (4 mL) with stirring, and cooled to -40 °C. Then, trifluoromethanesulfonic anhydride (0.3 mL, 1.8 mmol) was added. The mixture was reacted at -40 °C for 1 hour, warmed to room temperature overnight, diluted with DCM (10 mL), and neutralized with a saturated aqueous NaHCO<sub>3</sub> solution (10 mL). The organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated to dryness under reduced pressure. The residue was crystallized from a mixture of DCM and *tert*-butyl methyl ether (1:10, v/v) to give the corresponding arylsulfonium triflate (1c-d, 1f, 1k, 1m, 5b, and 5e-i).



**Procedure C**: Under a N<sub>2</sub> atmosphere, a 50 mL round-bottom flask was charged with diphenyl sulfoxide (1.1 mmol), arene (1.0 mmol), and MeCN (4 mL) with stirring, and cooled to -40 °C. Then, trifluoroacetic anhydride (TFAA, 0.42 mL, 3.0 mmol) and trifluoromethanesulfonic acid (TfOH, 0.26 mL, 3.0 mmol) were added. The mixture was reacted at -40 °C for 1 hour, warmed to room temperature overnight, diluted with DCM (10 mL), and neutralized with a saturated aqueous NaHCO<sub>3</sub> solution (10 mL). The organic layer was washed with an aqueous NaOTf solution (3 × 20 mL, 5% (w/w)), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated to dryness under reduced pressure. The residue was crystallized from a mixture of DCM and *tert*-butyl methyl ether (1:10, v/v) to give the corresponding arylsulfonium triflate (**6a**).



White solid (0.49 g, 85% yield from **Procedure B**), m.p.: 182.5-184.0 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.56 (d, *J* = 7.6 Hz, 2H), 7.84-7.79 (m, 4H), 7.74 (t, *J* = 6.8 Hz, 2H), 7.55 (d, *J* = 8.5 Hz, 2H), 7.44 (d, *J* = 8.4 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 7.12 (d, *J* = 8.5 Hz, 2H), 2.28 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -78.2 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  169.3, 151.4, 145.1, 136.6, 135.7, 135.3, 135.1, 130.4, 130.3, 129.0, 128.6, 128.3, 122.4, 122.3, 121.0 (q, *J* = 320.9 Hz), 118.6, 21.1. IR (KBr): 3081, 2998, 1755, 1608, 1484, 1448, 1375, 1270, 1197, 1155, 1029, 960, 913, 819, 768, 709, 673 cm<sup>-1</sup>. HRMS (ESI) m/z: [M]<sup>+</sup> Calcd for C<sub>26</sub>H<sub>19</sub>O<sub>2</sub>S<sub>2</sub> 427.0821; Found 427.0815.

5-(5-(2-Ethoxy-2-oxoethyl)-2-methylphenyl)-5*H*-thianthren-5-ium trifluoromethanesulfonate (**1d**)



Yellow solid (0.19 g, 35% yield from **Procedure B**), m.p.: 86.8-88.4 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.58 (d, *J* = 7.5 Hz, 2H), 7.86 (d, *J* = 7.6 Hz, 2H), 7.77 (t, *J* = 7.4 Hz, 2H), 7.73 (t, *J* = 7.2 Hz, 2H), 7.38 (d, *J* = 7.7 Hz, 1H), 7.33 (d, *J* = 7.7 Hz, 1H), 6.79 (s, 1H), 4.08 (q, *J* = 7.1 Hz, 2H), 3.46 (s, 2H), 2.69 (s, 3H), 1.20 (t, *J* = 7.2 Hz, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -78.2 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  170.1, 139.2, 137.5, 135.5, 134.8, 134.5, 134.0, 133.9, 130.4, 130.2, 129.5, 121.0, 120.6 (q, *J* = 321.3 Hz), 118.4, 61.3, 40.3, 20.3, 14.1. IR (KBr): 3075, 2985, 2937, 1732, 1569, 1445, 1369, 1266, 1224, 1157, 1030, 946, 835, 761, 638 cm<sup>-1</sup>. HRMS (ESI) m/z: [M]<sup>+</sup> Calcd for C<sub>23</sub>H<sub>21</sub>O<sub>2</sub>S<sub>2</sub> 393.0977; Found 393.0972.

5-(4-(2-(*N*-Benzylmethylsulfonamido)-5-nitrophenoxy)phenyl)-5*H*-thianthren-5-ium trifluoromethanesulfonate (**1g**)



Yellow solid (0.72 g, 94% yield from **Procedure A**), m.p.: 98.1-99.8 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.68 (d, *J* = 7.6 Hz, 2H), 7.91 (dd, *J* = 8.8 Hz, *J* = 2.5 Hz, 1H), 7.87 (dd, *J* = 7.6 Hz, *J* = 1.1 Hz, 2H), 7.83 (t, *J* = 7.3 Hz, 2H), 7.79 (t, *J* = 7.5 Hz, 2H), 7.62 (d, *J* = 2.4 Hz, 1H), 7.41 (d, *J* = 8.8 Hz, 1H), 7.32 (d, *J* = 8.9 Hz, 2H), 7.22-7.21 (m, 3H), 7.18 (m, 2H), 7.02 (d, *J* = 8.8 Hz, 2H), 4.78 (s, 2H), 2.96 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -78.1 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  159.3, 153.4, 147.7, 136.6, 136.4, 135.3, 135.1, 134.9, 133.4, 131.0, 130.5, 130.4, 128.8, 128.8, 128.4, 120.9 (q, *J* = 321.2 Hz), 120.7, 119.8, 119.2, 118.7, 114.6, 54.5, 40.5. IR (KBr): 3078, 2930, 1579, 1527, 1487, 1451, 1418, 1347, 1260, 1223, 1152, 1030, 951, 843, 762, 703, 637 cm<sup>-1</sup>. HRMS (ESI) m/z: [M]<sup>+</sup> Calcd for C<sub>32</sub>H<sub>25</sub>N<sub>2</sub>O<sub>5</sub>S<sub>3</sub> 613.0920; Found 613.0930.

