

## Supporting Information

### Structure-Activity Relationship of para-Carborane Selective Estrogen Receptor Beta Agonists

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## 1. Compound activity profiling in cell-based reporter assays for nuclear receptors:

Agonist and antagonist profiling of each compound from the library in selective luciferase reporter assays for select nuclear receptors<sup>1</sup>.

**Table S1. Agonist properties of the compound library**

| Compound <sup>a</sup> | Agonist activities of compounds at 1 μM (%) <sup>b</sup> |       |      |      |     |      |      |      |      |      |       |       |       |      |      |       |       |      |       |                      |
|-----------------------|--|-------|------|------|-----|------|------|------|------|------|-------|-------|-------|------|------|-------|-------|------|-------|----------------------|
|                       | ERα  | ERβ   | AR   | PR   | GR  | MR   | ERRγ | FXR  | LXRα | LXRβ | PPARα | PPARβ | PPARγ | VDR  | RARα | RARγ  | RXRα  | RXRβ | RXRγ  | control <sup>c</sup> |
| <b>BE120</b>          | 66.5   | 100.0 | -0.3 | -0.8 | 0.3 | -0.4 | 1.2  | -0.7 | -3.8 | -2.1 | 8.9   | -6.7  | -9.4  | -0.1 | -3.4 | -3.7  | -9.6  | 7.3  | 11.5  | 0.9                  |
| <b>3b</b>             | 59.8   | 95.2  | -0.3 | -0.7 | 0.3 | 0.2  | 5.1  | -0.4 | -2.6 | -1.5 | -2.0  | 6.2   | -3.5  | -0.5 | -0.5 | -7.0  | 1.4   | -3.8 | -5.1  | 0.9                  |
| <b>3c</b>             | 67.6   | 70.1  | -0.3 | -0.7 | 1.1 | -0.2 | 17.6 | -1.0 | -0.9 | -0.5 | 18.4  | -1.0  | -14.5 | -0.3 | -1.9 | -7.0  | 6.9   | 6.3  | 15.4  | 1.0                  |
| <b>3d</b>             | 56.6   | 85.3  | -0.3 | -0.8 | 0.6 | 1.4  | 4.4  | -1.1 | -1.5 | -1.7 | 1.0   | -2.4  | -11.3 | -0.4 | -1.4 | -3.7  | -11.0 | 4.0  | -2.6  | 0.8                  |
| <b>3e</b>             | 63.5   | 101.6 | -0.3 | -0.7 | 0.6 | 1.3  | 1.6  | -0.5 | -2.6 | -1.7 | 0.1   | -1.0  | -6.7  | -0.4 | -0.5 | -2.6  | 2.8   | -0.3 | 10.3  | 0.9                  |
| <b>3f</b>             | 65.6   | 72.6  | -0.3 | -0.6 | 0.2 | 0.5  | 5.9  | 0.2  | -3.2 | -1.7 | 5.3   | 1.9   | -10.2 | -0.4 | -1.4 | -0.4  | -11.0 | -3.8 | 3.8   | 1.2                  |
| <b>3g</b>             | 63.8   | 86.4  | -0.3 | -0.7 | 2.2 | 1.1  | 21.0 | -0.5 | -2.1 | -1.6 | 4.4   | 3.3   | -3.2  | -0.3 | -1.0 | -1.5  | -15.1 | 2.8  | 1.3   | 0.9                  |
| <b>3h</b>             | 60.5   | 87.3  | 0.2  | -0.7 | 1.2 | 0.5  | -1.6 | -0.7 | -2.1 | -1.6 | -12.4 | 10.5  | -8.6  | -0.4 | -2.9 | -0.4  | -1.4  | 1.5  | -10.3 | 1.1                  |
| <b>3i</b>             | 60.9   | 85.8  | -0.3 | -0.7 | 0.7 | 2.5  | 9.8  | -0.6 | -2.1 | -0.6 | -3.9  | -3.8  | -1.9  | -0.3 | -1.0 | -12.6 | 1.4   | -1.9 | 1.3   | 0.9                  |
| <b>3j</b>             | 17.2   | 94.1  | 0.2  | -0.4 | 0.6 | 1.4  | 1.9  | -0.6 | -2.1 | -0.7 | -9.6  | 7.6   | -7.5  | -0.2 | -1.9 | -3.7  | 1.4   | 1.5  | 6.4   | 1.1                  |
| <b>3k</b>             | 0.7  | 85.1  | 0.2  | -0.7 | 1.1 | 0.2  | 10.3 | -0.7 | -2.1 | 0.0  | 2.8   | -2.4  | -11.6 | -0.3 | -1.4 | -3.7  | -1.4  | 2.2  | 1.3   | 1.0                  |
| <b>8</b>              | 0.3  | 92.6  | 0.6  | -0.7 | 1.6 | 1.3  | 7.5  | -1.0 | -0.4 | -0.1 | -5.4  | -6.7  | -7.5  | -0.3 | -2.4 | -7.0  | -5.5  | 4.8  | -9.0  | 1.0                  |
| <b>7</b>              | 0.4  | 103.1 | 0.6  | -0.6 | 0.8 | 0.2  | 11.6 | -0.5 | -3.2 | -0.6 | -0.2  | -1.0  | -1.9  | -0.3 | -1.0 | -0.4  | 1.4   | 6.9  | 15.4  | 0.9                  |
| <b>9</b>              | 0.3  | 62.7  | 0.6  | -0.4 | 0.2 | 0.5  | 10.3 | -1.0 | -2.1 | -0.9 | 5.3   | 1.9   | -3.5  | -0.2 | -1.0 | -2.6  | -2.8  | 6.5  | 0.0   | 0.8                  |
| <b>3l</b>             | 0.2  | 66.1  | 0.2  | -0.7 | 1.6 | 0.9  | 7.6  | -1.0 | -2.6 | -1.9 | -5.1  | 7.6   | -8.9  | -0.5 | -1.9 | -1.5  | 0.0   | -3.9 | 6.4   | 1.0                  |
| <b>3m</b>             | 0.4  | 79.7  | -0.3 | -0.7 | 1.4 | 2.9  | 2.3  | -0.5 | -1.5 | 0.1  | -3.5  | -9.5  | 0.8   | 0.0  | -1.4 | -2.6  | 6.9   | 19.4 | 2.6   | 0.9                  |
| <b>3n</b>             | 16.8   | 84.9  | 0.2  | -0.6 | 1.5 | 0.9  | 7.3  | -0.3 | -2.1 | 0.2  | 6.2   | 7.6   | 0.0   | -0.2 | 1.4  | 0.7   | 4.1   | 9.0  | 1.3   | 0.9                  |
| <b>3o</b>             | 2.8  | 61.0  | 0.2  | -0.6 | 1.8 | 1.4  | 11.2 | -0.2 | -2.6 | -0.5 | 10.1  | 10.5  | 5.4   | -0.1 | 1.4  | -0.4  | 20.6  | -3.4 | 21.8  | 1.0                  |
| <b>3p</b>             | 0.5  | 73.1  | 0.6  | -0.6 | 0.3 | -0.4 | 1.5  | -0.5 | -2.1 | -2.4 | -10.9 | -1.0  | -5.9  | -0.3 | -1.9 | -4.8  | -9.6  | 2.7  | 2.6   | 0.9                  |
| <b>3q</b>             | 0.3  | 92.4  | -0.3 | -0.6 | 0.3 | 1.4  | 9.4  | -0.7 | -0.9 | -0.1 | -8.4  | -1.0  | -6.2  | -0.4 | -1.0 | -0.4  | -5.5  | 13.2 | 14.1  | 1.1                  |
| <b>3r</b>             | 8.2  | 42.4  | 0.6  | -0.5 | 0.8 | -1.1 | 2.8  | -0.3 | -2.1 | -0.8 | -3.2  | 1.9   | -4.6  | -0.7 | 0.5  | -1.5  | -8.3  | 9.4  | 6.4   | 1.0                  |
| <b>3s</b>             | 0.4  | 13.2  | -0.3 | -0.6 | 0.7 | 0.0  | 1.2  | -0.6 | -2.1 | -0.8 | 6.2   | 1.9   | 5.7   | -0.6 | -0.5 | -3.7  | -5.5  | 2.1  | 5.1   | 1.0                  |
| <b>3t</b>             | 34.0   | 66.0  | 0.6  | -0.5 | 1.5 | 0.4  | 7.4  | -0.4 | -2.1 | -0.3 | 0.4   | -2.4  | -12.7 | -0.1 | 0.5  | -1.5  | -11.0 | 12.0 | 9.0   | 1.0                  |
| <b>3u</b>             | 0.0  | 58.0  | 1.1  | -0.6 | 1.4 | 1.3  | 14.4 | -0.3 | -2.6 | -1.0 | 7.7   | 1.9   | 0.5   | -0.1 | -0.5 | -0.4  | -1.4  | 8.0  | -3.8  | 1.0                  |

| Compound <sup>a</sup> | Agonist activities of compounds at 1 μM (%) <sup>b</sup> |      |      |      |     |      |      |      |      |      |       |       |       |      |      |      |      |      |      |                      |
|-----------------------|--|------|------|------|-----|------|------|------|------|------|-------|-------|-------|------|------|------|------|------|------|----------------------|
|                       | ERα  | ERβ  | AR   | PR   | GR  | MR   | ERRγ | FXR  | LXRα | LXRβ | PPARα | PPARβ | PPARγ | VDR  | RARα | RARγ | RXRα | RXRβ | RXRγ | control <sup>c</sup> |
| <b>3v</b>             | 1.1  | 89.1 | -0.3 | -0.5 | 0.8 | -0.5 | 7.5  | -0.1 | -0.9 | -1.6 | 14.4  | -1.0  | -0.8  | -0.3 | -1.4 | -0.4 | -2.8 | 6.3  | 6.4  | 0.9                  |
| <b>3w</b>             | 0.2  | 62.4 | 0.2  | -0.6 | 2.2 | 2.1  | 46.1 | -0.4 | -2.1 | -1.2 | -3.5  | 10.5  | 3.8   | 0.0  | 0.5  | 0.7  | 0.0  | -5.5 | 9.0  | 1.0                  |
| <b>3x</b>             | 19.9   | 87.1 | 0.6  | -0.6 | 2.3 | 1.4  | 30.8 | -0.5 | -1.5 | -0.5 | 23.8  | 19.0  | 8.1   | -0.2 | 0.0  | -1.5 | 1.4  | -2.7 | -5.1 | 1.3                  |
| <b>28</b>             | 0.2  | 0.0  | -0.3 | -0.6 | 0.3 | 0.0  | 2.0  | -0.3 | -1.5 | -1.3 | 1.6   | -6.7  | -4.6  | -0.4 | -1.0 | -2.6 | -4.1 | -1.0 | -1.3 | 1.0                  |
| <b>2k</b>             | 0.2  | 0.0  | 0.2  | -0.5 | 0.7 | 0.5  | 0.7  | -0.5 | -2.6 | -1.1 | -2.6  | -1.0  | 0.0   | -0.2 | -1.0 | -0.4 | -8.3 | 3.4  | 1.3  | 1.0                  |
| <b>31</b>             | 0.1  | 71.9 | -0.3 | -0.5 | 0.4 | 0.0  | 5.6  | -0.5 | -2.1 | -1.2 | 4.1   | 1.9   | 9.2   | -0.4 | -1.9 | -1.5 | 5.5  | -6.4 | 11.5 | 1.0                  |

**Table S1.** Agonist properties of novel carborane-based compound library in transactivation assays for nuclear receptors. <sup>a</sup>Each compound was tested in selective luciferase reporter assays for different nuclear receptors at 1 μM. Compounds were tested in quadruplicates and were incubated with reporter cells for 14h after which, luciferase activity was measured. <sup>b</sup>The activity is expressed relative to maximal activity induced by reference compound or as a <sup>c</sup>fold induction compared to untreated cells for control assay using reporter alone.

**Table S2. Antagonist properties of the compound library**

| Compound <sup>a</sup> | Antagonist activities of compounds at 1 μM (%) <sup>b</sup> |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       | Cell viability <sup>c</sup> |       |       |
|-----------------------|---|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-----------------------------|-------|-------|
|                       | ERα   | ERβ   | AR    | PR    | GR    | MR    | ERRγ  | FXR   | LXRα  | LXRβ  | PPARα | PPARβ | PPARγ | VDR   | RARα  | RARγ  | RXRα  | RXRβ                        | RXRγ  |       |
| <b>BE120</b>          | 108.5   | 87.9  | 98.0  | 98.3  | 90.9  | 82.2  | 82.1  | 91.5  | 90.4  | 89.7  | 80.7  | 97.1  | 88.7  | 76.1  | 64.5  | 100.4 | 98.5  | 87.0                        | 108.5 | 87.9  |
| <b>3b</b>             | 101.4   | 77.4  | 97.5  | 100.8 | 98.0  | 82.6  | 79.6  | 98.2  | 86.7  | 106.9 | 142.2 | 97.4  | 96.1  | 82.3  | 120.4 | 75.4  | 73.7  | 93.0                        | 101.4 | 77.4  |
| <b>3c</b>             | 102.5   | 74.7  | 96.1  | 104.9 | 106.1 | 110.4 | 111.5 | 86.6  | 75.6  | 92.5  | 96.3  | 100.6 | 107.8 | 103.1 | 72.6  | 95.1  | 130.9 | 106.6                       | 102.5 | 74.7  |
| <b>3d</b>             | 94.5  | 101.5 | 89.9  | 102.0 | 92.0  | 83.6  | 94.5  | 72.5  | 75.6  | 104.1 | 103.6 | 88.6  | 111.3 | 84.1  | 83.1  | 86.0  | 96.9  | 87.6                        | 94.5  | 101.5 |
| <b>3e</b>             | 98.4  | 86.8  | 99.7  | 102.3 | 92.0  | 82.2  | 81.3  | 103.4 | 93.4  | 95.2  | 86.7  | 91.4  | 99.0  | 99.0  | 97.1  | 82.3  | 143.3 | 96.4                        | 98.4  | 86.8  |
| <b>3f</b>             | 103.1   | 97.5  | 87.7  | 102.7 | 98.9  | 80.5  | 89.0  | 103.4 | 93.4  | 99.2  | 95.8  | 119.8 | 67.1  | 94.9  | 113.4 | 105.6 | 87.6  | 101.1                       | 103.1 | 97.5  |
| <b>3g</b>             | 91.8  | 88.6  | 96.6  | 90.9  | 88.1  | 92.0  | 93.0  | 98.4  | 134.9 | 103.7 | 122.9 | 85.6  | 91.2  | 102.4 | 101.7 | 105.0 | 76.8  | 87.1                        | 91.8  | 88.6  |
| <b>3h</b>             | 92.0  | 95.2  | 108.8 | 95.9  | 103.5 | 88.9  | 103.2 | 102.0 | 94.9  | 89.7  | 119.7 | 121.4 | 109.8 | 95.1  | 73.8  | 109.2 | 67.5  | 111.1                       | 92.0  | 95.2  |
| <b>3i</b>             | 83.8  | 95.1  | 100.9 | 91.0  | 101.7 | 94.8  | 93.9  | 114.0 | 103.8 | 111.6 | 176.1 | 90.5  | 121.6 | 75.3  | 118.1 | 110.0 | 138.7 | 89.8                        | 83.8  | 95.1  |
| <b>3j</b>             | 94.0  | 99.8  | 101.9 | 97.1  | 92.0  | 111.8 | 95.7  | 105.2 | 90.4  | 100.5 | 131.6 | 97.8  | 89.7  | 110.7 | 83.1  | 98.3  | 104.6 | 99.2                        | 94.0  | 99.8  |
| <b>3k</b>             | 107.8   | 89.7  | 86.9  | 91.5  | 115.3 | 93.1  | 92.8  | 103.2 | 98.6  | 91.3  | 119.2 | 133.0 | 97.0  | 95.2  | 85.4  | 171.1 | 115.5 | 93.3                        | 107.8 | 89.7  |
| <b>8</b>              | 81.5  | 102.3 | 102.9 | 85.0  | 106.7 | 99.4  | 108.7 | 85.0  | 106.7 | 97.7  | 87.6  | 84.2  | 90.7  | 79.8  | 88.9  | 113.4 | 70.6  | 93.5                        | 81.5  | 102.3 |
| <b>7</b>              | 92.1  | 114.9 | 94.5  | 92.0  | 102.8 | 96.9  | 82.4  | 98.2  | 91.9  | 92.9  | 102.7 | 98.4  | 116.2 | 93.4  | 75.0  | 116.3 | 132.5 | 97.0                        | 92.1  | 114.9 |
| <b>9</b>              | 94.0  | 103.1 | 95.6  | 89.7  | 95.1  | 81.5  | 85.0  | 87.1  | 97.8  | 120.6 | 117.4 | 107.2 | 111.3 | 174.7 | 95.9  | 106.0 | 101.5 | 94.0                        | 94.0  | 103.1 |
| <b>3l</b>             | 92.5  | 101.2 | 85.7  | 98.1  | 94.5  | 85.2  | 98.2  | 90.5  | 84.5  | 105.1 | 127.5 | 121.1 | 120.6 | 153.4 | 102.9 | 94.5  | 89.2  | 100.9                       | 92.5  | 101.2 |
| <b>3m</b>             | 100.3   | 99.6  | 117.4 | 102.6 | 114.9 | 128.6 | 101.9 | 90.3  | 96.4  | 95.7  | 141.2 | 112.3 | 102.4 | 115.7 | 98.3  | 123.0 | 130.9 | 89.6                        | 100.3 | 99.6  |
| <b>3n</b>             | 90.7  | 120.4 | 113.2 | 100.1 | 116.2 | 116.7 | 99.1  | 101.3 | 99.3  | 106.3 | 110.5 | 83.2  | 157.4 | 88.6  | 108.7 | 125.5 | 129.4 | 79.6                        | 90.7  | 120.4 |
| <b>3o</b>             | 83.5  | 111.6 | 108.8 | 127.2 | 116.5 | 120.4 | 126.6 | 119.1 | 134.9 | 109.1 | 157.8 | 99.0  | 126.0 | 104.9 | 91.3  | 117.9 | 134.0 | 93.6                        | 83.5  | 111.6 |
| <b>3p</b>             | 94.7  | 96.5  | 94.6  | 92.2  | 89.3  | 111.3 | 84.6  | 74.5  | 103.8 | 98.2  | 88.0  | 112.6 | 138.3 | 100.5 | 85.4  | 104.7 | 90.7  | 92.6                        | 94.7  | 96.5  |
| <b>3q</b>             | 102.7   | 98.0  | 108.1 | 93.9  | 109.2 | 97.8  | 106.5 | 86.7  | 91.2  | 104.3 | 119.7 | 90.2  | 67.1  | 86.2  | 75.0  | 106.9 | 117.0 | 107.4                       | 102.7 | 98.0  |
| <b>3r</b>             | 112.8   | 99.5  | 96.1  | 98.3  | 101.8 | 78.7  | 94.9  | 104.8 | 110.4 | 109.0 | 181.6 | 98.1  | 84.8  | 89.7  | 90.1  | 108.9 | 98.5  | 104.9                       | 112.8 | 99.5  |
| <b>3s</b>             | 95.4  | 102.0 | 94.3  | 110.1 | 105.7 | 103.4 | 108.2 | 93.8  | 123.8 | 98.5  | 138.0 | 101.8 | 84.3  | 77.0  | 127.4 | 124.3 | 120.1 | 107.6                       | 95.4  | 102.0 |
| <b>3t</b>             | 96.8  | 106.1 | 117.2 | 106.4 | 101.4 | 122.5 | 103.8 | 102.5 | 96.4  | 110.5 | 147.2 | 84.5  | 100.0 | 74.3  | 62.1  | 140.9 | 107.7 | 103.8                       | 96.8  | 106.1 |
| <b>3u</b>             | 82.1  | 108.4 | 97.8  | 112.7 | 109.6 | 118.8 | 119.7 | 115.7 | 103.8 | 100.0 | 109.6 | 76.1  | 108.3 | 91.1  | 90.1  | 113.0 | 69.1  | 111.1                       | 82.1  | 108.4 |
| <b>3v</b>             | 78.9  | 99.6  | 107.4 | 95.5  | 105.7 | 100.3 | 112.9 | 100.1 | 120.8 | 104.5 | 128.4 | 93.1  | 112.8 | 105.9 | 128.5 | 90.0  | 107.7 | 109.2                       | 78.9  | 99.6  |
| <b>3w</b>             | 72.1  | 98.5  | 108.1 | 105.5 | 113.7 | 128.6 | 126.6 | 76.1  | 96.4  | 109.4 | 105.5 | 84.5  | 139.3 | 106.3 | 127.4 | 111.0 | 72.2  | 103.9                       | 72.1  | 98.5  |

| Compound <sup>a</sup> | Antagonist activities of compounds at 1 μM (%) <sup>b</sup> |       |       |       |       |       |       |       |       |       |       |       |       |       |       |       | Cell viability <sup>c</sup> |       |       |       |
|-----------------------|---|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-----------------------------|-------|-------|-------|
|                       | ERα   | ERβ   | AR    | PR    | GR    | MR    | ERRγ  | FXR   | LXRα  | LXRβ  | PPARα | PPARβ | PPARγ | VDR   | RARα  | RARγ  | RXRα                        | RXRβ  | RXRγ  |       |
| <b>3x</b>             | 74.5  | 101.9 | 118.6 | 119.2 | 143.5 | 108.3 | 143.3 | 115.9 | 85.2  | 96.0  | 129.3 | 101.6 | 112.3 | 106.1 | 105.2 | 103.0 | 96.9                        | 106.3 | 74.5  | 101.9 |
| <b>28</b>             | 104.6   | 98.7  | 87.9  | 106.4 | 93.9  | 101.7 | 102.1 | 99.9  | 88.9  | 102.1 | 133.4 | 115.9 | 103.4 | 99.5  | 104.1 | 98.6  | n.d.                        | 96.6  | 104.6 | 98.7  |
| <b>2k</b>             | 109.1   | 92.9  | 89.9  | 104.5 | 95.9  | 96.6  | 93.8  | 100.1 | 99.3  | 109.3 | 144.5 | 127.8 | 72.0  | 156.4 | 100.6 | 135.2 | 93.8                        | 111.9 | 109.1 | 92.9  |
| <b>31</b>             | 97.5  | 108.1 | 92.6  | 100.5 | 96.4  | 110.8 | 97.7  | 93.1  | 116.4 | 92.8  | 135.3 | 105.2 | 101.5 | 51.8  | 134.4 | 116.1 | 144.8                       | 104.3 | 97.5  | 108.1 |

**Table S2.** Antagonist properties of novel carborane-based compound library in transactivation assays for nuclear receptors. <sup>a</sup>Each compound was tested in selective luciferase reporter assays for different nuclear receptors at 1 μM. Compounds were tested in quadruplicates and were incubated with reporter cells for 30 min after which an agonist specific for the reporter assay was added and cells were incubated for further 14h. Then, luciferase activity was measured. <sup>b</sup>The activity is expressed relative to the activity induced by specific agonist alone set as 100%. <sup>c</sup>Cell viability in U2OS cells was used as a control experiment for the antagonistic activities and is expressed in scale of 0-100% relative to DMSO-treated cells.

**Table S3. Cell-based luciferase reporter assays for nuclear receptors**

| Nuclear Receptor <sup>a</sup> | Assay <sup>b</sup>           | Cell line | Reference agonist     | Reference agonist concentration |                              |
|-------------------------------|------------------------------|-----------|-----------------------|---------------------------------|------------------------------|
|                               |                              |           |                       | Maximal activity <sup>c</sup>   | Antagonist mode <sup>d</sup> |
| ER $\alpha$                   | f1-ER $\alpha$ /3xERE        | HEK-293   | 17 $\beta$ -estradiol | 100 nM                          | 10 nM                        |
| ER $\beta$                    | f1-ER $\beta$ /3xERE         | HEK-293   | 17 $\beta$ -estradiol | 100 nM                          | 10 nM                        |
| AR                            | f1-AR/MMTV                   | U2OS      | Dihydrotestosterone   | 100 nM                          | 10 nM                        |
| PR-B                          | f1-PR-B/MMTV                 | U2OS      | Progesterone          | 100 nM                          | 25 nM                        |
| GR                            | f1-GR/MMTV                   | U2OS      | Dexamethasone         | 1 $\mu$ M                       | 25 nM                        |
| MR                            | f1-MR MMTV                   | U2OS      | Aldosterone           | 100 nM                          | 10 nM                        |
| ERR $\gamma$                  | ERR $\gamma$ -LBD/UAS        | U2OS      | GSK4716               | 50 $\mu$ M                      | 10 $\mu$ M                   |
| FXR                           | FXR-LBD/UAS                  | U2OS      | GW4064                | 1 $\mu$ M                       | 5 $\mu$ M                    |
| LXR $\alpha$                  | LXR $\alpha$ -LBD/UAS        | U2OS      | T0901317              | 1 $\mu$ M                       | 500 nM                       |
| LXR $\beta$                   | LXR $\beta$ -LBD/UAS         | U2OS      | T0901317              | 5 $\mu$ M                       | 1 $\mu$ M                    |
| PPAR $\alpha$                 | PPAR $\alpha$ -LBD/UAS       | U2OS      | GW7647                | 100 nM                          | 5 nM                         |
| PPAR $\beta/\delta$           | PPAR $\beta/\delta$ -LBD/UAS | U2OS      | GW0742                | 100 nM                          | 5 nM                         |
| PPAR $\gamma$                 | PPAR $\gamma$ -LBD/UAS       | U2OS      | Rosiglitazone         | 1 $\mu$ M                       | 100 nM                       |
| VDR                           | VDR-LBD/UAS                  | U2OS      | Calcitriol            | 100 nM                          | 50 nM                        |
| RAR $\alpha$                  | RAR $\alpha$ -LBD/UAS        | U2OS      | ATRA                  | 100 nM                          | 10 nM                        |
| RAR $\gamma$                  | RAR $\gamma$ -LBD/UAS        | U2OS      | ATRA                  | 100 nM                          | 10 nM                        |
| RXR $\alpha$                  | RXR $\alpha$ -LBD/UAS        | U2OS      | ATRA                  | 100 nM                          | 100 nM                       |
| RXR $\beta$                   | RXR $\beta$ -LBD/UAS         | U2OS      | ATRA                  | 10 nM                           | 10 nM                        |
| RXR $\gamma$                  | RXR $\gamma$ -LBD/UAS        | U2OS      | ATRA                  | 100 nM                          | 100 nM                       |

**Table S3.** Cell-based luciferase reporter assays for nuclear receptors. <sup>a</sup>Reporter cell-based assays selective for different nuclear receptors. <sup>b</sup>Reporter assays for individual steroid receptors rely on the expression of full-length (f1) steroid receptors recognizing either synthetic estrogen response elements in the promoter of the reporter vector (ER $\alpha$  and ER $\beta$ ), or response elements occurring in the LTR sequences from the MMTV virus (AR, PR, GR, MR). Reporter assays for remaining nuclear receptors are based on chimeric NRs created by replacement of the N-terminally located DNA binding domain (DBD) by a DBD from the yeast transcription factor Gal4. The chimeric Gal4-DBD/NR-LBD (LBD stands for ligand binding domain) binds to multiple upstream activating sequences (UAS) in the promoter of the reporter construct controlling the expression of the firefly luciferase from the pGL4.35 reporter vector (Promega, USA). <sup>c</sup>Reference agonist was used at the specified concentration to determine 100% reporter activity for the specific assay. Compound activities were then normalized relative to this value. <sup>d</sup>Concentration of the reference compound used for the competition with test compounds in the antagonist mode.

## 2. Inhibition of cytochrome P450 isoforms

Enzyme activities of individual CYP forms were measured according to established protocols. The following microsomal CYP activities were tested: CYP1A2, 7-ethoxyresorufin O-deethylation<sup>2</sup>; CYP2A6, coumarin 7-hydroxylation<sup>3</sup>; CYP2B6, 7-ethoxy-4-(trifluoromethyl)coumarin 7-deethylation<sup>4</sup>; CYP2C8, paclitaxel 6α-hydroxylation<sup>5</sup>; CYP2C9, diclofenac 4'-hydroxylation<sup>6</sup>; CYP2C19, mephenytoin 4'-hydroxylation<sup>7</sup>; CYP2D6, bufuralol 1'-hydroxylation<sup>8</sup>; CYP2E1, chlorzoxazone 6-hydroxylation<sup>9</sup>, and CYP3A4, midazolam 1'-hydroxylation<sup>10,11</sup>.

Incubation mixtures contained 100 mM potassium phosphate buffer (pH 7.4), NADPH-generating system (0.8 mM NADP+, 5.8 mM isocitrate, 0.3 unit/ml of isocitrate dehydrogenase, and 8 mM MgCl<sub>2</sub>), human liver microsomes and individual probe substrate. Compounds tested were dissolved in DMSO to a concentration 200 mM and then were diluted in the same phosphate buffer. Concentrations of the tested compounds in the experiments ranged from 0.01 to 100 μM. Assay conditions are listed in **Table S4**. The concentrations of probe substrates for individual CYP forms were chosen in the range of the respective K<sub>m</sub> determined separately as described [1-10] for each CYP enzyme and for its respective substrate.

Activities were measured using HPLC Prominence system (Shimadzu; Kyoto, Japan) with a UV/fluorescence detection. Data were analyzed using Sigma Plot v.12.0 graphing software (Jandel Scientific, Chicago, IL, USA).

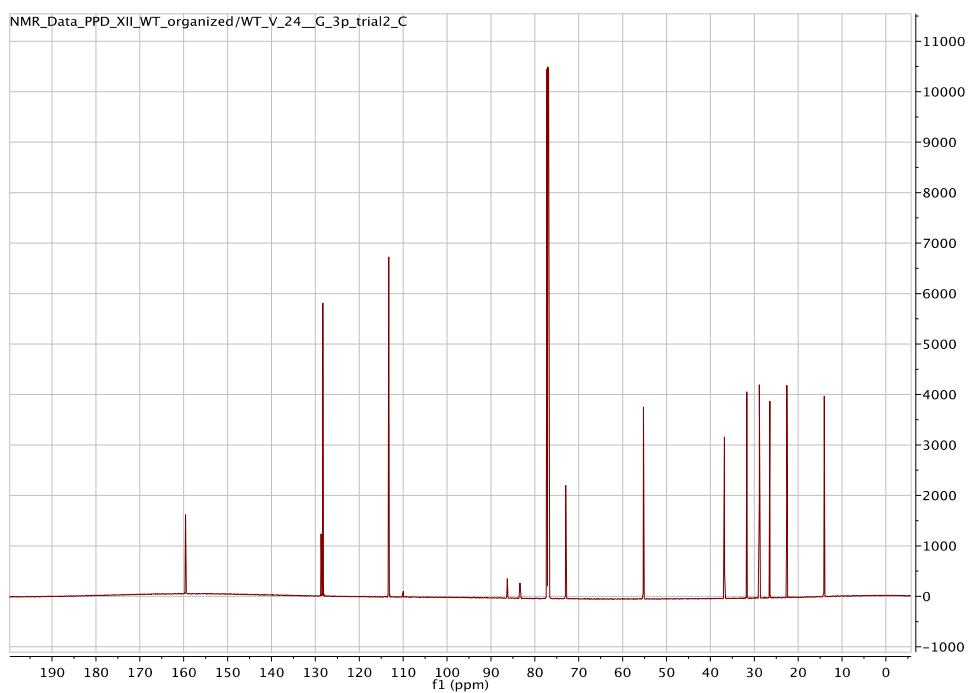
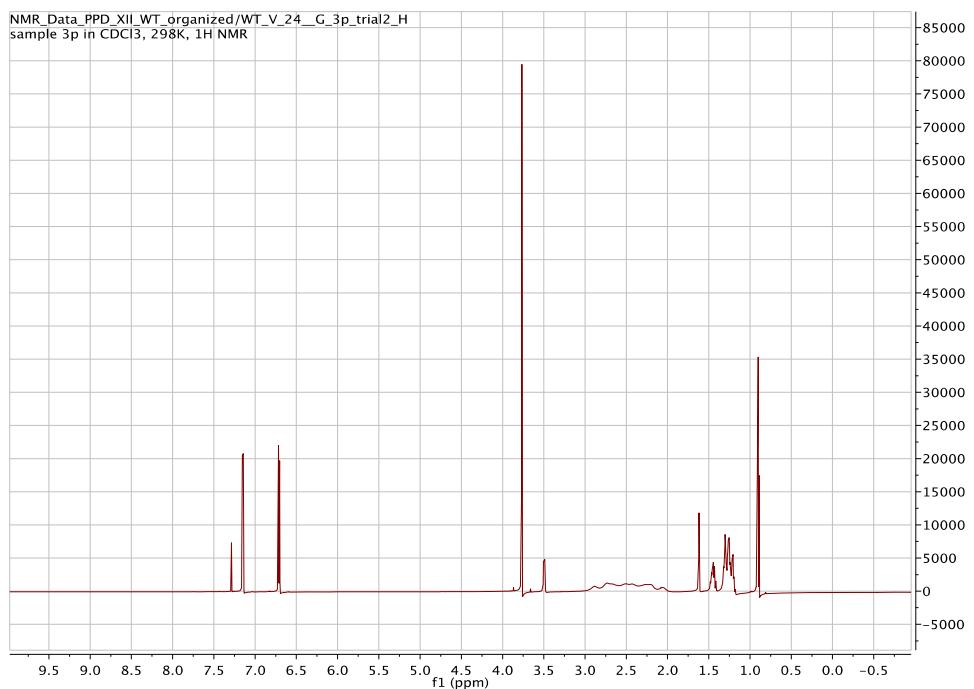
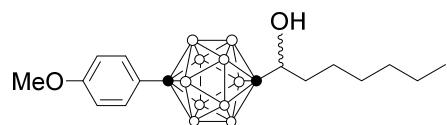
**Table S4. Details of the CYP450 enzymatic assay conditions.**

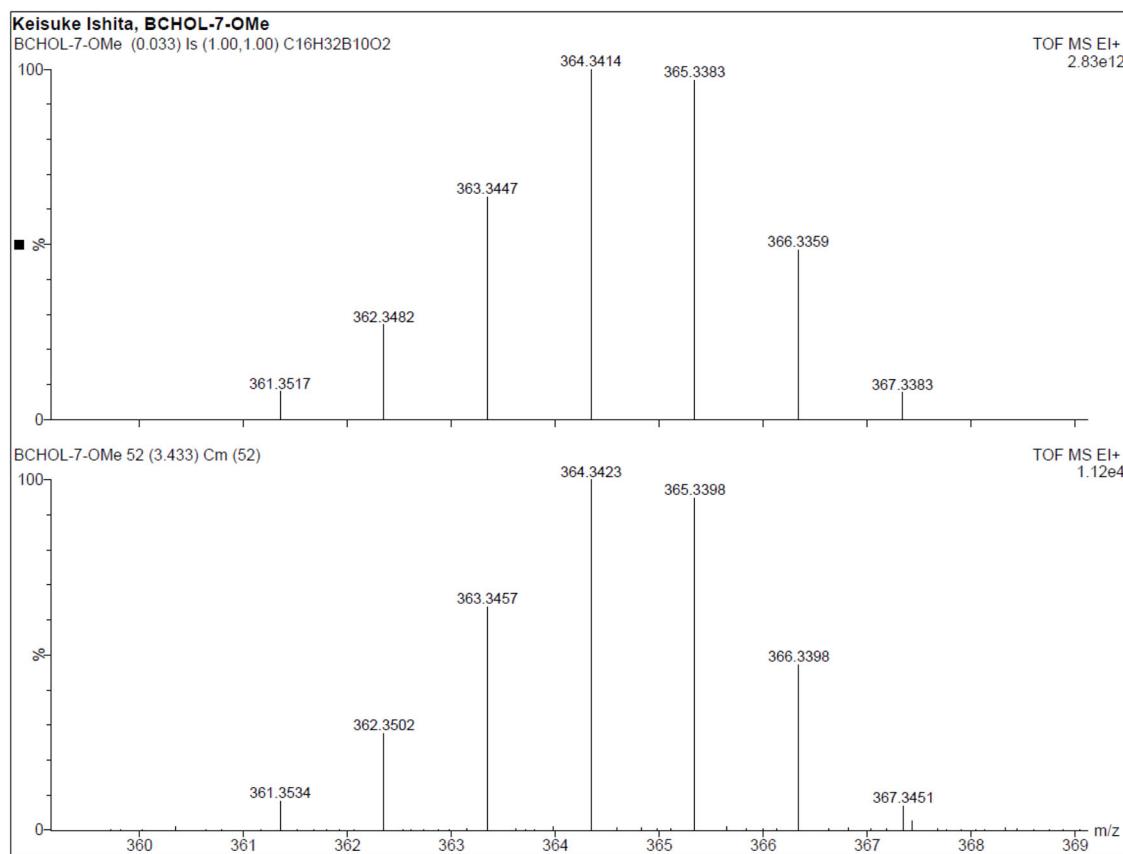
| CYP450 isoform | Substrate                            | Substrate conc. (μM) | CYP/volume (pmol/μl) | Quench agent                             | Incubation time (min) |
|----------------|--------------------------------------|----------------------|----------------------|--|-----------------------|
| CYP1A2         | 7-ethoxyresorufin                    | 2.5                  | 25/100               | 0.2 ml Methanol                          | 15                    |
| CYP2A6         | Coumarin                             | 15                   | 35/100               | 0.2 ml Methanol                          | 15                    |
| CYP2B6         | 7-ethoxy-4-(trifluoromethyl)coumarin | 15                   | 35/100               | 0.2 ml Methanol                          | 15                    |
| CYP2C8         | Paclitaxel                           | 45                   | 70/200               | 0.05 ml Acetonitrile                     | 15                    |
| CYP2C9         | Diclofenac                           | 15                   | 35/200               | 0.05 ml 94% Acetonitrile, 6% acetic acid | 30                    |
| CYP2C19        | S-mephenytoin                        | 25                   | 50/200               | 0.02 ml Hydrochloric acid                | 30                    |
| CYP2D6         | Bufuralol                            | 20                   | 70/200               | 0.02 ml 70% Perchloric acid              | 30                    |
| CYP2E1         | Chlorzoxazone                        | 25                   | 160/1000             | 0.05 ml 42.5% Phosphoric acid            | 30                    |
| CYP3A4         | Midazolam                            | 2.5                  | 15/100               | 0.1 ml Methanol                          | 10                    |

### 3. References

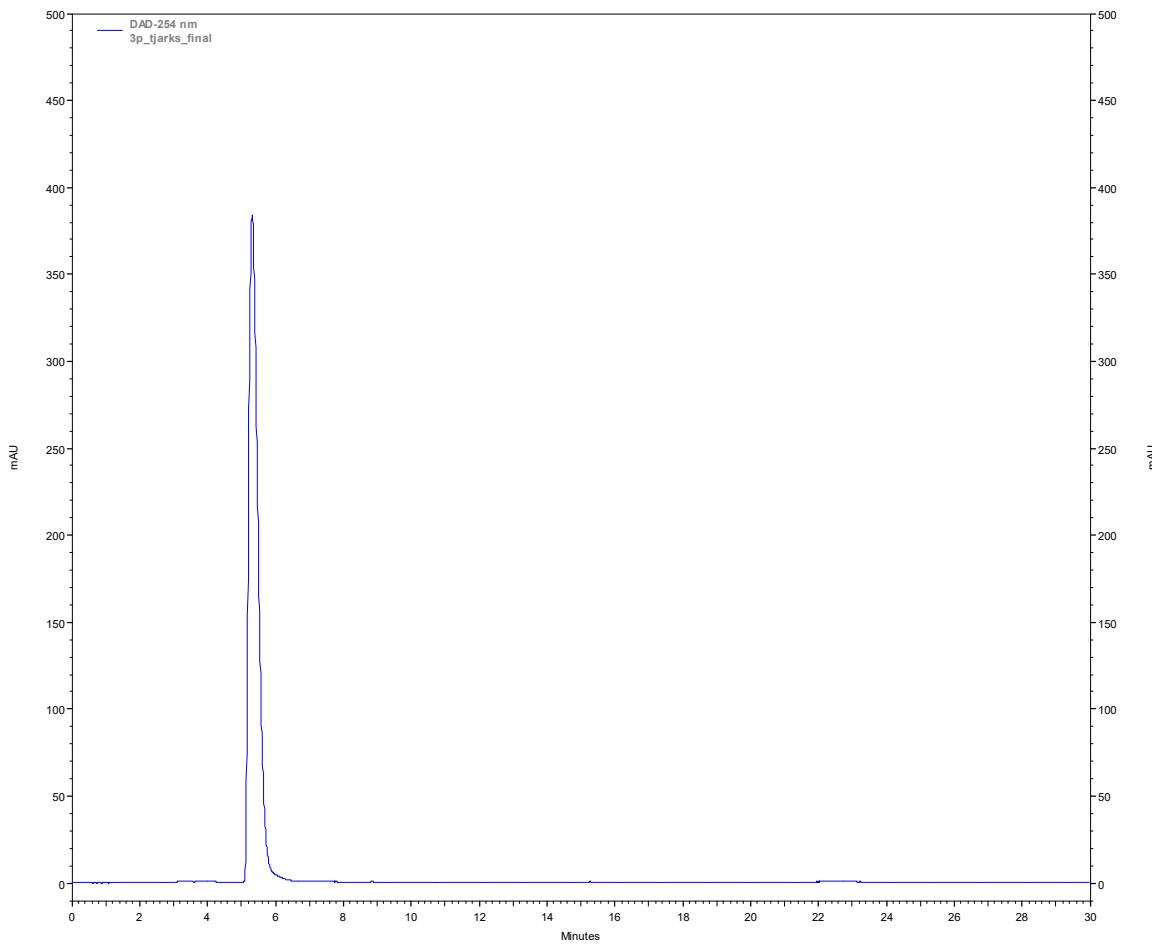
- [1] Sedlak, D.; Paguio, A.; Bartunek, P. Two Panels of Steroid Receptor Luciferase Reporter Cell Lines for Compound Profiling. *Comb Chem High Throughput Screen* 2011, 14 (4), 248-266. doi: 10.2174/138620711795222446
- [2] Chang TKH, Waxman DJ. Enzymatic analysis of cDNA-expressed human CYP1A1, CYP1A2, and CYP1B1 with 7-ethoxyresorufin as substrate. In: Phillips IR, Shephard EA, editors. Cytochrome P450 Protocols. Totowa NJ: Humana; 1998. p. 103-122.
- [3] Soucek P. Novel sensitive high-performance liquid chromatographic method for assay of coumarin 7-hydroxylation. *J Chromatogr B* 1999;734: 23-29.
- [4] Donato MT, Jimenez N, Castell JV, Gomez-Lechon MJ. Fluorescence-based assays for screening nine cytochrome P450 (P450) activities in intact cells expressing individual human P450 enzymes. *Drug Metab Dispos.* 2004;32: 699-706.
- [5] Crespi CL, Chang TKH, Waxman DJ. High-performance liquid chromatography analysis of CYP2C8-catalyzed paclitaxel 6 $\alpha$ -hydroxylation. In: Phillips IR, Shephard EA, editors. Cytochrome P450 Protocols. Totowa NJ: Humana; 1998. p. 123-127.
- [6] Crespi CL, Chang TKH, Waxman DJ. Determination of CYP2C9-catalyzed diclofenac 4'-hydroxylation by high-performance liquid chromatography. In: Phillips IR, Shephard EA, editors. Cytochrome P450 Protocols. Totowa NJ: Humana; 1998. p. 129-133.
- [7] Crespi CL, Chang TKH, Waxman DJ. CYP2C19-mediated (S)-mephentoin 4'-hydroxylation assayed by high-performance liquid chromatography with radiometric detection. In: Phillips IR, Shephard EA, editors. Cytochrome P450 Protocols. Totowa NJ: Humana; 1998. p. 135-139.
- [8] Crespi CL, Chang TKH, Waxman DJ. CYP2D6-dependent bufuralol 1'-hydroxylation assayed by reversed-phase ion-pair high-performance liquid chromatography with fluorescence detection. In: Phillips IR, Shephard EA, editors. Cytochrome P450 Protocols. Totowa NJ: Humana; 1998. p. 141-145.
- [9] Lucas D, Menez JF, Berthou F. Chlorzoxazone: An *in vitro* and *in vivo* substrate probe for liver CYP2E1. *Methods Enzymol.* 1996;272: 115-123.
- [10] Ghosal A, Satoh H, Thomas PE, Bush E, Moore D. Inhibition and kinetics of cytochrome P4503A activity in microsomes from rat, human, and CDNA-expressed human cytochrome P450. *Drug Metab. Dispos.* 1996, 24(9): 940-947.
- [11] Kronbach T, Mathys D, Umeno M, Gonzalez FJ, Meyer UA. Oxidation of midazolam and triazolam by human liver cytochrome P450IIIA4. *Mol. Pharmacol.* 1989, 36(1): 89-96.

