

# **Oxidative addition of cyclic 1-oxa-5,6-ditellurasprirooctane to platinum(0) complexes**

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## 1. Experimental

### 1.1 General Methods

All reactions were performed under a dry argon atmosphere using standard Schlenk techniques. 1-oxa-5,6-ditellurasprirooctane (**1**), as well as [Pt(PPh<sub>3</sub>)<sub>2</sub>(η<sup>2</sup>-nb)] (**2**) and [Pt(dppn)(η<sup>2</sup>-nb)] (**5**) were prepared according to the literature procedures.<sup>1,2</sup> <sup>1</sup>H and <sup>13</sup>C{<sup>1</sup>H} spectra were recorded on a Bruker AC400 spectrometer, and <sup>31</sup>P{<sup>1</sup>H}, <sup>125</sup>Te, and <sup>195</sup>Pt- spectra on a Bruker DPX400 spectrometer operating at 400.25, 100.65, and 161.98, 126.23, and 85.53 MHz, respectively. The typical respective spectral widths were 3.53, 12.56, 11.36, 126.58, and 85.47 kHz and pulse widths 10.08, 9.85, 7.50, 10.00, and 10.00 μs. The pulse delays were 1.00, 2.00, 2.00, 1.60, and 0.01 s for <sup>1</sup>H, <sup>13</sup>C{<sup>1</sup>H}, <sup>31</sup>P{<sup>1</sup>H}, <sup>125</sup>Te, and <sup>195</sup>Pt, respectively. Orthophosphoric acid (85%), a solution of Ph<sub>2</sub>Te<sub>2</sub> in CDCl<sub>3</sub>, and a D<sub>2</sub>O solution of [PtCl<sub>6</sub>]<sup>2-</sup> were used as external standards. <sup>1</sup>H and <sup>13</sup>C{<sup>1</sup>H} NMR spectra were recorded in CD<sub>2</sub>Cl<sub>2</sub> that also served as an internal <sup>2</sup>H lock. <sup>31</sup>P{<sup>1</sup>H}, <sup>125</sup>Te, and <sup>195</sup>Pt NMR spectra were recorded unlocked in CH<sub>2</sub>Cl<sub>2</sub>. <sup>31</sup>P and <sup>195</sup>Pt chemical shifts are reported relative to external standards and <sup>125</sup>Te to relative to neat Me<sub>2</sub>Te [ $\delta$  (Me<sub>2</sub>Te) =  $\delta$  (Ph<sub>2</sub>Te<sub>2</sub>) + 422].<sup>3</sup> <sup>1</sup>H-<sup>1</sup>H COSY and <sup>1</sup>H-<sup>13</sup>C HSQC experiments were recorded on a Bruker AC400 spectrometer in CD<sub>2</sub>Cl<sub>2</sub>.

### 1.2 Syntheses of [Pt<sub>2</sub>(Te<sub>2</sub>C<sub>5</sub>H<sub>8</sub>O)<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub>] (**4**)