5-(2-Methoxy-5-(2-oxopyrrolidine-1-carbonyl)phenyl)-5*H*-thianthren-5-ium trifluoromethanesulfonate (**1n**)



White solid (0.42 g, 72% yield from **Procedure A**), m.p.: 179.8-181.7 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.43 (d, *J* = 7.3 Hz, 2H), 7.88 (d, *J* = 8.3 Hz, 1H), 7.83 (d, *J* = 7.5 Hz, 2H), 7.77 (t, *J* = 7.2 Hz, 2H), 7.73 (t, *J* = 7.2 Hz, 2H), 7.10 (d, *J* = 8.6 Hz, 1H), 6.86 (s, 1H), 4.02 (s, 3H), 3.82 (t, *J* = 7.2 Hz, 2H), 2.51 (t, *J* = 7.2 Hz, 2H), 2.07 (m, 2H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -78.1 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  174.9, 167.2, 160.6, 137.7, 137.6, 135.4, 134.8, 130.7, 130.4, 130.1, 127.4, 120.9 (q, *J* = 320.0 Hz), 116.7, 112.7, 108.5, 57.5, 46.7, 33.1, 17.4. IR (KBr): 3075, 2998, 1727, 1668, 1597, 1570, 1498, 1452, 1363, 1317, 1274, 1224, 1148, 1031, 1002, 914, 843, 763, 702, 637 cm<sup>-1</sup>. HRMS (ESI) m/z: [M]<sup>+</sup> Calcd for C<sub>24</sub>H<sub>20</sub>NO<sub>3</sub>S<sub>2</sub> 434.0879; Found 434.0881.

5-(3-Iodo-4-methoxyphenyl)-5*H*-thianthren-5-ium trifluoromethanesulfonate (5h)



White solid (0.45 g, 80% yield from **Procedure B**), m.p.: 199.7-201.7 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.21 (d, *J* = 7.7 Hz, 2H), 8.09-8.07 (m, 3H), 7.84 (t, *J* = 7.5 Hz, 2H), 7.61 (t, *J* = 7.7 Hz, 2H), 7.49 (d, *J* = 2.0 Hz, 1H), 6.99 (d, *J* = 8.7 Hz, 1H), 3.90 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -78.1 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  163.6, 139.3, 138.6, 135.1, 134.6, 132.0, 131.8, 128.5, 124.3, 120.9 (q, *J* = 320.2 Hz), 116.1, 112.8, 88.6, 57.3. IR (KBr): 3084, 3023, 2951, 2846, 1570, 1475, 1465, 1431, 1392, 1266, 1156, 1081, 1030, 1011, 876, 812, 761, 635 cm<sup>-1</sup>. HRMS (ESI) m/z: [M]<sup>+</sup> Calcd for C<sub>19</sub>H<sub>14</sub>IOS 416.9805; Found 416.9794.

4. General procedures for the photoinduced trifluoromethyltellurolation of diverse arylsulfonium salts with [Me<sub>4</sub>N][TeCF<sub>3</sub>] (2).



**Procedure A**: In a nitrogen-filled glovebox, a sealed reaction vial was charged with 1 (0.1 mmol),  $[Me_4N][TeCF_3]$  (2, 37.9 mg, 0.14 mmol), and DMF (0.5 mL) with stirring. The reaction vial was taken out of glovebox and irradiated with green LEDs (5 W) at ambient temperature (30-32 °C) for 12 hours. The resulting mixture was diluted with ethyl acetate (5 mL), washed with water (2 × 5 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated to dryness under reduced pressure. The residue was purified by flash column chromatography or preparative TLC plate on silica gel using a mixture of petroleum ether and ethyl acetate as eluents to give the desired product (**3**).

**Procedure B**: In a nitrogen-filled glovebox, a sealed reaction vial was charged with 5 (0.1 mmol), [Me<sub>4</sub>N][TeCF<sub>3</sub>] (**2**, 37.9 mg, 0.14 mmol), and DMF (0.5 mL) with stirring. The reaction vial was taken out of glovebox and irradiated with green LEDs (5 W) at ambient temperature (30-32 °C) for 12 hours. The resulting mixture was diluted with ethyl acetate (5 mL), washed with water ( $2 \times 5$  mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated to dryness under reduced pressure. The residue

was purified by flash column chromatography or preparative TLC plate on silica gel using a mixture of petroleum ether and ethyl acetate as eluents to give the desired product (**3**).

4-(4-((Trifluoromethyl)tellanyl)phenoxy)benzonitrile (3a)

Yellow oil (32.4 mg, 83% yield from **Procedure A**), petroleum ether/ethyl acetate = 20:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.02 (d, *J* = 8.5 Hz, 2H), 7.66 (d, *J* = 8.7 Hz, 2H), 7.08 (d, *J* = 8.7 Hz, 2H), 7.02 (d, *J* = 8.5 Hz, 2H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -25.7 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  160.3, 157.3, 143.9, 134.3, 121.2, 119.0, 118.5, 107.2, 104.6, 102.7 (q, *J* = 353.6 Hz). IR (KBr): 3072, 2228, 1735, 1606, 1580, 1486, 1451, 1250, 1171, 1152, 1030, 873, 836, 759, 701, 637 cm<sup>-1</sup>. HRMS (APCI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>14</sub>H<sub>9</sub>F<sub>3</sub>NOTe 393.9693; Found 393.9686.

(4-(4-Nitrophenoxy)phenyl)(trifluoromethyl)tellane (3b)

Yellow oil (27.5 mg, 67% yield from **Procedure A**), petroleum ether/ethyl acetate = 20:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.25 (d, *J* = 9.2 Hz, 2H), 8.04 (d, *J* = 8.7 Hz, 2H), 7.09 (d, *J* = 9.2 Hz, 2H), 7.05 (d, *J* = 8.6 Hz, 2H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -25.6 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  162.0, 157.1, 143.9, 143.5, 126.1, 121.4, 118.2, 104.9, 102.8 (q, *J* = 354.3 Hz). IR (KBr): 3111, 3082, 1597, 1576, 1519, 1483, 1344, 1246, 1169, 1111, 1082, 1010, 877, 846, 750 cm<sup>-1</sup>. HRMS (ESI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>13</sub>H<sub>9</sub>F<sub>3</sub>NO<sub>3</sub>Te 413.9591; Found 413.9590.

(4-(2-Methyl-4-nitrophenoxy)phenyl)(trifluoromethyl)tellane (3c)



Yellow oil (25.6 mg, 60% yield from **Procedure B**), petroleum ether/ethyl acetate = 20:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.19 (d, *J* = 2.3 Hz, 1H), 8.05 (dd, *J* = 8.9 Hz, *J* = 2.6 Hz, 1H), 8.01 (d, *J* = 8.7 Hz, 2H),

6.96 (d, J = 8.7 Hz, 2H), 6.92 (d, J = 8.9 Hz, 1H), 2.38 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -25.8 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  159.9, 157.9, 144.0, 143.8, 130.8, 127.2, 123.4, 120.5, 118.0, 104.3, 102.9 (q, J = 352.5 Hz), 16.5. IR (KBr): 3074, 2929, 2854, 1732, 1696, 1576, 1520, 1482, 1344, 1247, 1212, 1169, 1083, 1010, 932, 844, 826, 802, 746 cm<sup>-1</sup>. HRMS (ESI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>14</sub>H<sub>11</sub>F<sub>3</sub>NO<sub>3</sub>Te 427.9748; Found 427.9752.