#### 4. NMR spectra, MS spectra, HPLC chromatograms of target compounds





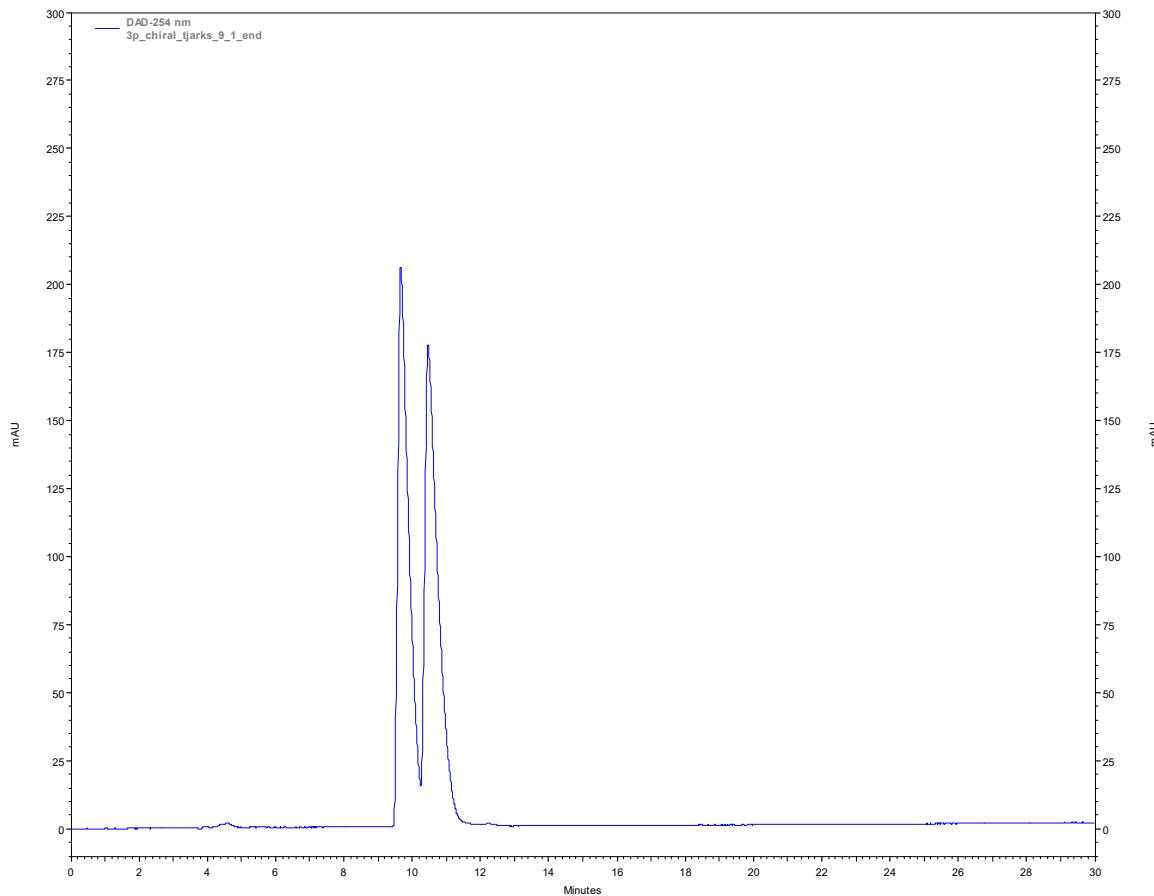
MS of Compound (*RS*)-1-[1-(4-methoxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol



#### DAD-254 nm Results

| Retention Time | Area     | Area % |
|----------------|----------|--------|
| 4.053          | 3533     | 0.01   |
| 4.633          | 723      | 0.00   |
| 4.767          | 1976     | 0.01   |
| 4.847          | 513      | 0.00   |
| 5.320          | 29415641 | 99.87  |
| 6.527          | 8555     | 0.03   |
| 22.253         | 22440    | 0.08   |
| 22.813         | 339      | 0.00   |
| 22.907         | 1307     | 0.00   |
| Totals         |          | 100.00 |
|                | 29455027 |        |

HPLC of Compound (*RS*)-1-[1-(4-methoxyphenyl)-1,12-dicarba-*clos*-dodecaborane-12-yl]heptan-1-ol

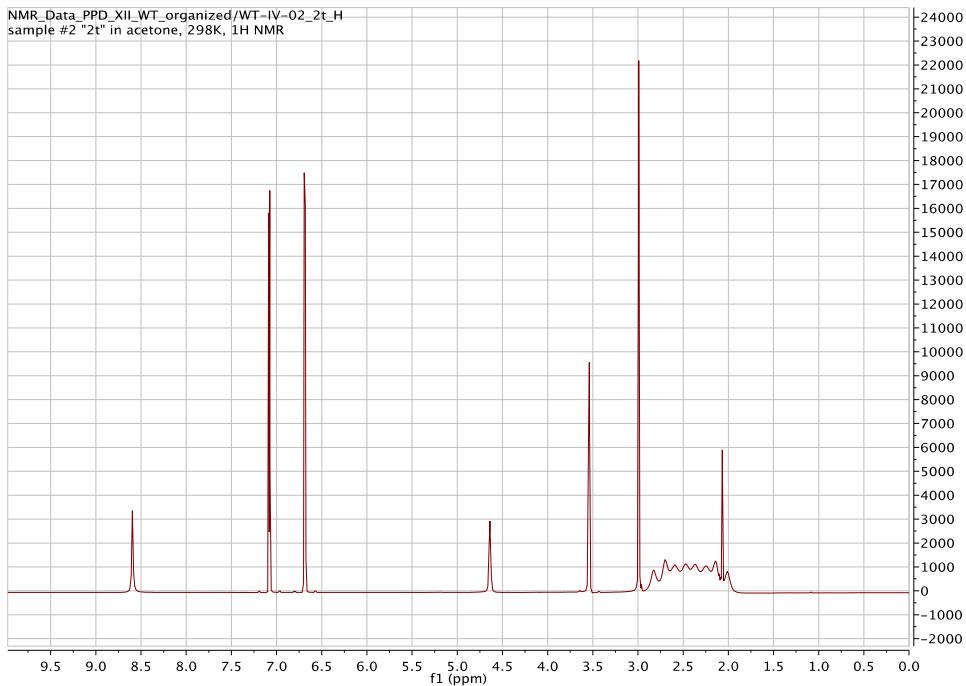
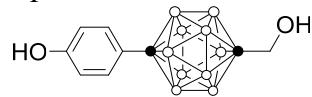


#### DAD-254 nm Results

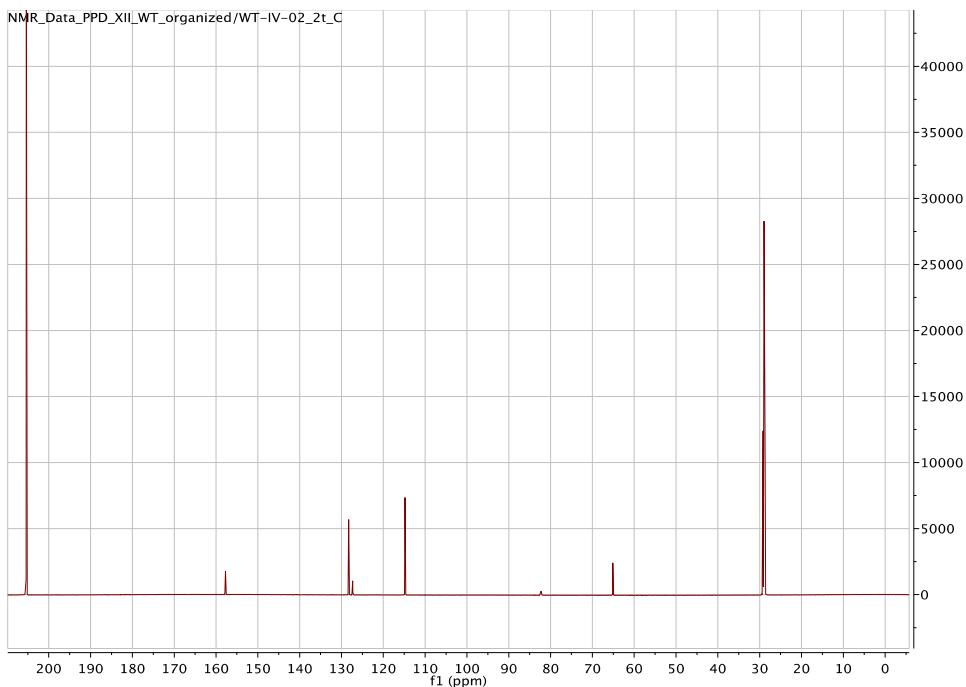
| Retention Time | Area     | Area % |
|----------------|----------|--------|
| 9.667          | 16729944 | 49.99  |
| 10.467         | 16700168 | 49.90  |
| 12.233         | 35029    | 0.10   |
| Totals         | 33465141 | 100.00 |

Chiral HPLC of Compound (*RS*)-1-[1-(4-methoxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol

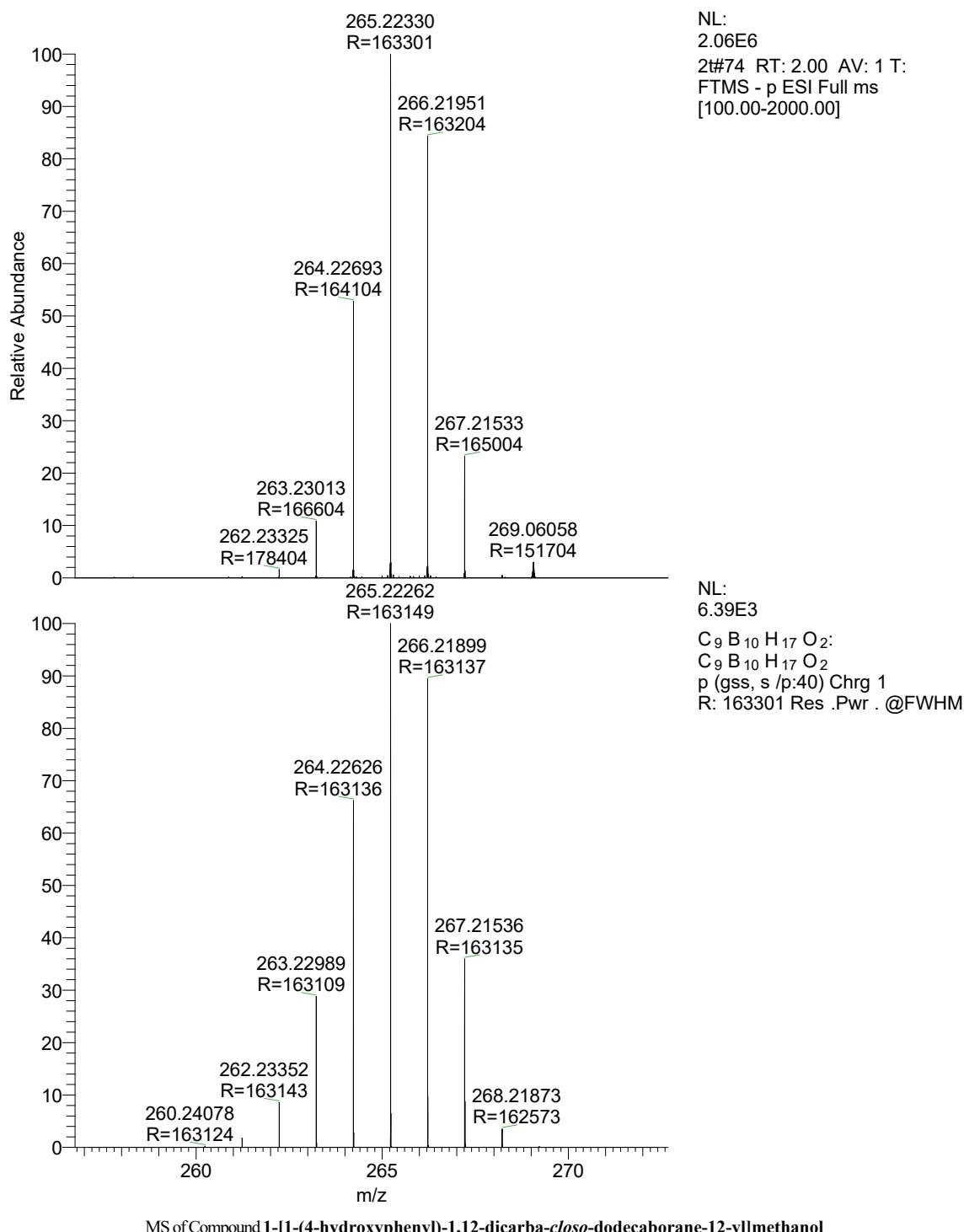
## Characterization Data for Target WT Compounds

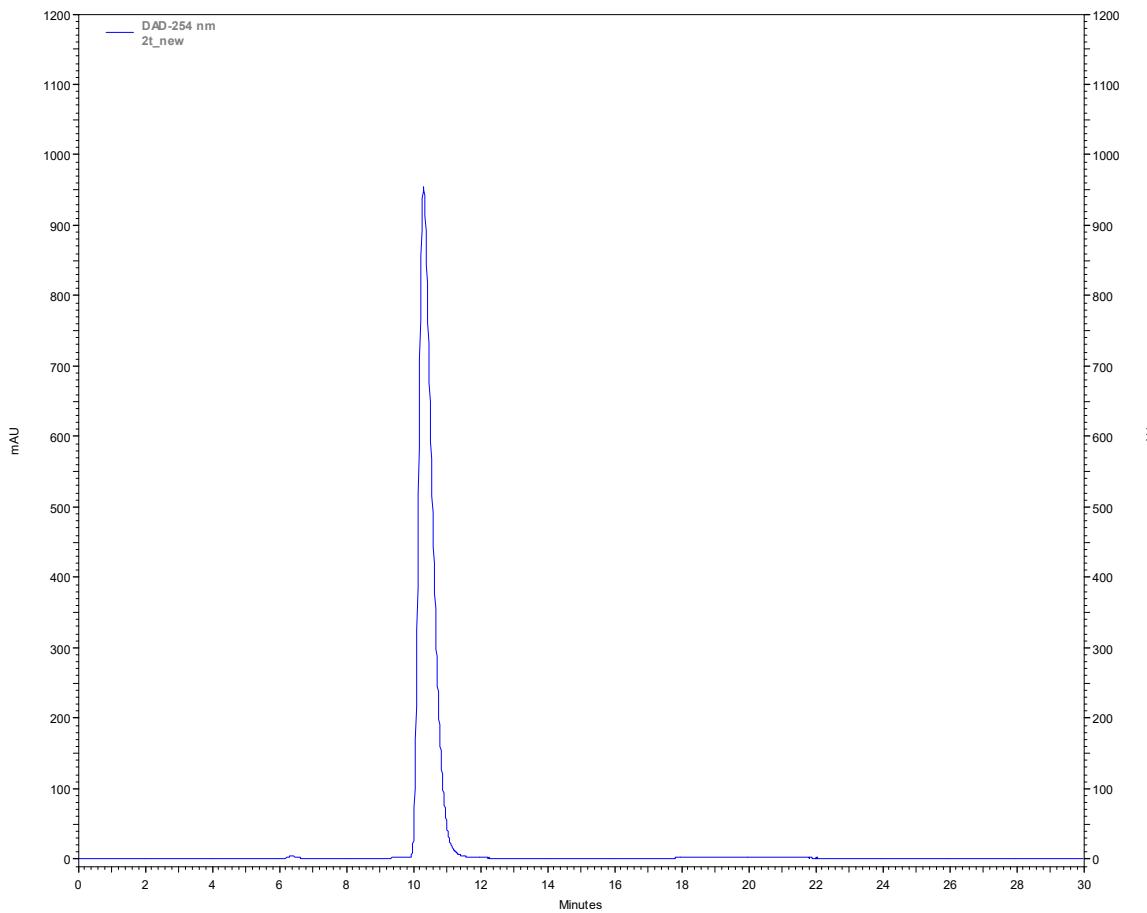


<sup>1</sup>H NMR of Compound 1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]methanol



<sup>13</sup>C NMR of Compound 1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]methanol

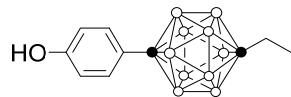




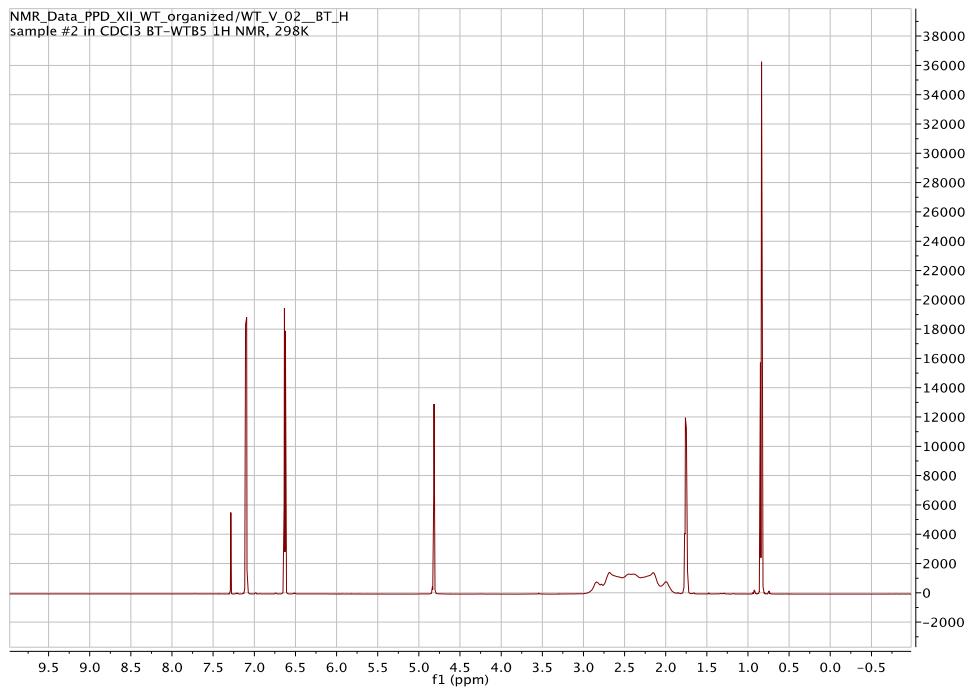
#### DAD-254 nm Results

| Retention Time | Area      | Area %           |
|----------------|-----------|------------------|
| 6.340          | 231253    | 0.21             |
| 9.633          | 3200      | 0.00             |
| 10.307         | 107621486 | 99.71            |
| 19.153         | 73232     | 0.07             |
| 20.600         | 1259      | 0.00             |
| 20.833         | 564       | 0.00             |
| <b>Totals</b>  |           | <b>107930994</b> |
|                |           | 100.00           |

HPLC of Compound 1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]methanol

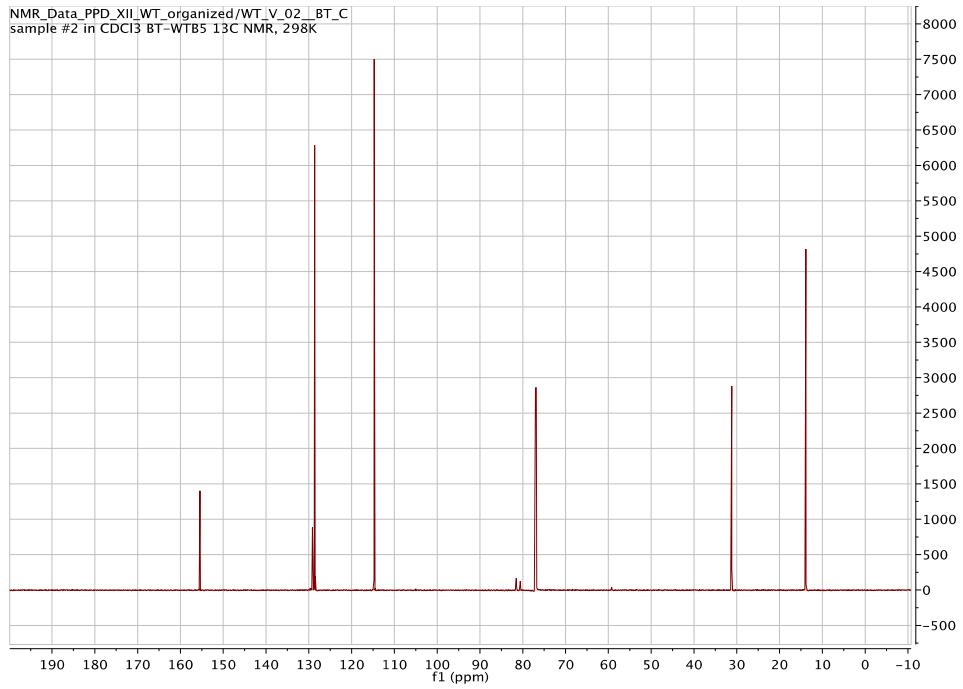


NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_02\_BT\_H  
sample #2 in CDCl<sub>3</sub> BT-WTB5 1H NMR, 298K



<sup>1</sup>H NMR of Compound 1-(4-hydroxyphenyl)-12-ethyl-1,12-dicarba-closo-dodecaborane

NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_02\_BT\_C  
sample #2 in CDCl<sub>3</sub> BT-WTB5 <sup>13</sup>C NMR, 298K

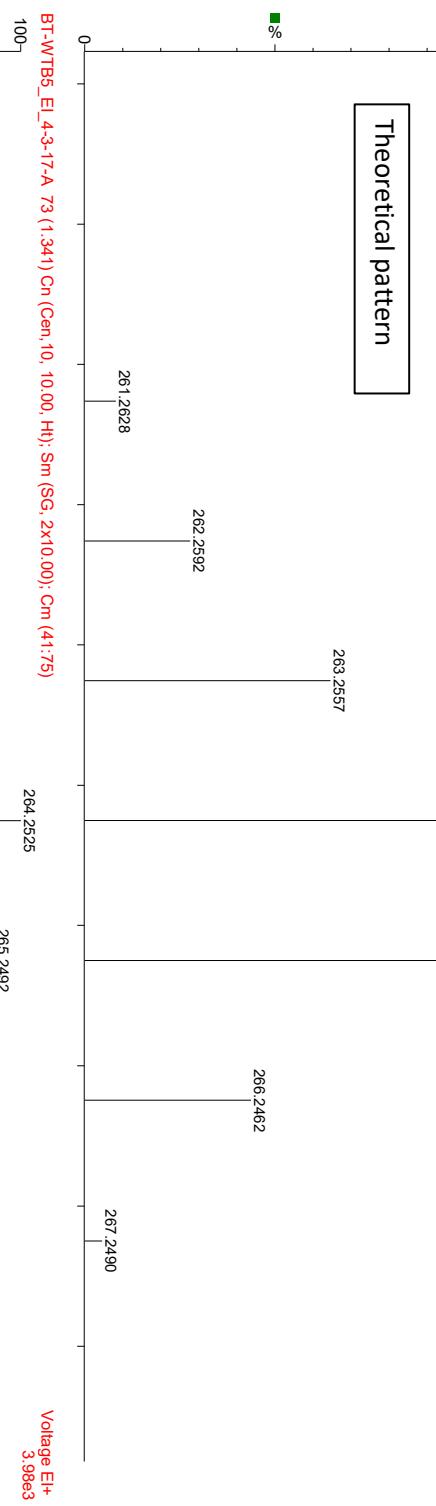


<sup>13</sup>C NMR of Compound 1-(4-hydroxyphenyl)-12-ethyl-1,12-dicarba-closo-dodecaborane

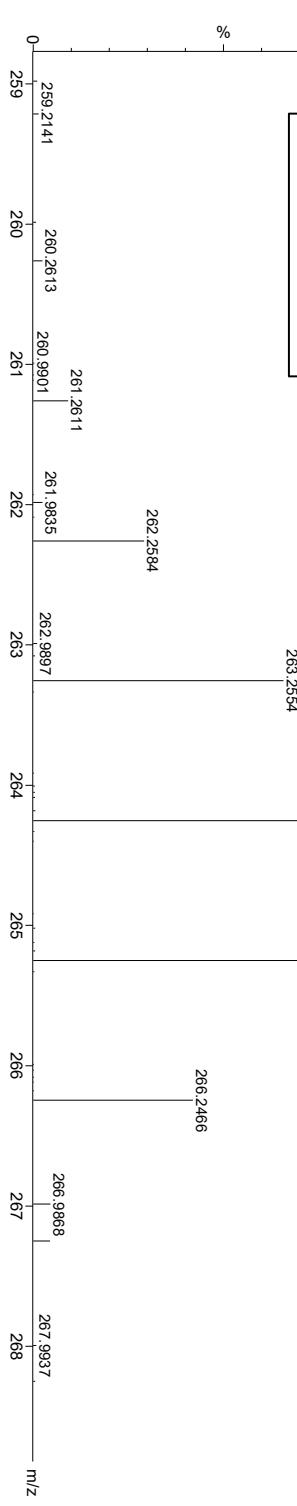
Werner Tjarks BT-WTB5 EI 70 eV



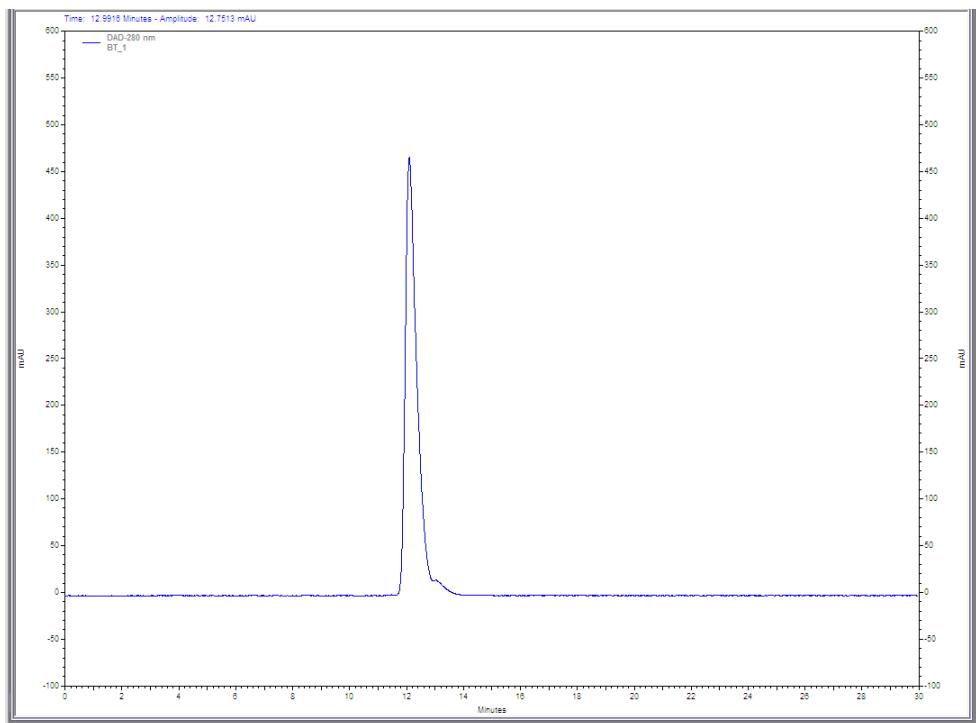
Theoretical pattern



Measured pattern



MS of Compound 1-(4-hydroxyphenyl)-12-ethyl-1,12-dicarba-closo-dodecaborane

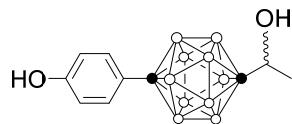


### DAD-280 nm

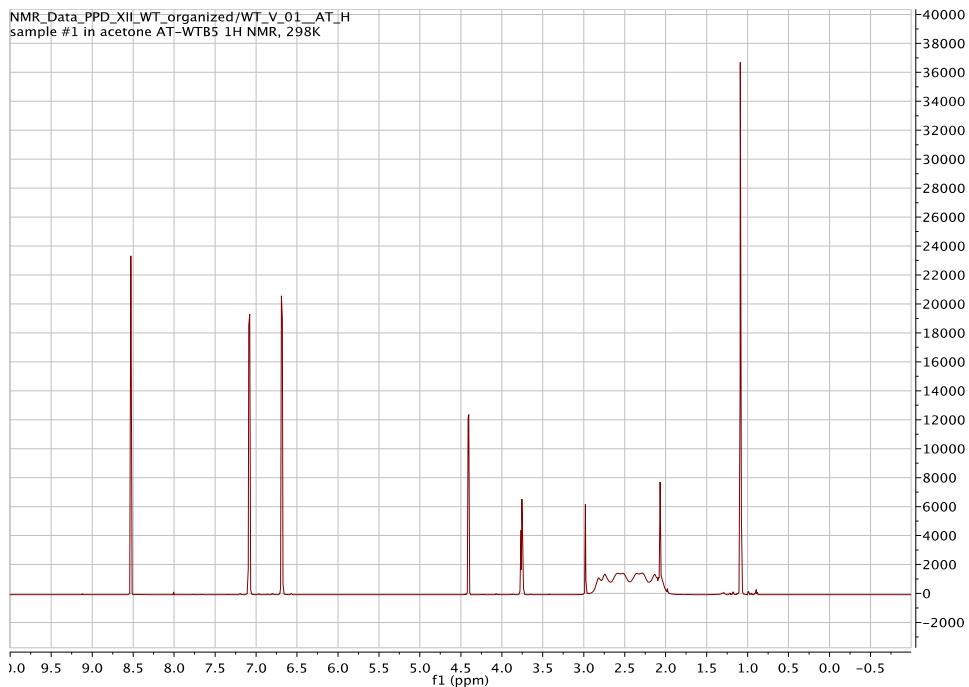
#### Results

| Retention Time | Area            | Area %        | Height         | Height %      |
|----------------|-----------------|---------------|----------------|---------------|
| 12.093         | 50057811        | 99.61         | 1850129        | 99.45         |
| 13.020         | 195020          | 0.39          | 10236          | 0.55          |
| <b>Totals</b>  | <b>50252831</b> | <b>100.00</b> | <b>1860365</b> | <b>100.00</b> |

HPLC of Compound 1-(4-hydroxyphenyl)-12-ethyl-1,12-dicarba-*clos*o-dodecaborane

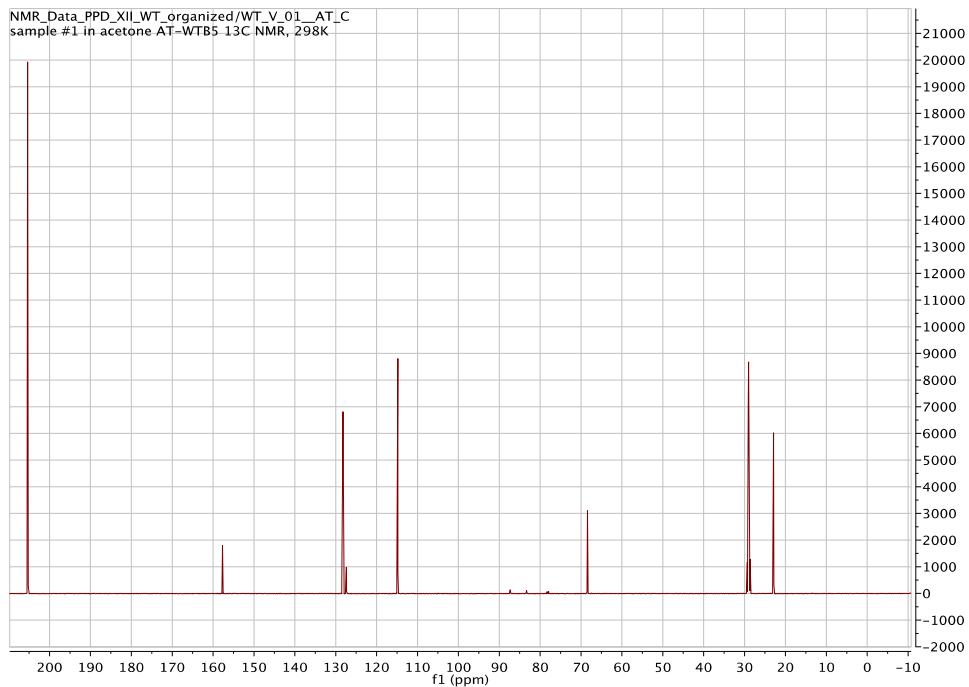


NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_01\_AT\_H  
sample #1 in acetone AT-WTB5 1H NMR, 298K

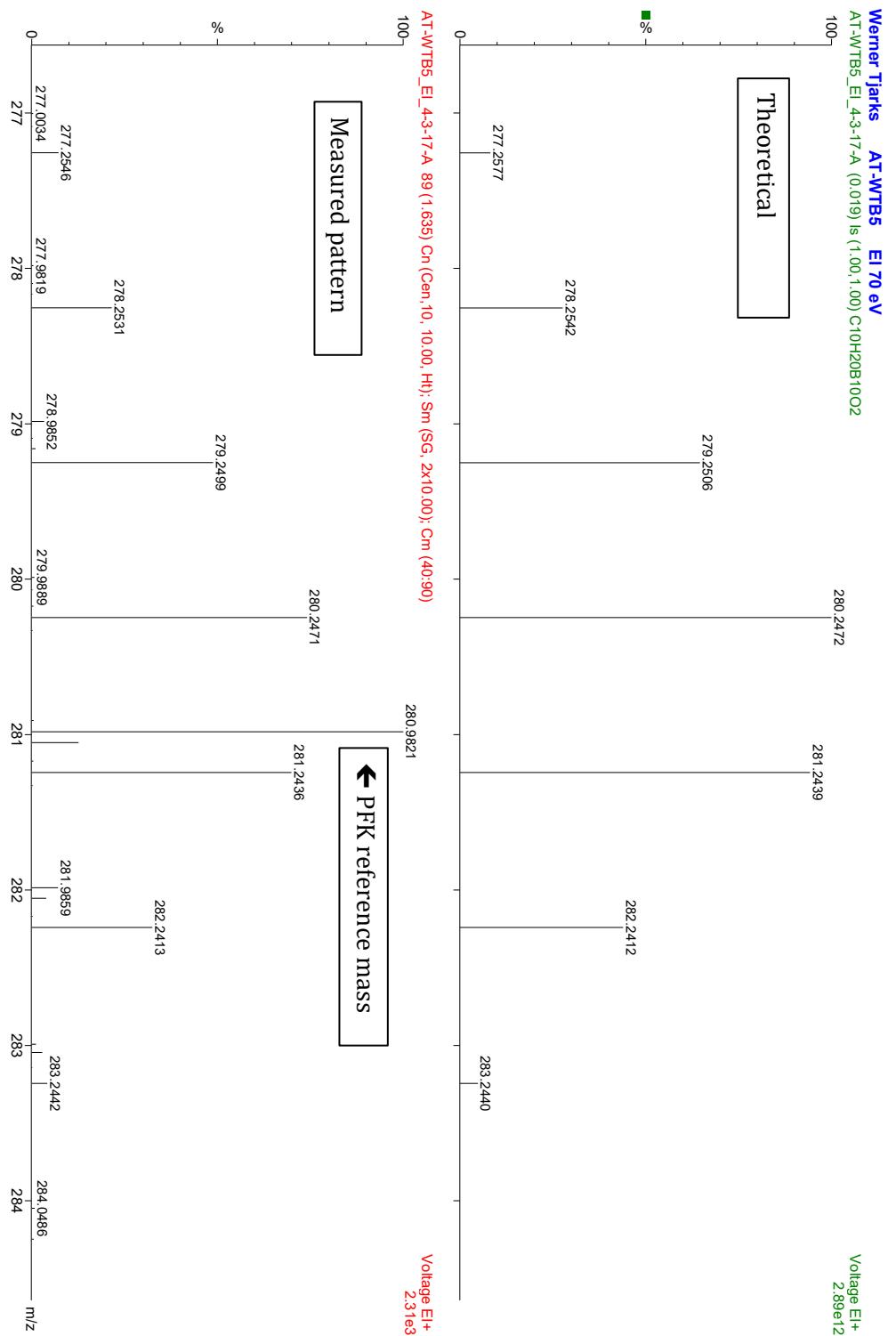


<sup>1</sup>H NMR of Compound (RS)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]ethan-1-ol

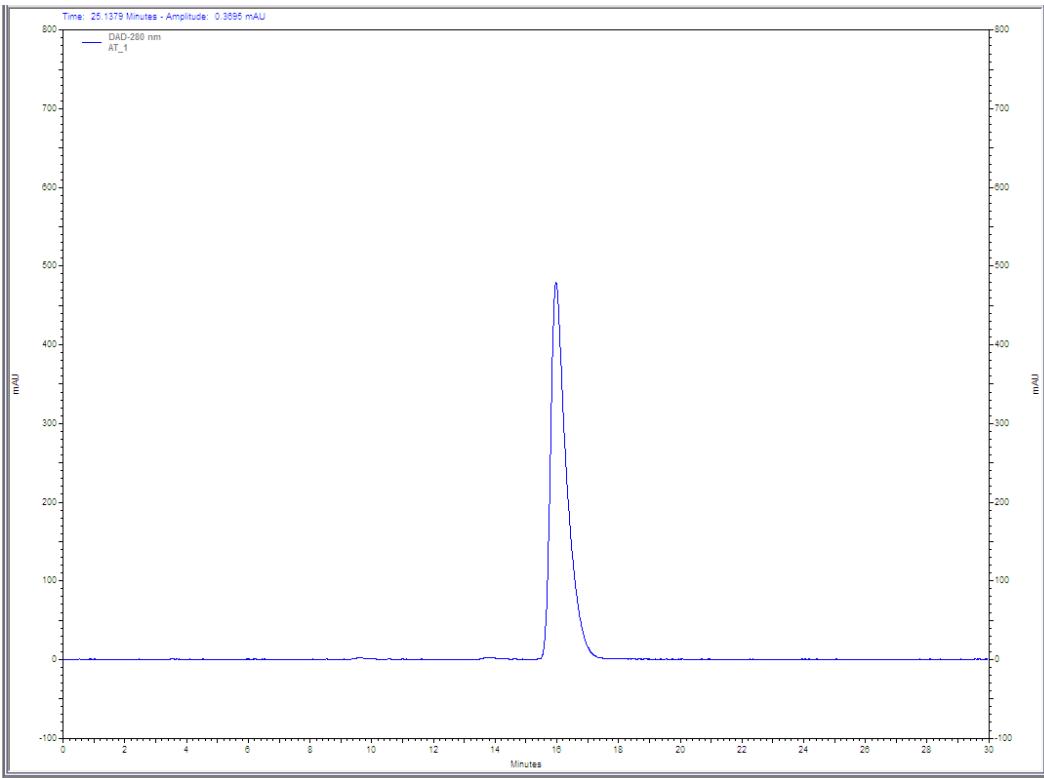
NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_01\_AT\_C  
sample #1 in acetone AT-WTB5 13C NMR, 298K



<sup>13</sup>C NMR of Compound (RS)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]ethan-1-ol



MS of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]ethan-1-ol

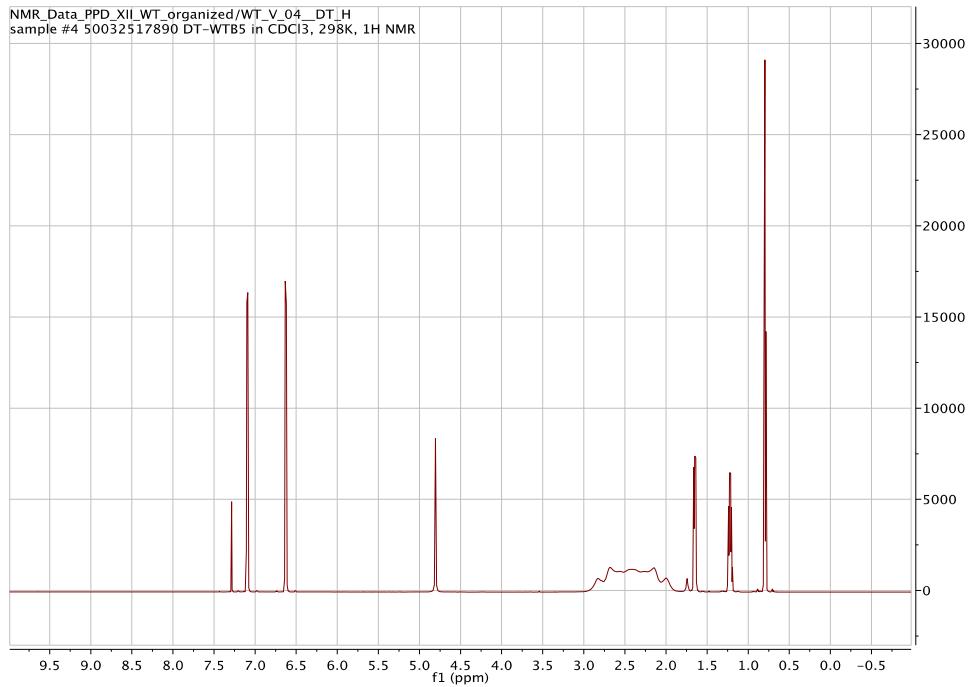
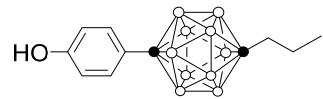


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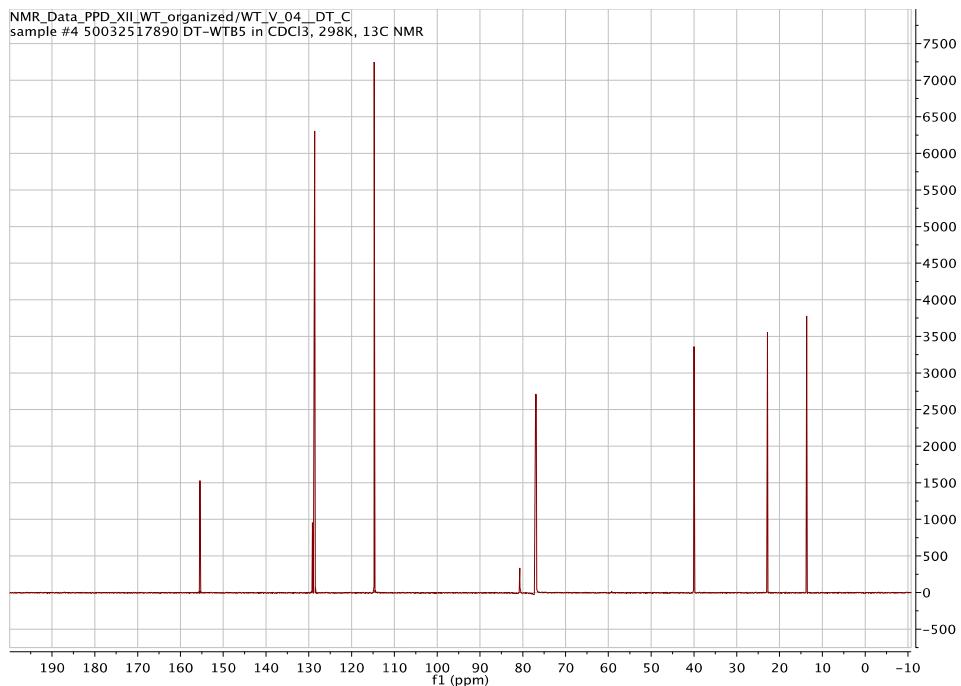
#### Results

| Retention Time | Area            | Area %        | Height         | Height %      |
|----------------|-----------------|---------------|----------------|---------------|
| 9.580          | 154580          | 0.22          | 6955           | 0.36          |
| 13.840         | 309408          | 0.45          | 9889           | 0.51          |
| 15.973         | 68574848        | 99.33         | 1915760        | 99.13         |
| <b>Totals</b>  | <b>69038836</b> | <b>100.00</b> | <b>1932604</b> | <b>100.00</b> |

HPLC of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]ethan-1-ol

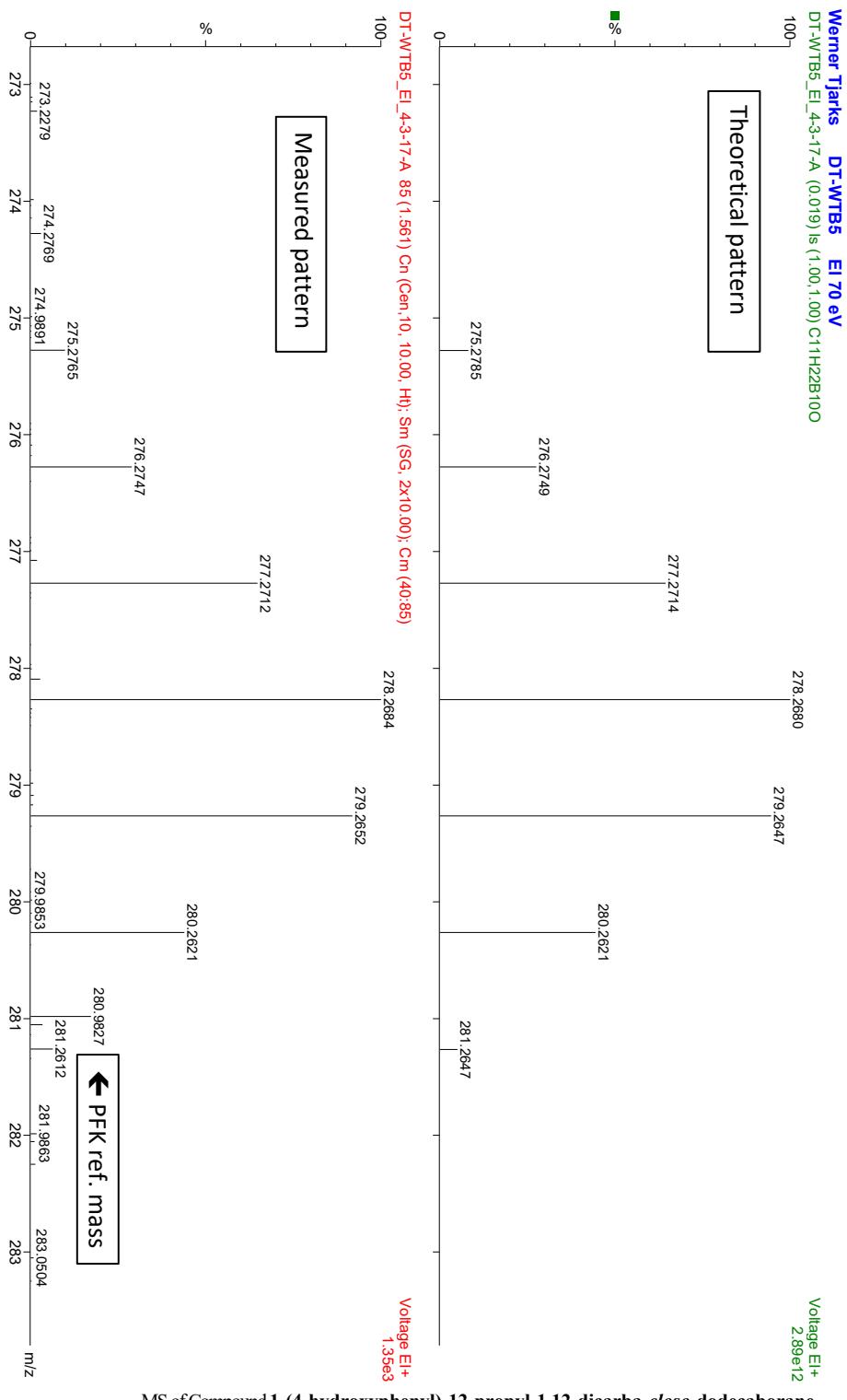


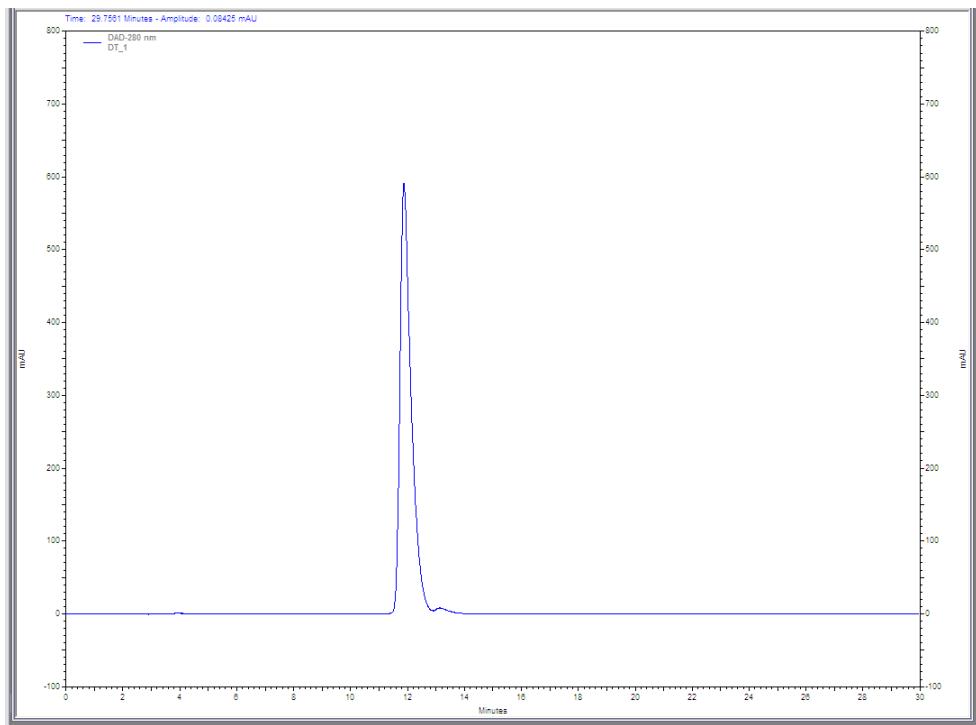
<sup>1</sup>H NMR of Compound 1-(4-hydroxyphenyl)-12-propyl-1,12-dicarba-closo-dodecaborane