A solution of **1** (30 mg, 0.09 mmol) dissolved in dry toluene (5 ml) was added with a syringe to a stirred solution of **2** (75 mg, 0.09 mmol) in dry toluene (5 ml) at -10°C under an inert atmosphere. The reaction mixture was stirred for three hours at room temperature and concentrated by partial removal of the solvent. The product **4** was precipitated by addition of pentane. The yellow solid (60 mg, 84 %) was filtered and washed with pentane. Found: C 35.22, H 2.98% Anal. Calc. for C<sub>46</sub>H<sub>46</sub>O<sub>2</sub>P<sub>2</sub>Pt<sub>2</sub>Te<sub>4</sub> (1593.4): C 34.67, H 2.91 %; <sup>31</sup>P NMR (400 MHz, CH<sub>2</sub>Cl<sub>2</sub>, H<sub>3</sub>PO<sub>4</sub>):  $\delta$  = 13.8 ppm (<sup>1</sup>J<sub>Pt-P</sub> = 3355 Hz), <sup>195</sup>Pt NMR (400 MHz, CH<sub>2</sub>Cl<sub>2</sub>, Na<sub>2</sub>PtCl<sub>6</sub>):  $\delta$  = -5748 ppm (<sup>1</sup>J<sub>Pt-P</sub> = 3355 Hz), <sup>125</sup>Te NMR (400 MHz, CH<sub>2</sub>Cl<sub>2</sub>, (CH<sub>3</sub>)<sub>2</sub>Te):  $\delta$  = 75, -313 ppm, <sup>1</sup>H NMR (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>):  $\delta$  = 7.2 – 7.8 ppm (30 H, m), 4.1 (1 H, d, <sup>2</sup>J<sub>H-H</sub> = 5.6 Hz), 4.0 (1 H, d, <sup>2</sup>J<sub>H-H</sub> = 5.8 Hz), 3.7 (1 H, d, <sup>2</sup>J<sub>H-H</sub> = 5.6 Hz), 3.6 (1 H, d, <sup>2</sup>J<sub>H-H</sub> = 5.6 Hz), 3.4 (1 H, d, <sup>2</sup>J<sub>H-H</sub> = 12.8 Hz, <sup>3</sup>J<sub>H-Pt</sub> = 71 Hz), 2.9 (1 H, d, <sup>2</sup>J<sub>H-H</sub> = 12.8 Hz, <sup>3</sup>J<sub>H-Pt</sub> = 36 Hz), 2.7 (1 H, m), and 2.3 (1 H, m). <sup>13</sup>C NMR (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>):  $\delta$  = 7.1 (s), 23.6 (s), 43.5 (s), 81.4 (s), 82.3 (s), 128.3 (d, J<sub>C-P</sub> = 10.6 Hz), 131.2 (s), 132.0 (d, J<sub>C-P</sub> = 54 Hz), and 134.9 (d, J<sub>C-P</sub> = 11.4 Hz) ppm.

### 1.3 Syntheses of [Pt(Te<sub>2</sub>C<sub>5</sub>H<sub>8</sub>O)(dppn)] (**6**)

A solution of **1** (30 mg, 0.09 mmol) dissolved in dry toluene (5 ml) was added by a syringe to a stirred solution of **5** (71 mg, 0.09 mmol) in dry toluene (5 ml) at -10°C and the mixture was stirred for 30 min. at room temperature and concentrated by partial removal of the solvent. The product **6** was precipitated by addition of pentane. The yellow precipitate (65 mg, 70 %) was filtered and washed with pentane. Found: C 47.05, H 3.36% Anal. Calc. for C<sub>39</sub>H<sub>34</sub>OP<sub>2</sub>PtTe<sub>2</sub> (1030.9): C 45.44, H 3.32% (NMR spectroscopy indicates that the crystalline sample contains a small amount of pentane that was used in the precipitation). <sup>31</sup>P NMR (400 MHz, CH<sub>2</sub>Cl<sub>2</sub>, H<sub>3</sub>PO<sub>4</sub>):  $\delta$  = -2.3 ppm, (<sup>1</sup>J<sub>Pt-P</sub> = 2554 Hz, <sup>2</sup>J<sub>P-Te</sub> = 70 Hz) <sup>195</sup>Pt NMR (400 MHz, CH<sub>2</sub>Cl<sub>2</sub>, Na<sub>2</sub>PtCl<sub>6</sub>):  $\delta$  = -5955 ppm (<sup>1</sup>J<sub>Pt-P</sub> = 2554 Hz, <sup>1</sup>J<sub>Te-Pt</sub> = 820 Hz), <sup>125</sup>Te NMR (400 MHz, CH<sub>2</sub>Cl<sub>2</sub>,

(CH<sub>3</sub>)<sub>2</sub>Te):  $\delta = 71$  ppm ( $^1J_{\text{Te-Pt}} = 820$  Hz),  $^1\text{H}$  NMR (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>):  $\delta = 7.2$  (2 H, m), 7.5 (2 H, m), 8.2 (30 H, m), 4.4 (4 H, s), 3.5 (4 H, s,  $^3J_{\text{H-Pt}} = 39$  Hz),  $^{13}\text{C}$  NMR (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>):  $\delta = 15.4$  (s), 42.2 (s), 82.5 (s); 127.9 (m), 130.7 (s), 134.4 (m) ppm.

## 2. References

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