4'-((Trifluoromethyl)tellanyl)-[1,1'-biphenyl]-4-yl acetate (**3d**)



Yellow oil (14.6 mg, 36% yield from **Procedure A**), petroleum ether/ethyl acetate = 15:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.05 (d, *J* = 8.2 Hz, 2H), 7.59 (d, *J* = 8.6 Hz, 2H), 7.53 (d, *J* = 8.2 Hz, 2H), 7.19 (d, *J* = 8.6 Hz, 2H), 2.33 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -25.4 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  169.5, 150.9, 142.5, 142.1, 137.8, 128.7, 128.4, 122.2, 108.7, 102.8 (q, *J* = 353.4 Hz), 21.3. IR (KBr): 3060, 2925, 2853, 1756, 1645, 1604, 1518, 1483, 1371, 1214, 1197, 1168, 1085, 1002, 914, 824, 802, 723, 665, 606 cm<sup>-1</sup>. HRMS (APCI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>15</sub>H<sub>12</sub>F<sub>3</sub>O<sub>2</sub>Te 410.9846; Found 410.9841.

Ethyl 2-(4-methyl-3-((trifluoromethyl)tellanyl)phenyl)acetate (3e)

Yellow oil (20.0 mg, 54% yield from **Procedure A**), petroleum ether/ethyl acetate = 20:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.02 (s, 1H), 7.36 (d, *J* = 7.8 Hz, 1H), 7.31 (dd, *J* = 7.8 Hz, *J* = 1.6 Hz, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 3.58 (s, 2H), 2.60 (s, 3H), 1.25 (t, *J* = 7.1 Hz, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -24.8 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  171.1, 144.1, 143.7, 133.1, 132.1, 129.5, 114.6, 102.7 (q, *J* = 354.8 Hz), 61.0, 40.4, 27.6, 14.1. IR (KBr): 2981, 2961, 2927, 2854, 1736, 1646, 1594, 1483, 1448, 1369, 1334, 1253, 1223, 1156, 1083, 1033, 942, 827, 723, 669 cm<sup>-1</sup>. HRMS (APCI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>12</sub>H<sub>14</sub>F<sub>3</sub>O<sub>2</sub>Te 377.0003; Found 376.9996.

9-Methyl-3-((trifluoromethyl)tellanyl)-9H-carbazole (3f)



Light yellow solid (31.0 mg, 82% yield from **Procedure B**), m.p.:139.6-141.5 °C, petroleum ether as eluent for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.75 (s, 1H), 8.12 (d, *J* = 7.9 Hz, 1H), 8.09 (d, *J* = 8.4 Hz, 1H), 7.53 (t, *J* =7.5 Hz, 1H), 7.43 (d, *J* = 8.2 Hz, 1H), 7.36 (dd, *J* = 8.2 Hz, *J* = 2.1 Hz, 1H), 7.29 (t, *J* =7.5 Hz, 1H), 3.86 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -26.7 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  141.7, 141.3, 139.2, 135.0, 126.7, 124.7, 122.2, 120.7, 120.0, 110.2, 108.9, 102.7 (q, *J* = 354.6 Hz), 97.8, 29.3. IR (KBr): 3055, 2936, 1585, 1495, 1475, 1455, 1426, 1354, 1334, 1274, 1247, 1155, 1100, 1084, 1019, 887, 853, 802, 749, 727 cm<sup>-1</sup>. HRMS (APCI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>14</sub>H<sub>11</sub>F<sub>3</sub>NTe 379.9900; Found 379.9894.

2-Isobutoxy-5-((trifluoromethyl)tellanyl)benzonitrile (3g)



Yellow oil (14.1 mg, 38% yield from **Procedure A** or 21.9 mg, 59% yield from **Procedure B**), petroleum ether/ethyl acetate = 40:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.16 (s, 1H), 8.11 (d, *J* = 8.6 Hz, 1H), 6.91 (d, *J* = 8.7 Hz, 1H), 3.88 (d, *J* = 6.4 Hz, 2H), 2.19 (m, 1H), 1.08 (d, *J* = 6.7 Hz, 6H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -25.9 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  162.3, 147.8, 146.9, 114.9, 113.8, 104.3, 103.8 (q, *J* = 354.2 Hz), 98.6, 75.6, 28.1, 19.0. IR (KBr): 3074, 2965, 2934, 2877, 2229, 1585, 1562, 1492, 1469, 1387, 1292, 1268, 1135, 1083, 1013, 968, 899, 816, 724, 672 cm<sup>-1</sup>. HRMS (APCI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>12</sub>H<sub>13</sub>F<sub>3</sub>NOTe 374.0006; Found 373.9999.

(3-Fluoro-4-methoxyphenyl)(trifluoromethyl)tellane (3h)

Yellow oil (11.0 mg, 34% yield from **Procedure B**), petroleum ether as eluent for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.72-7.70 (m, 2H), 6.92 (m, 1H), 3.93 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -26.2 (s, 3F), -133.1 (m, 1F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  152.2 (d, *J* = 251.3 Hz), 149.9 (d, *J* = 10.4 Hz), 138.7 (d, *J* = 3.8 Hz), 129.1 (d, *J* = 18.4 Hz), 114.6, 102.7 (q, *J* = 357.3 Hz), 98.4 (d, *J* = 5.3 Hz),

56.2. IR (KBr): 2928, 2849, 1638, 1598, 1503, 1463, 1442, 1404, 1309, 1272, 1212, 1137, 1084, 1024, 869, 807, 760, 671 cm<sup>-1</sup>. HRMS (ESI) m/z:  $[M + H]^+$  Calcd for C<sub>8</sub>H<sub>7</sub>F<sub>4</sub>OTe 324.9490; Found 324.9489.