<sup>13</sup>C NMR of Compound 1-(4-hydroxyphenyl)-12-propyl-1,12-dicarba-closo-dodecaborane

Werner Tjarks DT-WTB5 EI 70 eV



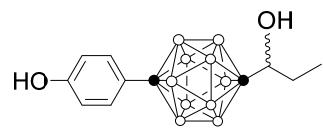


### DAD-280 nm

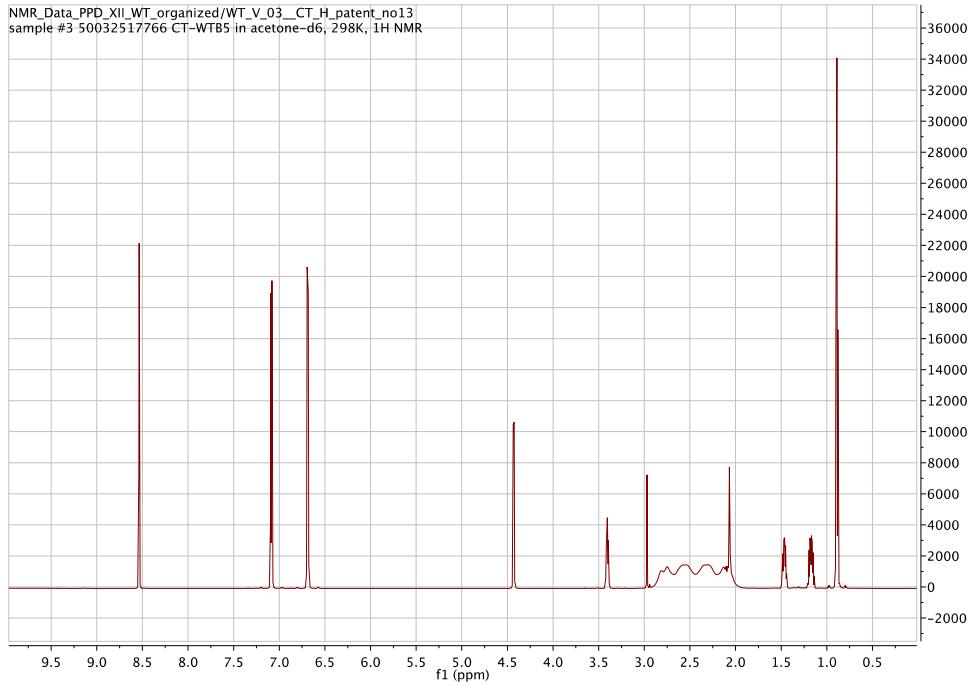
#### Results

| Retention Time | Area            | Area %        | Height         | Height %      |
|----------------|-----------------|---------------|----------------|---------------|
| 11.873         | 63784269        | 99.29         | 2359583        | 99.19         |
| 13.127         | 458512          | 0.71          | 19172          | 0.81          |
| <b>Totals</b>  | <b>64242781</b> | <b>100.00</b> | <b>2378755</b> | <b>100.00</b> |

HPLC of Compound 1-(4-hydroxyphenyl)-12-propyl-1,12-dicarba-*clos*o-dodecaborane

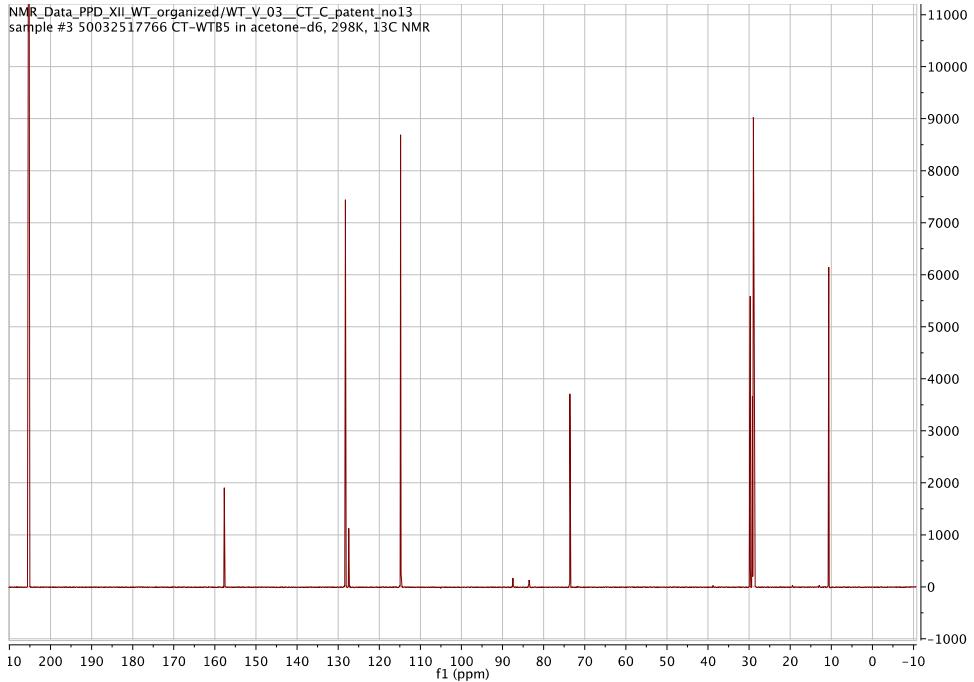


NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_03\_CT\_H\_patent\_no13  
sample #3 50032517766 CT-WTB5 in acetone-d<sub>6</sub>, 298K, 1H NMR

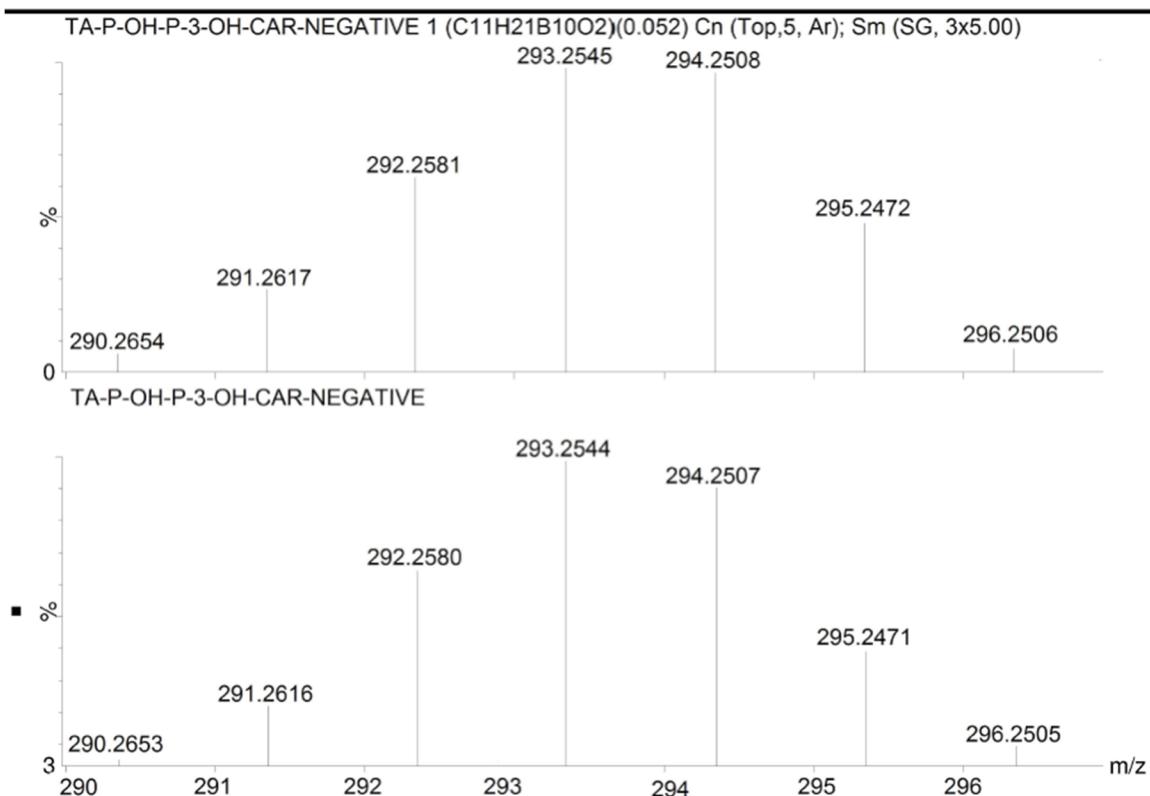


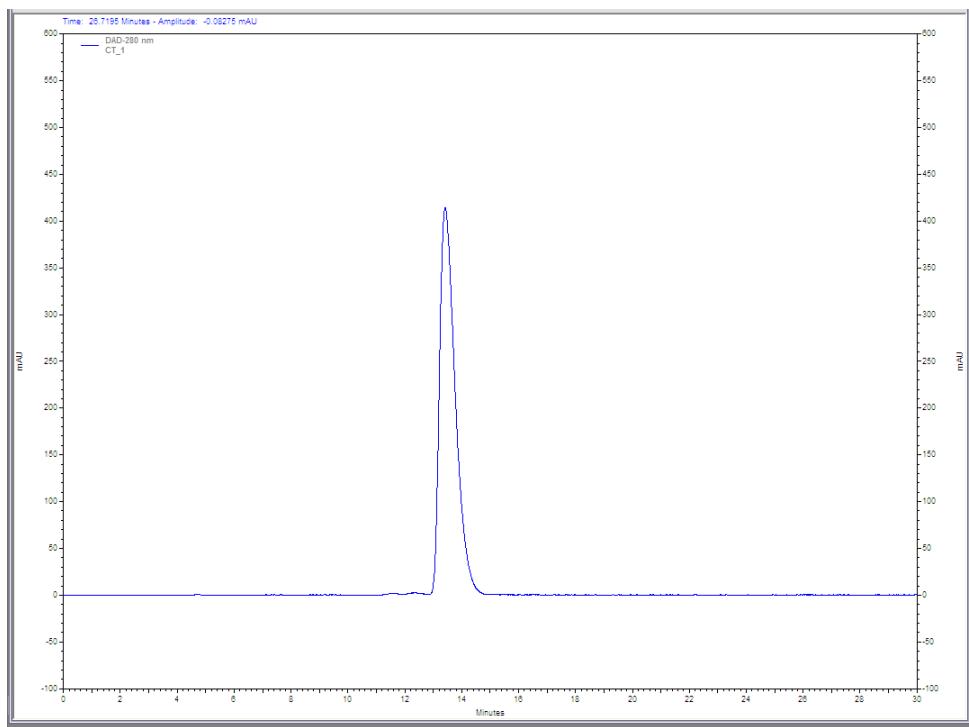
<sup>1</sup>H NMR of Compound 1-(4-Hydroxyphenyl)-12-(1-hydroxypropyl)-1,12-dicarba-closo-dodecaborane

NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_03\_CT\_C\_patent\_no13  
sample #3 50032517766 CT-WTB5 in acetone-d<sub>6</sub>, 298K, <sup>13</sup>C NMR



<sup>13</sup>C NMR of Compound 1-(4-Hydroxyphenyl)-12-(1-hydroxypropyl)-1,12-dicarba-closo-dodecaborane



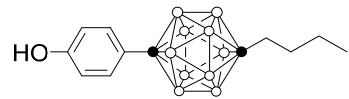


### DAD-280 nm

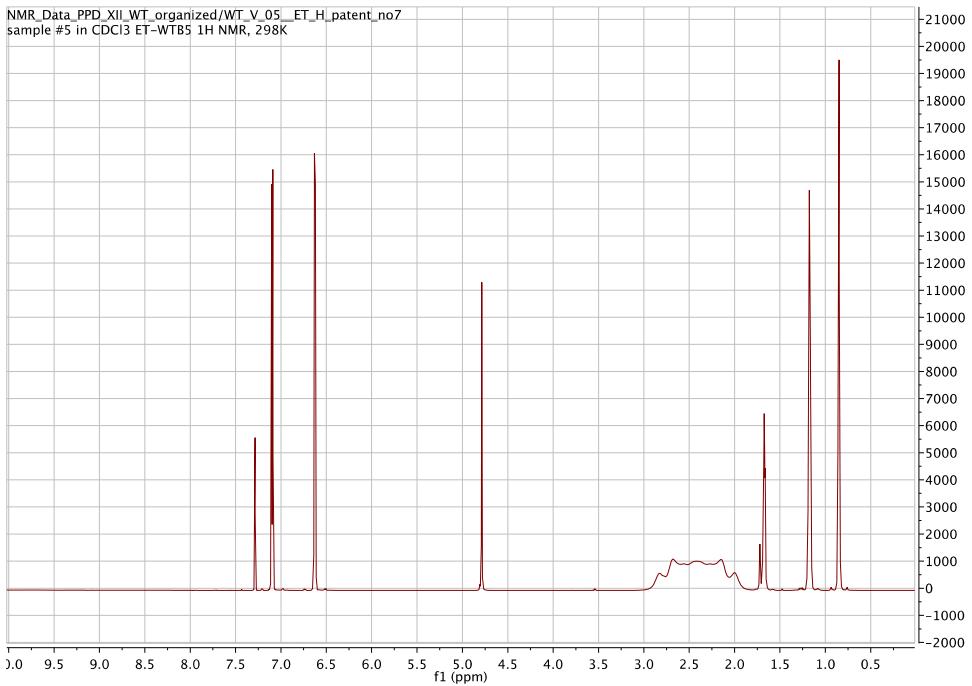
#### Results

| Retention Time | Area     | Area % | Height  | Height % |
|----------------|----------|--------|---------|----------|
| 12.380         | 449826   | 0.71   | 8840    | 0.53     |
| 13.420         | 63251111 | 99.29  | 1655018 | 99.47    |
| Totals         | 63700937 | 100.00 | 1663858 | 100.00   |

HPLC of Compound 1-(4-Hydroxyphenyl)-12-(1-hydroxypropyl)-1,12-dicarba-*clos*-dodecaborane

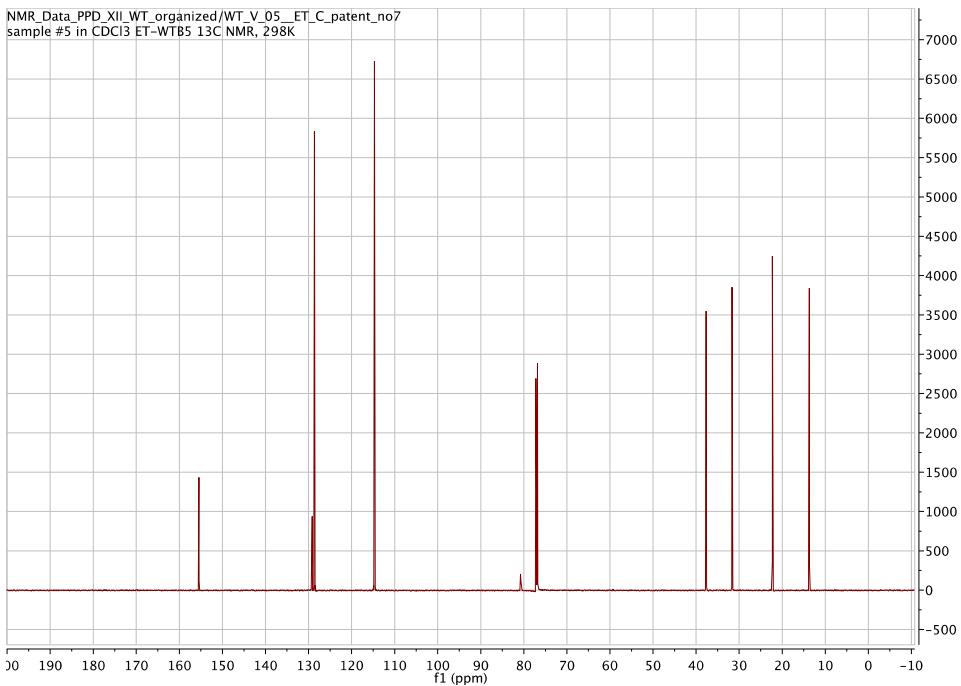


NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_05\_ET\_H\_patent\_no7  
sample #5 in CDCl<sub>3</sub> ET-WTB5 1H NMR, 298K

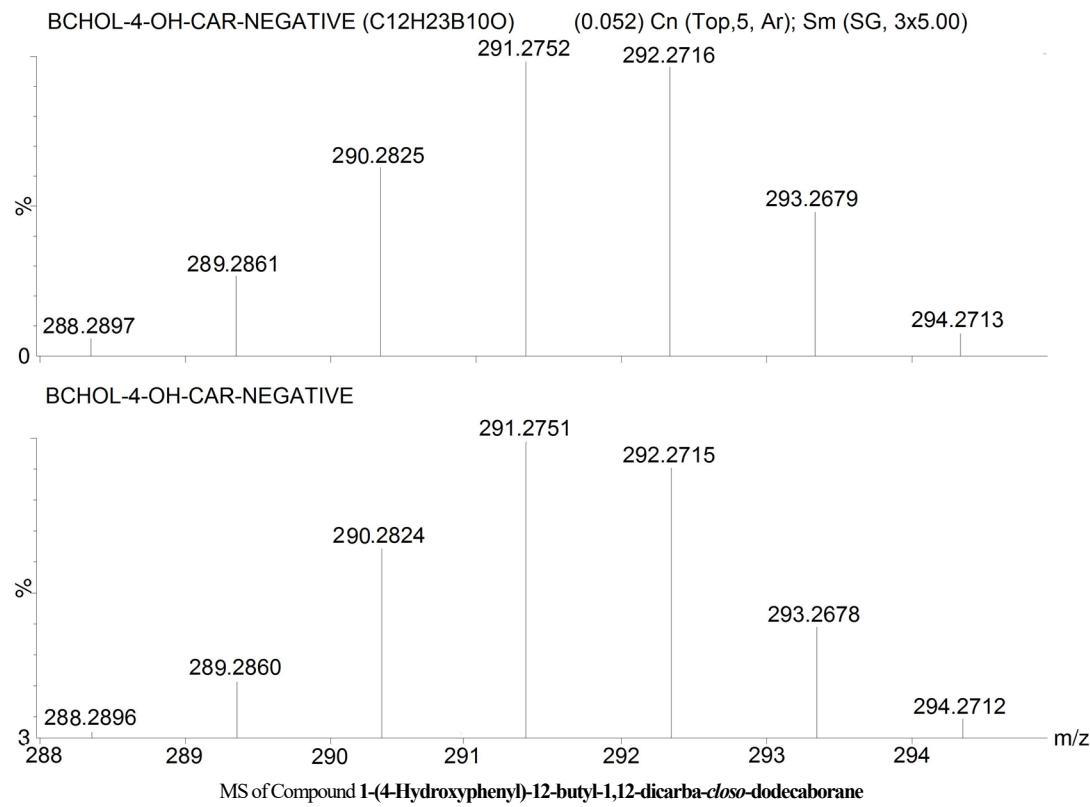


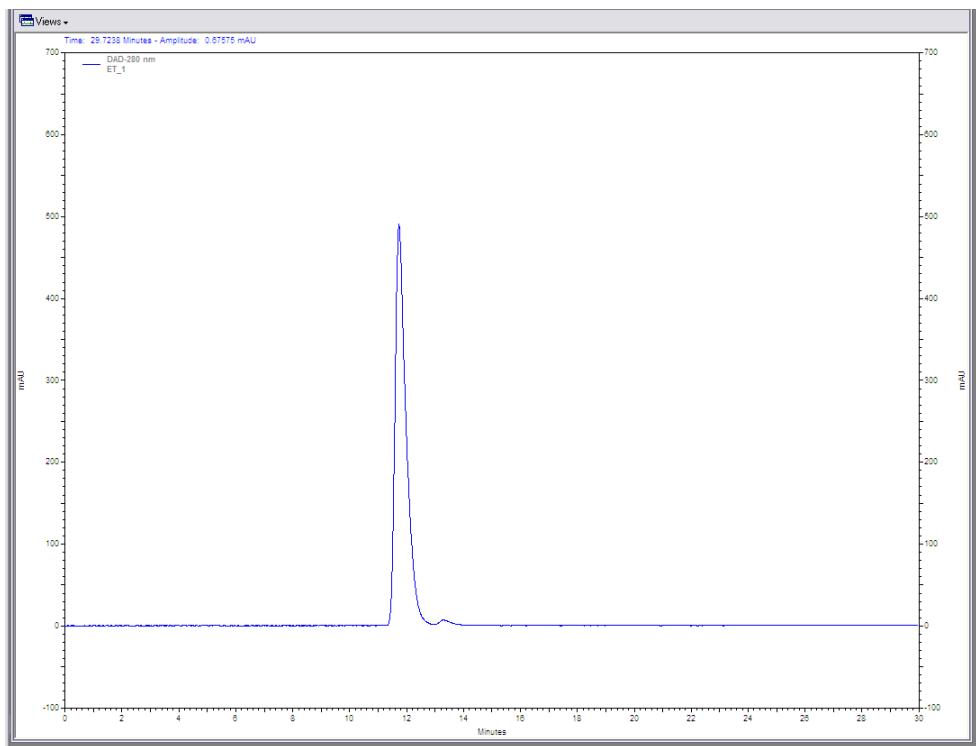
<sup>1</sup>H NMR of Compound 1-(4-Hydroxyphenyl)-12-butyl-1,12-dicarba-closo-dodecaborane

NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_05\_ET\_C\_patent\_no7  
sample #5 in CDCl<sub>3</sub> ET-WTB5 13C NMR, 298K



<sup>13</sup>C NMR of Compound 1-(4-Hydroxyphenyl)-12-butyl-1,12-dicarba-closo-dodecaborane



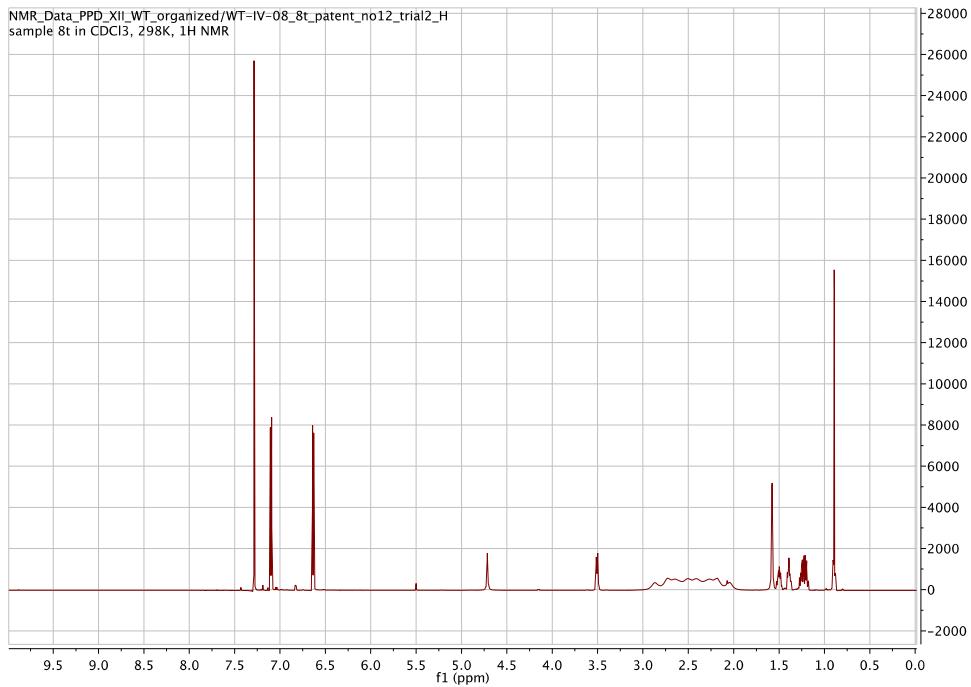
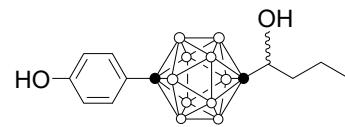


### DAD-280 nm

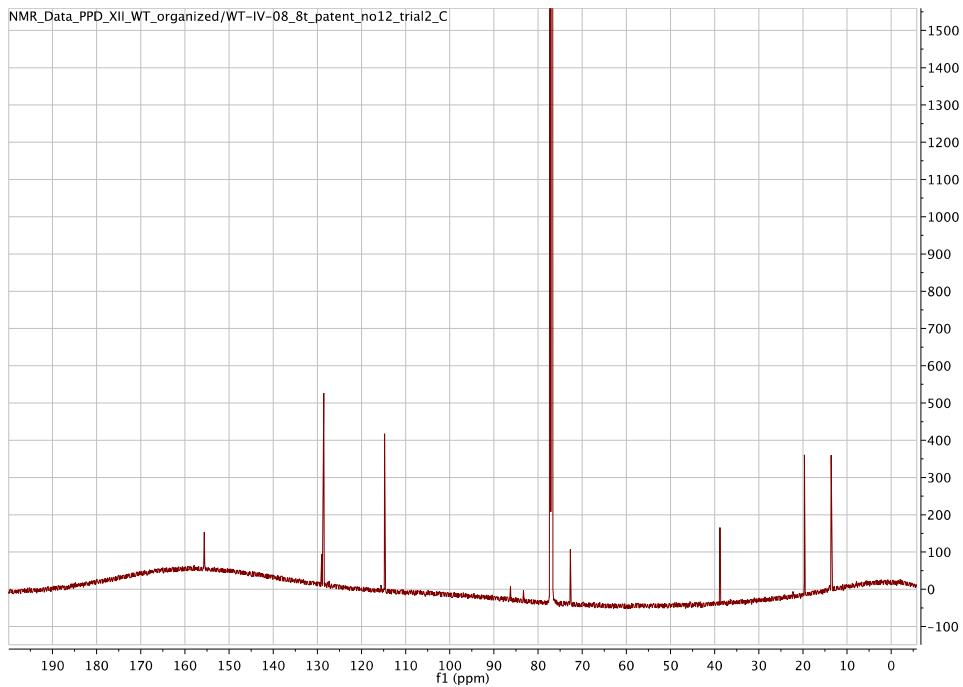
#### Results

| Retention Time | Area     | Area % | Height  | Height % |
|----------------|----------|--------|---------|----------|
| 11.733         | 52464432 | 98.83  | 1959245 | 98.85    |
| 13.287         | 620034   | 1.17   | 22871   | 1.15     |
| Totals         | 53084466 | 100.00 | 1982116 | 100.00   |

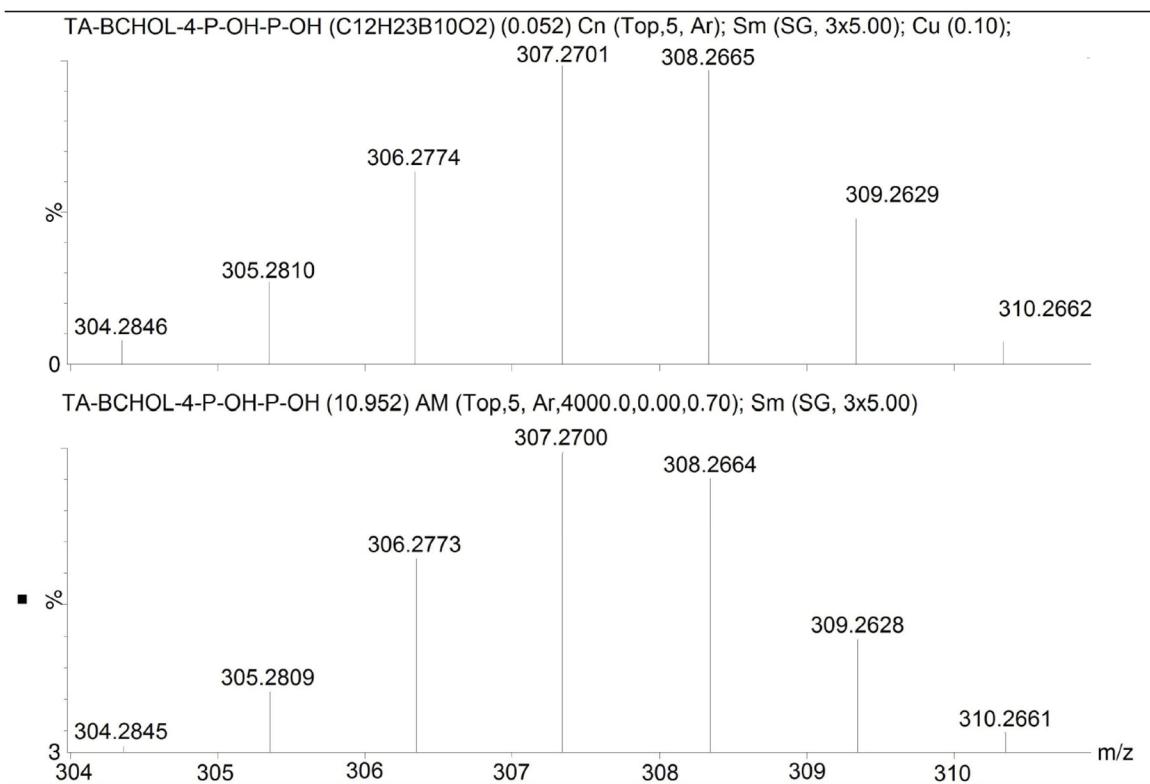
HPLC of Compound 1-(4-Hydroxyphenyl)-12-butyl-1,12-dicarba-*clos*-dodecaborane



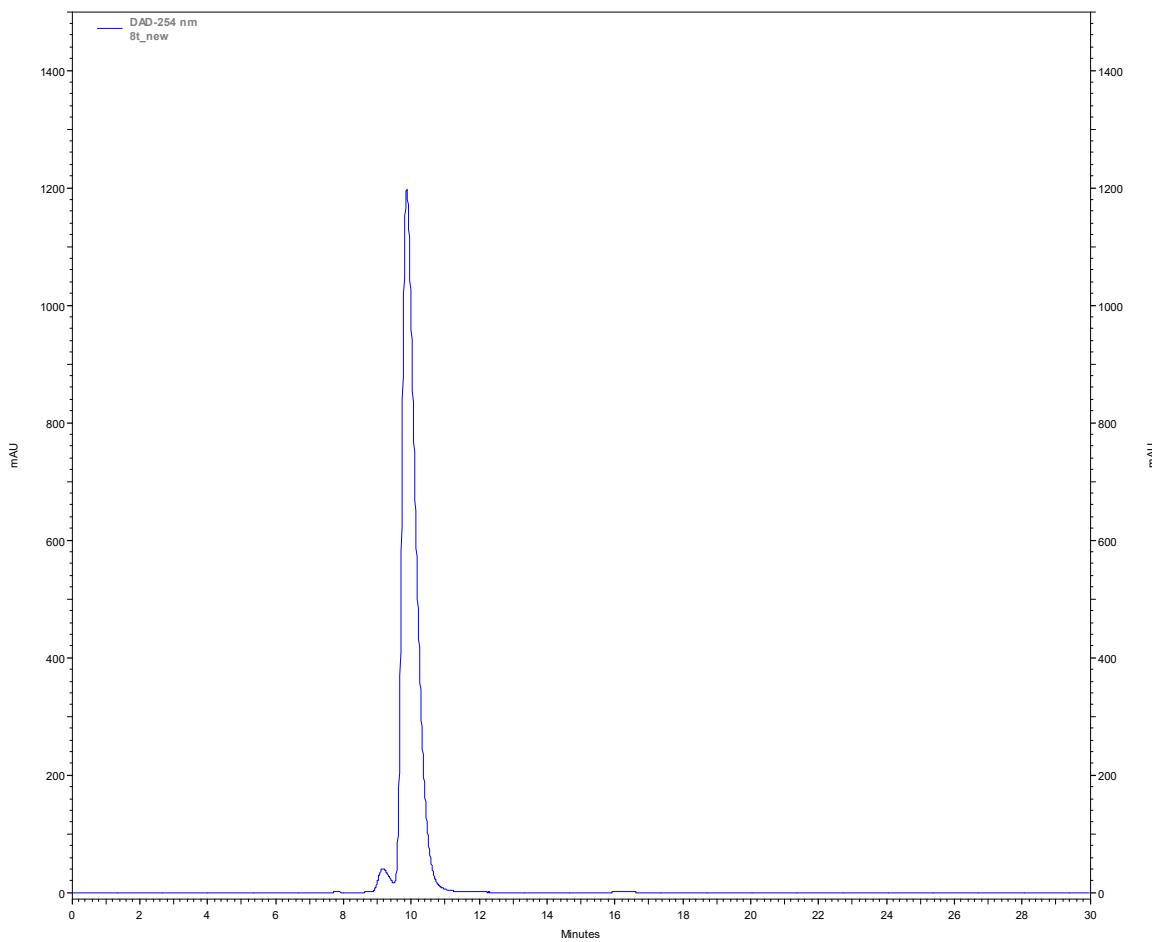
<sup>1</sup>H NMR of Compound 1-(4-Hydroxyphenyl)-12-(1-hydroxybutyl)-1,12-dicarba-closo-dodecaborane



<sup>13</sup>C NMR of Compound 1-(4-Hydroxyphenyl)-12-(1-hydroxybutyl)-1,12-dicarba-closo-dodecaborane



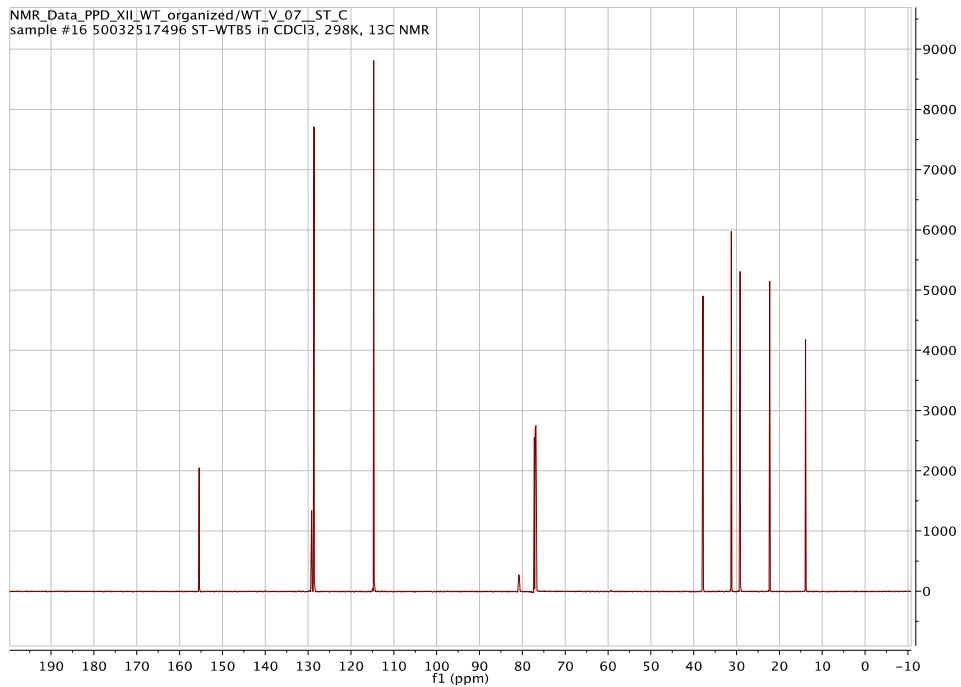
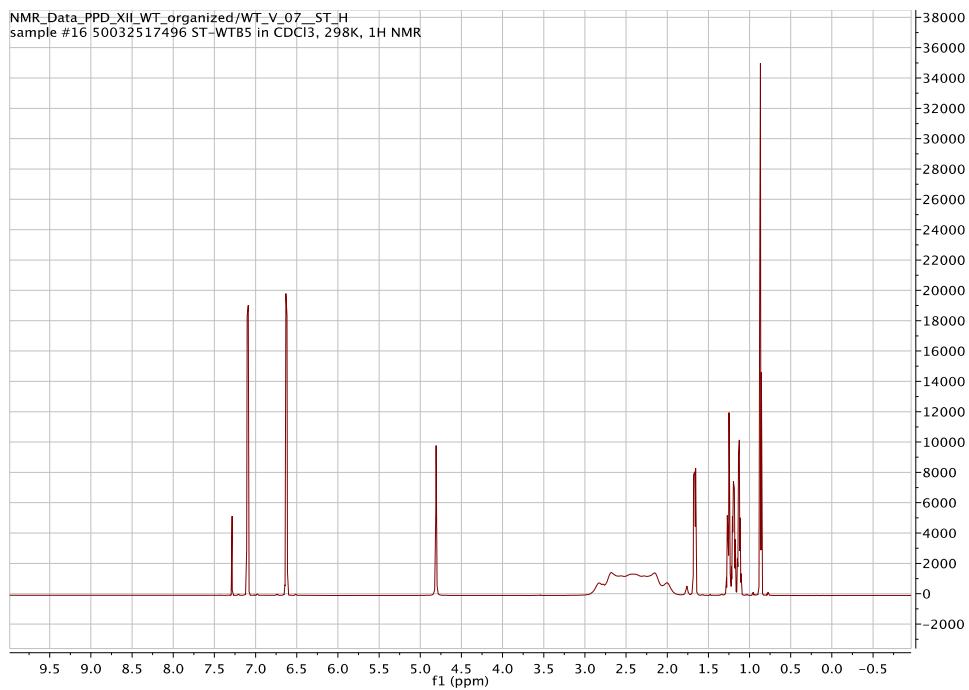
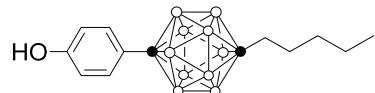
MS of Compound 1-(4-Hydroxyphenyl)-12-(1-hydroxybutyl)-1,12-dicarba-*clos*-dodecaborane



#### DAD-254 nm Results

| Retention Time | Area             | Area %        |
|----------------|------------------|---------------|
| 7.773          | 28460            | 0.02          |
| 9.167          | 2389747          | 1.77          |
| 9.867          | 132327460        | 98.10         |
| 16.147         | 143283           | 0.11          |
| <b>Totals</b>  | <b>134888950</b> | <b>100.00</b> |

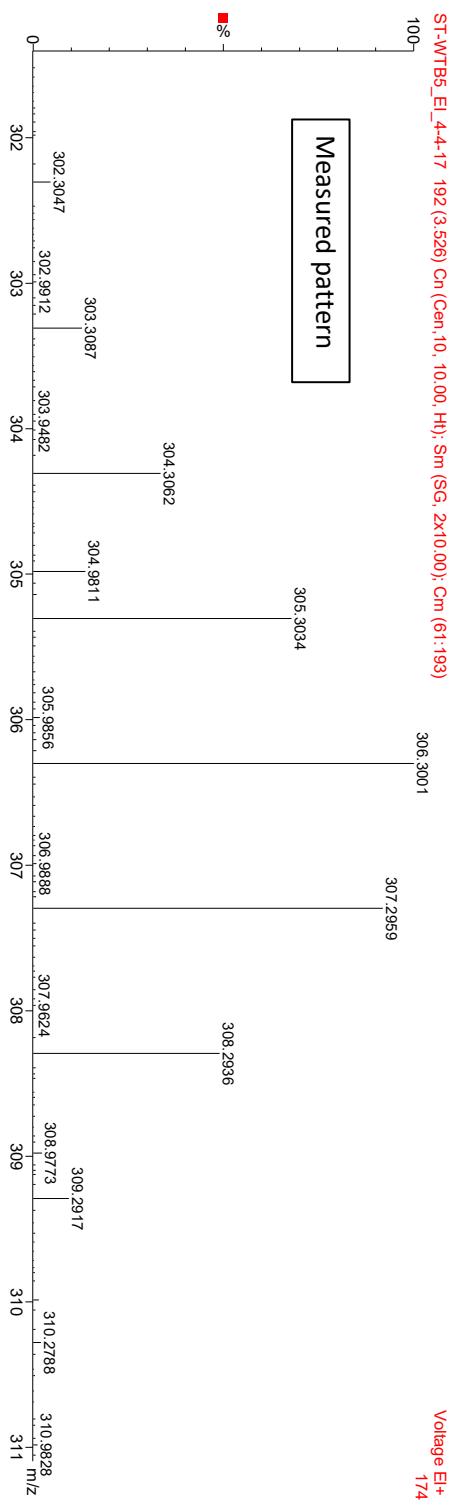
HPLC of Compound 1-(4-Hydroxyphenyl)-12-(1-hydroxybutyl)-1,12-dicarba-*clos*o-dodecaborane



Werner Tjarks ST-WTB5 EI 70 eV

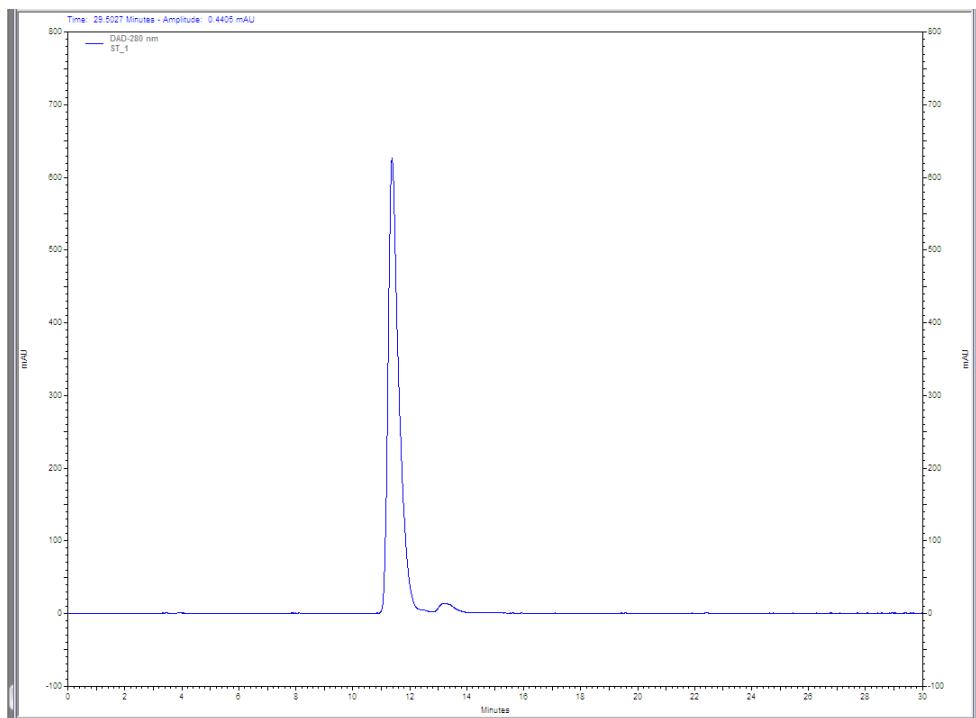


Theoretical pattern



Measured pattern

MS of Compound 1-(4-hydroxyphenyl)-12-pentyl-1,12-dicarba-*clos*o-dodecaborane

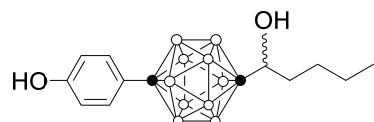


### DAD-280 nm

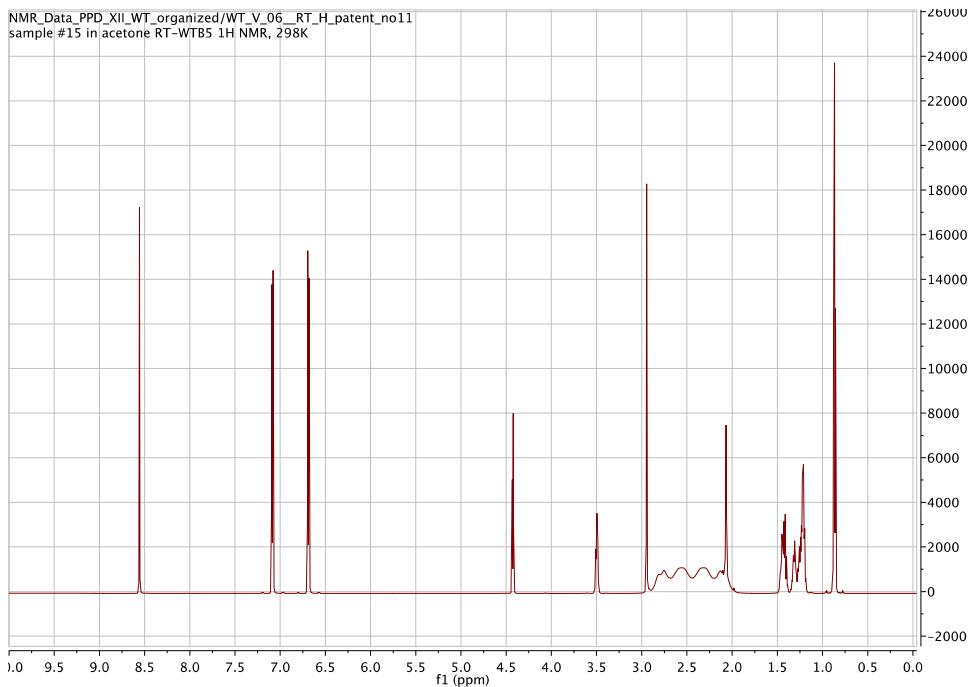
#### Results

| Retention Time | Area     | Area % | Height  | Height % |
|----------------|----------|--------|---------|----------|
| 11.373         | 65360761 | 97.39  | 2499818 | 98.11    |
| 13.160         | 1750915  | 2.61   | 48252   | 1.89     |
| Totals         | 67111676 | 100.00 | 2548070 | 100.00   |