(3-Chloro-4-methoxyphenyl)(trifluoromethyl)tellane (3i)

Yellow oil (19.3 mg, 57% yield from **Procedure B**), petroleum ether as eluent for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.00 (d, *J* = 1.8 Hz, 1H), 7.86 (dd, *J* = 8.4 Hz, *J* = 1.8 Hz, 1H), 6.89 (d, *J* = 8.5 Hz, 1H), 3.94 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -26.1 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  157.1, 143.2, 141.9, 124.0, 113.6, 102.9 (q, *J* = 354.7 Hz), 99.5, 56.3. IR (KBr): 2980, 2944, 2848, 1575, 1485, 1461, 1440, 1375, 1293, 1254, 1084, 1065, 1015, 886, 808, 704, 670 cm<sup>-1</sup>. HRMS (ESI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>8</sub>H<sub>7</sub>ClF<sub>3</sub>OTe 340.9194; Found 340.9206.

(3-Bromo-4-methoxyphenyl)(trifluoromethyl)tellane (3j)

Yellow oil (21.4 mg, 56% yield from **Procedure B**), petroleum ether as eluent for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.17 (d, *J* = 1.8 Hz, 1H), 7.90 (dd, *J* = 8.4 Hz, *J* = 1.8 Hz, 1H), 6.86 (d, *J* = 8.4 Hz, 1H), 3.93 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -26.1 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  157.8, 145.9, 142.5, 113.3, 113.1, 102.7 (q, *J* = 355.4 Hz), 100.0, 56.3. IR (KBr): 3063, 3015, 2938, 2843, 1573, 1480, 1461, 1438, 1375, 1291, 1274, 1256, 1084, 1050, 1017, 888, 806, 722, 676 cm<sup>-1</sup>. HRMS (ESI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>8</sub>H<sub>7</sub>BrF<sub>3</sub>OTe 384.8689; Found 384.8701.

(3-Iodo-4-methoxyphenyl)(trifluoromethyl)tellane (3k)

Yellow oil (14.9 mg, 35% yield from **Procedure B**), petroleum ether as eluent for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.40 (s, 1H), 7.93 (d, *J* = 8.2 Hz, 1H), 6.78 (d, *J* = 8.2 Hz, 1H), 3.92 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -26.1 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  159.9, 151.8, 143.5, 112.3, 102.8 (q, *J* = 356.1

Hz), 100.9, 87.6, 56.4. IR (KBr): 3010, 2931, 2848, 1649, 1566, 1473, 1436, 1370, 1286, 1254, 1082, 1038, 887, 808, 723, 661 cm<sup>-1</sup>. HRMS (ESI) m/z:  $[M + H]^+$  Calcd for C<sub>8</sub>H<sub>7</sub>F<sub>3</sub>IOTe 432.8550; Found 432.8548.

2-Methoxy-5-((trifluoromethyl)tellanyl)phenyl trifluoromethanesulfonate (31)

Yellow oil (18.4 mg, 41% yield from **Procedure B**), petroleum ether/ethyl acetate = 20:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.95 (d, *J* = 8.5 Hz, 1H), 7.84 (s, 1H), 7.01 (d, *J* = 8.4 Hz, 1H), 3.96 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -26.0 (s, 3F), -73.7 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  153.5, 143.2, 138.8, 135.5, 118.7 (q, *J* = 321.3 Hz), 114.6, 102.8 (q, *J* = 355.2 Hz), 98.5, 56.3. IR (KBr): 3020, 2951, 2849, 1731, 1595, 1497, 1425, 1299, 1247, 1213, 1140, 1117, 1083, 1021, 905, 806, 748, 723, 639, 614 cm<sup>-1</sup>. HRMS (ESI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>9</sub>H<sub>7</sub>F<sub>6</sub>O<sub>4</sub>STe 454.9026; Found 454.9040.

(4-Methoxyphenyl)(trifluoromethyl)tellane  $(3m)^8$ 

Yellow oil (19.7 mg, 65% yield from **Procedure A**), petroleum ether as eluent for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.91 (d, *J* = 8.6 Hz, 2H), 6.86 (d, *J* = 8.6 Hz, 2H), 3.83 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -26.5 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  161.4, 143.6, 115.8, 102.5 (q, *J* = 352.8 Hz), 99.6, 55.2.

N-Benzyl-N-(4-nitro-2-(4-

((trifluoromethyl)tellanyl)phenoxy)phenyl)methanesulfonamide (**3n**)

Yellow oil (35.0 mg, 59% yield from **Procedure A**), petroleum ether/ethyl acetate = 2:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.07 (d, *J* = 8.5 Hz, 2H), 7.87 (dd, *J* = 8.7 Hz, *J* = 2.4 Hz, 1H), 7.67 (d, *J* = 2.3 Hz, 1H),  $\delta$  7.39 (d, *J* = 8.8 Hz, 1H), 7.27-7.26 (m, 5H), 7.00 (d, *J* = 8.4 Hz, 2H), 4.88 (s, 2H), 3.04 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -25.4 (s, 3F). <sup>13</sup>C NMR (126 MHz,

CDCl<sub>3</sub>)  $\delta$  156.7, 154.6, 148.1, 144.3, 135.4, 135.3, 134.3, 129.0, 128.9, 128.6, 121.0, 118.8, 113.6, 105.7, 103.0 (q, *J* = 352.9 Hz), 54.4, 40.8. IR (KBr): 3086, 3032, 2929, 2854, 1579, 1528, 1484, 1418, 1347, 1246, 1216, 1154, 1084, 961, 876, 841, 761, 731, 701 cm<sup>-1</sup>. HRMS (APCI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>21</sub>H<sub>18</sub>F<sub>3</sub>N<sub>2</sub>O<sub>5</sub>STe 596.9945; Found 596.9934.

Methyl 2-(2-fluoro-4'-((trifluoromethyl)tellanyl)-[1,1'-biphenyl]-4-yl)propanoate (30)



Yellow oil (30.0 mg, 66% yield from **Procedure A**), petroleum ether/ethyl acetate = 10:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.05 (d, *J* = 8.2 Hz, 2H), 7.51 (d, *J* = 8.3 Hz, 2H), 7.39 (t, *J* = 8.0 Hz, 1H), 7.18-7.13 (m, 2H), 3.77 (q, *J* = 7.1 Hz, 1H), 3.71 (s, 3H), 1.55 (d, *J* = 7.2 Hz, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -25.3 (s, 3F), -117.2 (m, 1F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  174.3, 159.7 (d, *J* = 248.7 Hz), 142.7 (d, *J* = 7.8 Hz), 141.5, 137.5, 130.7 (d, *J* = 3.6 Hz), 130.3 (d, *J* = 3.1 Hz), 126.7 (d, *J* = 13.4 Hz), 123.8 (d, *J* = 3.4 Hz), 115.4 (d, *J* = 23.5 Hz), 109.0, 102.8 (q, *J* = 353.9 Hz), 52.3, 45.0, 18.4. IR (KBr): 3062, 2982, 2952, 2852, 1737, 1624, 1572, 1513, 1483, 1428, 1389, 1335, 1199, 1172, 1085, 1005, 921, 875, 820, 736, 723, 671 cm<sup>-1</sup>. HRMS (APCI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>17</sub>H<sub>15</sub>F<sub>4</sub>O<sub>2</sub>Te 457.0065; Found 457.0050.

Methyl 5-(2,5-dimethyl-4-((trifluoromethyl)tellanyl)phenoxy)-2,2dimethylpentanoate (**3p**)



Yellow oil (30.8 mg, 67% yield from **Procedure A**), petroleum ether/ethyl acetate = 20:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.84 (s, 1H), 6.80 (s, 1H), 3.95 (t, *J* = 6.0 Hz, 2H), 3.66 (s, 3H), 2.59 (s, 3H), 2.17 (s, 3H), 1.76-1.70 (m, 4H), 1.22 (s, 6H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -25.9 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  178.2, 159.6, 145.9, 144.6, 126.0, 111.9, 103.7, 102.6 (q, *J* = 355.7 Hz), 67.9, 51.7, 42.1, 37.0, 28.4, 25.2, 25.0, 15.3. IR (KBr): 2952, 2927, 2874, 1732, 1596, 1557, 1493, 1474, 1384, 1362, 1304, 1246, 1198, 1149, 1083, 1047, 888,

842, 722 cm<sup>-1</sup>. HRMS (APCI) m/z:  $[M + H]^+$  Calcd for  $C_{17}H_{24}F_3O_3Te$  463.0734; Found 463.0723.

Methyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-6-((trifluoromethyl)tellanyl)-1*H*-indol-3-yl)acetate (**3q**)



Light yellow solid (26.1 mg, 46% yield from **Procedure A**), m.p.: 130.1-132.1 °C, petroleum ether/ethyl acetate = 10:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.66 (d, *J* = 8.5 Hz, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 7.24 (s, 1H), 6.98 (s, 1H), 3.90 (s, 3H), 3.71 (s, 3H), 3.68 (s, 2H), 2.45 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -25.6 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  171.3, 168.3, 155.1, 139.9, 137.1, 133.5, 132.6, 132.0, 131.2, 129.4, 122.9, 112.4, 103.5 (q, *J* = 353.1 Hz), 99.3, 98.9, 56.8, 52.3, 30.3, 13.3. IR (KBr): 3090, 3067, 2951, 2930, 2850, 1738, 1687, 1592, 1462, 1419, 1400, 1350, 1306, 1264, 1223, 1169, 1089, 1014, 933, 836, 754, 737, 649 cm<sup>-1</sup>. HRMS (APCI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>21</sub>H<sub>18</sub>ClF<sub>3</sub>NO<sub>4</sub>Te 569.9933; Found 569.9916.

Isopropyl 2-(4-(4-chlorobenzoyl)-2-((trifluoromethyl)tellanyl)phenoxy)-2methylpropanoate (**3r**)



Yellow oil (45.6 mg, 82% yield from **Procedure A**), petroleum ether/ethyl acetate = 10:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.19 (s, 1H), 7.74 (dd, J = 8.6 Hz, J = 1.8 Hz, 1H), 7.71 (d, J = 8.5 Hz, 2H), 7.46 (d, J = 8.5 Hz, 2H), 6.79 (d, J = 8.7 Hz, 1H), 5.07 (m, 1H), 1.69 (s, 6H), 1.20 (d, J = 6.3 Hz, 6H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -24.8 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  193.4, 172.3, 158.7, 139.7, 139.0, 135.8, 132.5, 132.4, 131.4, 128.8, 117.4, 114.2, 104.2 (q, J = 353.9 Hz), 81.6, 69.9, 25.4, 21.6. IR (KBr): 3067, 2984, 2939, 2877, 1786, 1735, 1657, 1588, 1472, 1389, 1290, 1265, 1179, 1147, 1090, 960, 941, 849, 823, 760, 681 cm<sup>-1</sup>. HRMS (APCI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>21</sub>H<sub>21</sub>ClF<sub>3</sub>O<sub>4</sub>Te

559.0137; Found 559.0124.

2-((1-(4-((Trifluoromethyl)tellanyl)phenoxy)propan-2-yl)oxy)pyridine (3s)



Yellow oil (30.1 mg, 58% yield from **Procedure A**), petroleum ether/ethyl acetate = 10:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.16 (dd, *J* = 4.9 Hz, *J* = 1.2 Hz, 1H), 7.89 (d, *J* = 8.7 Hz, 2H), 7.57 (m, 1H), 7.00-6.95 (m, 4H), 6.87-6.85 (m, 3H), 6.75 (d, *J* = 8.3 Hz, 1H), 5.60 (m, 1H), 4.21 (dd, *J* = 9.9 Hz, *J* = 5.3 Hz, 1H), 4.09 (dd, *J* = 9.9 Hz, *J* = 4.9 Hz, 1H), 1.49 (d, *J* = 6.4 Hz, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -26.2 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  163.3, 160.9, 156.1, 148.9, 146.9, 143.7, 138.9, 121.7, 118.6, 116.9, 116.1, 111.8, 102.7 (q, *J* = 354.5 Hz), 101.4, 71.2, 69.3, 17.1. IR (KBr): 3057, 2979, 2932, 2873, 1595, 1571, 1504, 1483, 1471, 1433, 1397, 1309, 1286, 1229, 1197, 1171, 1083, 1045, 990, 957, 873, 825, 779, 723 cm<sup>-1</sup>. HRMS (APCI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>21</sub>H<sub>19</sub>F<sub>3</sub>NO<sub>3</sub>Te 520.0374; Found 520.0369.