HPLC of Compound 1-(4-hydroxyphenyl)-12-pentyl-1,12-dicarba-*clos*o-dodecaborane

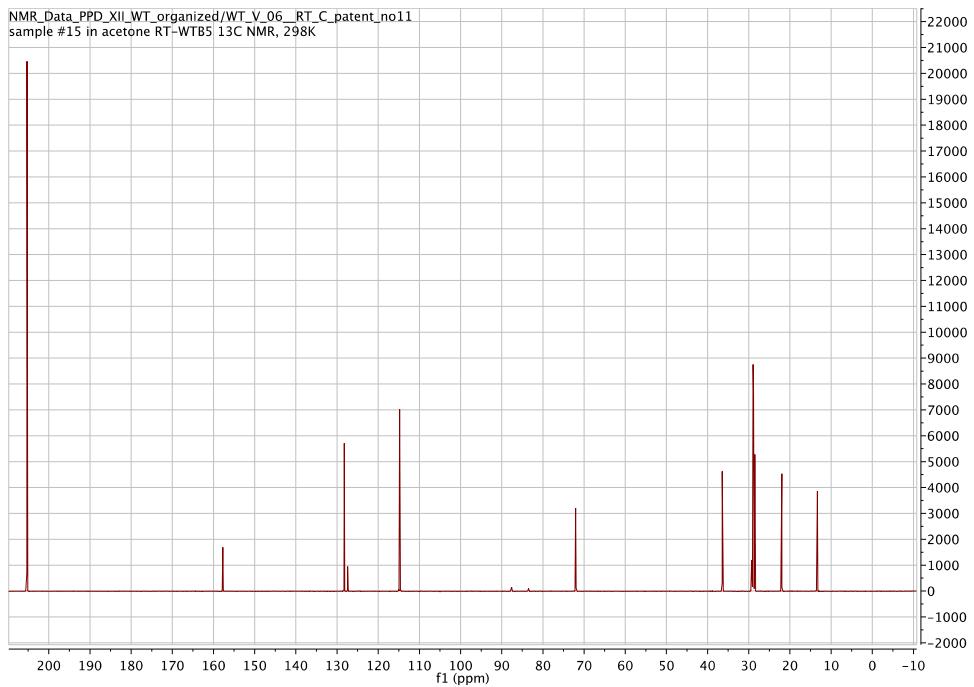


NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_06\_RT\_H\_patent\_no11  
sample #15 in acetone RT-WTB5 1H NMR, 298K

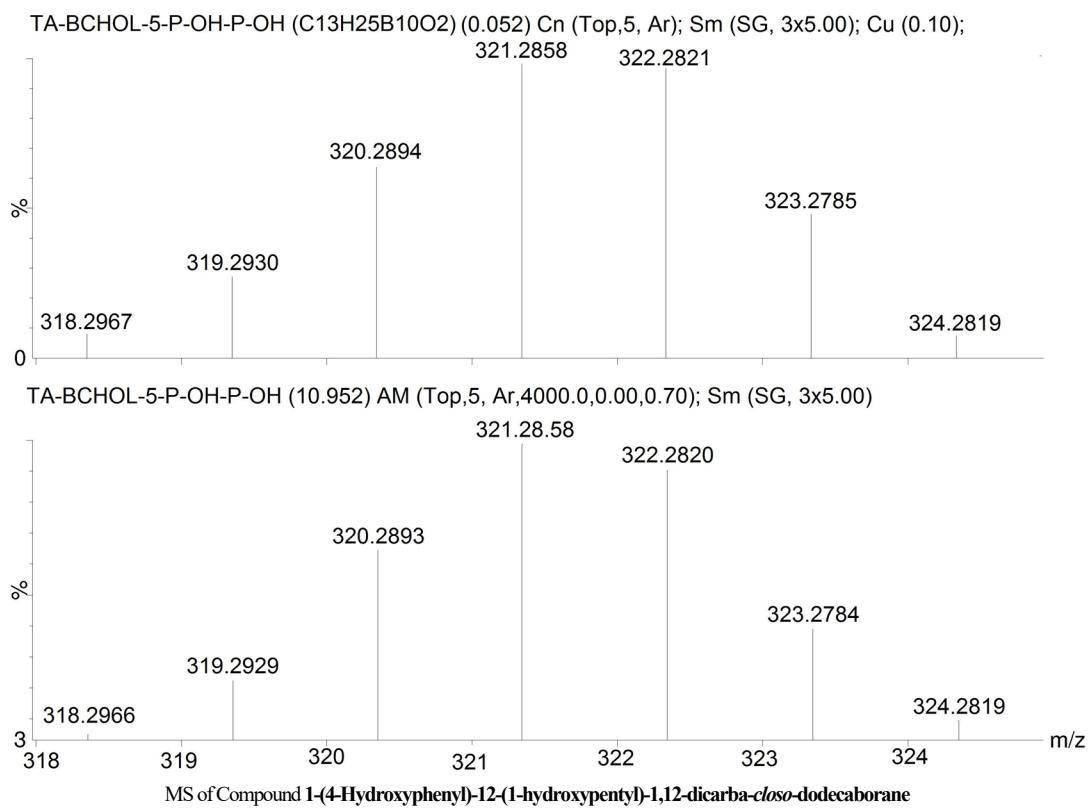


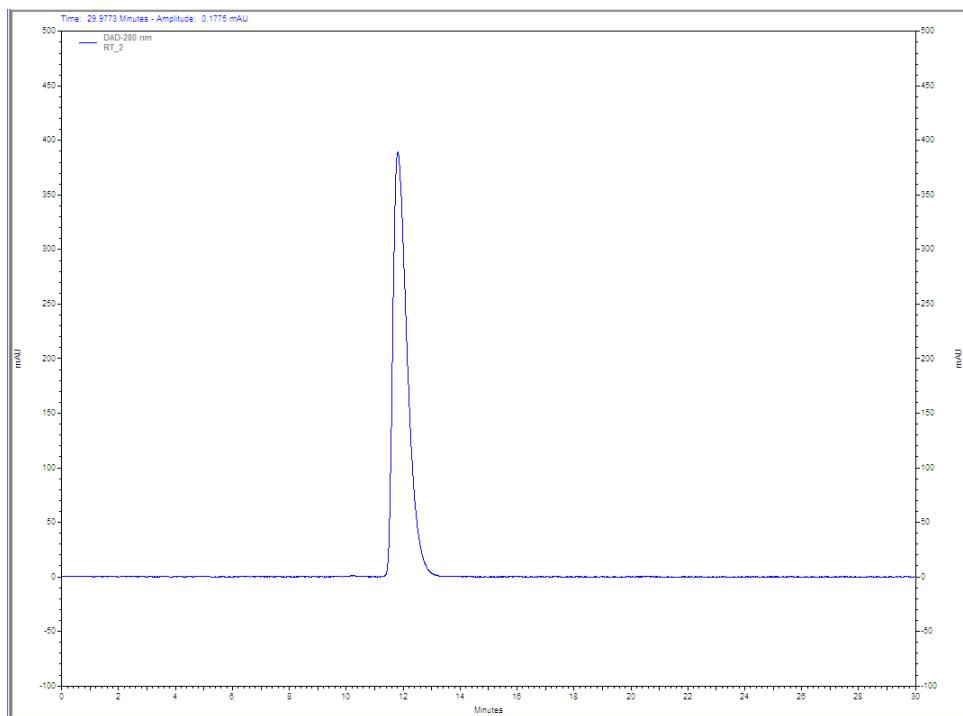
<sup>1</sup>H NMR of Compound 1-(4-Hydroxyphenyl)-12-(1-hydroxypentyl)-1,12-dicarba-closo-dodecaborane

NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_06\_RT\_C\_patent\_no11  
sample #15 in acetone RT-WTB5 <sup>13</sup>C NMR, 298K



<sup>13</sup>C NMR of Compound 1-(4-Hydroxyphenyl)-12-(1-hydroxypentyl)-1,12-dicarba-closo-dodecaborane



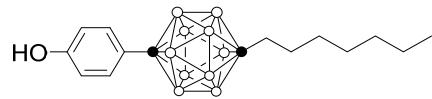


### DAD-280 nm

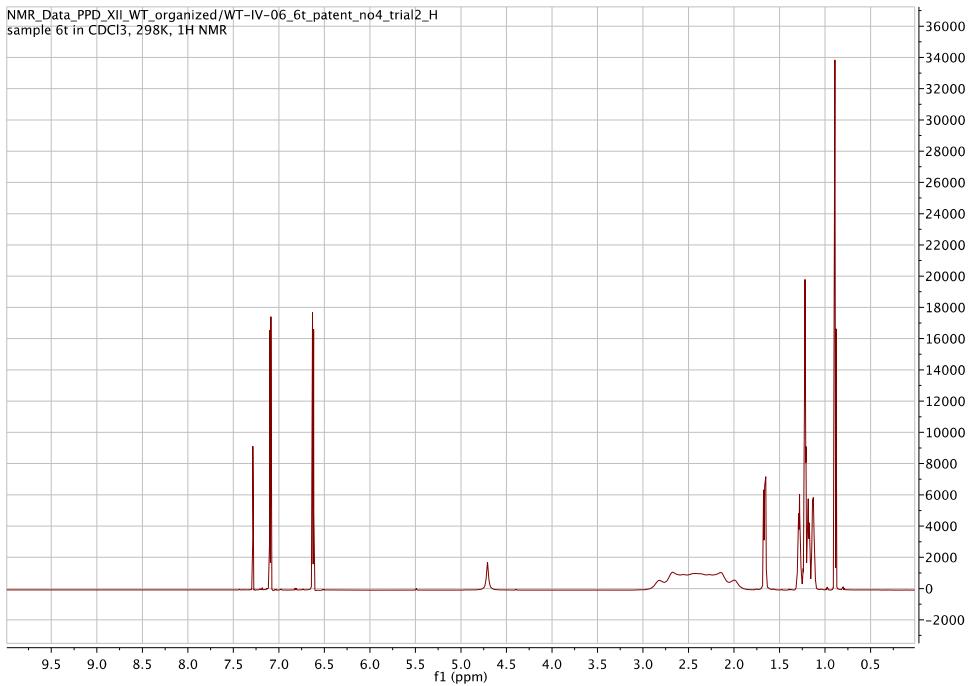
#### Results

| Retention Time | Area     | Area % | Height  | Height % |
|----------------|----------|--------|---------|----------|
| 10.267         | 127840   | 0.23   | 4509    | 0.29     |
| 11.813         | 54844477 | 99.77  | 1556891 | 99.71    |
| Totals         | 54972317 | 100.00 | 1561400 | 100.00   |

HPLC of Compound 1-(4-Hydroxyphenyl)-12-(1-hydroxypentyl)-1,12-dicarba-*clos*o-dodecaborane

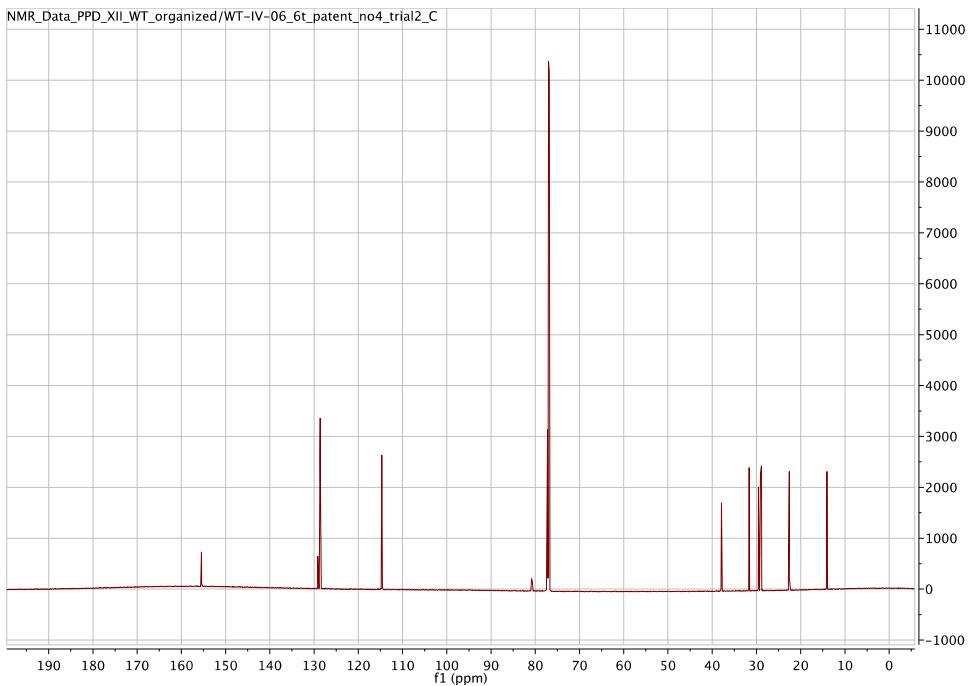


NMR\_Data\_PPD\_XII\_WT\_organized/WT-IV-06\_6t\_patent\_no4\_trial2\_H  
sample 6t in CDCl<sub>3</sub>, 298K, 1H NMR

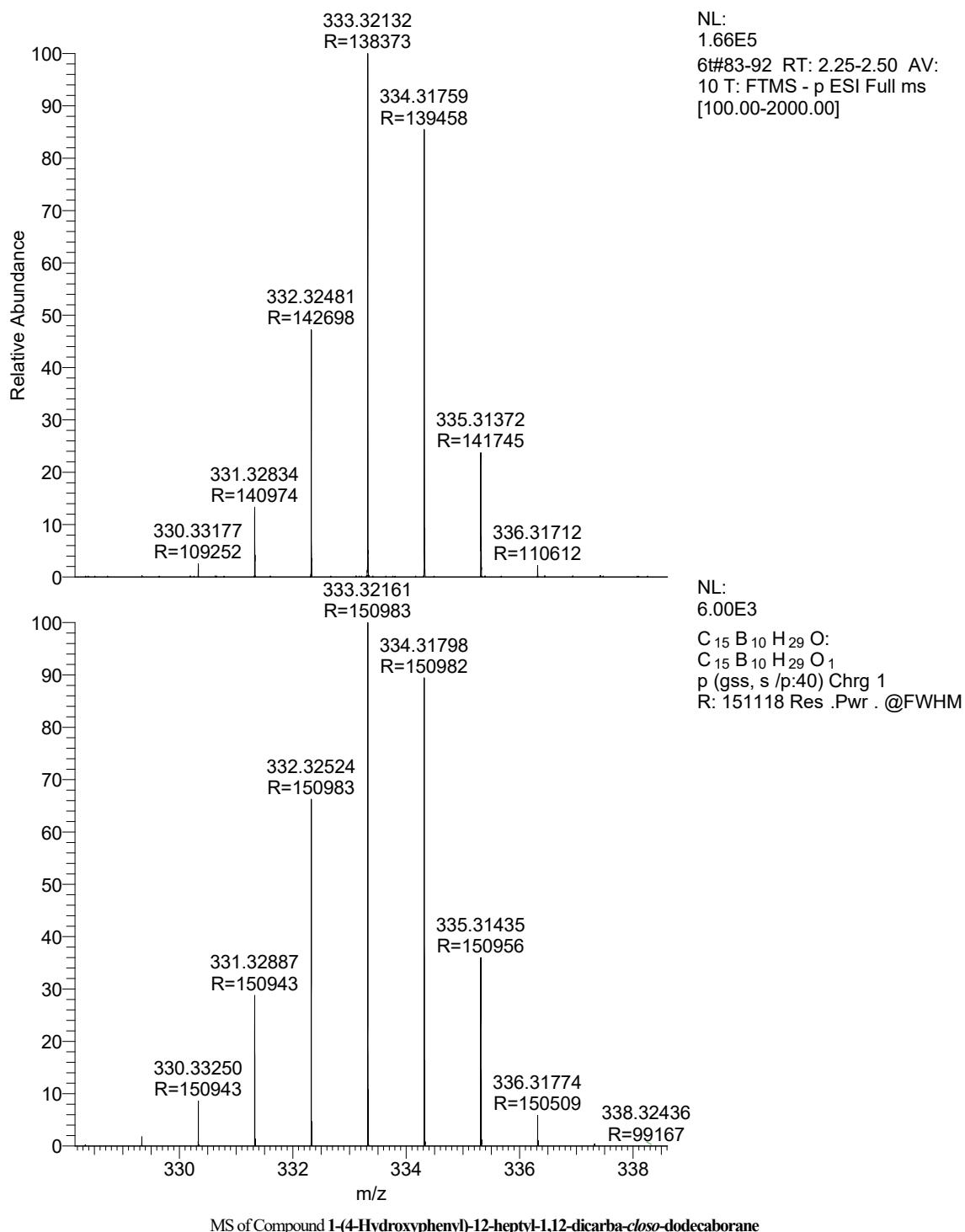


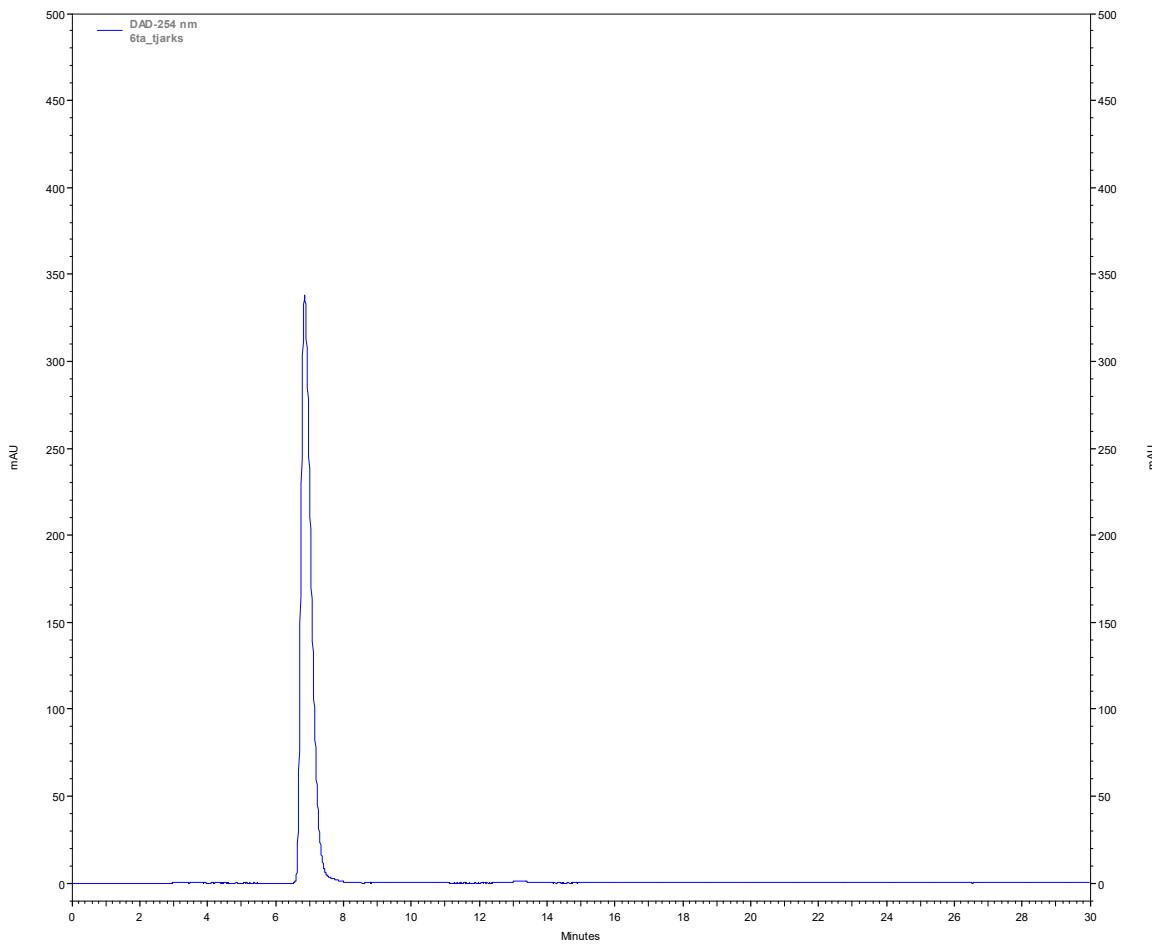
<sup>1</sup>H NMR of Compound 1-(4-Hydroxyphenyl)-12-heptyl-1,12-dicarba-closo-dodecaborane

NMR\_Data\_PPD\_XII\_WT\_organized/WT-IV-06\_6t\_patent\_no4\_trial2\_C



<sup>13</sup>C NMR of Compound 1-(4-Hydroxyphenyl)-12-heptyl-1,12-dicarba-closo-dodecaborane

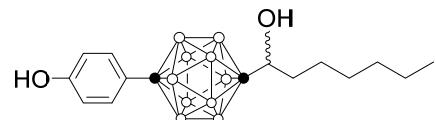




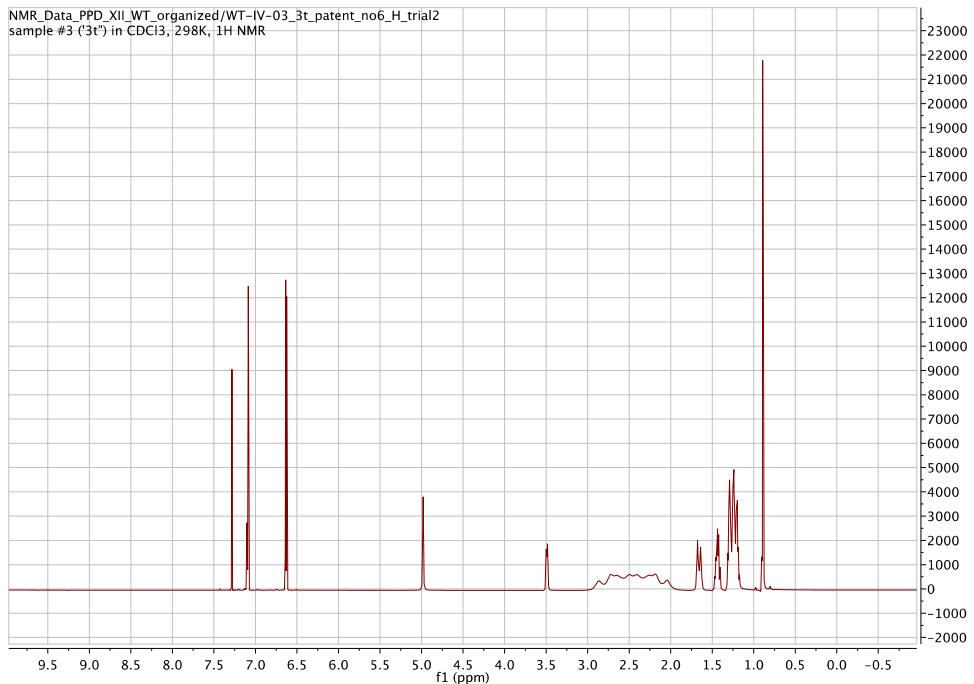
#### DAD-254 nm Results

| Retention Time | Area     | Area % |
|----------------|----------|--------|
| 6.860          | 28663162 | 99.52  |
| 13.193         | 136216   | 0.47   |
| 15.827         | 2217     | 0.01   |
| Totals         | 28801595 | 100.00 |

HPLC of Compound 1-(4-Hydroxyphenyl)-12-heptyl-1,12-dicarba-*clos*o-dodecaborane

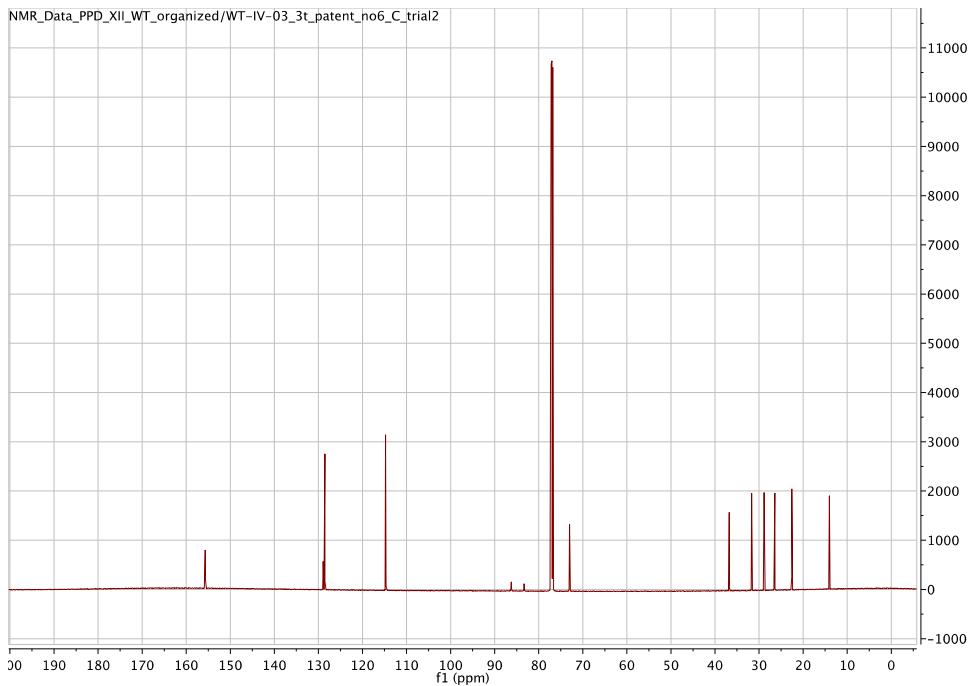


NMR\_Data\_PPD\_XII\_WT\_organized/WT-IV-03\_3t\_patent\_no6\_H\_trial2  
sample #3 ('3t') in CDCl<sub>3</sub>, 298K, <sup>1</sup>H NMR

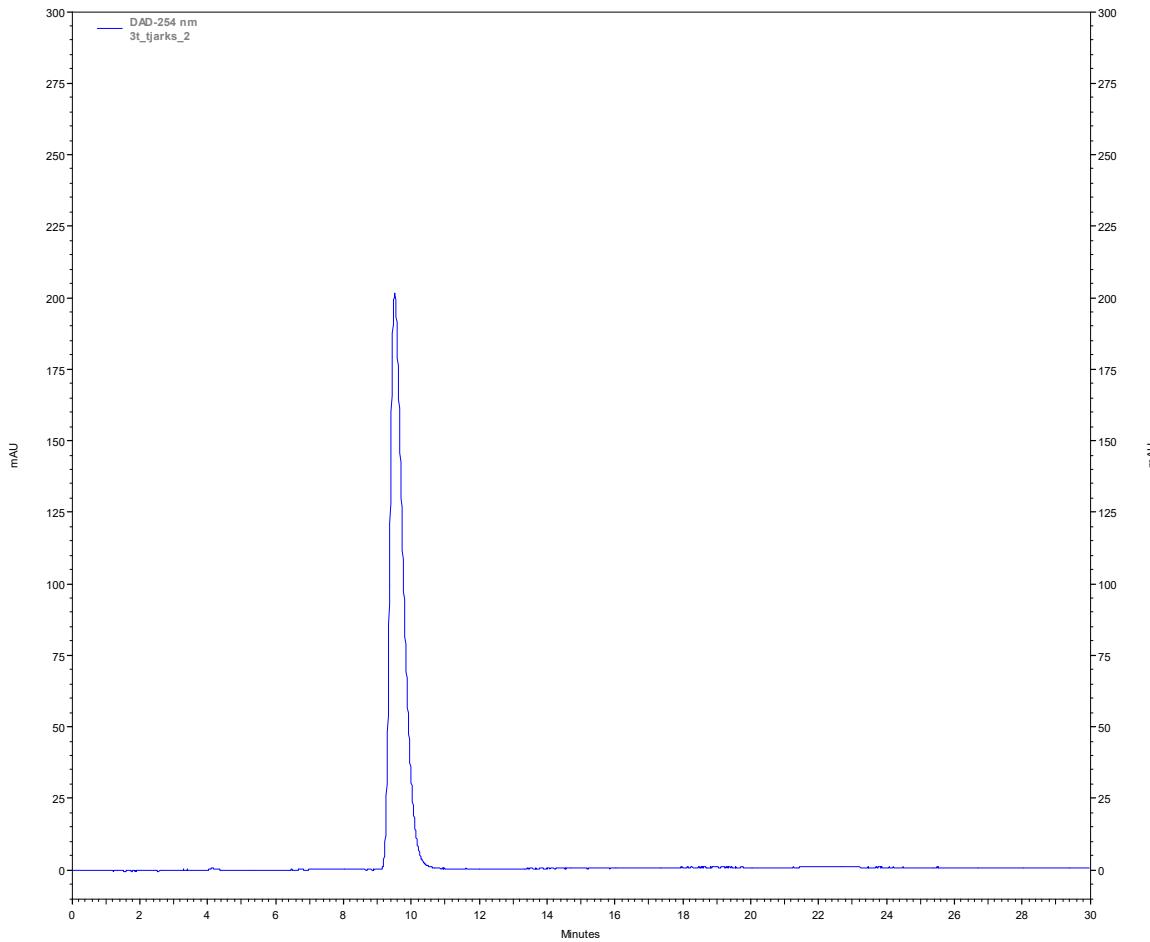
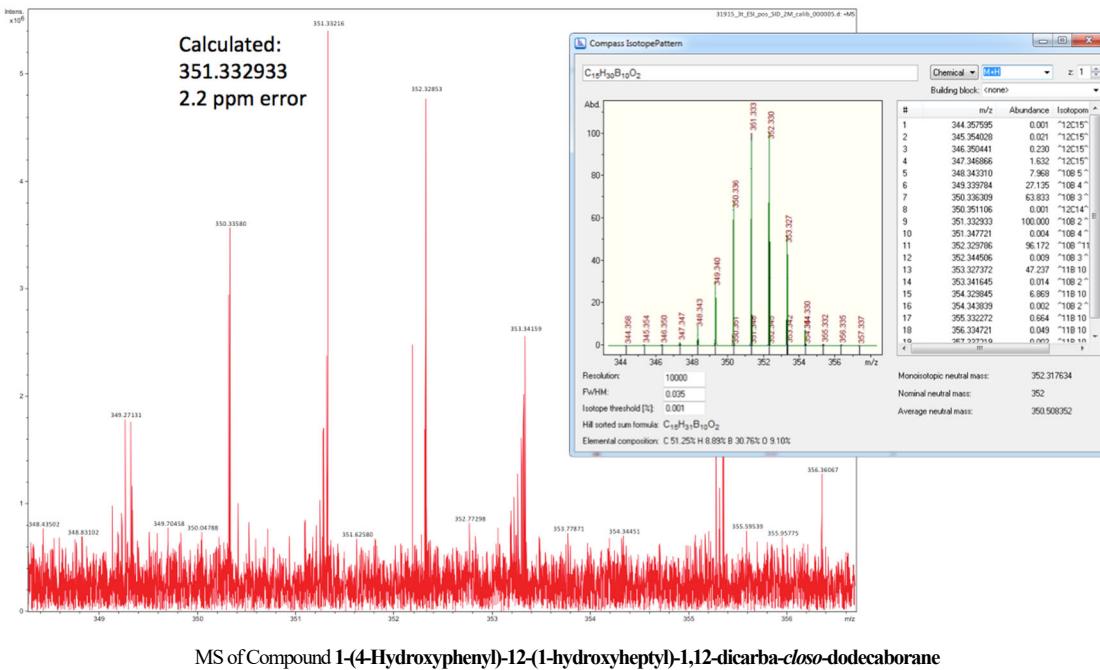


<sup>1</sup>H NMR of Compound 1-(4-Hydroxyphenyl)-12-(1-hydroxyheptyl)-1,12-dicarba-closo-dodecaborane

NMR\_Data\_PPD\_XII\_WT\_organized/WT-IV-03\_3t\_patent\_no6\_C\_trial2



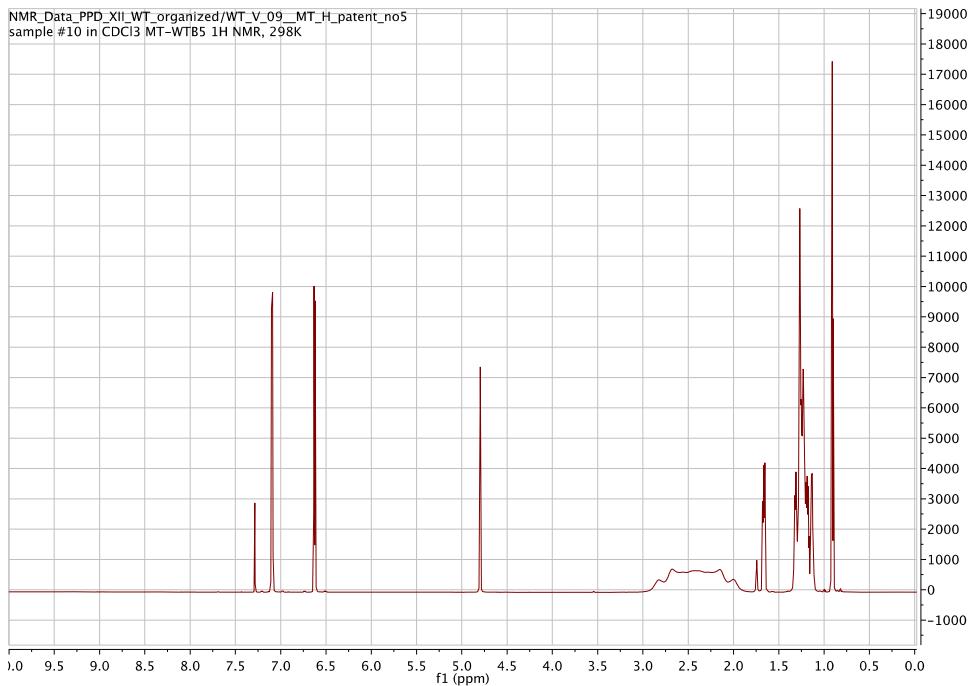
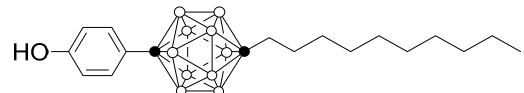
<sup>13</sup>C NMR of Compound 1-(4-Hydroxyphenyl)-12-(1-hydroxyheptyl)-1,12-dicarba-closo-dodecaborane



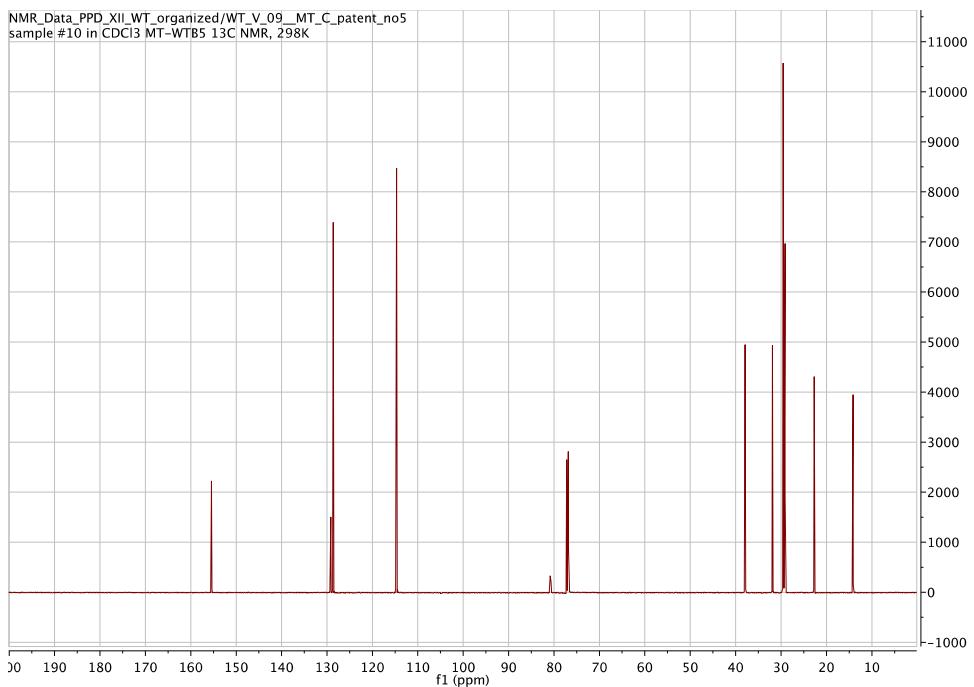
**DAD-254 nm Results**

| Retention Time | Area     | Area % |
|----------------|----------|--------|
| 9.520          | 22009765 | 99.94  |
| 21.467         | 471      | 0.00   |
| 21.500         | 16       | 0.00   |
| 22.093         | 9021     | 0.04   |
| 22.653         | 810      | 0.00   |
| 22.787         | 2098     | 0.01   |
| Totals         | 22022181 | 100.00 |

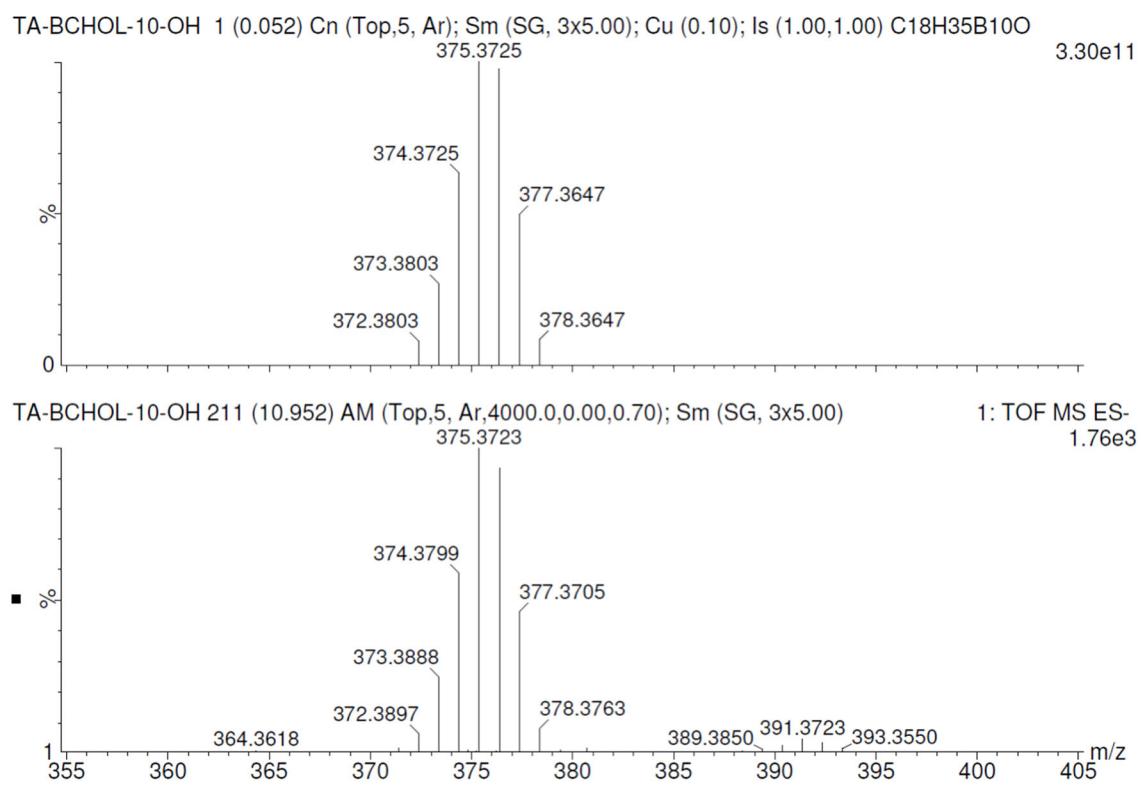
HPLC of Compound 1-(4-Hydroxyphenyl)-12-(1-hydroxyheptyl)-1,12-dicarba-*clos*o-dodecaborane

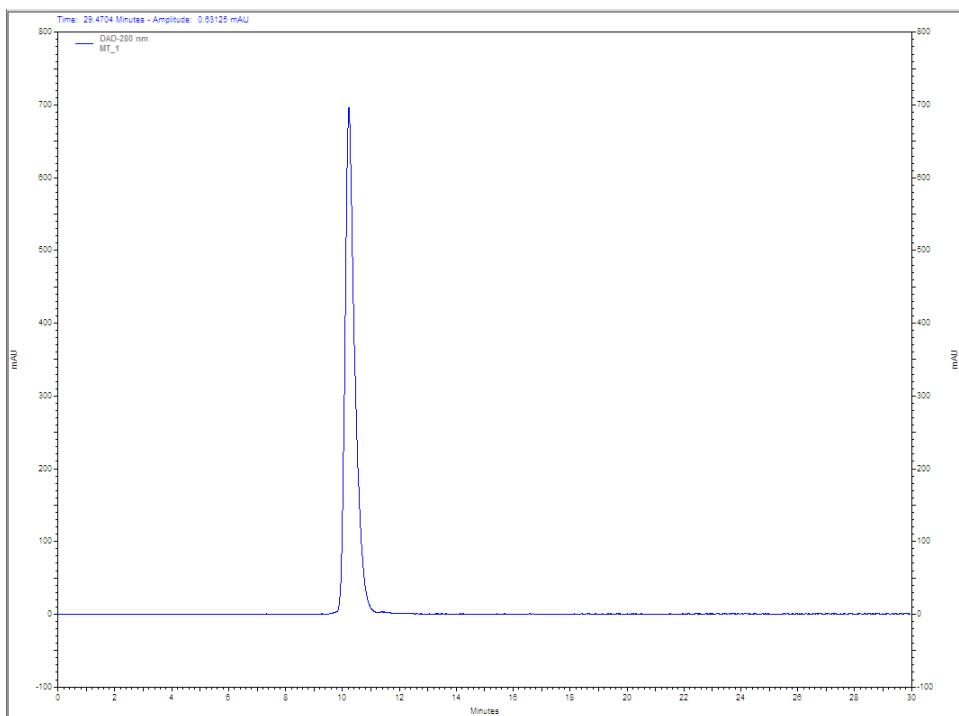


<sup>1</sup>H NMR of Compound 1-(4-Hydroxyphenyl)-12-decyl-1,12-dicarba-closo-dodecaborane



<sup>13</sup>C NMR of Compound 1-(4-Hydroxyphenyl)-12-decyl-1,12-dicarba-closo-dodecaborane



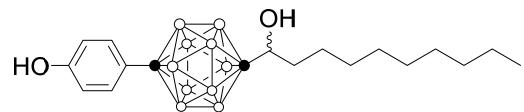


### DAD-280 nm

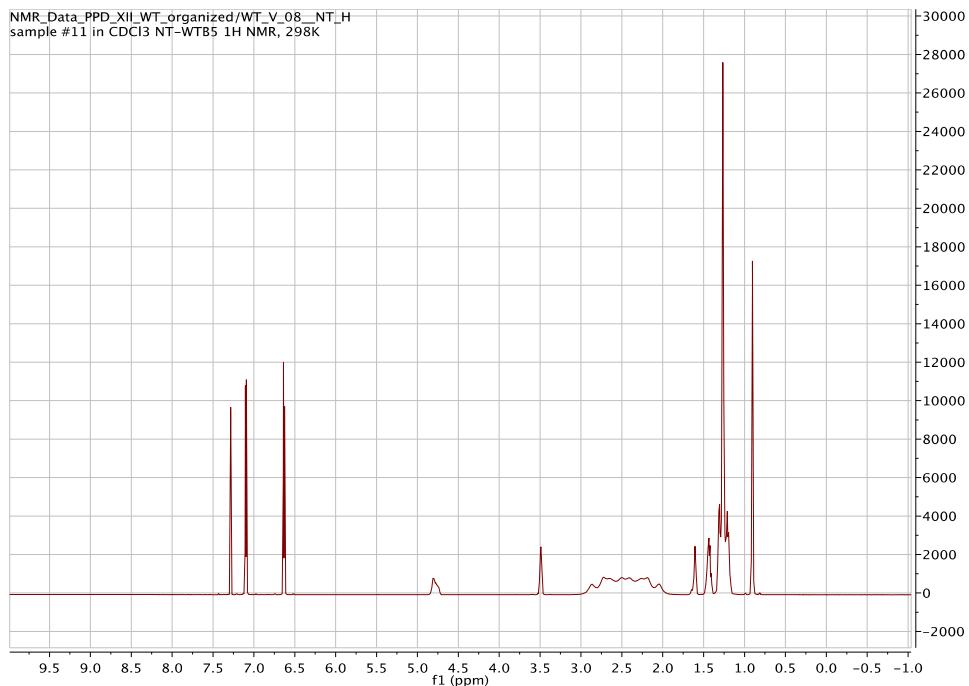
#### Results

| Retention Time | Area     | Area % | Height  | Height % |
|----------------|----------|--------|---------|----------|
| 10.227         | 66894742 | 100.00 | 2779851 | 100.00   |
| Totals         | 66894742 | 100.00 | 2779851 | 100.00   |