Ethyl 2-(4-chloro-2-((trifluoromethyl)tellanyl)phenoxy)-2-methylpropanoate (3t)



Yellow oil (40.2 mg, 92% yield from **Procedure A**), petroleum ether/ethyl acetate = 20:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.63 (d, *J* = 2.3 Hz, 1H), 7.18 (dd, *J* = 8.7 Hz, *J* = 2.4 Hz, 1H), 6.67 (d, *J* = 8.8 Hz, 1H), 4.23 (q, *J* = 7.2 Hz, 2H), 1.62 (s, 6H), 1.24 (t, *J* = 7.1 Hz, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -25.0 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  173.3, 153.4, 135.5, 129.5, 129.1, 116.5, 109.6, 104.6 (q, *J* = 355.6 Hz), 81.4, 62.0, 25.3, 14.2. IR (KBr): 2988, 2940, 1739, 1573, 1490, 1464, 1386, 1368, 1283, 1235, 1179, 1141, 1089, 1038, 1023, 966, 814, 723, 654 cm<sup>-1</sup>. HRMS (APCI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>13</sub>H<sub>15</sub>ClF<sub>3</sub>O<sub>3</sub>Te 440.9719; Found 440.9709.

1-(4-Methoxy-3-((trifluoromethyl)tellanyl)benzoyl)pyrrolidin-2-one (3u)



Yellow oil (31.0 mg, 75% yield from **Procedure A**), petroleum ether/ethyl acetate = 10:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.06 (d, *J* = 1.9 Hz, 1H), 7.70 (dd, *J* = 8.6 Hz, *J* = 2.1 Hz, 1H), 6.92 (d, *J* = 8.5 Hz, 1H), 3.96-3.93 (m, 5H), 2.62 (t, *J* = 7.9 Hz, 2H), 2.15 (m, 2H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -24.5 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  174.7, 169.1, 162.0, 140.1, 133.3, 128.6, 109.2, 103.6 (q, *J* = 353.9 Hz), 102.7, 56.6, 46.9, 33.5, 17.8. IR (KBr): 3080, 2926, 2851, 1741, 1669, 1591, 1487, 1460, 1442, 1362, 1311, 1267, 1188, 1084, 1044, 1015, 915, 890, 822, 805, 764, 671 cm<sup>-1</sup>. HRMS (APCI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>13</sub>H<sub>13</sub>F<sub>3</sub>NO<sub>3</sub>Te 417.9904; Found 417.9896.

(2-Ethoxy-5-(2-methyl-1-((3-phenoxybenzyl)oxy)propan-2-

yl)phenyl)(trifluoromethyl)tellane (**3v**)

Yellow oil (37.0 mg, 65% yield from **Procedure A**), petroleum ether/ethyl acetate = 40:1 (v/v) as eluents for column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.73 (d, *J* = 2.2 Hz, 1H), 7.35-7.30 (m, 3H), 7.27 (t, *J* = 7.8 Hz, 1H), 7.11 (t, *J* = 7.4 Hz, 1H), 7.02-6.98 (m, 3H), 6.93-6.89 (m, 2H), 6.79 (d, *J* = 8.7 Hz, 1H), 4.45 (s, 2H), 4.07 (q, *J* = 7.0 Hz, 2H), 3.42 (s, 2H), 1.41 (t, *J* = 7.0 Hz, 3H), 1.31 (s, 6H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)  $\delta$  -24.8 (s, 3F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  157.5, 157.4, 156.4, 142.2, 141.0, 135.3, 129.9, 129.7, 128.5, 127.2, 123.4, 122.2, 119.1, 117.9, 117.8, 114.1, 103.6 (q, *J* = 354.8 Hz), 80.1, 72.9, 65.0, 38.8, 26.3, 14.9. IR (KBr): 3065, 3039, 2977, 2931, 2870, 1584, 1488, 1445, 1393, 1361, 1251, 1215, 1162, 1088, 1049, 960, 927, 877, 812, 756, 723, 692 cm<sup>-1</sup>. HRMS (APCI) m/z: [M + H]<sup>+</sup> Calcd for C<sub>26</sub>H<sub>28</sub>F<sub>3</sub>O<sub>3</sub>Te 575.1047; Found 575.1037.

## 5. The control experiments for mechanistic insights.

5.1. Photoinduced trifluoromethyltellurolation of 1a with 2 in DMF in the presence of radical traps under green LEDs irradiation.



In a nitrogen-filled glovebox, a sealed reaction vial was charged with **1a** (56.0 mg, 0.1 mmol), [Me<sub>4</sub>N][TeCF<sub>3</sub>] (**2**, 37.9 mg, 0.14 mmol), 1,1-diphenylethylene (54.1 mg, 0.3 mmol), and DMF (0.5 mL) with stirring. The reaction vial was taken out of the glovebox and irradiated with green LEDs (5 W) at ambient temperature (30-32 °C) for 12 h. The resulting mixture was purified by preparative TLC plate on silica gel using a mixture of petroleum ether and ethyl acetate = 20 : 1 (v/v) as eluents to give **3a** as a yellow oil (20.3 mg, 52%).



In a nitrogen-filled glovebox, a sealed reaction vial was charged with **1a** (56.0 mg, 0.1 mmol), [Me<sub>4</sub>N][TeCF<sub>3</sub>] (**2**, 37.9 mg, 0.14 mmol), 2,2,6,6-tetramethylpiperidin-1-oxyl (TEMPO, 46.9 mg, 0.3 mmol), and DMF (0.5 mL) with stirring. The reaction vial was taken out of the glovebox and irradiated with green LEDs (5 W) at ambient temperature (30-32 °C) for 12 hours. The resulting mixture was purified by preparative TLC plate on silica gel using a mixture of petroleum ether and ethyl acetate = 20 : 1 (v/v) as eluents to give **3a** (16.0 mg, 41%) and **11** (10.5 mg, 30%) as yellow oils

#### 4-(4-((2,2,6,6-Tetramethylpiperidin-1-yl)oxy)phenoxy)benzonitrile (11)



Yellow oil. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.56 (d, J = 8.5 Hz, 2H),  $\delta$  7.21 (d, J = 8.4 Hz, 2H),  $\delta$  6.95 (d, J = 8.5 Hz, 2H),  $\delta$  6.91 (d, J = 8.5 Hz, 2H), 1.63-1.59 (m, 5H), 1.42 (m, 1H), 1.24 (s, 6H), 1.03 (s, 6H). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  162.8, 161.1, 147.2, 134.2, 121.1, 119.2, 117.2, 115.2, 105.1, 60.6, 39.9, 32.7, 20.6, 17.1. IR (KBr):

3002, 2966, 2916, 2871, 2221, 1645, 1607, 1594, 1489, 1379, 1363, 1246, 1210, 1182, 1165, 1144, 1092, 1049, 954, 936, 874, 854, 835, 799, 720, 635 cm<sup>-1</sup>. HRMS (ESI) m/z:  $[M + H]^+$  Calcd for C<sub>22</sub>H<sub>27</sub>N<sub>2</sub>O<sub>2</sub> 351.2067; Found 351.2067.

### 5.2. The light on-off experiment.



The light on-off experiments were carried out under the standard conditions by using a reaction of **1a** (0.6 mmol) with [Me<sub>4</sub>N][TeCF<sub>3</sub>] (**2**, 0.84 mmol) in DMF (3.0 mL). The green light was switched on and off with the intervals of 20 minutes. The reaction mixture was monitored by HPLC after each period using pure **3a** as an external standard (water / methanol = 20 / 80 (v/v), flow rate = 0.7 mL/min,  $\lambda_m = 253$  nm,  $t_R = 7.55$  min). The yields of **3a** for each period were reported in the following figure.





5.3. <sup>19</sup>F NMR analysis of the standard reaction mixtures at different times.



**Procedure**: In a nitrogen-filled glovebox, each of the five sealed reaction vials was charged with **1a** (0.1 mmol),  $[Me_4N]$ [TeCF<sub>3</sub>] (**2**, 0.14 mmol), and DMF (0.5 mL) with stirring. The reaction vials were taken out of glovebox and irradiated with green LEDs (5 W) at ambient temperature (30-32 °C) for 0, 2, 4, 8, and 12 hours, respectively. The reaction mixtures (under N<sub>2</sub>) were analyzed by <sup>19</sup>F NMR spectroscopy using PhOCF<sub>3</sub> as an internal standard. The yields of **3a** were calculated according to PhOCF<sub>3</sub>.

**Figure S2**. The <sup>19</sup>F NMR spectrum of  $[Me_4N]$ [TeCF<sub>3</sub>] (37.9 mg, 0.14 mmol) in DMF (0.5 mL) at ambient temperature using PhOCF<sub>3</sub> (16.2 mg, 0.10 mmol) as an internal standard.



**Figure S3**. The <sup>19</sup>F NMR spectrum of a mixture of **1a** (56.0 mg, 0.1 mmol) and  $[Me_4N]$ [TeCF<sub>3</sub>] (37.9 mg, 0.14 mmol) in DMF (0.5 mL) at ambient temperature with no light irradiation using PhOCF<sub>3</sub> (27.5 mg, 0.17 mmol) as an internal standard.



Figure S4. The combined <sup>19</sup>F NMR spectra of Figure S2 and Figure S3.



**Figure S5**. The <sup>19</sup>F NMR spectrum of a mixture of **1a** (56.0 mg, 0.1 mmol) and  $[Me_4N][TeCF_3]$  (37.9 mg, 0.14 mmol) in DMF (0.5 mL) at ambient temperature under green LEDs (5 W) irradiation for 2 h using PhOCF<sub>3</sub> (17.5 mg, 0.11 mmol) as an internal standard. 46% yield of **3a** was determined.



**Figure S6**. The <sup>19</sup>F NMR spectrum of a mixture of **1a** (56.0 mg, 0.1 mmol) and  $[Me_4N]$ [TeCF<sub>3</sub>] (37.9 mg, 0.14 mmol) in DMF (0.5 mL) at ambient temperature under green LEDs (5 W) irradiation for 4 h using PhOCF<sub>3</sub> (21.0 mg, 0.13 mmol) as an internal standard. 56% yield of **3a** was determined.



**Figure S7**. The <sup>19</sup>F NMR spectrum of a mixture of **1a** (56.0 mg, 0.1 mmol) and  $[Me_4N]$ [TeCF<sub>3</sub>] (37.9 mg, 0.14 mmol) in DMF (0.5 mL) at ambient temperature under

green LEDs (5 W) irradiation for 8 h using PhOCF<sub>3</sub> (22.0 mg, 0.14 mmol) as an internal standard. 60% yield of **3a** was determined.



**Figure S8**. The <sup>19</sup>F NMR spectrum of a mixture of **1a** (56.0 mg, 0.1 mmol) and  $[Me_4N]$ [TeCF<sub>3</sub>] (37.9 mg, 0.14 mmol) in DMF (0.5 mL) at ambient temperature under green LEDs (5 W) irradiation for 12 h using PhOCF<sub>3</sub> (17.0 mg, 0.11 mmol) as an internal standard. 82% yield of **3a** was determined.





Figure S9. The combined <sup>19</sup>F NMR spectra of the above reaction mixtures.

#### 5.4. The UV-visible absorption spectra of the reactants and their mixtures.

All UV-visible absorption experiments were performed on an AOE instrument (UVvis S22 Spectrophotometer A360) using sealed standard quartz cuvettes (l = 1.0 cm). The samples were prepared in a nitrogen-filled glovebox. The UV-visible spectra of 5-(4-(4-cyanophenoxy)phenyl)-5*H*-thianthren-5-ium trifluoromethanesulfonate (**1a**, 0.01 M), [Me<sub>4</sub>N][TeCF<sub>3</sub>] (**2**, 0.01 M), and a mixture of **1a** (0.01 M)/**2** (0.01 M) in MeCN were recorded, respectively.

Figure S10. The combined UV-visible absorption spectra of 1a (0.01 M), 2 (0.01 M), and a mixture of 1a (0.01 M)/2 (0.01 M) using MeCN as the solvent.



# 6. Decomposition of [Me<sub>4</sub>N][TeCF<sub>3</sub>] (2) under light radiation.

**Procedure**: In a nitrogen-filled glovebox, 37.9 mg of pure [Me<sub>4</sub>N][TeCF<sub>3</sub>] solid and a solution of [Me<sub>4</sub>N][TeCF<sub>3</sub>] (37.9 mg) in CD<sub>3</sub>CN (0.5 mL) were placed in the reaction vials, respectively. The reaction vials were sealed, taken out of the glovebox, and irradiated with green LEDs (5 W) at ambient temperature for 0, 2, 4, 6, 8, 10, 12, 24, 36, and 48 hours. The color change of the samples was recorded (see the following pictures). The samples irradiated after 48 h were also analyzed by <sup>1</sup>H and <sup>19</sup>F NMR spectroscopy.

*Note*: The color change and NMR analysis of the irradiated samples indicated that the [Me<sub>4</sub>N][TeCF<sub>3</sub>] reagent was slowly degraded with time under the green LEDs (5 W) radiation.

**Figure S11**. The color change of pure [Me<sub>4</sub>N][TeCF<sub>3</sub>] solid and its solution in CD<sub>3</sub>CN under green LEDs irradiation with times.