HPLC of Compound 1-(4-Hydroxyphenyl)-12-decyl-1,12-dicarba-*clos*-dodecaborane

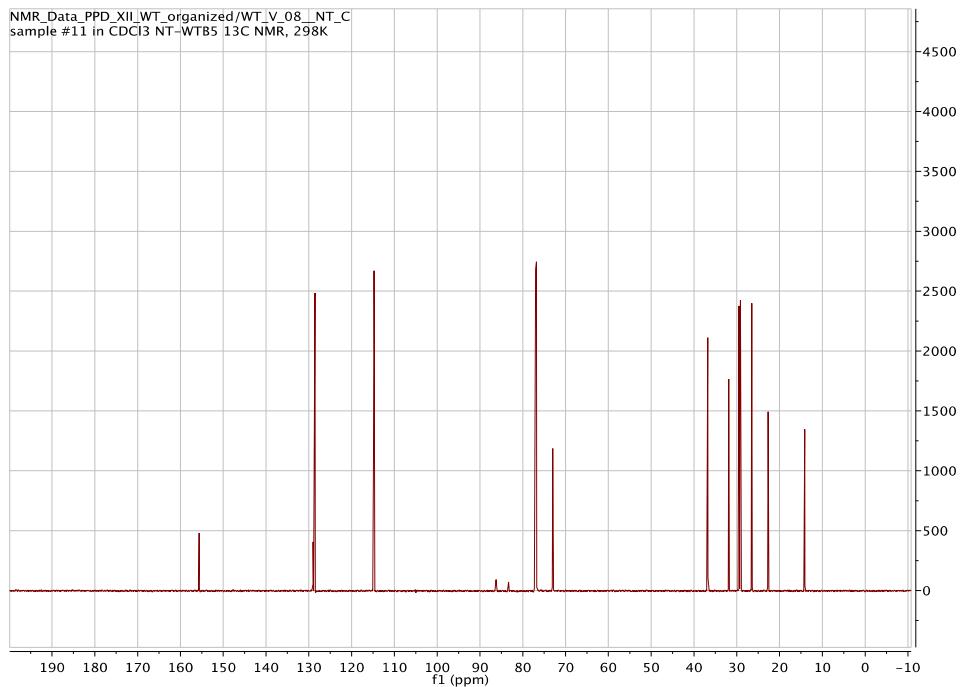


NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_08\_NT\_H  
sample #11 in CDCl<sub>3</sub> NT-WTB5 1H NMR, 298K



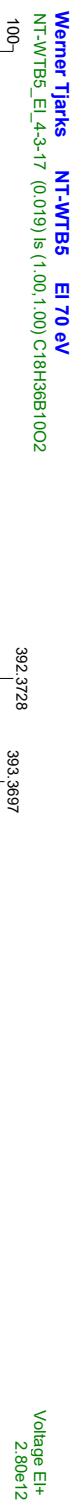
<sup>1</sup>H NMR of Compound (RS)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]decan-1-ol

NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_08\_NT\_C  
sample #11 in CDCl<sub>3</sub> NT-WTB5 13C NMR, 298K

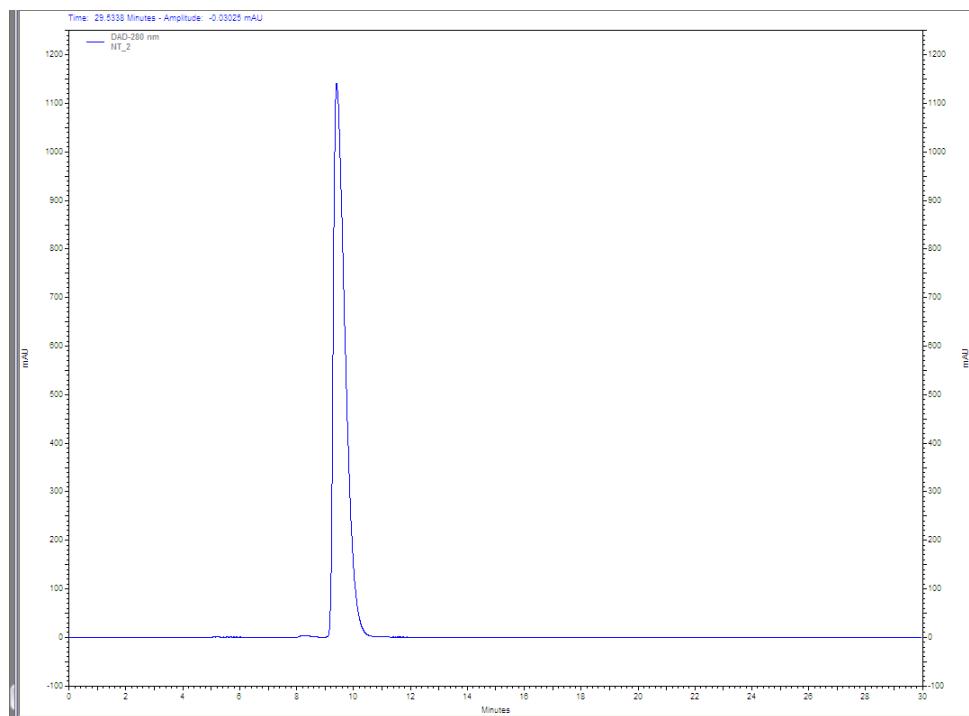


<sup>13</sup>C NMR of Compound (RS)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]decan-1-ol

Werner Tjarks NT-WTB5 EI 70 eV



MS of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*-dodecaborane-12-yl]decan-1-ol

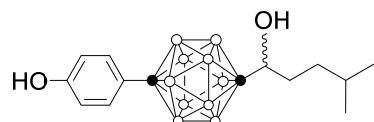


### DAD-280 nm

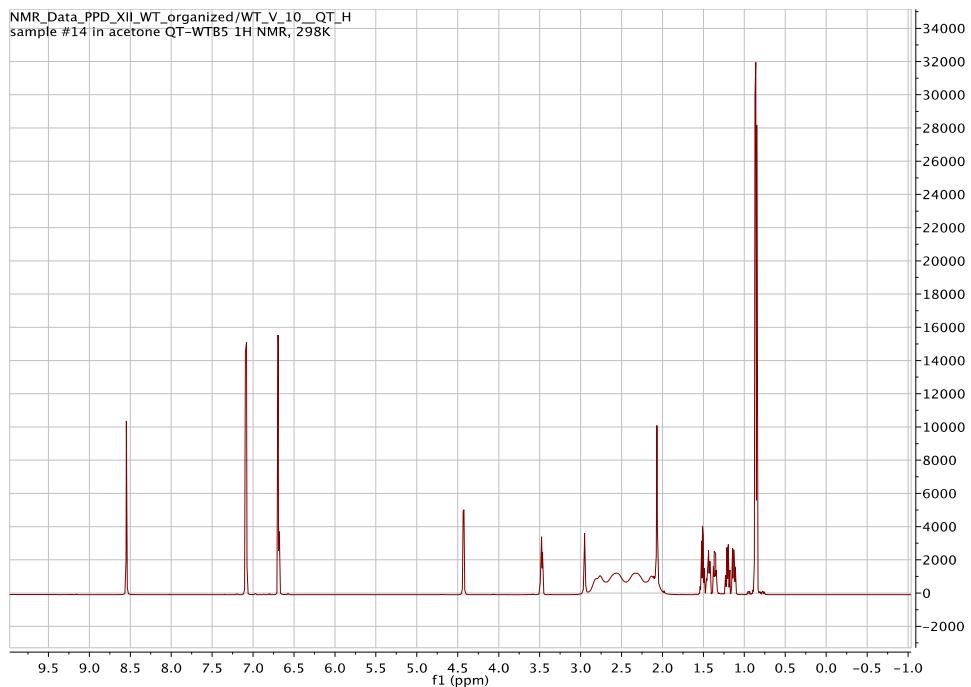
#### Results

| Retention Time | Area      | Area % | Height  | Height % |
|----------------|-----------|--------|---------|----------|
| 8.213          | 352950    | 0.27   | 16057   | 0.35     |
| 9.407          | 131569639 | 99.73  | 4564912 | 99.65    |
| Totals         | 131922589 | 100.00 | 4580969 | 100.00   |

HPLC of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]decan-1-ol

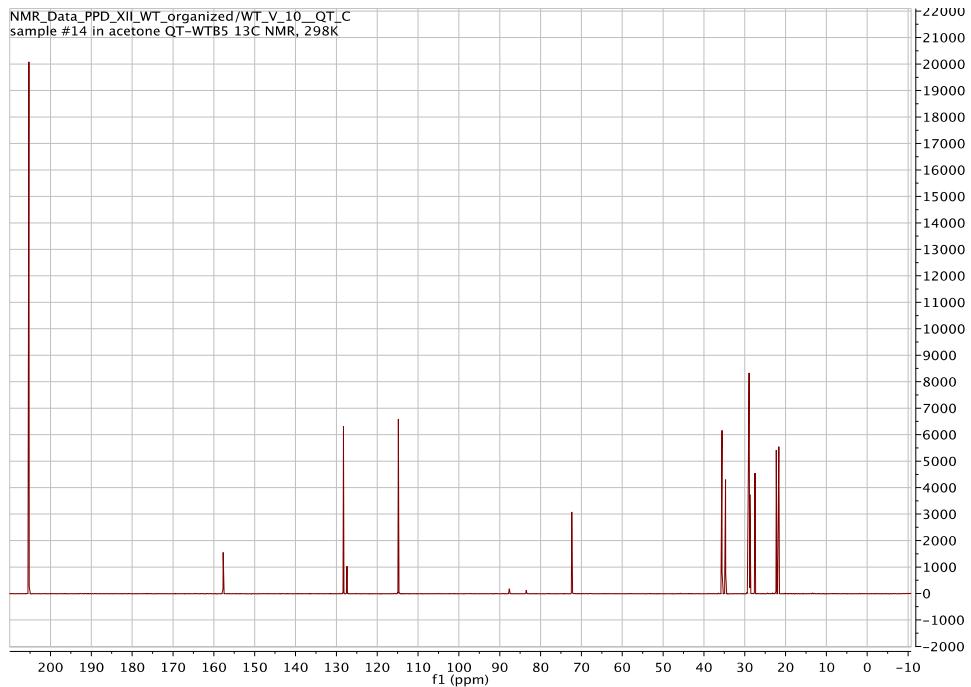


NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_10\_QT\_H  
sample #14 in acetone QT-WTB5 1H NMR, 298K



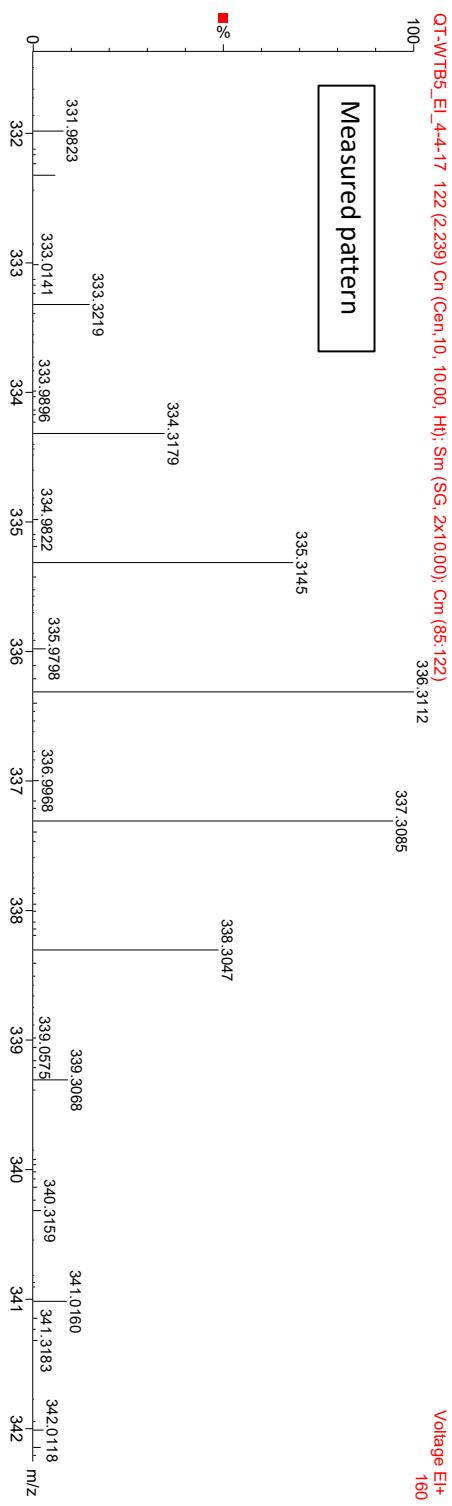
<sup>1</sup>H NMR of Compound (RS)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]-4-methylpentan-1-ol

NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_10\_QT\_C  
sample #14 in acetone QT-WTB5 13C NMR, 298K

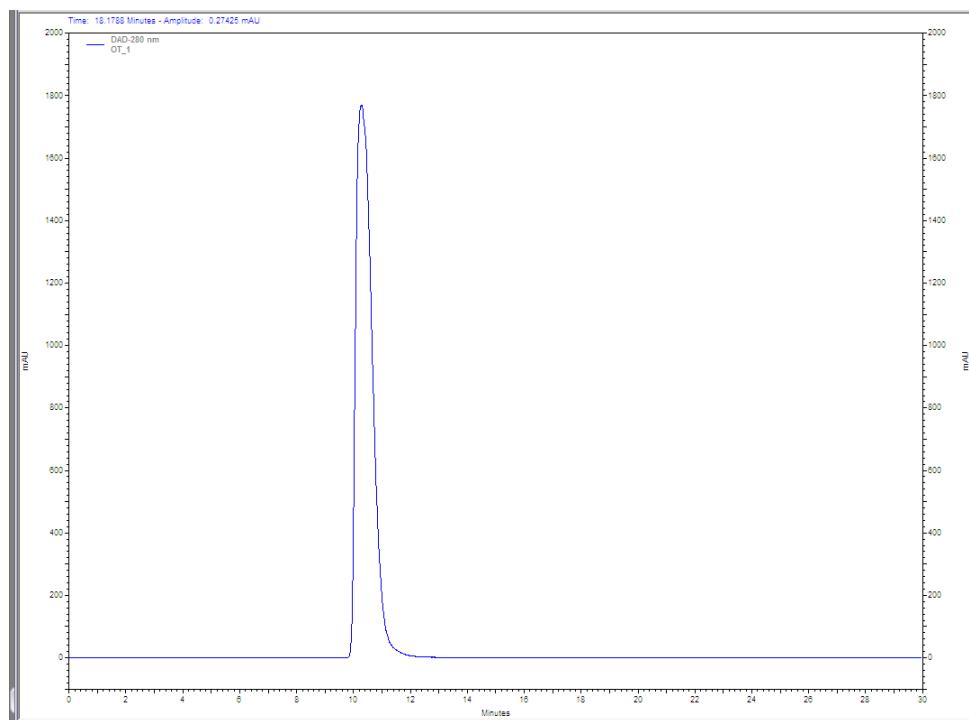


<sup>13</sup>C NMR of Compound (RS)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]-4-methylpentan-1-ol

Werner Tjarks QT-WTB5 EI 70 eV



MS of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]-4-methylpentan-1-ol

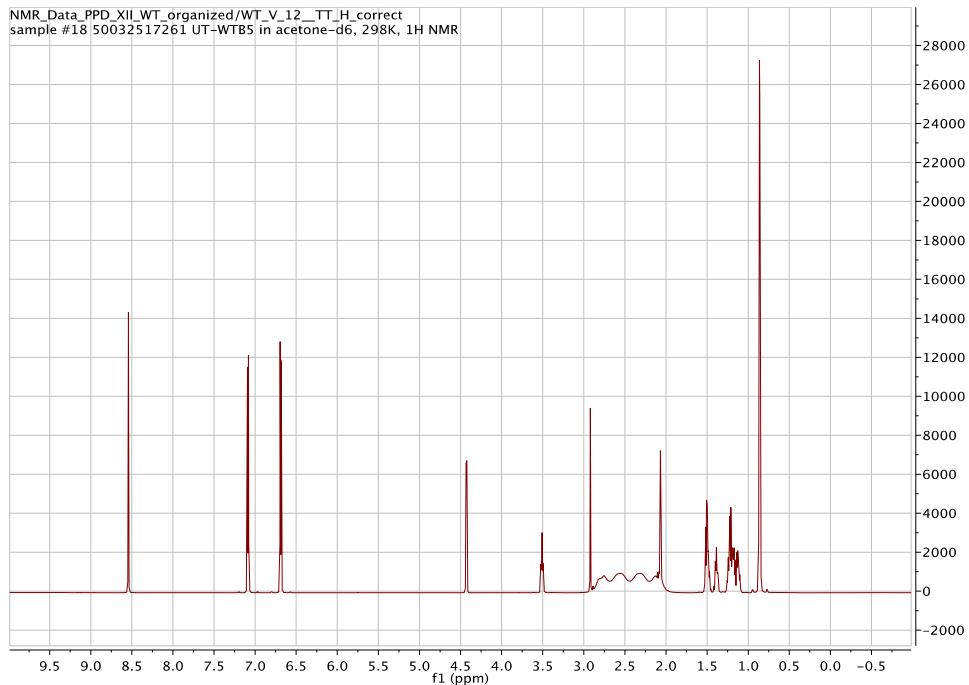
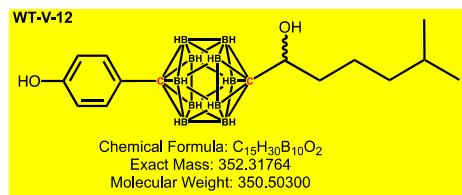


### DAD-280 nm

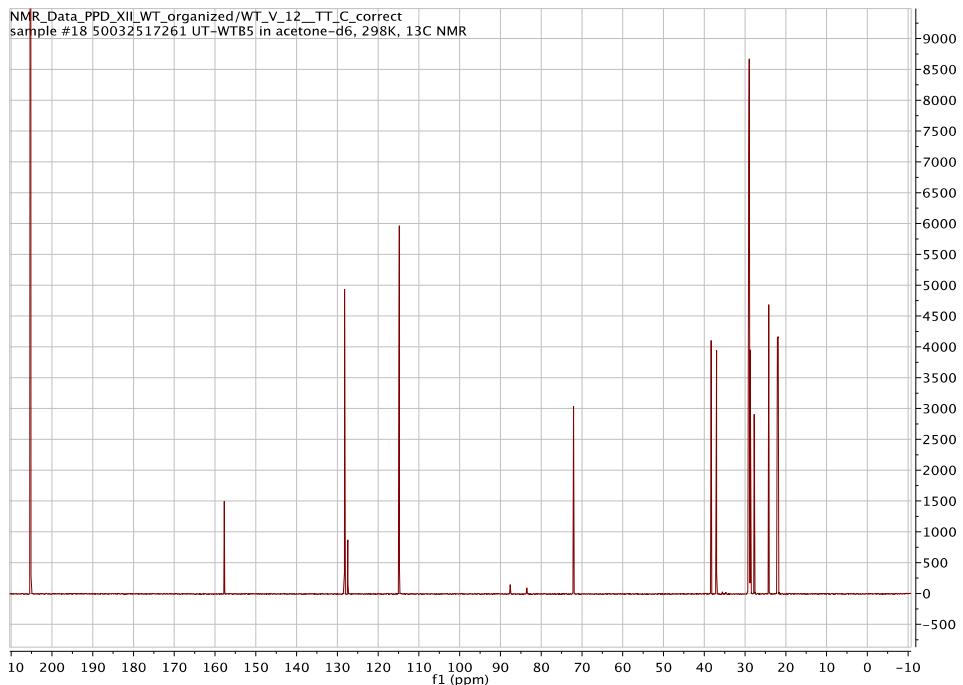
#### Results

| Retention Time | Area     | Area % | Height  | Height % |
|----------------|----------|--------|---------|----------|
| 9.613          | 342209   | 0.40   | 9516    | 0.48     |
| 11.073         | 85583352 | 99.60  | 1965175 | 99.52    |
| Totals         | 85925561 | 100.00 | 1974691 | 100.00   |

HPLC of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*-dodecaborane-12-yl]-4-methylpentan-1-ol



$^1\text{H}$  NMR of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl] -5-methylhexan-1-ol



$^{13}\text{C}$  NMR of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl] -5-methylhexan-1-ol

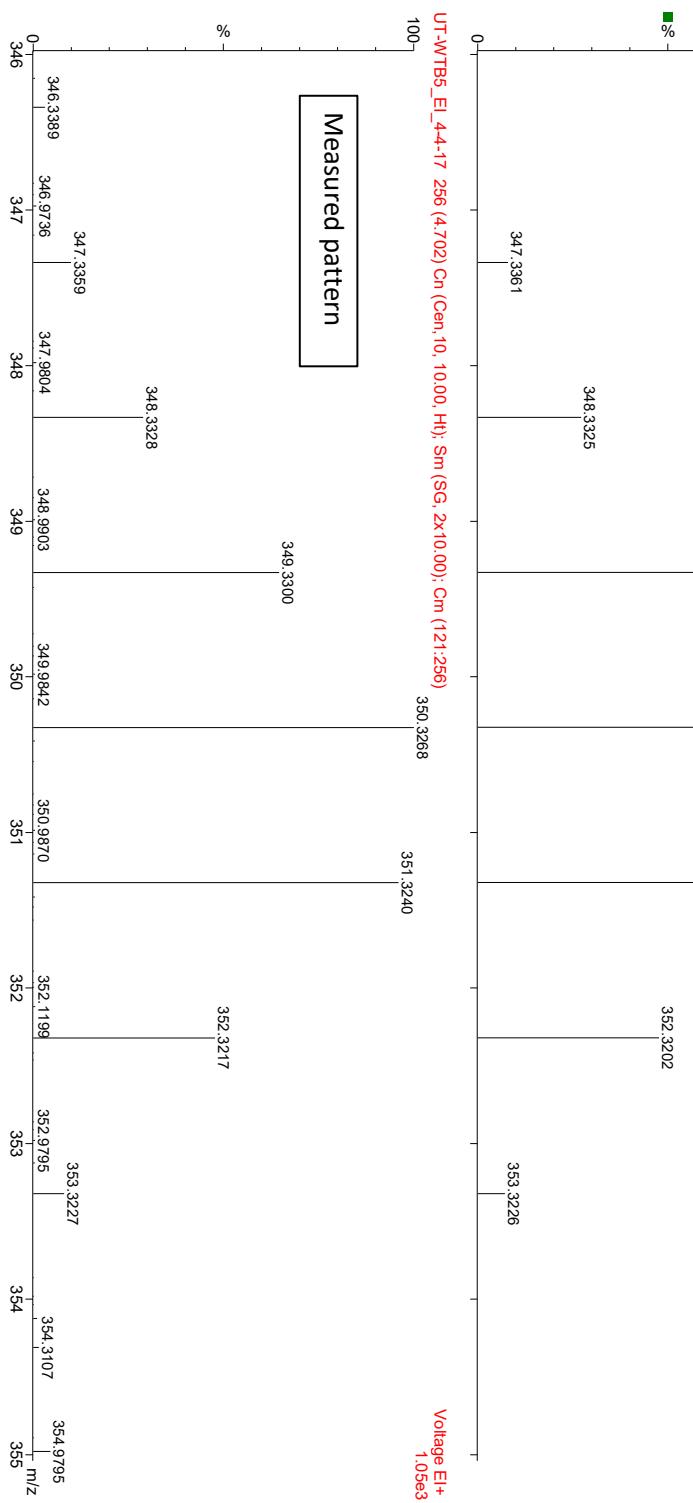
Werner Tjarks TT-WTB5 EI 70 eV

Werner Tjarks UT-WTB5 EI 70 eV  
UT-WTB5\_EI\_4-4-17 (0.019) ls (1.00,1.00) C15H30B10O2

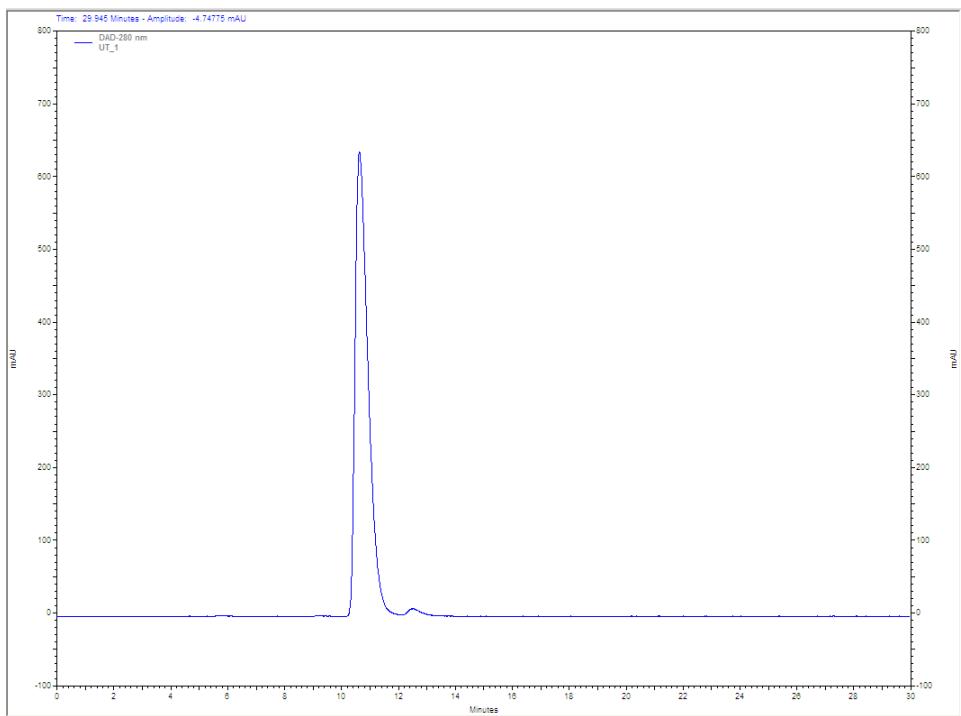
Voltage El<sup>+</sup>  
2.84e12

Voltage El<sup>+</sup>  
1.05e3

Theoretical pattern



MS of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*-dodecaborane-12-yl]-5-methylhexan-1-ol

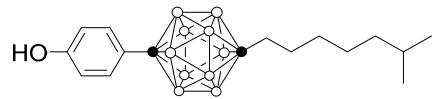


### DAD-280 nm

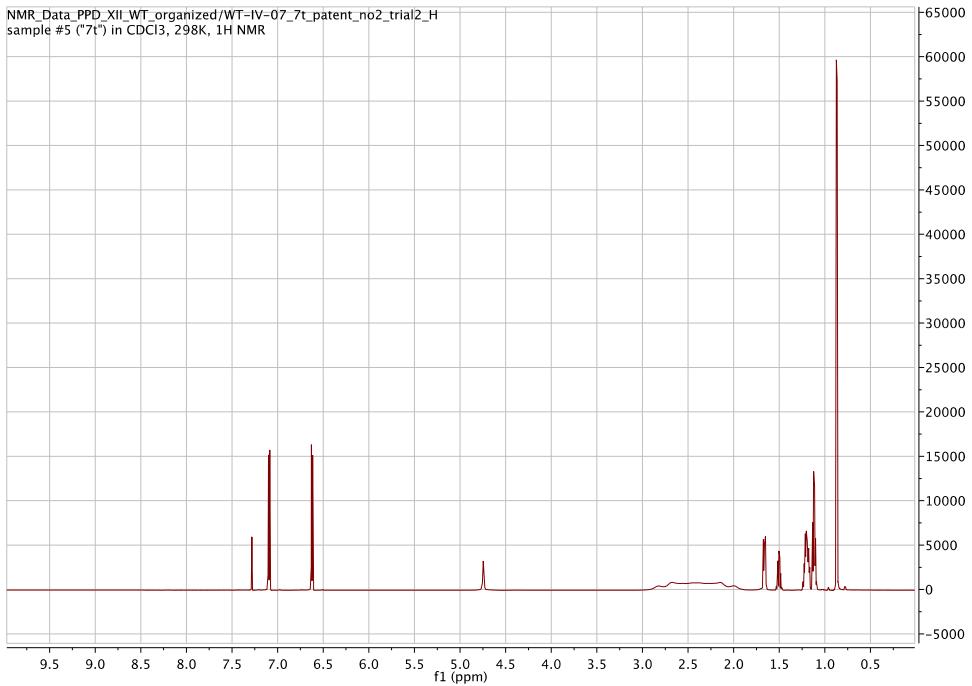
#### Results

| Retention Time | Area            | Area %        | Height         | Height %      |
|----------------|-----------------|---------------|----------------|---------------|
| 5.767          | 133837          | 0.16          | 4040           | 0.16          |
| 9.253          | 67789           | 0.08          | 2902           | 0.11          |
| 10.627         | 80517401        | 98.39         | 2552447        | 98.34         |
| 12.520         | 1116293         | 1.36          | 36152          | 1.39          |
| <b>Totals</b>  | <b>81835320</b> | <b>100.00</b> | <b>2595541</b> | <b>100.00</b> |

HPLC of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*-dodecaborane-12-yl] |-5-methylhexan-1-ol

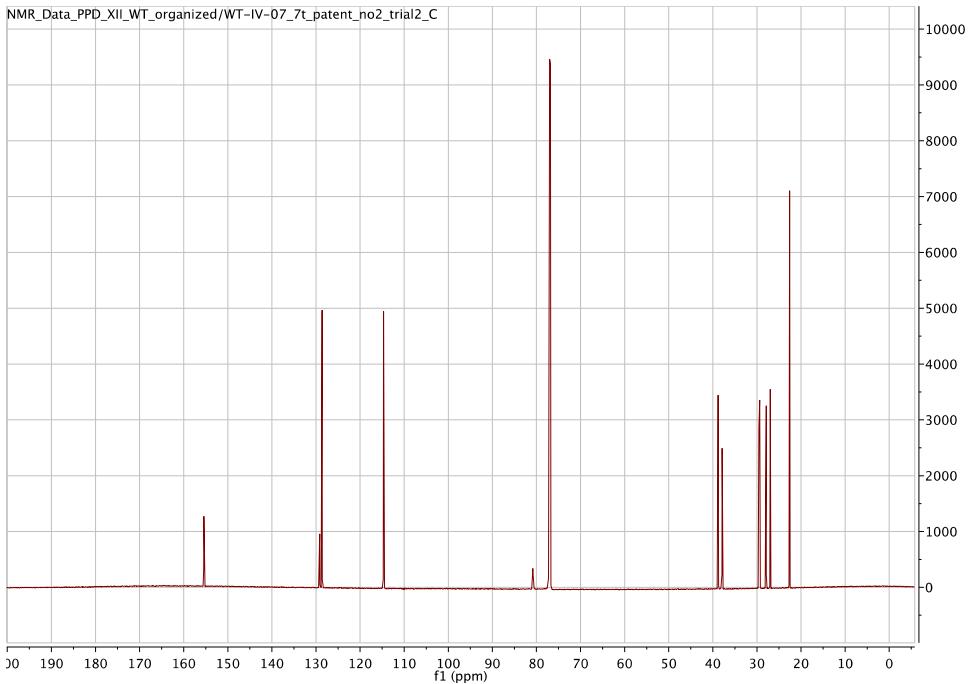


NMR\_Data\_PPD\_XII\_WT\_organized/WT-IV-07\_7t\_patent\_no2\_trial2\_H  
sample #5 ("7t") in CDCl<sub>3</sub>, 298K, 1H NMR

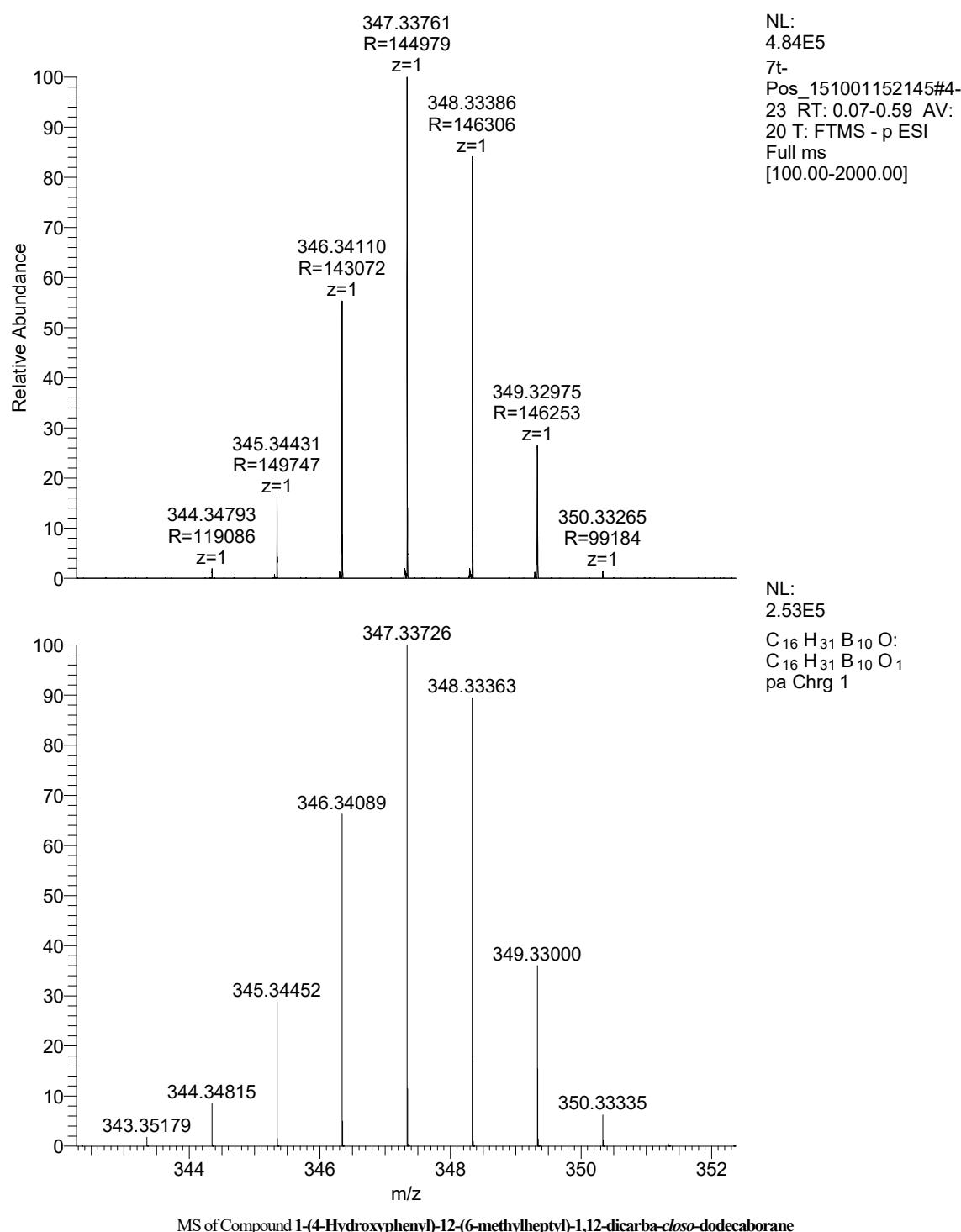


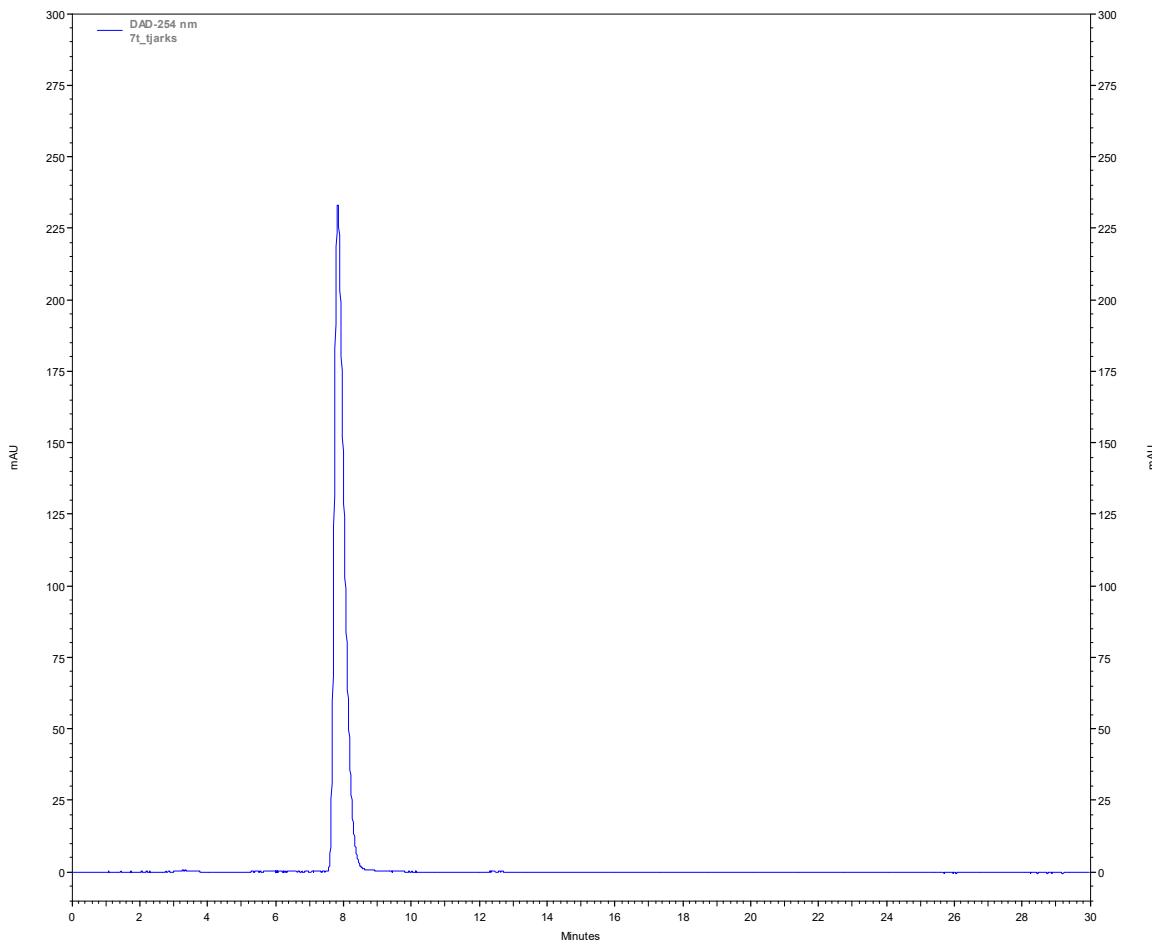
<sup>1</sup>H NMR of Compound 1-(4-Hydroxyphenyl)-12-(6-methylheptyl)-1,12-dicarba-closo-dodecaborane

NMR\_Data\_PPD\_XII\_WT\_organized/WT-IV-07\_7t\_patent\_no2\_trial2\_C



<sup>13</sup>C NMR of Compound 1-(4-Hydroxyphenyl)-12-(6-methylheptyl)-1,12-dicarba-closo-dodecaborane

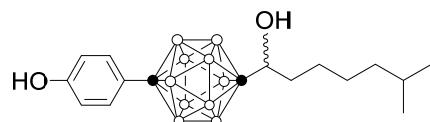




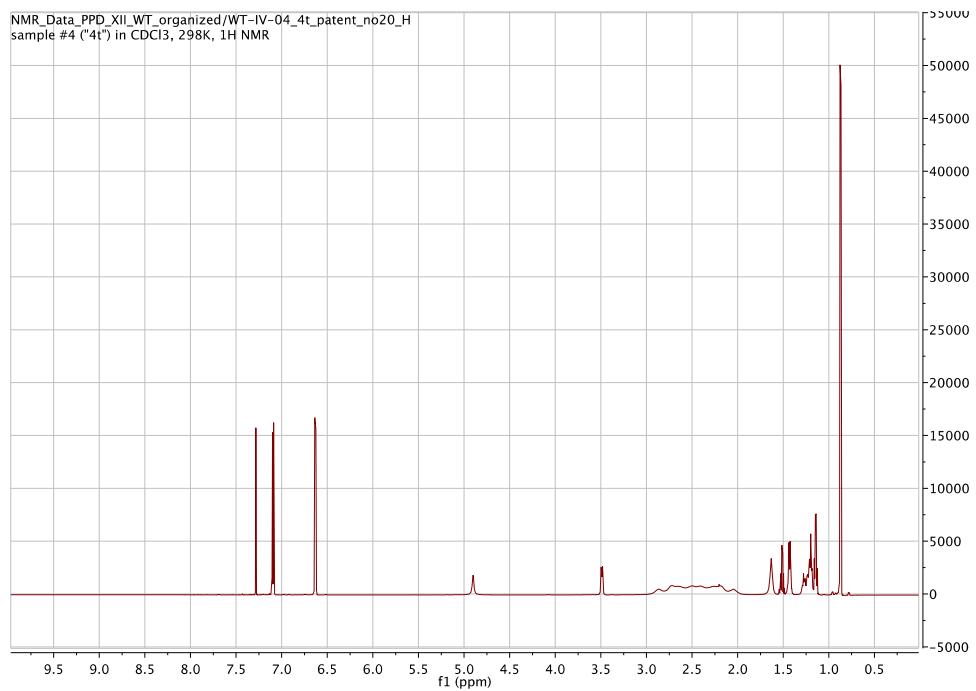
#### DAD-254 nm Results

| Retention Time | Area     | Area % |
|----------------|----------|--------|
| 7.840          | 19022561 | 100.00 |
| Totals         | 19022561 | 100.00 |

HPLC of Compound 1-(4-Hydroxyphenyl)-12-(6-methylheptyl)-1,12-dicarba-*clos*o-dodecaborane

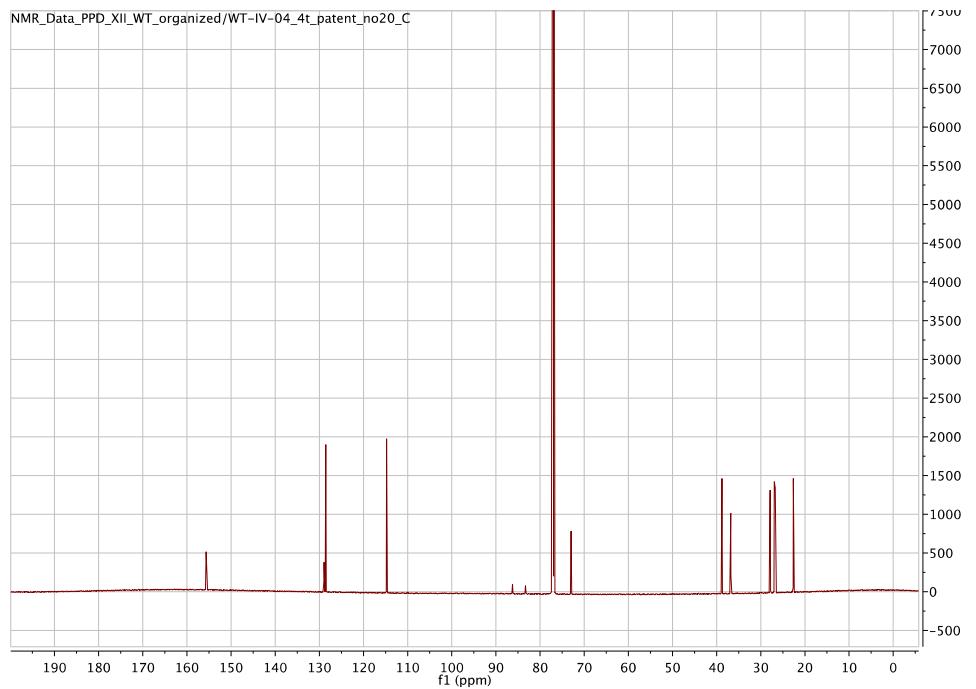


NMR\_Data\_PPD\_XII\_WT\_organized/WT-IV-04\_4t\_patent\_no20\_H  
sample #4 ("4t") in CDCl<sub>3</sub>, 298K, 1H NMR

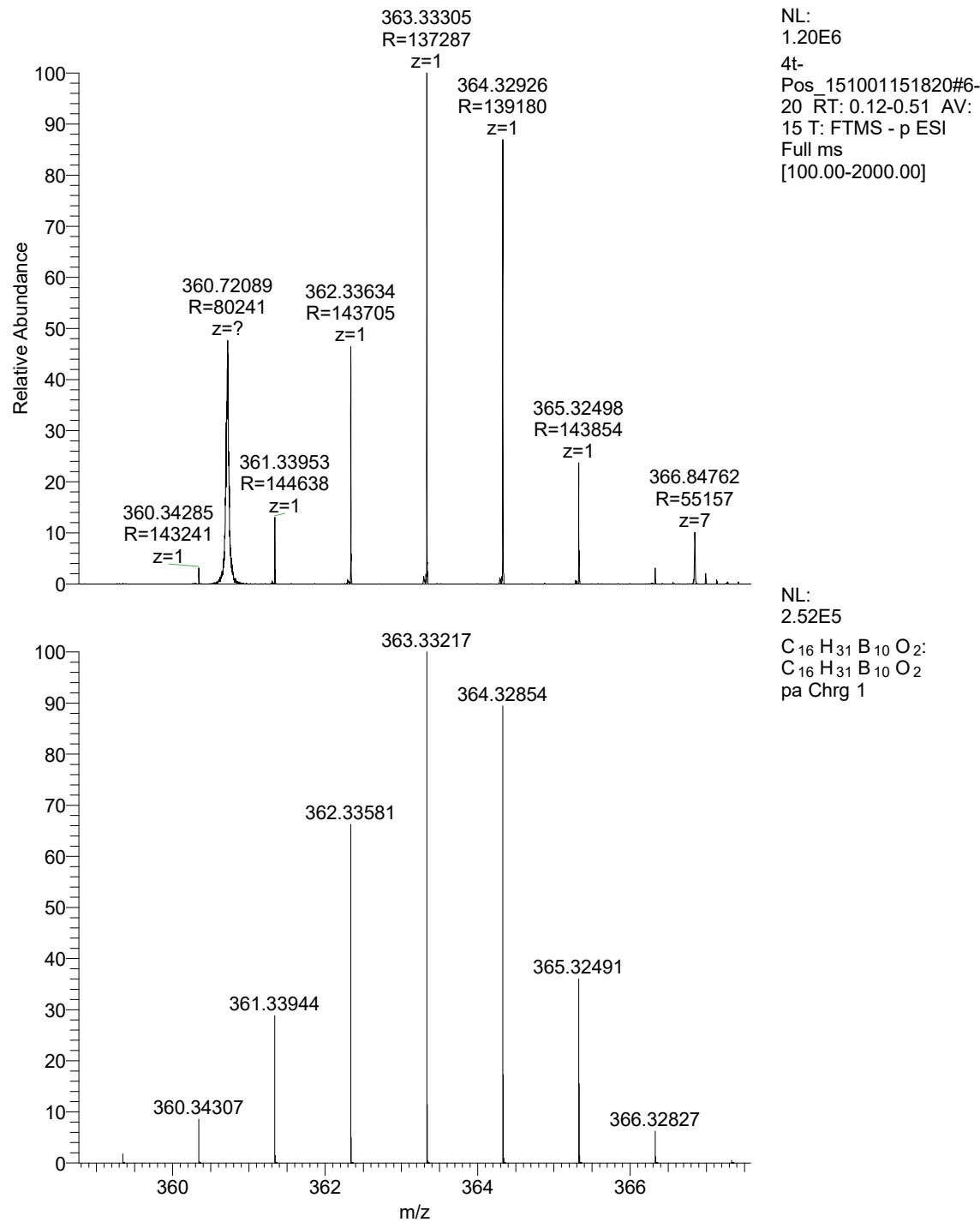


<sup>1</sup>H NMR of Compound (RS)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]-6-methylheptan-1-ol

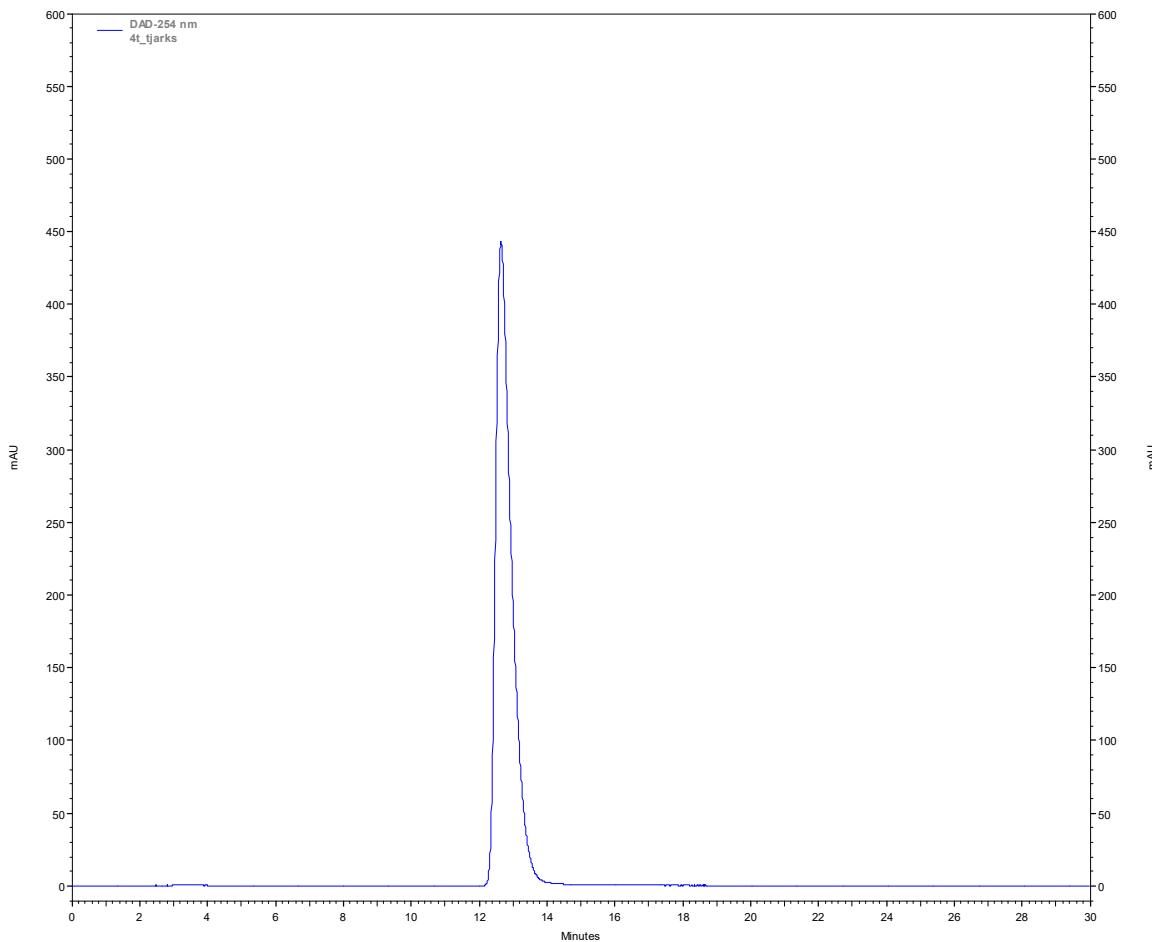
NMR\_Data\_PPD\_XII\_WT\_organized/WT-IV-04\_4t\_patent\_no20\_C



<sup>13</sup>C NMR of Compound (RS)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]-6-methylheptan-1-ol



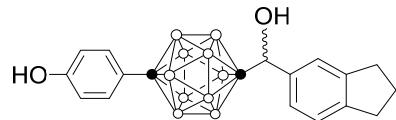
MS of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]-6-methylheptan-1-ol



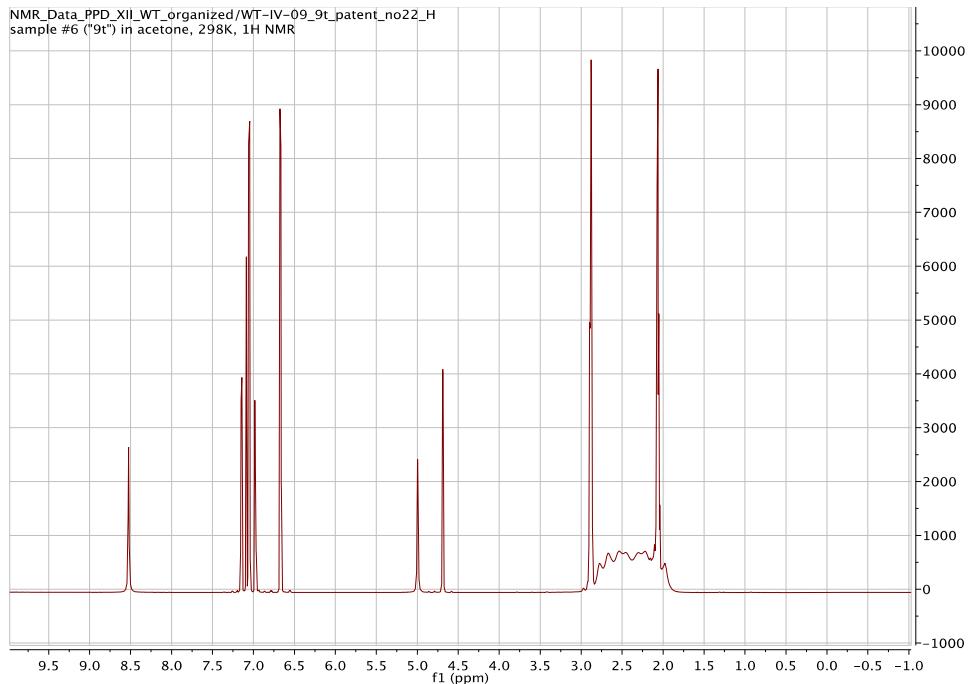
#### DAD-254 nm Results

| Retention Time | Area     | Area %          |
|----------------|----------|-----------------|
| 12.647         | 57748741 | 99.99           |
| 15.400         | 3101     | 0.01            |
| 15.827         | 733      | 0.00            |
| 15.913         | 835      | 0.00            |
| 15.973         | 53       | 0.00            |
| <b>Totals</b>  |          | <b>57753463</b> |
|                |          | 100.00          |

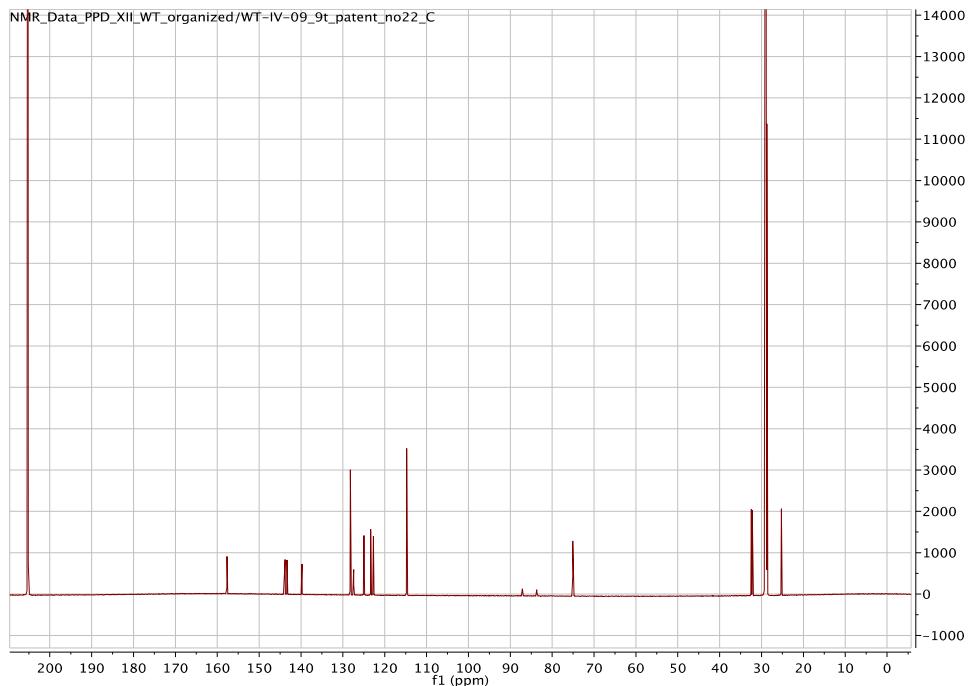
HPLC of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]-6-methylheptan-1-ol



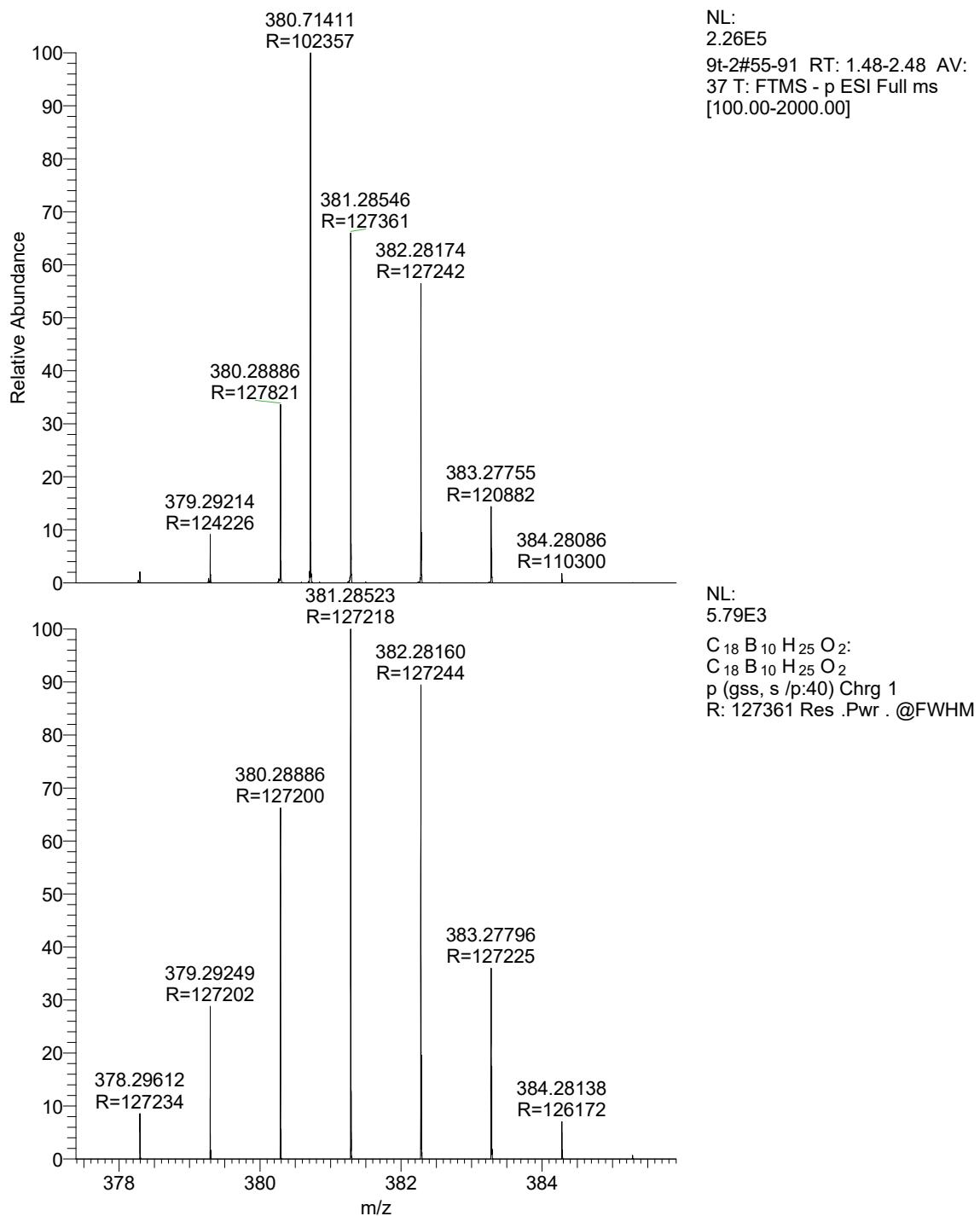
NMR\_Data\_PPD\_XII\_WT\_organized/WT-IV-09\_9t\_patent\_no22\_H  
sample #6 ("9t") in acetone, 298K, 1H NMR



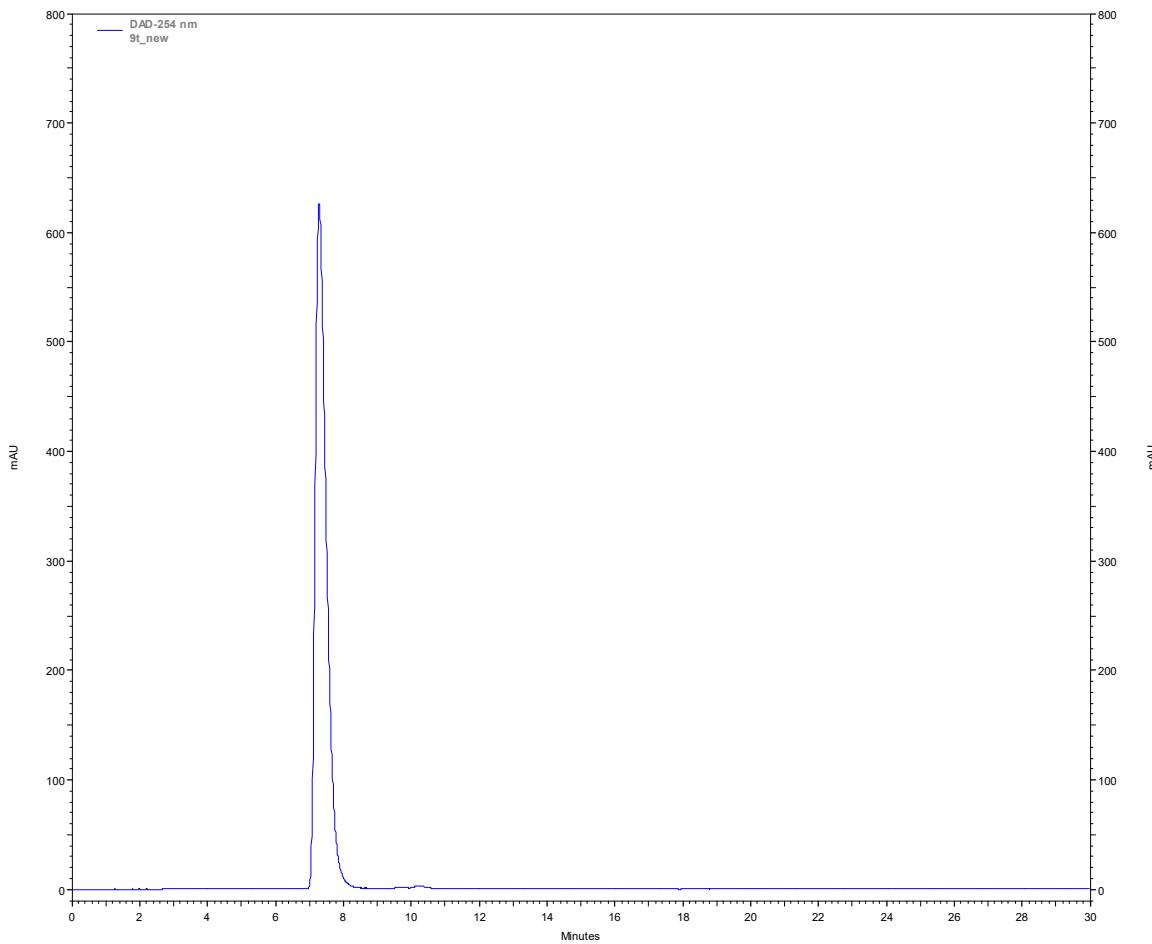
<sup>1</sup>H NMR of Compound (RS)-(2,3-dihydro-1*H*-inden-5-yl)-[1-(4-hydroxyphenyl)-1,12-dicarba-*creso*-dodecaborane-12-yl]methanol



<sup>13</sup>C NMR of Compound (RS)-(2,3-dihydro-1*H*-inden-5-yl)-[1-(4-hydroxyphenyl)-1,12-dicarba-*creso*-dodecaborane-12-yl]methanol



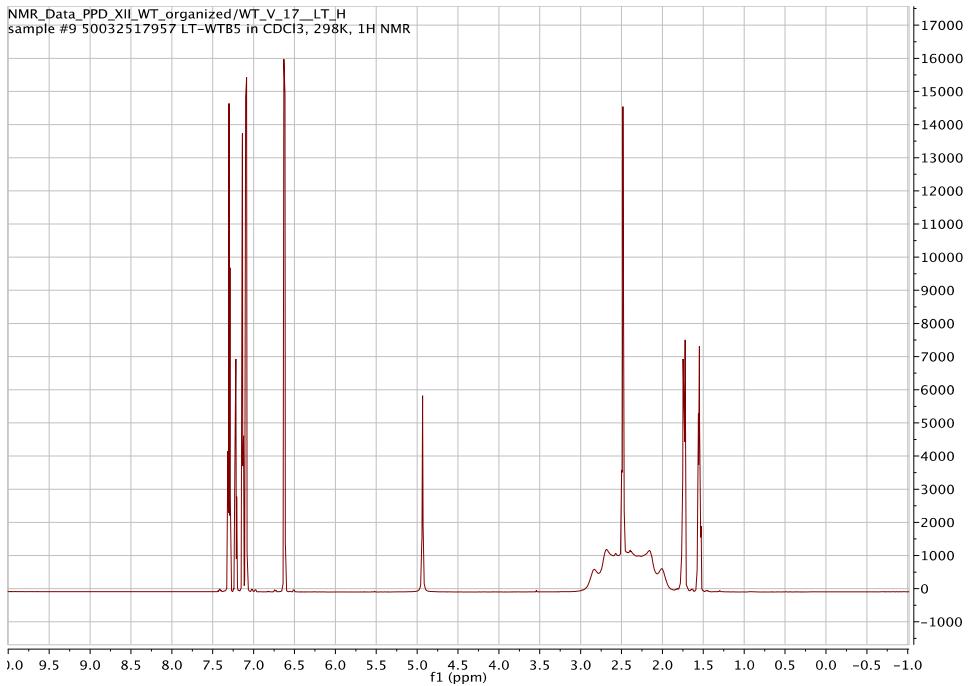
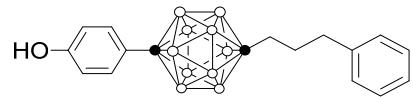
MS of Compound (*RS*)-(2,3-dihydro-1*H*-inden-5-yl)-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]methanol



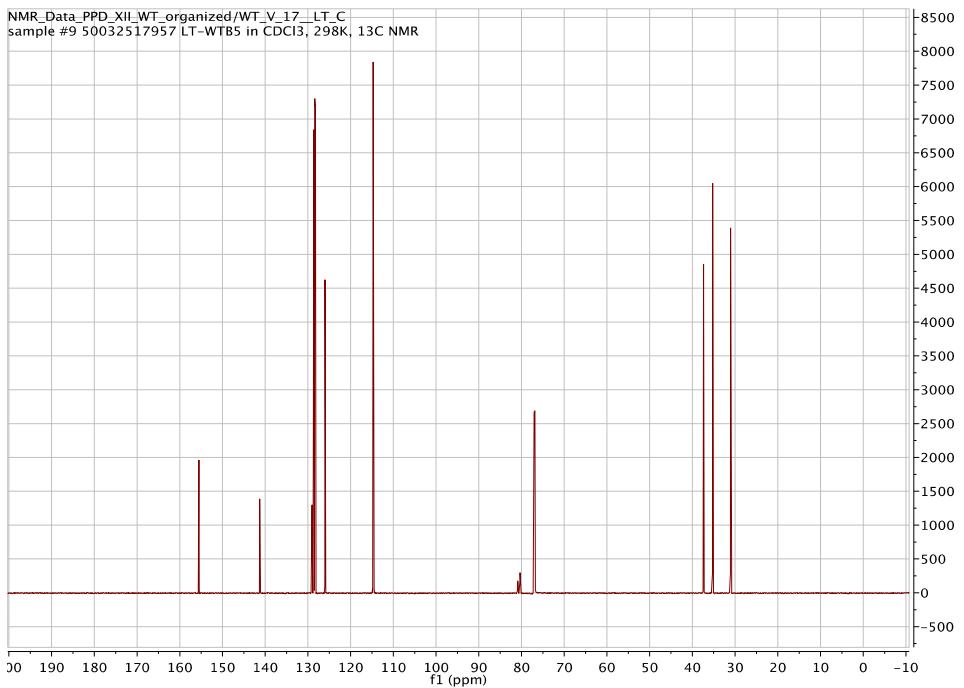
#### DAD-254 nm Results

| Retention Time | Area     | Area %   |
|----------------|----------|----------|
| 7.287          | 57122831 | 99.69    |
| 10.200         | 178819   | 0.31     |
| Totals         |          | 57301650 |
|                |          | 100.00   |

HPLC of Compound (*RS*)-(2,3-dihydro-1*H*-inden-5-yl)-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]methanol



<sup>1</sup>H NMR of Compound 1-(4-hydroxyphenyl)-12-(3-phenylpropyl)-1,12-dicarba-closo-dodecaborane



<sup>13</sup>C NMR of Compound 1-(4-hydroxyphenyl)-12-(3-phenylpropyl)-1,12-dicarba-closo-dodecaborane

Werner Tjarks LT-WTB5 EI 70 eV

Werner Tjarks LT-WTB5 EI 70 eV  
LT-WTB5\_EI\_44-17 (0.019) ls (1.00,1.00) C17H26B10O

Voltage El<sup>+</sup>  
2.82e12

Theoretical pattern

354.2996  
355.2984

353.3029

356.2942

352.3063

351.3099

LT-WTB5\_EI\_44-17 84 (1.543) Cn (Cen,10, 10.00, Ht); Sm (SG, 2x10.00); Cm (60:109)

354.2975  
355.2949

Voltage El<sup>+</sup>  
166

Measured pattern

353.3025

356.2935

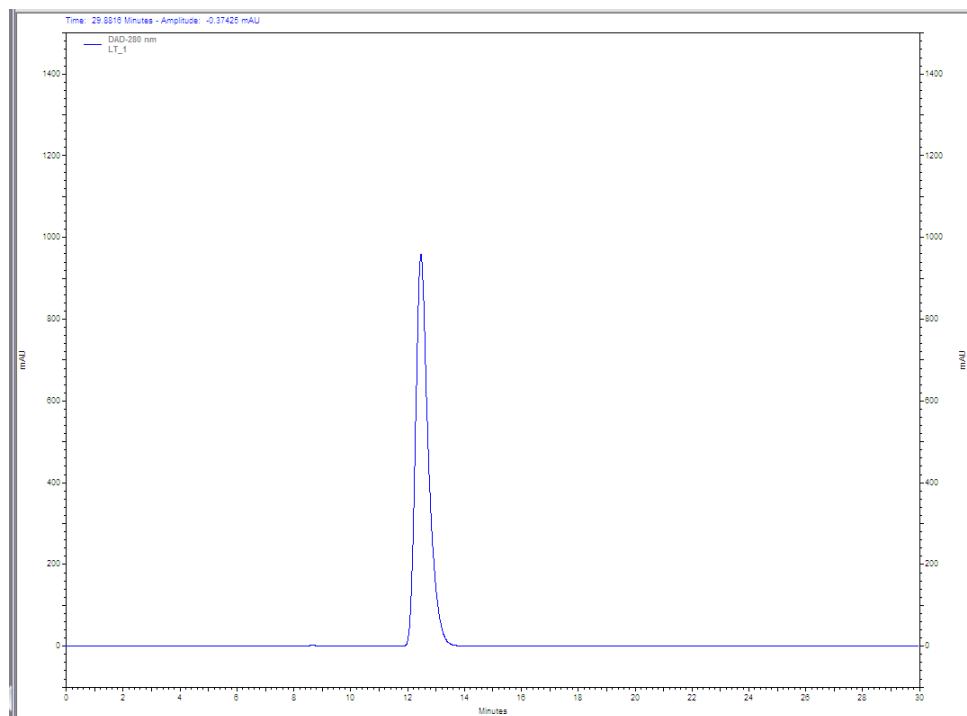
352.3053

351.3088

349.3374  
348.9799  
350.3243  
350.9780  
351  
352  
353  
354  
355  
356  
357  
358  
359  
360

%

MS of Compound 1-(4-hydroxyphenyl)-12-(3-phenylpropyl)-1,12-dicarba-*clos*-dodecaborane



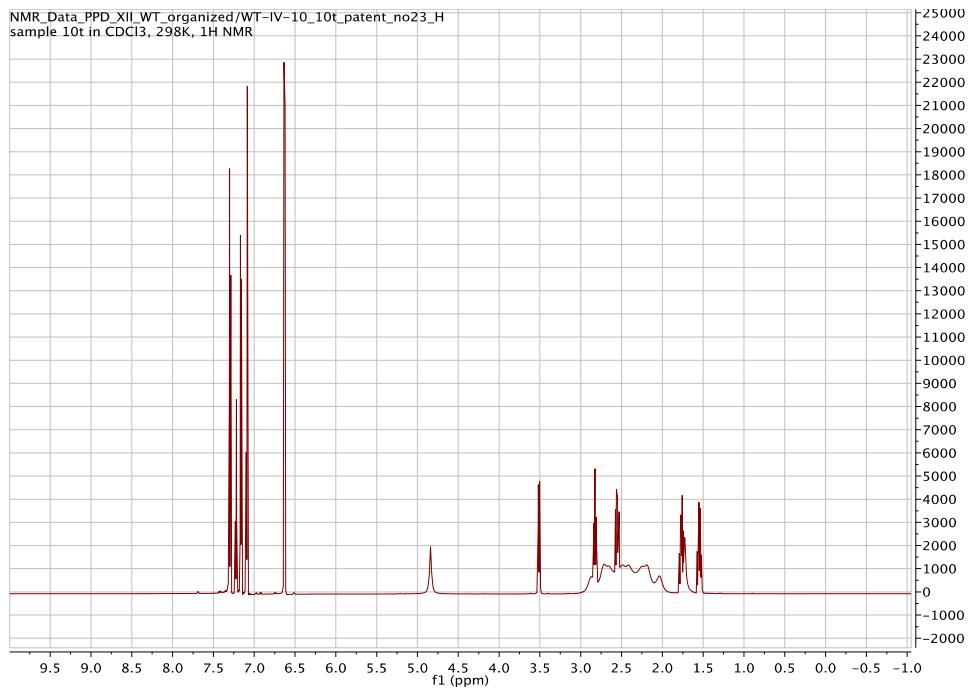
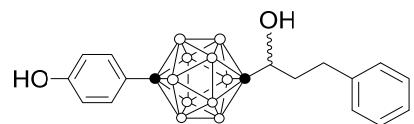
### DAD-280 nm

#### Results

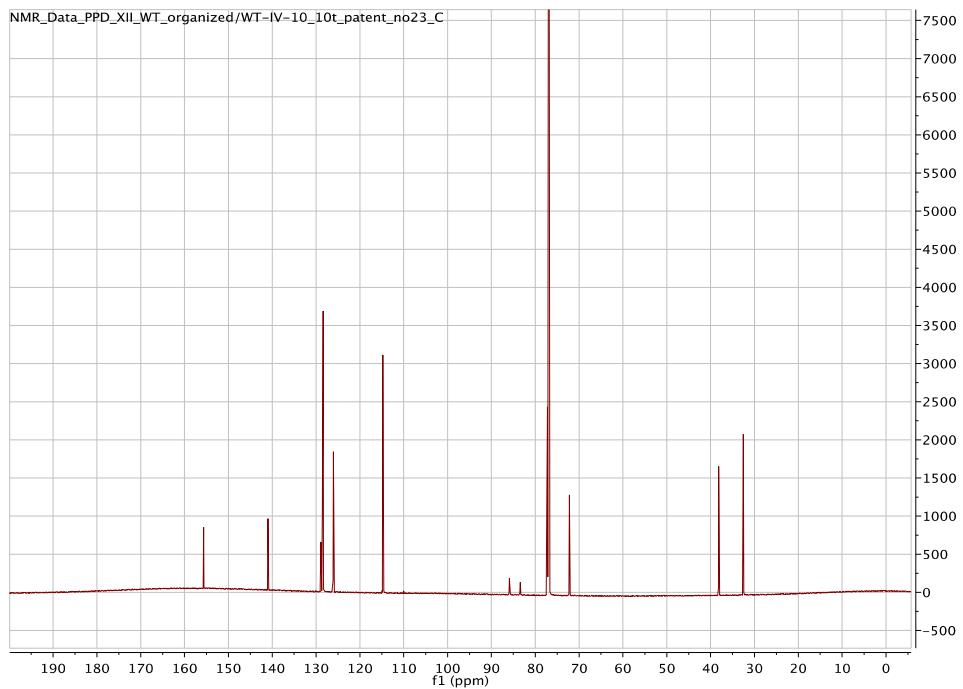
| Retention Time | Area      | Area % | Height  | Height % |
|----------------|-----------|--------|---------|----------|
| 8.667          | 280902    | 0.23   | 9533    | 0.25     |
| 12.467         | 119996840 | 99.77  | 3831965 | 99.75    |
|                |           |        |         |          |

|        |           |        |         |        |
|--------|-----------|--------|---------|--------|
| Totals | 120277742 | 100.00 | 3841498 | 100.00 |
|--------|-----------|--------|---------|--------|

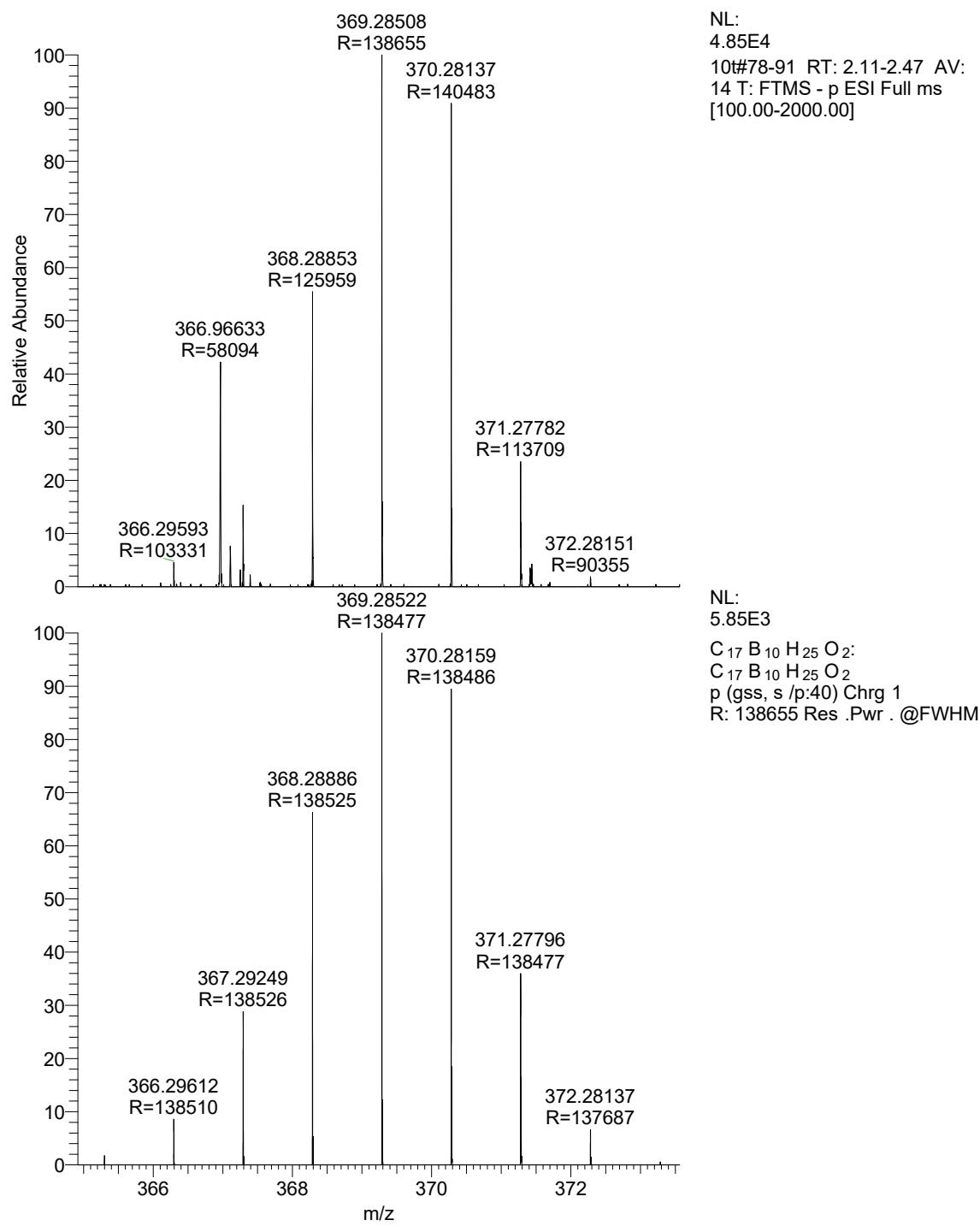
HPLC of Compound 1-(4-hydroxyphenyl)-12-(3-phenylpropyl)-1,12-dicarba-*clos*o-dodecaborane

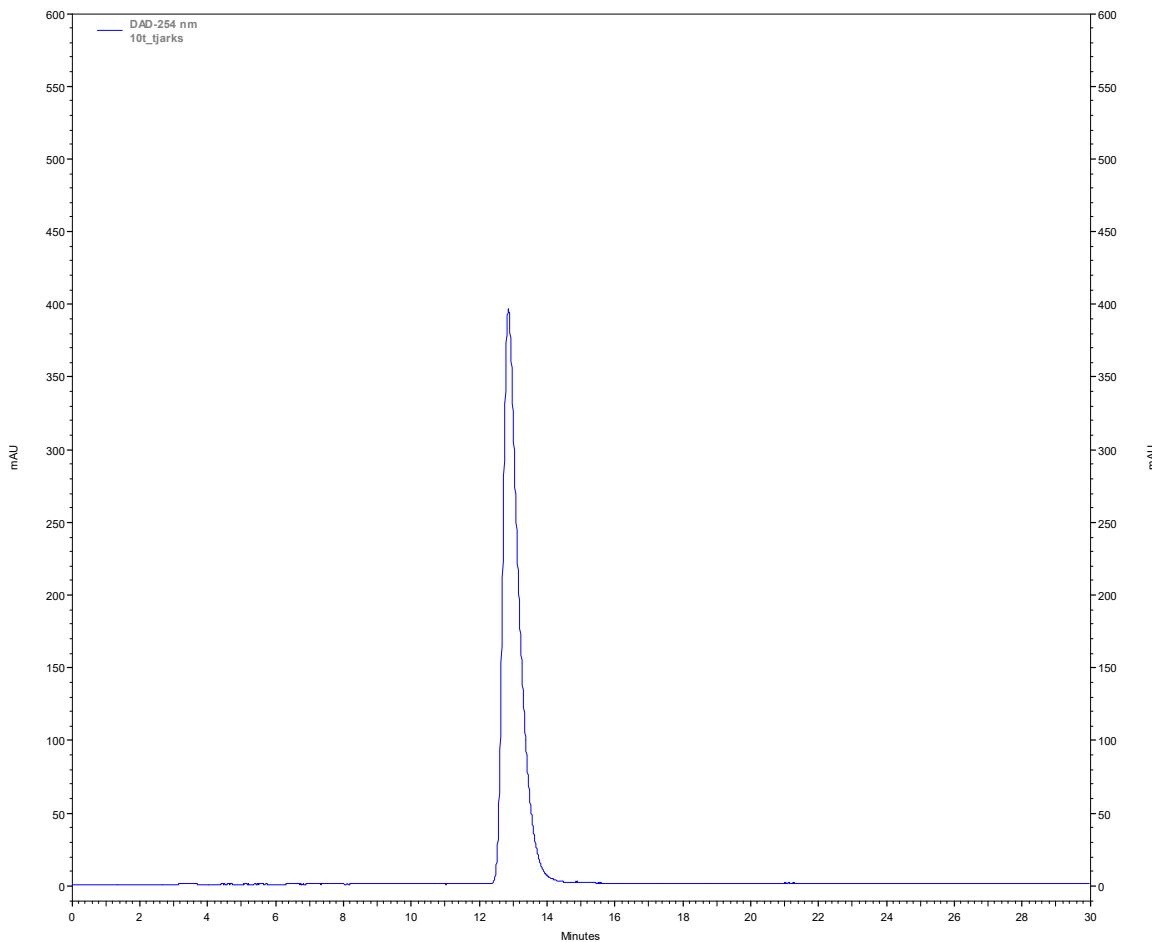


<sup>1</sup>H NMR of Compound (RS)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]-3-phenylpropan-1-ol



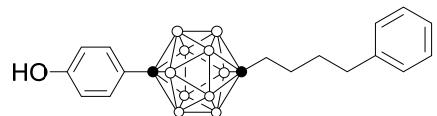
<sup>13</sup>C NMR of Compound (RS)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]-3-phenylpropan-1-ol

MS of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]-3-phenylpropan-1-ol

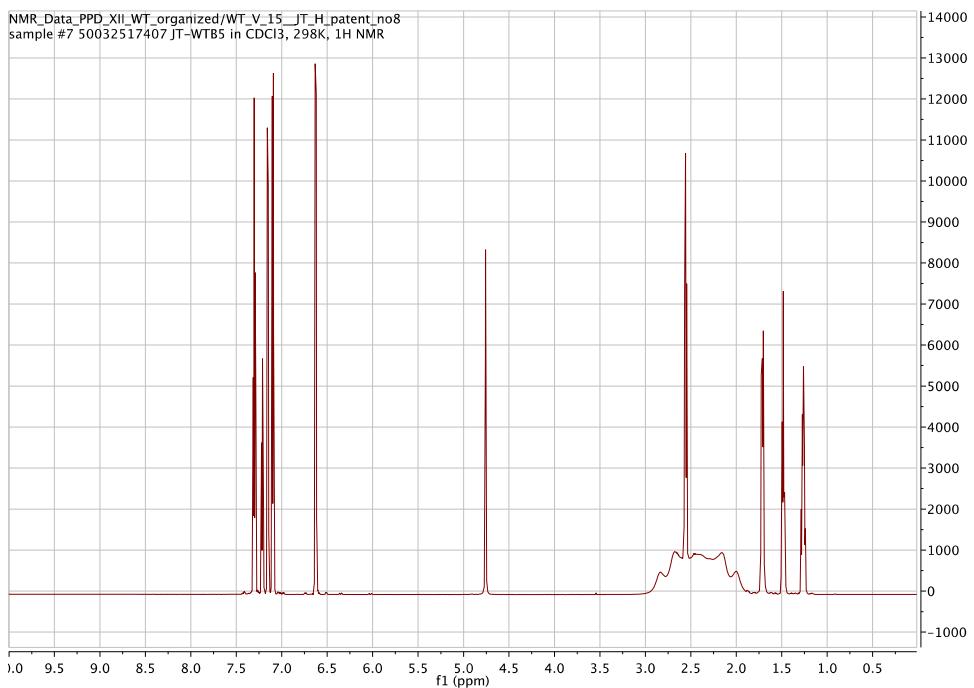
**DAD-254 nm Results**

| Retention Time | Area     | Area % |
|----------------|----------|--------|
| 12.867         | 52266702 | 100.00 |
| Totals         | 52266702 | 100.00 |

HPLC of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*closو*-dodecaborane-12-yl]-3-phenylpropan-1-ol

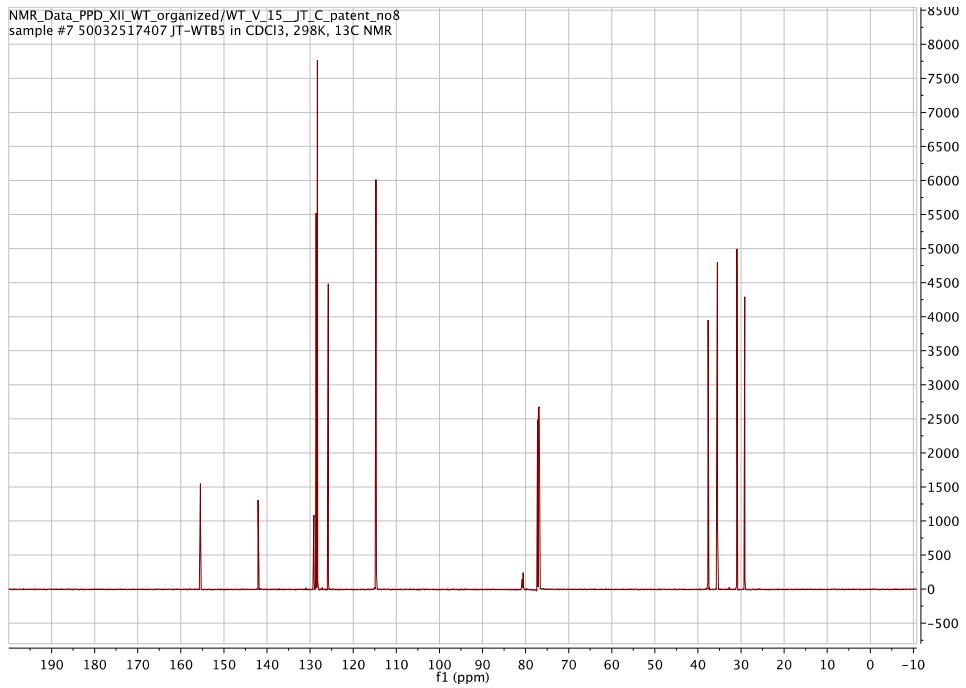


NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_15\_JT\_H\_patent\_no8  
Sample #7 50032517407 JT-WTB5 in CDCl<sub>3</sub>, 298K, 1H NMR