A (left for each picture): The pure [Me<sub>4</sub>N][TeCF<sub>3</sub>] solid in the reaction vial.

**B** (right for each picture): A solution of [Me<sub>4</sub>N][TeCF<sub>3</sub>] in CD<sub>3</sub>CN in the reaction vial.



Figure S12. The <sup>1</sup>H NMR spectrum of  $[Me_4N]$ [TeCF<sub>3</sub>] (A) after irradiated for 48 h.



Figure S13. The  ${}^{19}$ F NMR spectrum of [Me<sub>4</sub>N][TeCF<sub>3</sub>] (A) after irradiated for 48 h.



**Figure S14**. The <sup>1</sup>H NMR spectrum of [Me<sub>4</sub>N][TeCF<sub>3</sub>] (**B**) after irradiated for 48 h in CD<sub>3</sub>CN.



Figure S15. The <sup>19</sup>F NMR spectrum of [Me<sub>4</sub>N][TeCF<sub>3</sub>] (B) after irradiated for 48 h in



#### 7. The *in vitro* anti-tumor activity evaluation.

A 100 µL aliquot of cell suspension (5 × 10<sup>4</sup> cells/mL) from either 4T1 or HeLa cell lines was evenly distributed into the wells of a 96-well plate and incubated for 24 hours. After this incubation, the culture medium was replaced with 100 µL of drugcontaining (**3a-3v**) medium at various concentrations, and the cells were further incubated for an additional 24 hours. Subsequently, 100 µL of phosphate-buffered saline (PBS) was added twice to each well, followed by 100 µL of MTT solution. The plates were incubated for 3 hours to allow for the conversion of MTT into formazan. Finally, 100 µL of dimethyl sulfoxide (DMSO) was added to each well to dissolve the formazan crystals. The optical density (OD) was measured at 570 nm using a microplate reader. Each condition was performed in triplicate (five wells per group), and the experiment was repeated three times to ensure reproducibility. The halfmaximal inhibitory concentration (IC<sub>50</sub>) values were calculated to assess the antitumor activity of the tested compounds.

The IC<sub>50</sub> values were calculated using GraphPad Prism 10.1.2 software and determined by the concentration causing a half-maximal percent activity. Data are expressed as the mean  $\pm$  standard deviation (SD) from three independent experiments.

Statistical differences between groups were determined using the Student's t-test, with p < 0.05 considered statistically significant.

Compounds **3m-SeCF**<sup>3</sup> and **3m-SCF**<sup>3</sup> employed for the comparison with **3m** were synthesized according to the literature.<sup>9</sup> Compounds **3m-OCF**<sup>3</sup> and **3m-H** employed for the comparison with **3m** were purchased from the commercial sources and used without further purification.

Commound ID	Antitumor activities (IC <sub>50</sub> , µM)	
	HeLa	4T1
<b>3</b> a	$36.86 \pm 1.15$	$69.30 \pm 1.30$
3b	$193.4 \pm 1.11$	$168.2 \pm 1.04$
3c	$55.17 \pm 1.34$	$195.4 \pm 1.37$
3d	$43.64 \pm 1.32$	$134.1\pm1.14$
3e	$118.4 \pm 1.11$	$16.81 \pm 1.38$
<b>3f</b>	$102.7\pm1.06$	$93.47 \pm 1.03$
3g	$19.75\pm1.40$	$177.4 \pm 1.21$
3h	$8.885 \pm 1.14$	$40.87 \pm 1.04$
<b>3</b> i	$20.95 \pm 1.09$	$46.14 \pm 1.03$
3ј	$4.027 \pm 1.29$	$43.88 \pm 1.04$
3k	$26.84 \pm 1.24$	$62.08 \pm 1.08$
31	$5.040 \pm 1.32$	$45.83 \pm 1.14$
3m	$13.34 \pm 1.05$	$16.37 \pm 1.08$
<b>3</b> n	$17.17 \pm 1.28$	$352.3 \pm 1.10$
30	$715.1\pm1.05$	$740.7 \pm 1.09$
<b>3</b> p	$167.9 \pm 1.14$	$63.29 \pm 1.04$
3q	$29.02 \pm 1.03$	$200.2 \pm 1.12$
3r	$745.2\pm1.04$	$100.2 \pm 1.23$
3s	$421.1\pm1.04$	$126.8\pm1.04$
3t	$594.7 \pm 1.09$	$106.9 \pm 1.04$
<b>3</b> u	$1.093 \pm 1.09$	$98.05 \pm 1.26$
3v	$122.2\pm1.05$	$53.11 \pm 1.03$
3m-SeCF <sub>3</sub>	$443.8\pm1.02$	$707.6 \pm 1.07$

Table S7. The antitumor activity of compounds (3a-3v).

3m-SCF <sub>3</sub>	$539.8 \pm 1.02$	$653.3 \pm 1.06$
3m-OCF <sub>3</sub>	$595.7 \pm 1.07$	$1995 \pm 1.17$
3m-H	$574.7 \pm 1.06$	$5652 \pm 1.10$

The plots of IC50-titration of compounds (3a-3v).











The plots of IC50-titration of compounds 3m, 3m-H, and 3m-XCF3 (X = Se, S, O).




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#### 8. NMR spectra of the products.

<sup>1</sup>H NMR spectrum of 1c



# <sup>19</sup>F NMR spectrum of **1c**



---78.15



<sup>1</sup>H NMR spectrum of **1d** 











### <sup>19</sup>F NMR spectrum of **1g**







<sup>19</sup>F NMR spectrum of **1n** 











<sup>1</sup>H NMR spectrum of **3a** 











<sup>1</sup>H NMR spectrum of **3c** 





90 80 70 60 50 40 30 20 10 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280 fl (ppm)



S50











<sup>1</sup>H NMR spectrum of **3g** 







S57



S58



S59













# <sup>1</sup>H NMR spectrum of **3n**





S67

6 fl (ppm)

8 8 8 8

14

13

12

11

10

9

<sup>4</sup> 3.00

3

2

i

2

-1

0

-2



-60 -80 -100 -120 fl (ppm)















<sup>1</sup>H NMR spectrum of **3r** 



S72


<sup>1</sup>H NMR spectrum of **3s** 















S76







<sup>19</sup>F NMR spectrum of **3v** 





<sup>1</sup>H NMR spectrum of 11













The <sup>19</sup>F NMR spectrum of the reaction mixture of 6a and 2 under the standard conditions using 30.0 mg of PhOCF<sub>3</sub> as an internal standard.