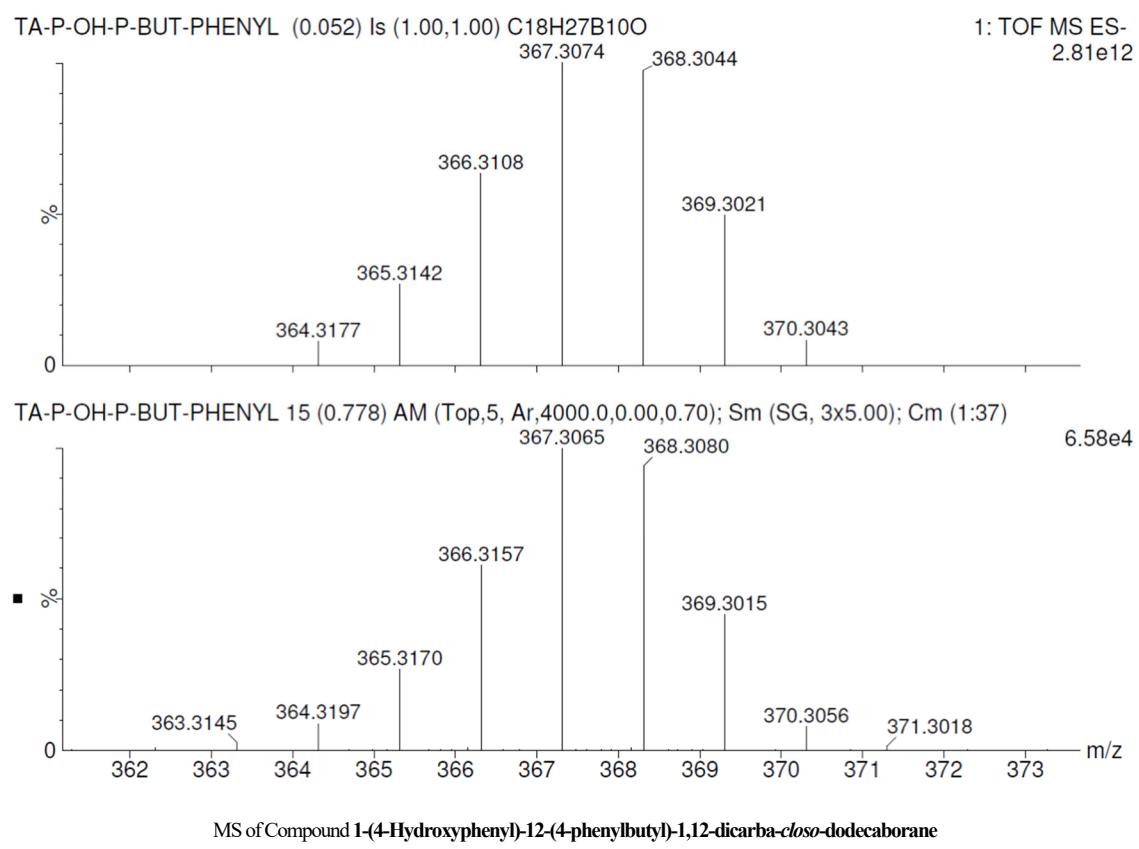


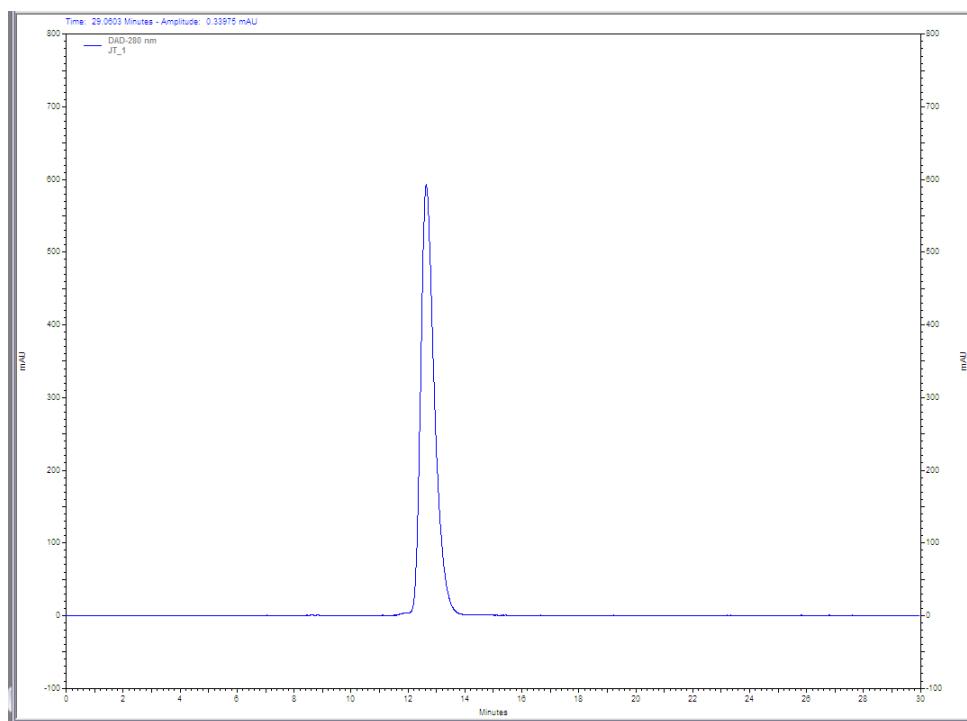
<sup>1</sup>H NMR of Compound 1-(4-Hydroxyphenyl)-12-(4-phenylbutyl)-1,12-dicarba-closo-dodecaborane

NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_15\_JT\_C\_patent\_no8  
Sample #7 50032517407 JT-WTB5 in CDCl<sub>3</sub>, 298K, 13C NMR



<sup>13</sup>C NMR of Compound 1-(4-Hydroxyphenyl)-12-(4-phenylbutyl)-1,12-dicarba-closo-dodecaborane



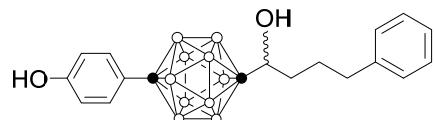


### DAD-280 nm

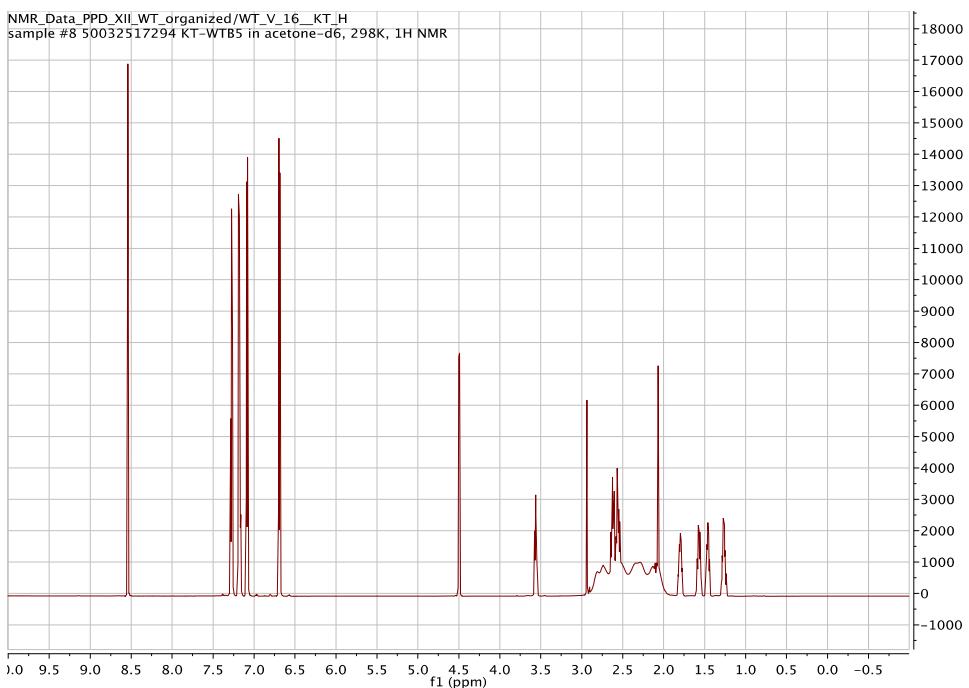
#### Results

| Retention Time | Area            | Area %        | Height         | Height %      |
|----------------|-----------------|---------------|----------------|---------------|
| 11.987         | 52486           | 0.07          | 1175           | 0.05          |
| 12.640         | 78257189        | 99.93         | 2358543        | 99.95         |
| <b>Totals</b>  | <b>78309675</b> | <b>100.00</b> | <b>2359718</b> | <b>100.00</b> |

HPLC of Compound 1-(4-Hydroxyphenyl)-12-(4-phenylbutyl)-1,12-dicarba-*clos*-dodecaborane

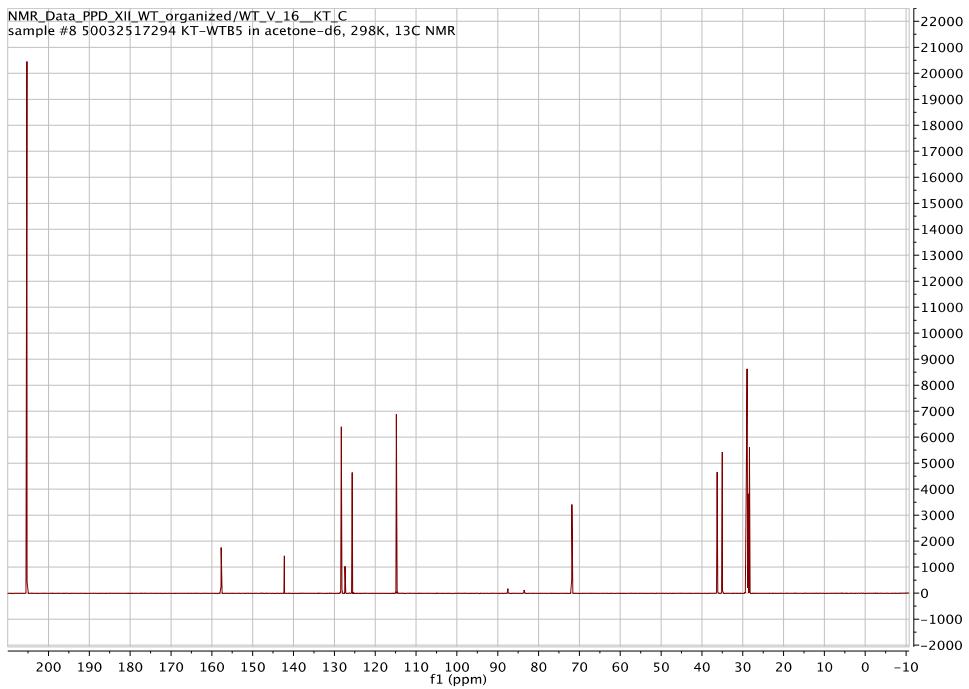


NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_16\_KT\_H  
sample #8 50032517294 KT-WTB5 in acetone-d<sub>6</sub>, 298K, 1H NMR



<sup>1</sup>H NMR of Compound (RS)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]-4-phenylbutan-1-ol

NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_16\_KT\_C  
sample #8 50032517294 KT-WTB5 in acetone-d<sub>6</sub>, 298K, 13C NMR



<sup>13</sup>C NMR of Compound (RS)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]-4-phenylbutan-1-ol

Werner Tjarks KT-WTB5 EI 70 eV

**Werner Tjarks KT-WTB5 EI 70 eV**  
KT-WTB5\_EI\_4-4-17 (0.019)ls (1.00,1.00) C18H28B10O2

Voltage EI<sup>+</sup>  
2.80e12

Theoretical pattern

383.3135

386.3049

387.3072

388.3034

389.8705

390.9472

391.3061

392.9804

393.9741

394.9818

395.9804

396.9741

397.3105

398.3034

399.8705

381.3180

382.3146

383.9829

384.9818

385.9804

386.9741

387.3105

388.9472

389.8705

380.9682

381.5760

382.9901

383.9829

384.9818

385.9804

386.9741

387.3105

388.9472

389.8705

381.3204

382.3169

383.3135

384.3102

385.3071

386.3049

387.3072

388.3034

389.8705

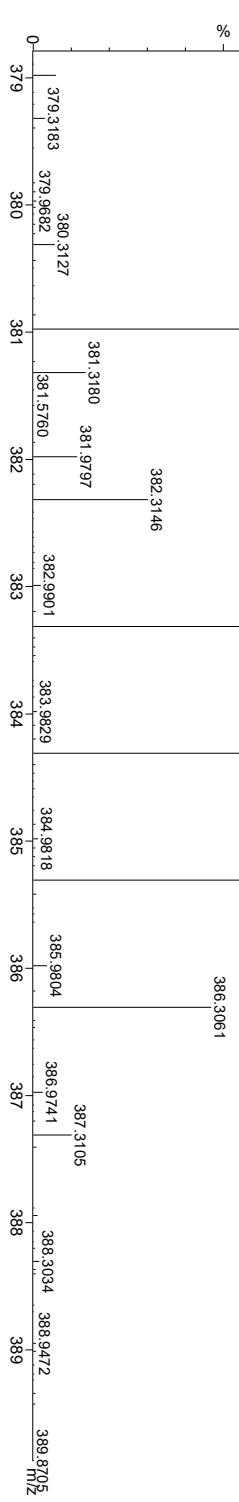
380.9758

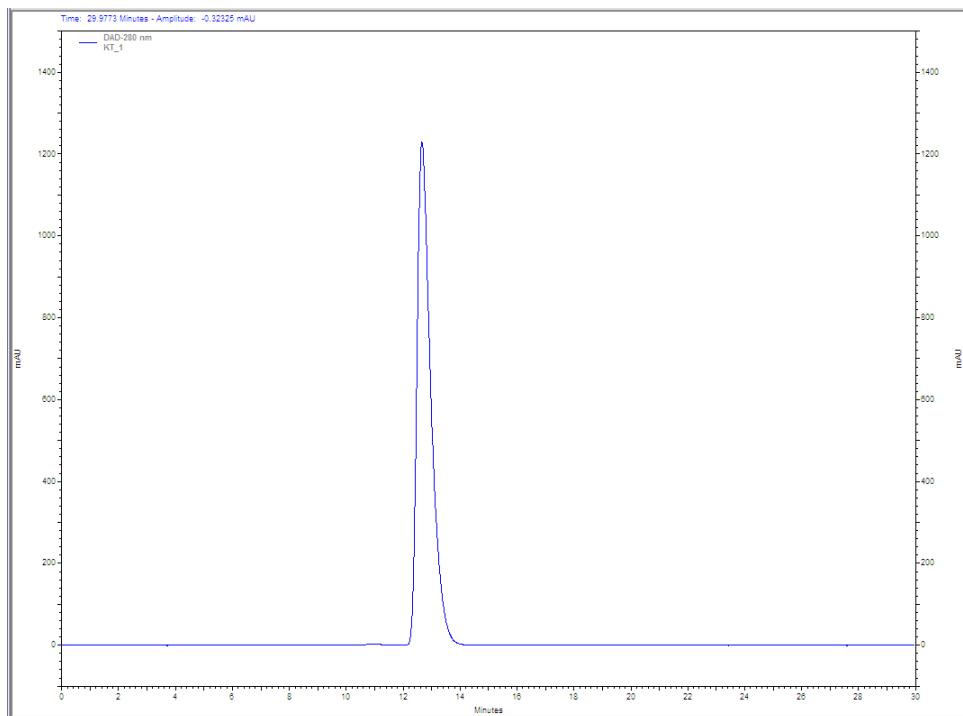
KT-WTB5\_EI\_4-4-17 65 (1.193) Cn(Cen,10,10,00, Ht); Sm(SG,2x10,00); Cm(50:80)

Voltage EI<sup>+</sup>  
158

◀ PFK ref. mass

Measured pattern



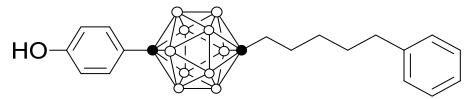


### DAD-280 nm

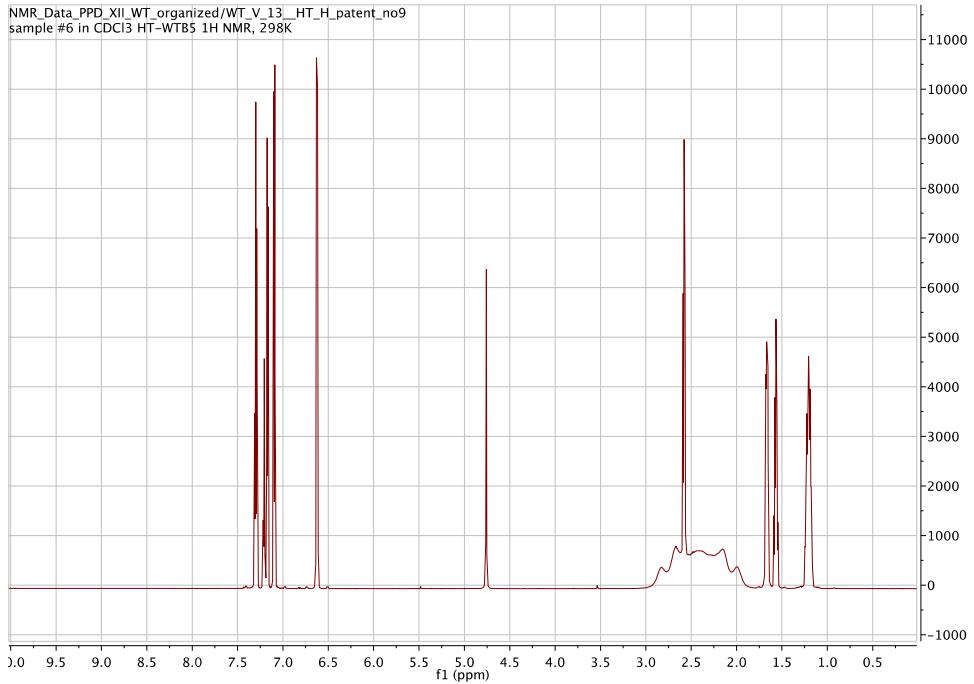
#### Results

| Retention Time | Area      | Area % | Height  | Height % |
|----------------|-----------|--------|---------|----------|
| 10.913         | 362389    | 0.22   | 12866   | 0.26     |
| 12.653         | 165382334 | 99.78  | 4917065 | 99.74    |
| Totals         | 165744723 | 100.00 | 4929931 | 100.00   |

HPLC of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]-4-phenylbutan-1-ol

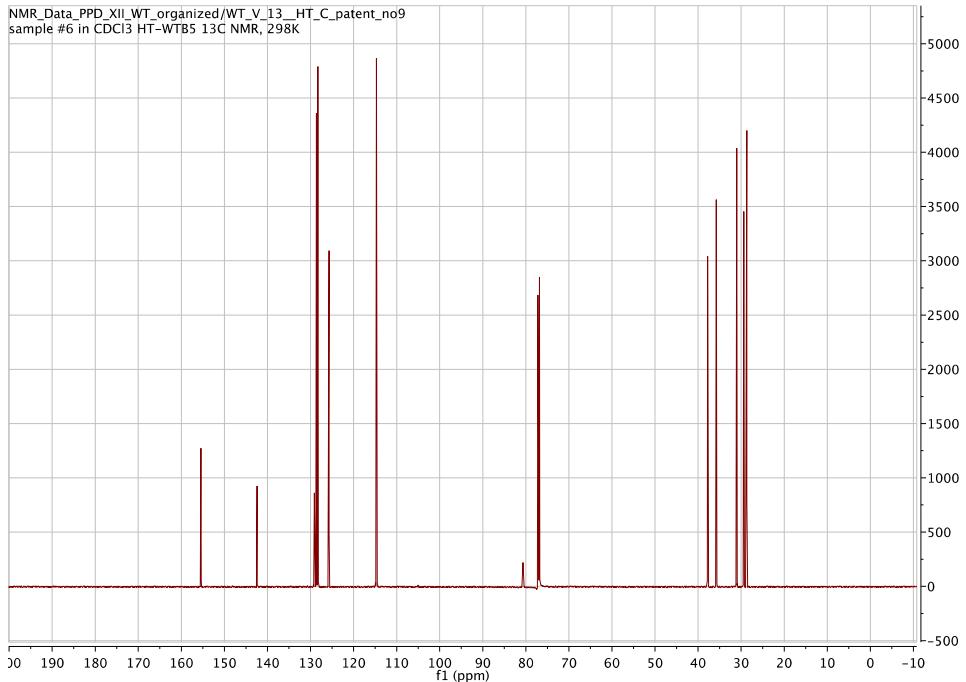


NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_13\_\_HT\_H\_patent\_no9  
sample #6 in CDCl<sub>3</sub> HT-WTB5 1H NMR, 298K

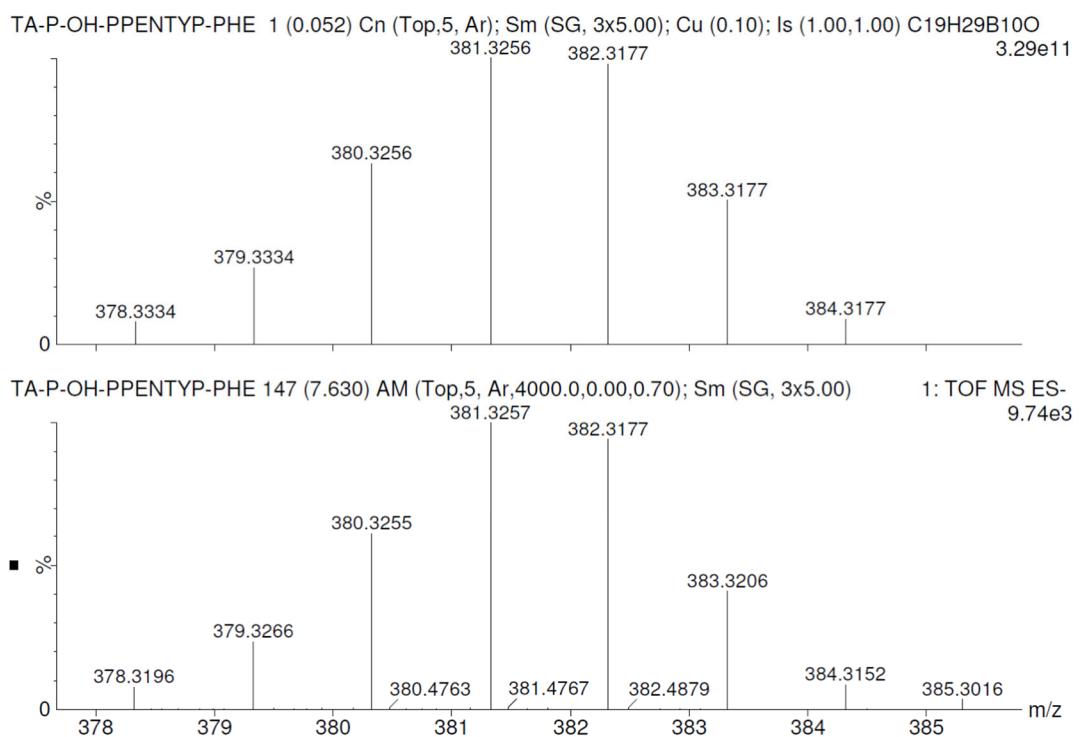


<sup>1</sup>H NMR of Compound 1-(4-Hydroxyphenyl)-12-(5-phenylpentyl)-1,12-dicarba-closo-dodecaborane

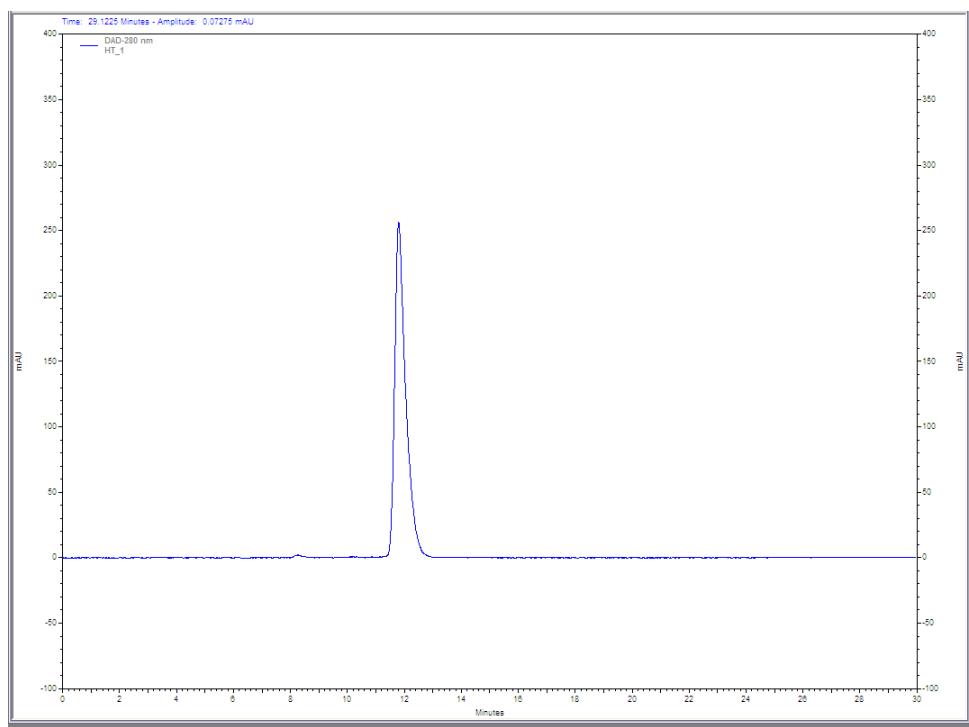
NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_13\_\_HT\_C\_patent\_no9  
sample #6 in CDCl<sub>3</sub> HT-WTB5 13C NMR, 298K



<sup>13</sup>C NMR of Compound 1-(4-Hydroxyphenyl)-12-(5-phenylpentyl)-1,12-dicarba-closo-dodecaborane



MS of Compound 1-(4-Hydroxyphenyl)-12-(5-phenylpentyl)-1,12-dicarba-*clos**o*-dodecaborane



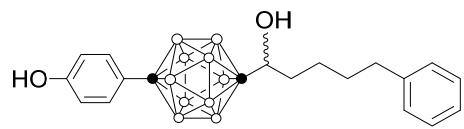
### DAD-280 nm

#### Results

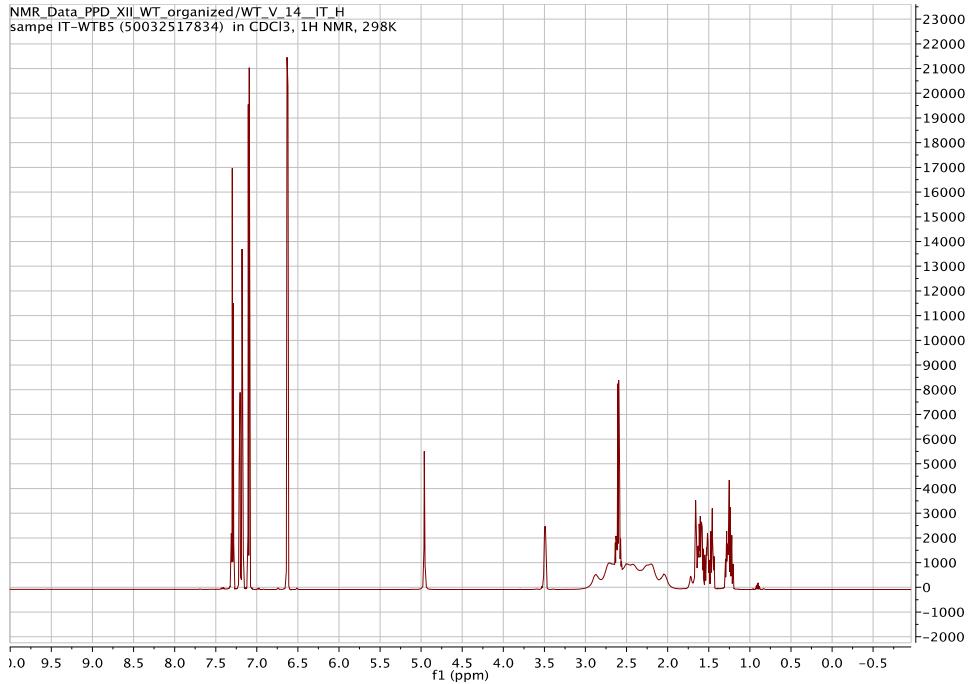
| Retention Time | Area     | Area % | Height  | Height % |
|----------------|----------|--------|---------|----------|
| 8.267          | 209756   | 0.76   | 8831    | 0.86     |
| 11.800         | 27340968 | 99.24  | 1021604 | 99.14    |

|        |          |        |         |        |
|--------|----------|--------|---------|--------|
| Totals | 27550724 | 100.00 | 1030435 | 100.00 |
|--------|----------|--------|---------|--------|

HPLC of Compound 1-(4-Hydroxyphenyl)-12-(5-phenylpentyl)-1,12-dicarba-*clos*o-dodecaborane

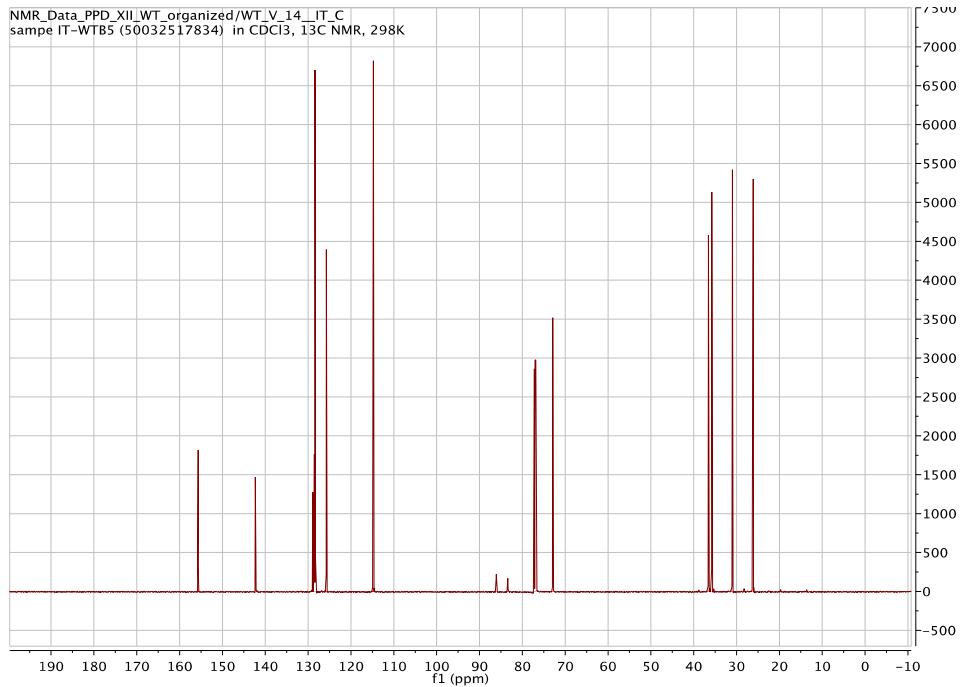


NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_14\_IT\_H  
sample IT-WTB5 (50032517834) in CDCl<sub>3</sub>, <sup>1</sup>H NMR, 298K



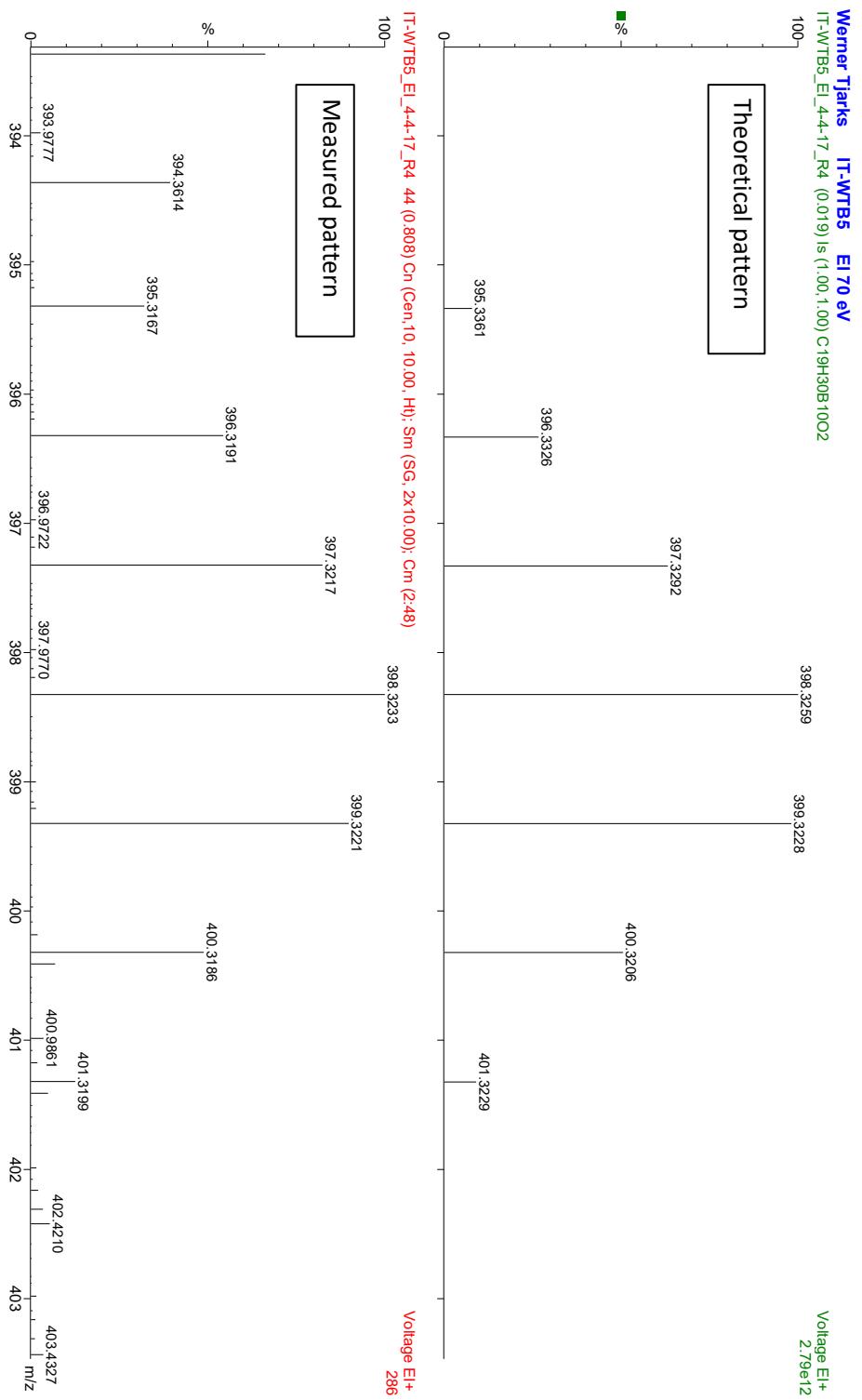
<sup>1</sup>H NMR of Compound (RS)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]-5-phenypentan-1-ol

NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_14\_IT\_C  
sample IT-WTB5 (50032517834) in CDCl<sub>3</sub>, <sup>13</sup>C NMR, 298K

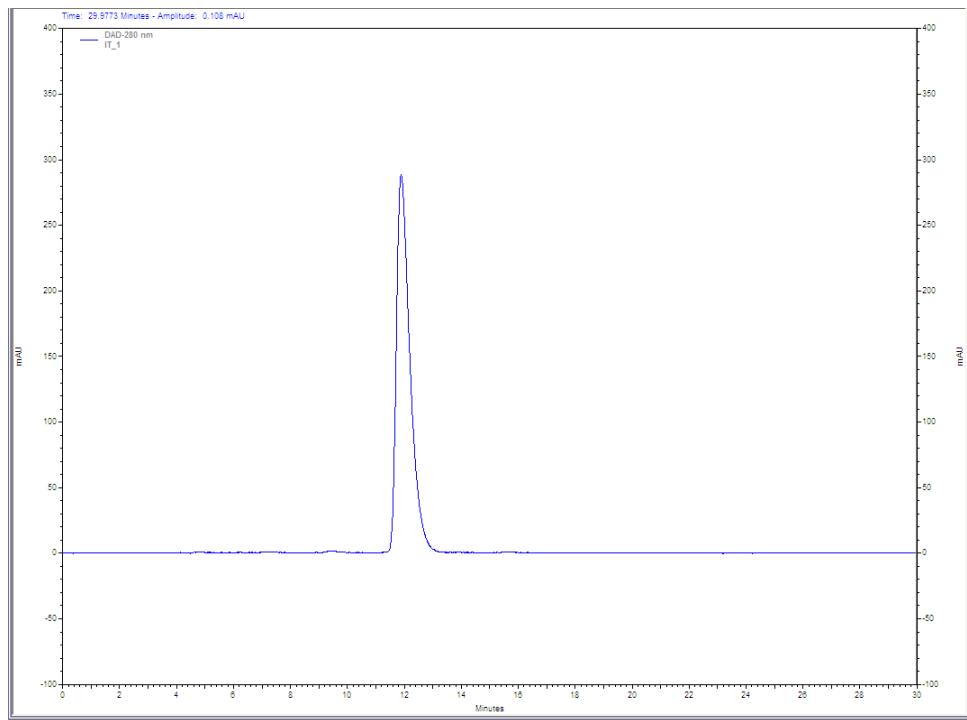


<sup>13</sup>C NMR of Compound (RS)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]-5-phenypentan-1-ol

Werner Tjarks IT-WTB5 EI 70 eV



MS of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]-5-phenylpentan-1-ol

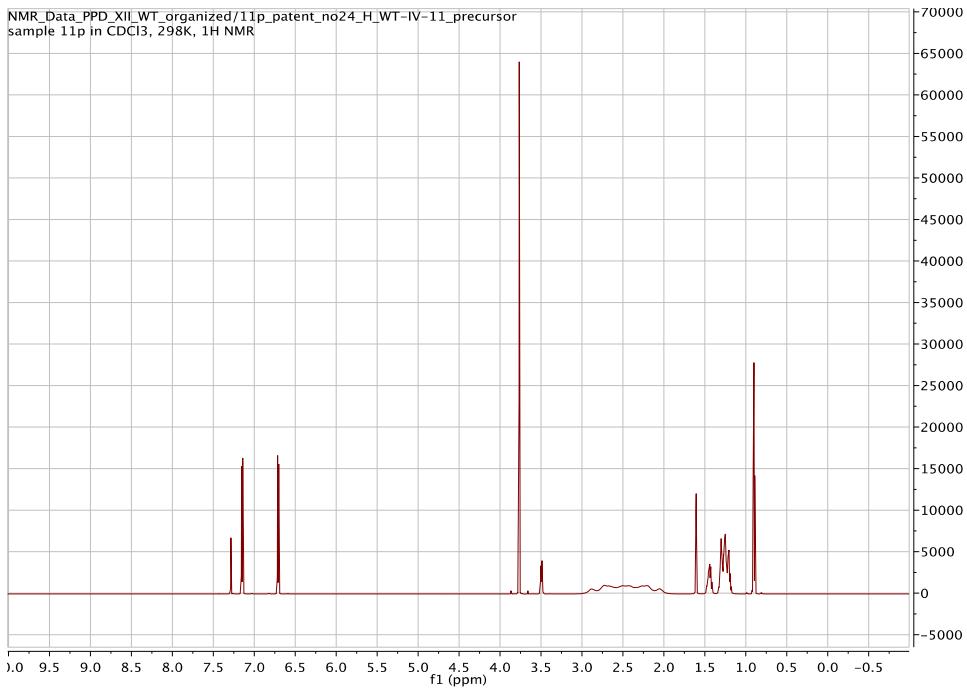
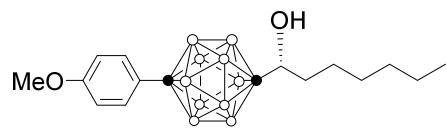


### DAD-280 nm

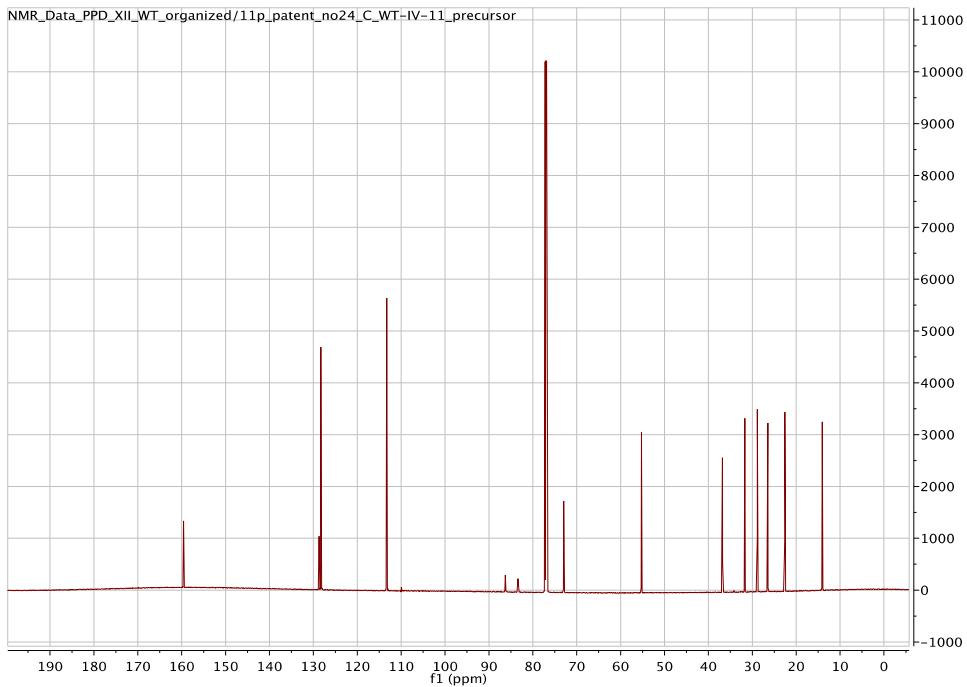
#### Results

| Retention Time | Area            | Area %        | Height         | Height %      |
|----------------|-----------------|---------------|----------------|---------------|
| 4.727          | 150488          | 0.39          | 4533           | 0.39          |
| 7.207          | 99436           | 0.26          | 2734           | 0.23          |
| 9.427          | 143612          | 0.37          | 5844           | 0.50          |
| 11.887         | 38129954        | 98.56         | 1151868        | 98.54         |
| 15.633         | 162999          | 0.42          | 3914           | 0.33          |
| <b>Totals</b>  | <b>38686489</b> | <b>100.00</b> | <b>1168893</b> | <b>100.00</b> |

HPLC of Compound (*RS*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]-5-phenylpentan-1-ol



<sup>1</sup>H NMR of Compound (S)-1-[1-(4-methoxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]heptan-1-ol



<sup>13</sup>C NMR of Compound (S)-1-[1-(4-methoxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]heptan-1-ol

**University of Illinois SCS Mass Spectrometry Laboratory**

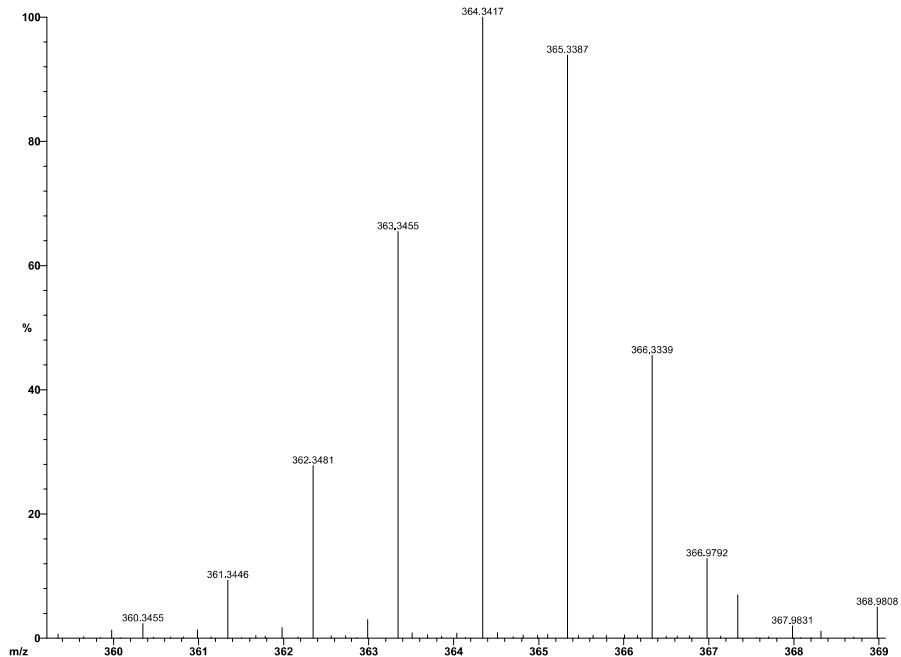
File: 11p-3-HR  
Sample:

Date Run: 02-09-2016 (Time Run: 10:08:34)

Ionization mode: EI+  
Instrument: VG 70-VSE(A)

Scan: 23-26

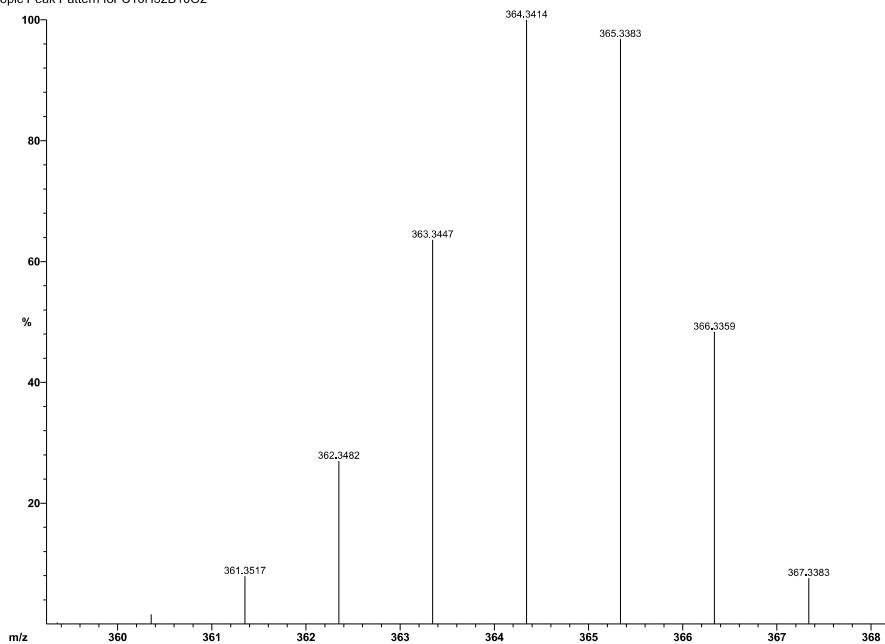
Base: m/z 364; 9.6%FS



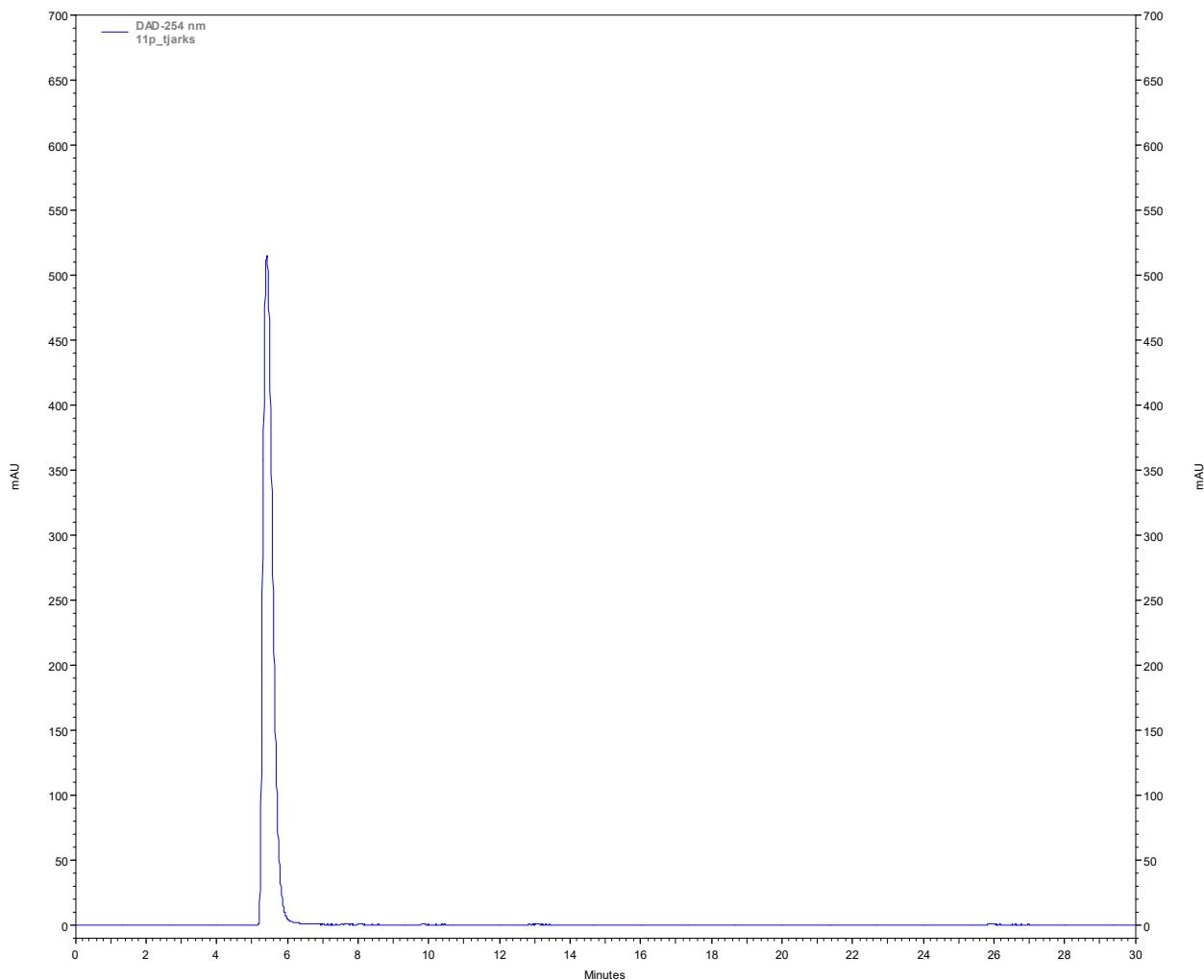
**University of Illinois SCS Mass Spectrometry Laboratory**

Isotopic Peak Pattern for C<sub>16</sub>H<sub>32</sub>B<sub>10</sub>O<sub>2</sub>

Base: m/z 364



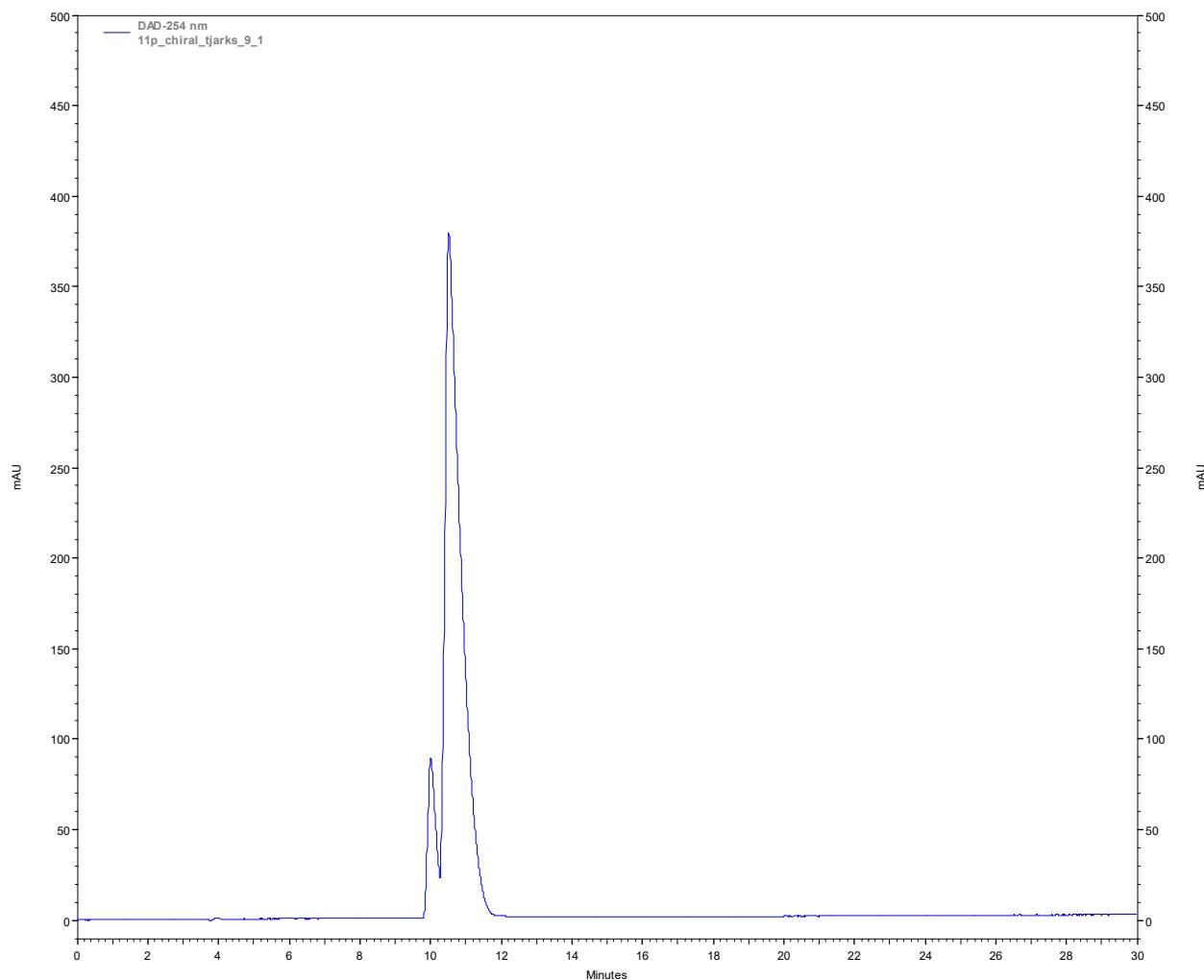
MS of Compound (*S*)-1-[1-(4-methoxyphenyl)-1,12-dicarba-*clos*-dodecaborane-12-yl]heptan-1-ol



**DAD-254 nm Results**

| Retention Time | Area     | Area % |
|----------------|----------|--------|
| 5.427          | 38987830 | 100.00 |
| Totals         | 38987830 | 100.00 |

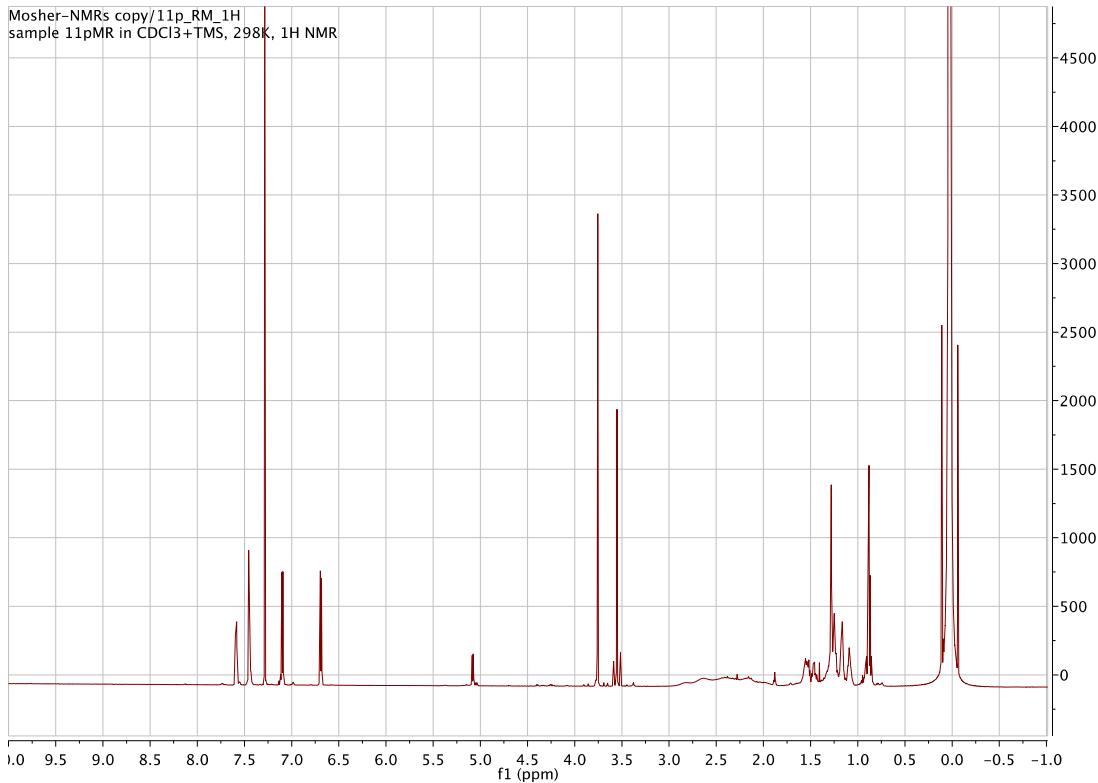
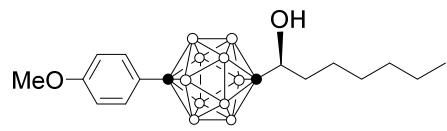
HPLC of Compound (*S*)-1-[1-(4-methoxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol



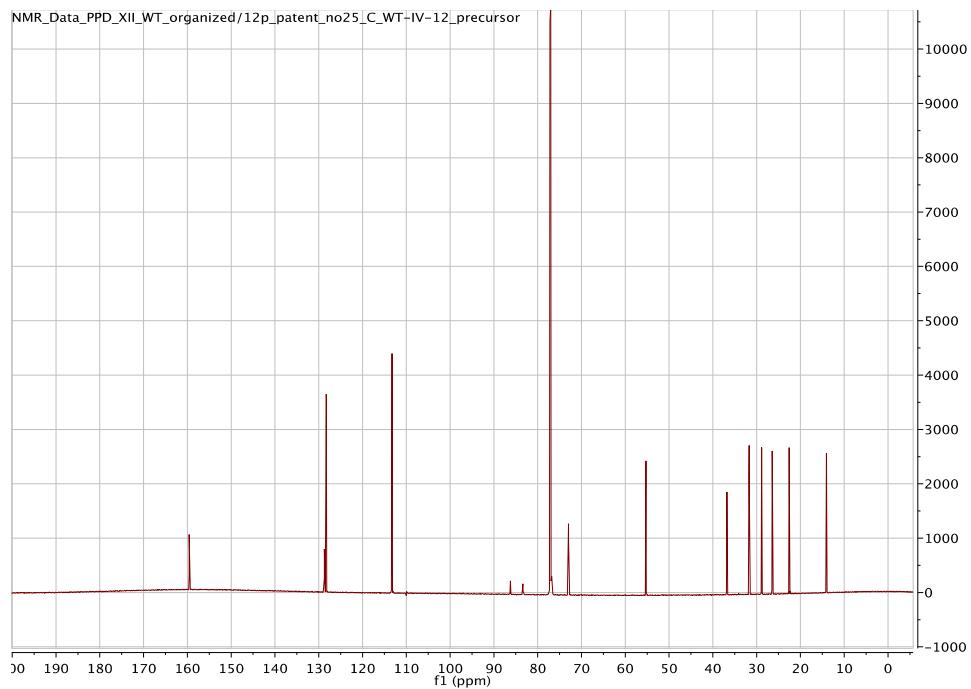
#### DAD-254 nm Results

| Retention Time | Area     | Area % |
|----------------|----------|--------|
| 10.007         | 4219319  | 8.74   |
| 10.520         | 44038907 | 91.26  |
| Totals         | 48258226 | 100.00 |

Chiral HPLC of Compound (S)-1-[1-(4-methoxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol



<sup>1</sup>H NMR of Compound (R)-1-[1-(4-methoxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]heptan-1-ol



<sup>13</sup>C NMR of Compound **(R)-1-[1-(4-methoxyphenyl)-1,12-dicarba-*clos*-dodecaborane-12-yl]heptan-1-ol**

**University of Illinois SCS Mass Spectrometry Laboratory**

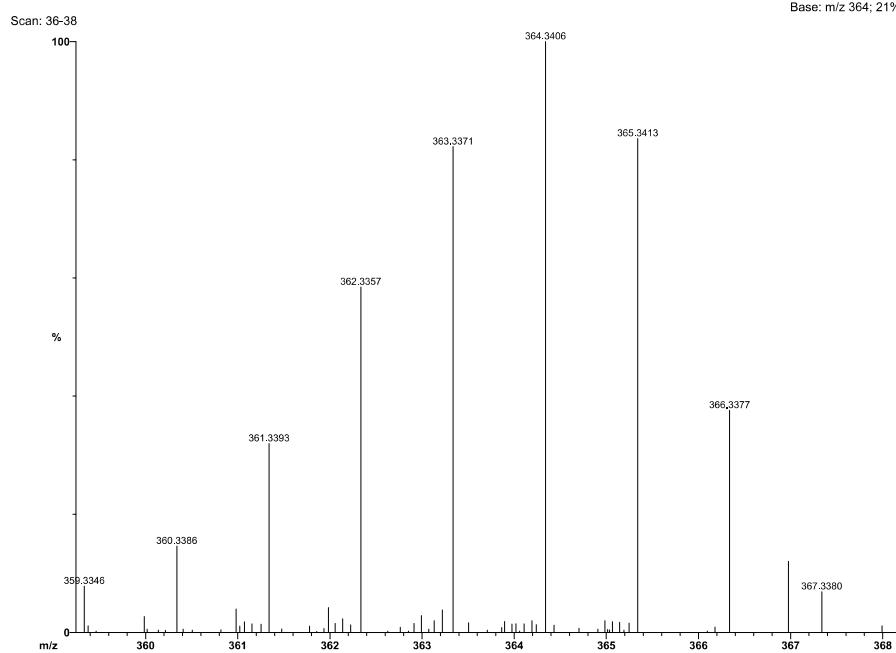
File: 12P\_HR\_RERun  
Sample:

Date Run: 11-11-2015 (09:19:19)

Ionization mode: EI+

Instrument: 70-VSE(C)

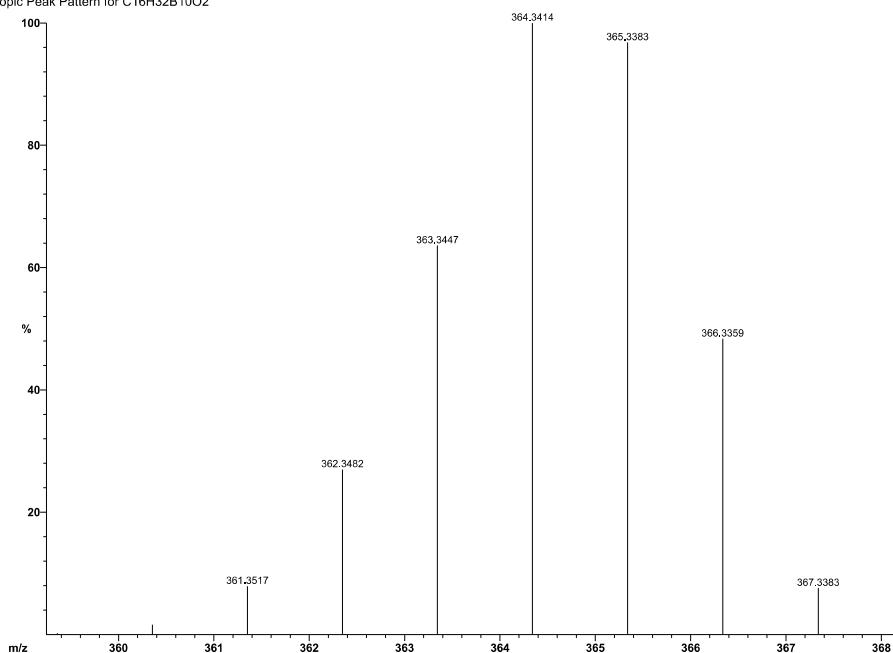
Base: m/z 364; 21%FS



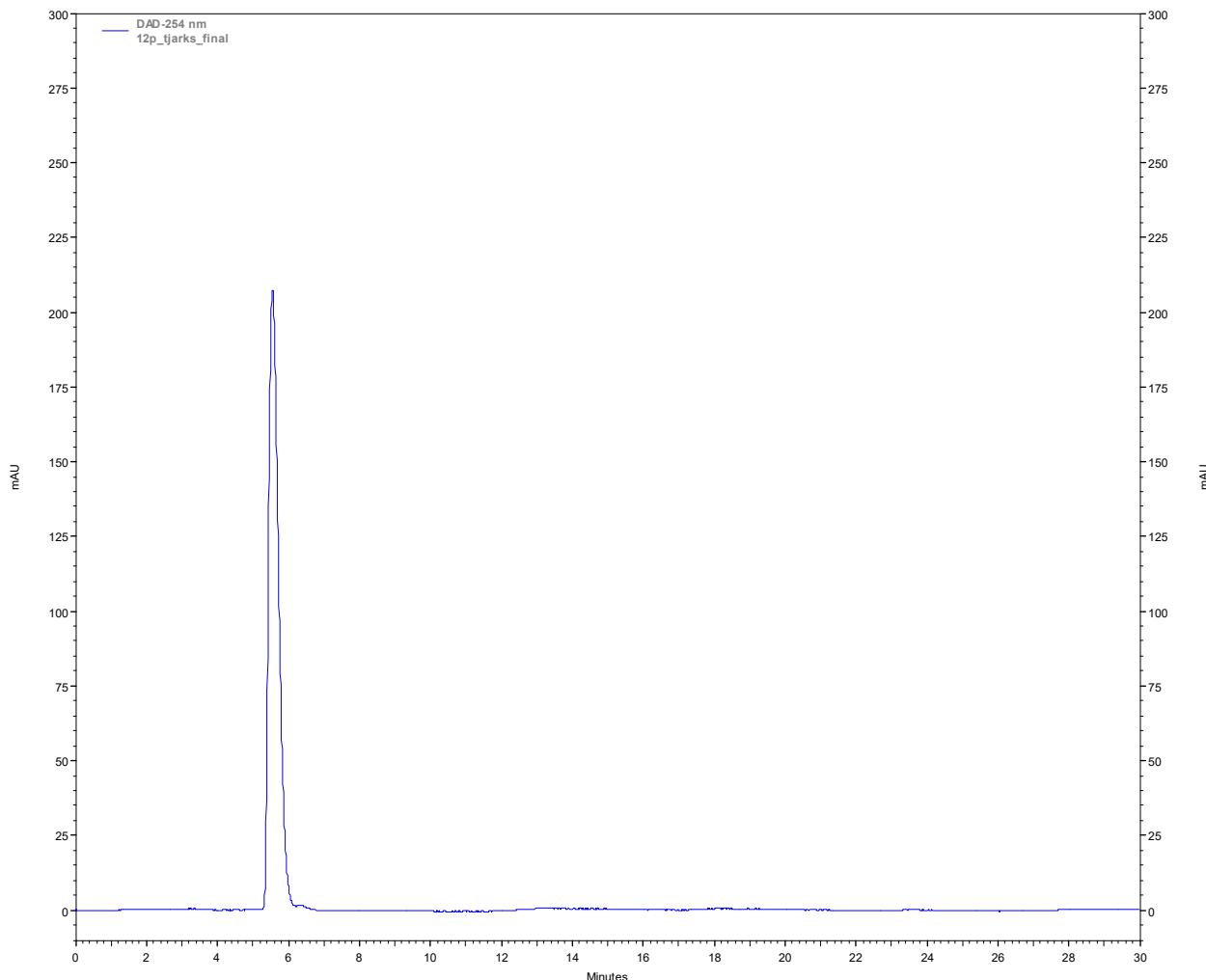
**University of Illinois SCS Mass Spectrometry Laboratory**

Isotopic Peak Pattern for C<sub>16</sub>H<sub>32</sub>B<sub>10</sub>O<sub>2</sub>

Base: m/z 364



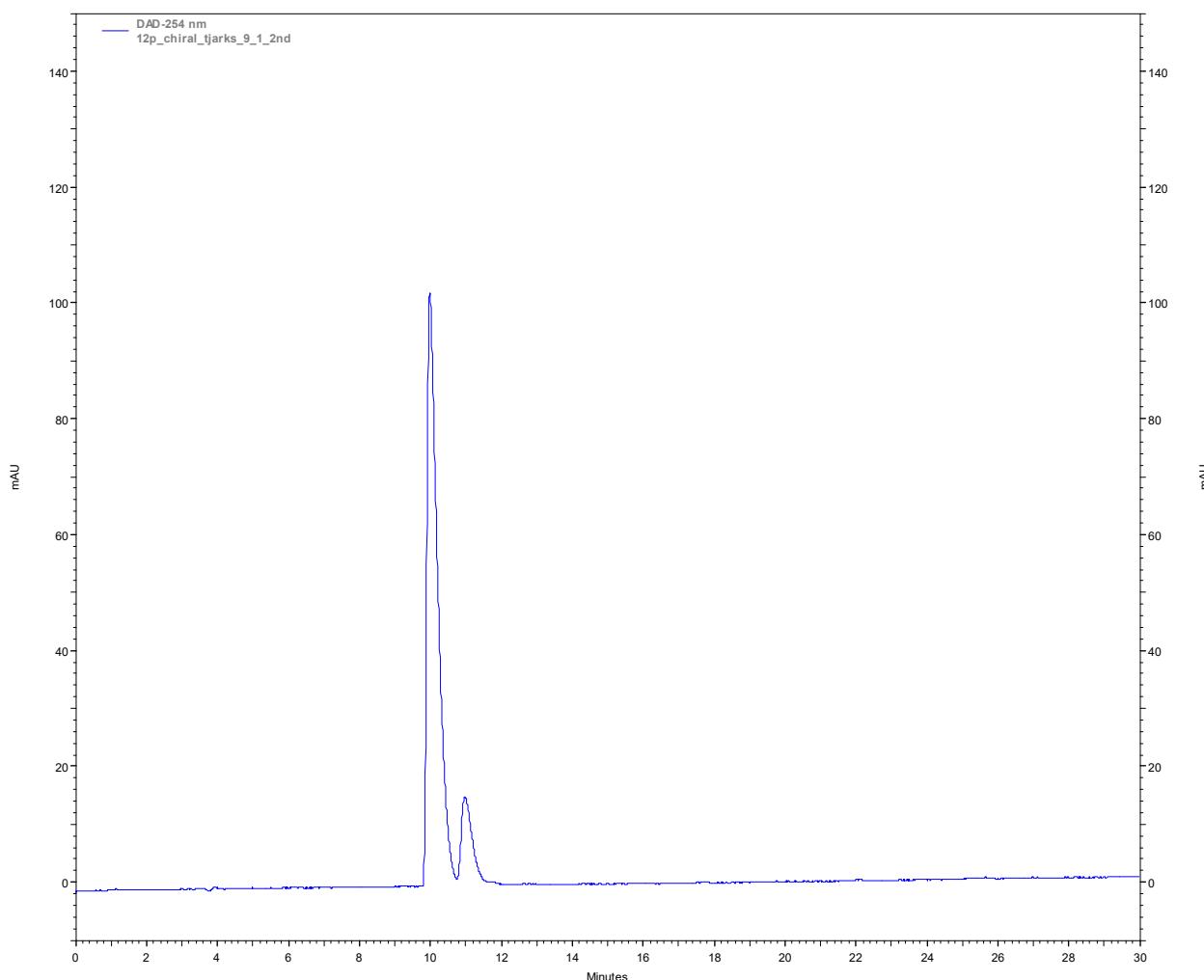
MS of Compound (*R*)-1-[1-(4-methoxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol



#### DAD-254 nm Results

| Retention Time | Area     | Area % |
|----------------|----------|--------|
| 5.553          | 16194772 | 99.81  |
| 6.347          | 31401    | 0.19   |
| Totals         | 16226173 | 100.00 |

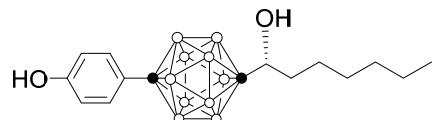
HPLC of Compound (*R*)-1-[1-(4-methoxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol



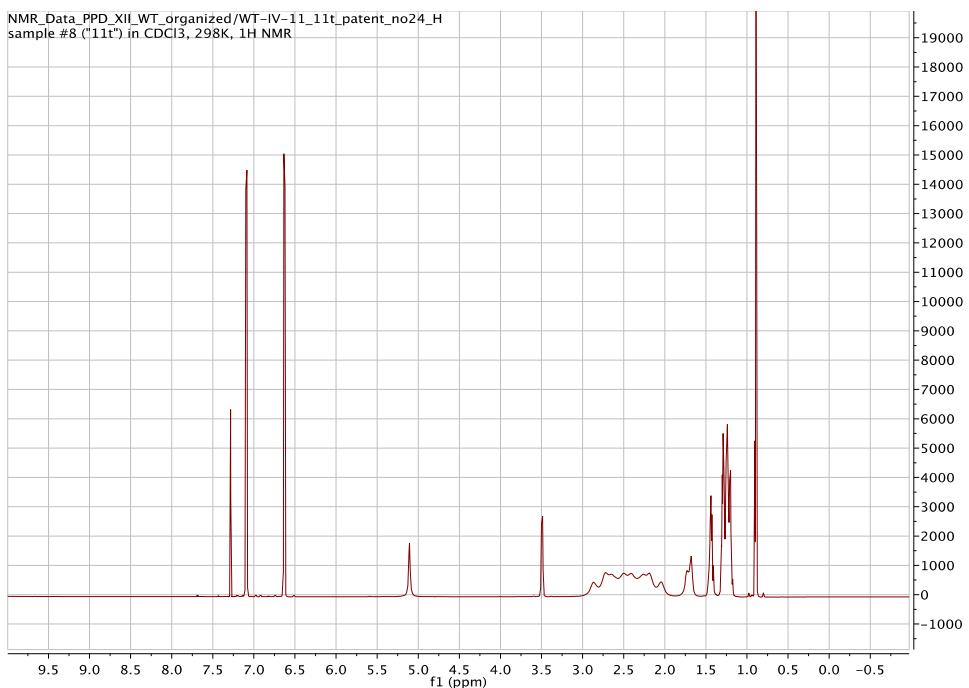
#### DAD-254 nm Results

| Retention Time | Area    | Area % |
|----------------|---------|--------|
| 9.987          | 8567975 | 87.90  |
| 10.967         | 1179215 | 12.10  |
| Totals         | 9747190 | 100.00 |

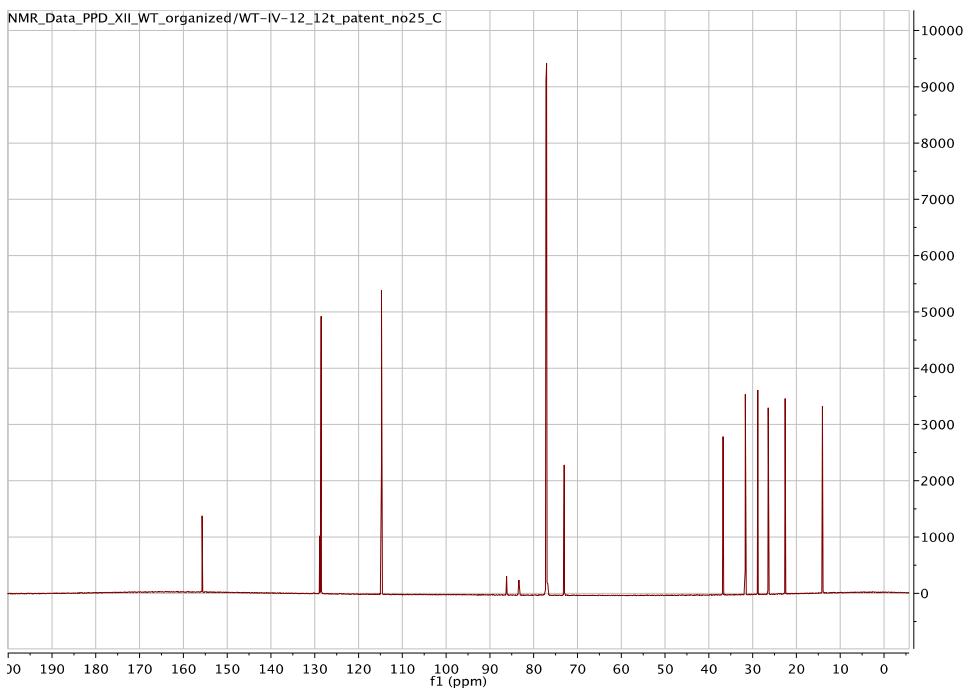
Chiral HPLC of Compound (*R*)-1-[1-(4-methoxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol



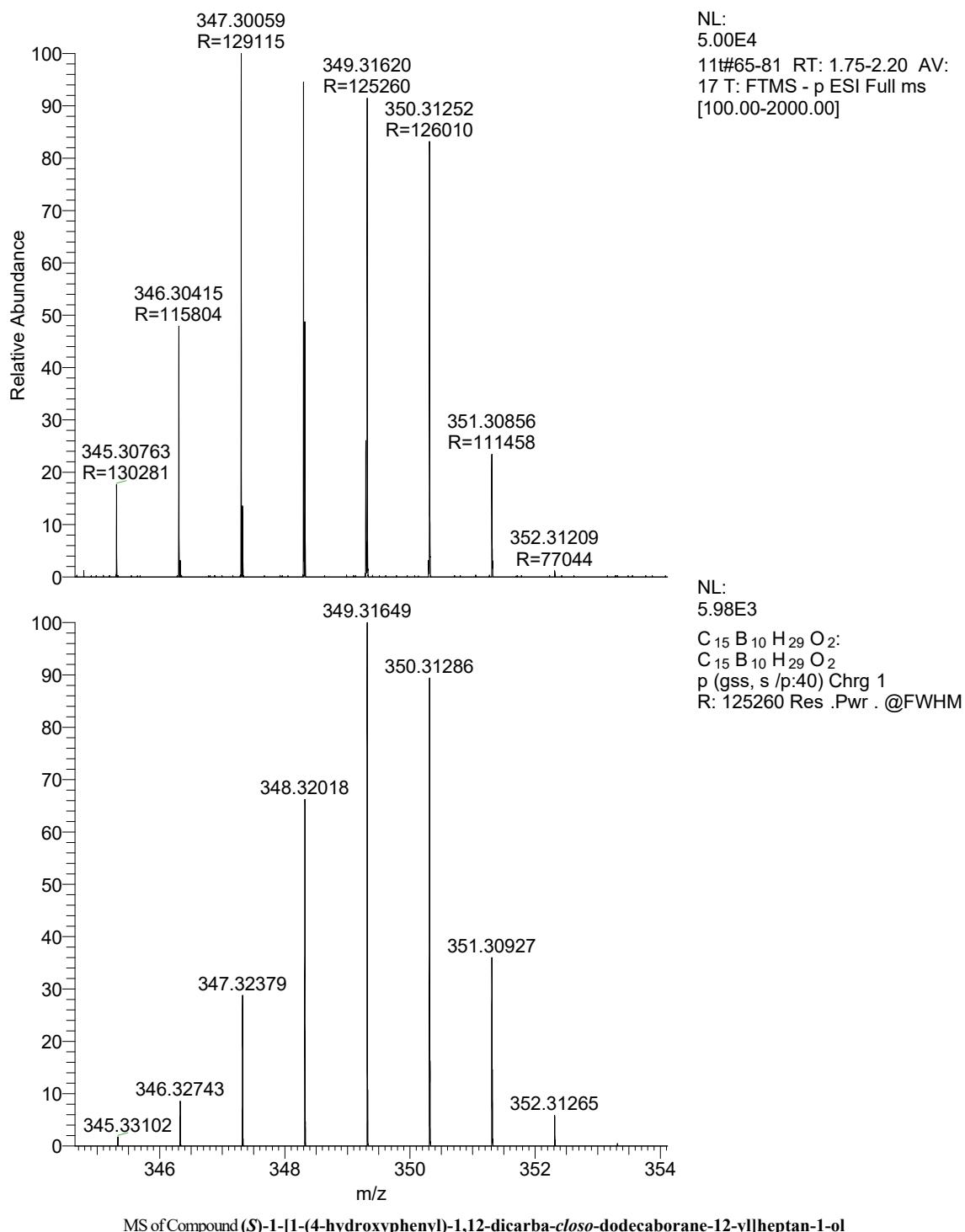
NMR\_Data\_PPD\_XII\_WT\_organized/WT-IV-11\_11t\_patent\_no24\_H  
sample #8 ("11t") in CDCl<sub>3</sub>, 298K, 1H NMR

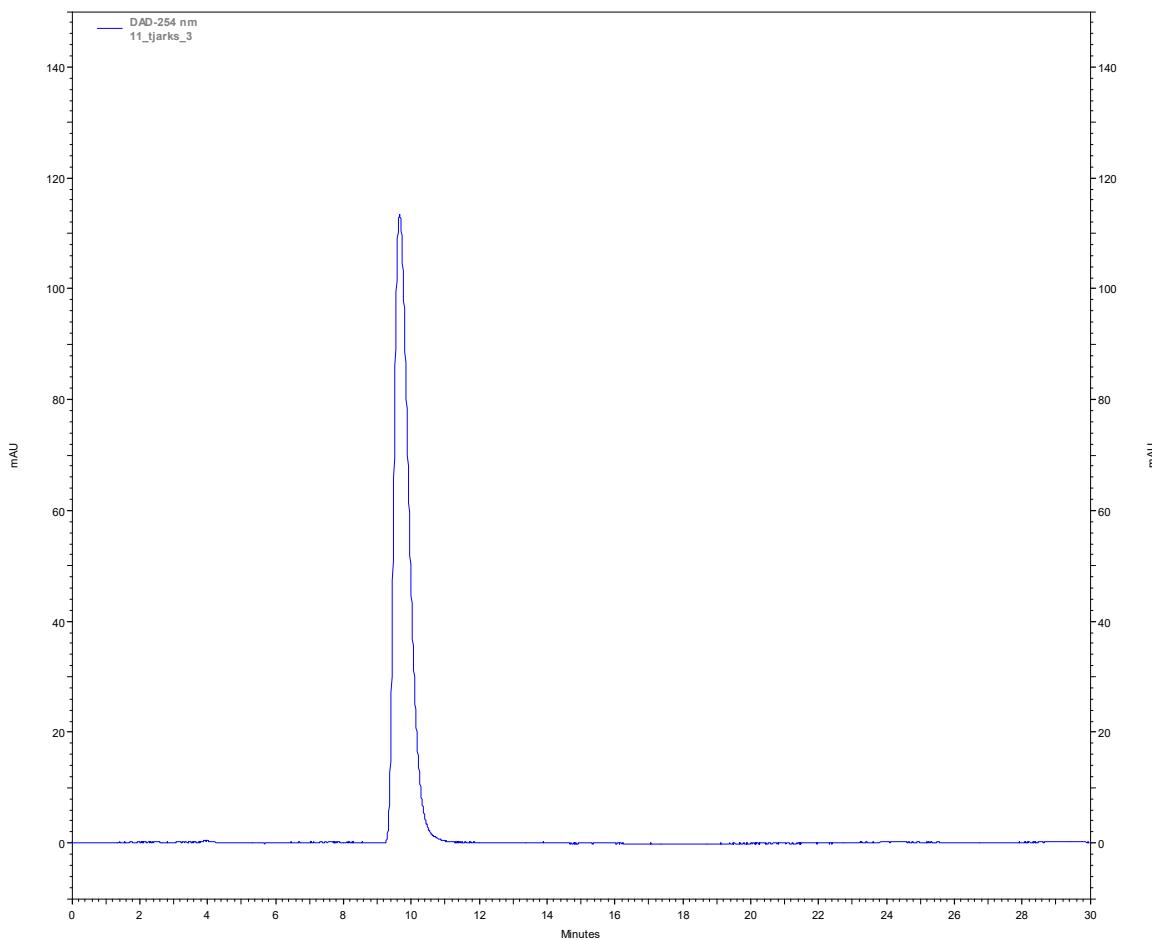


<sup>1</sup>H NMR of Compound (S)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]heptan-1-ol



<sup>13</sup>C NMR of Compound (S)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]heptan-1-ol

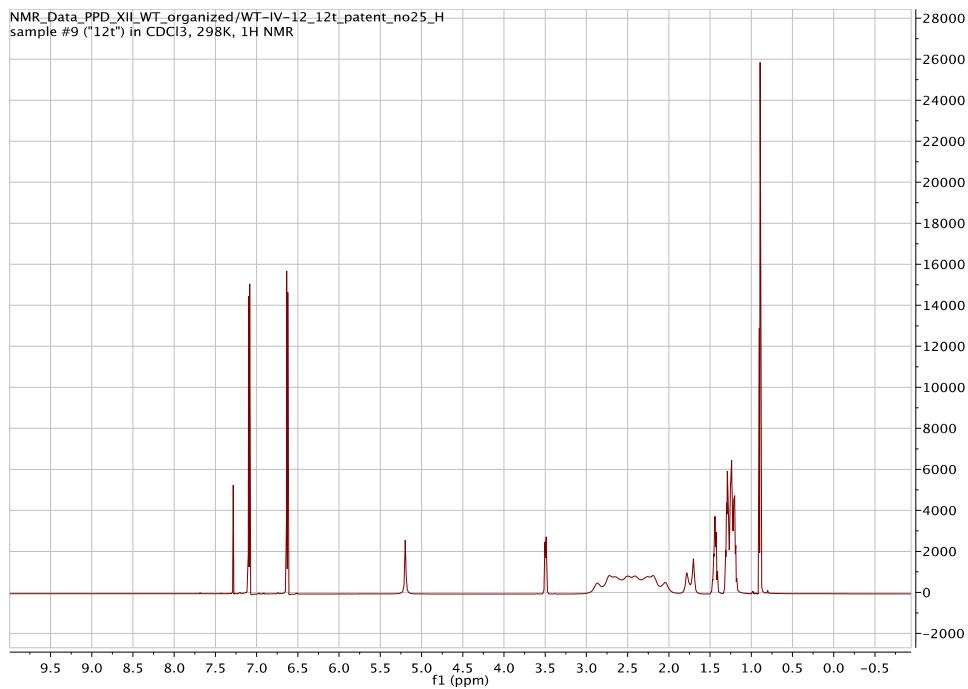
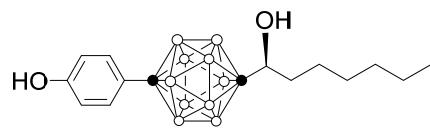




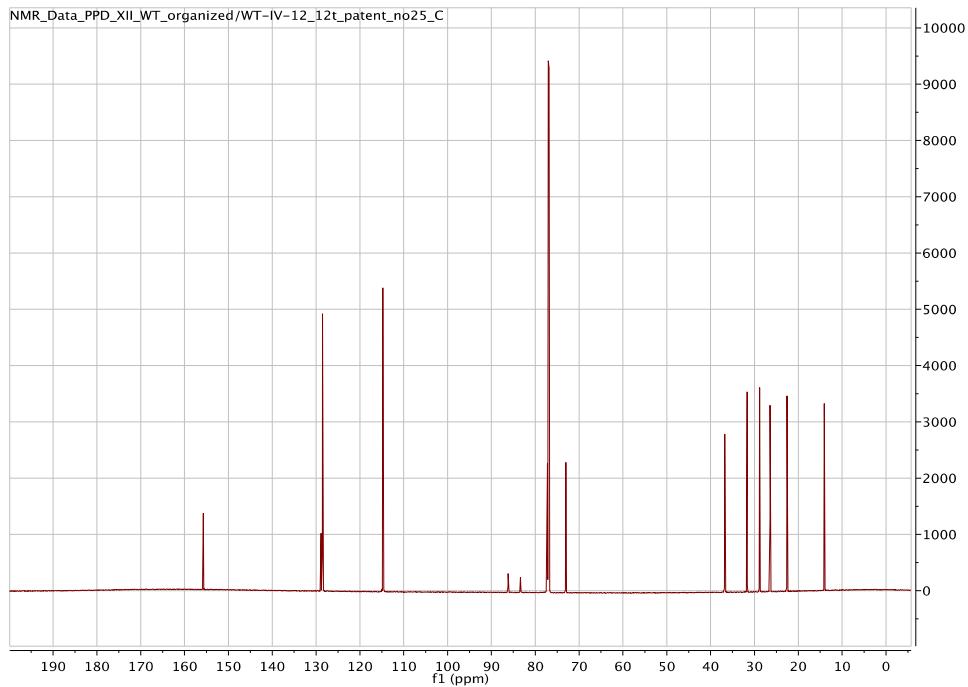
**DAD-254 nm Results**

| Retention Time | Area     | Area % |
|----------------|----------|--------|
| 9.660          | 13963846 | 100.00 |
| Totals         | 13963846 | 100.00 |

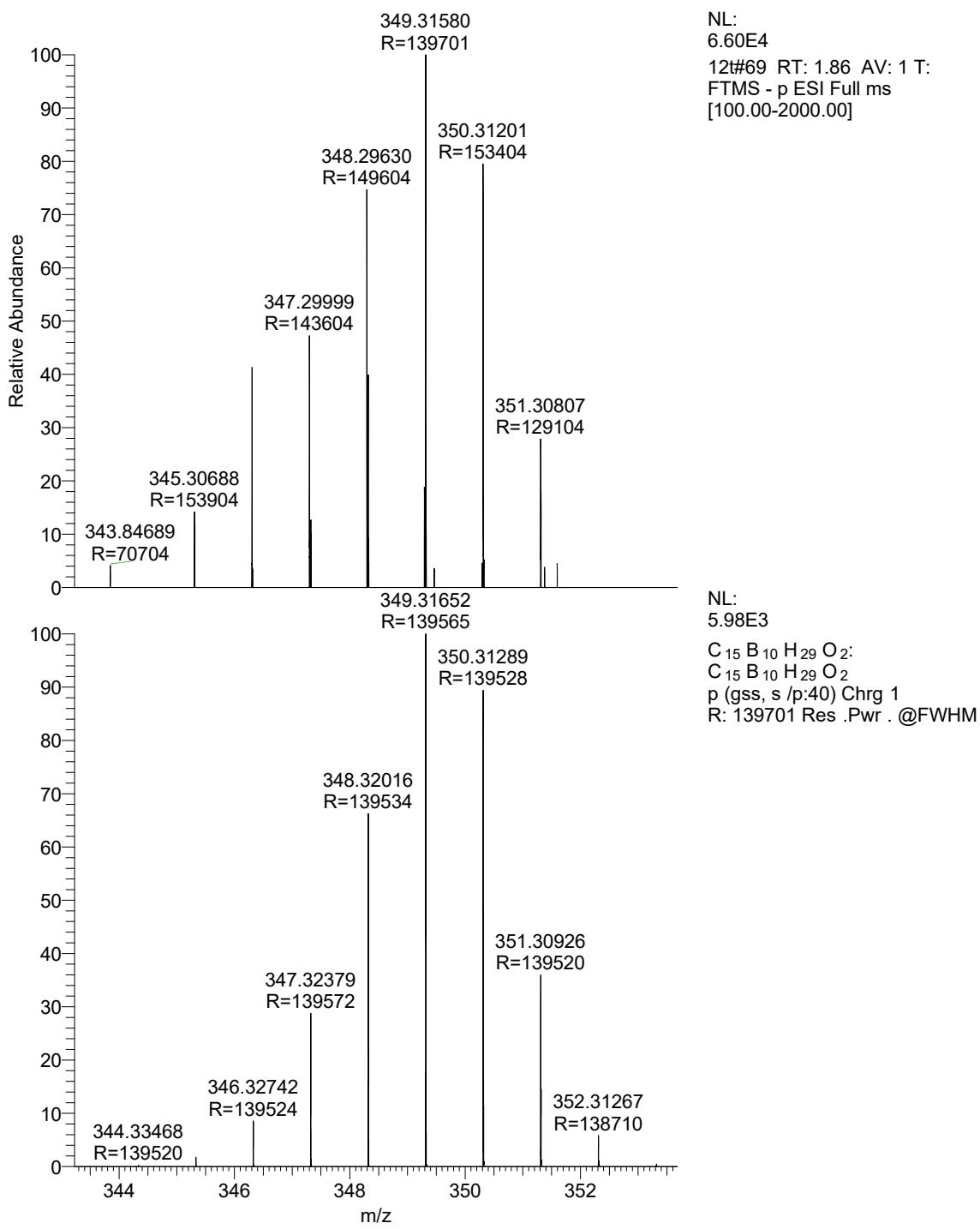
HPLC of Compound (*S*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol

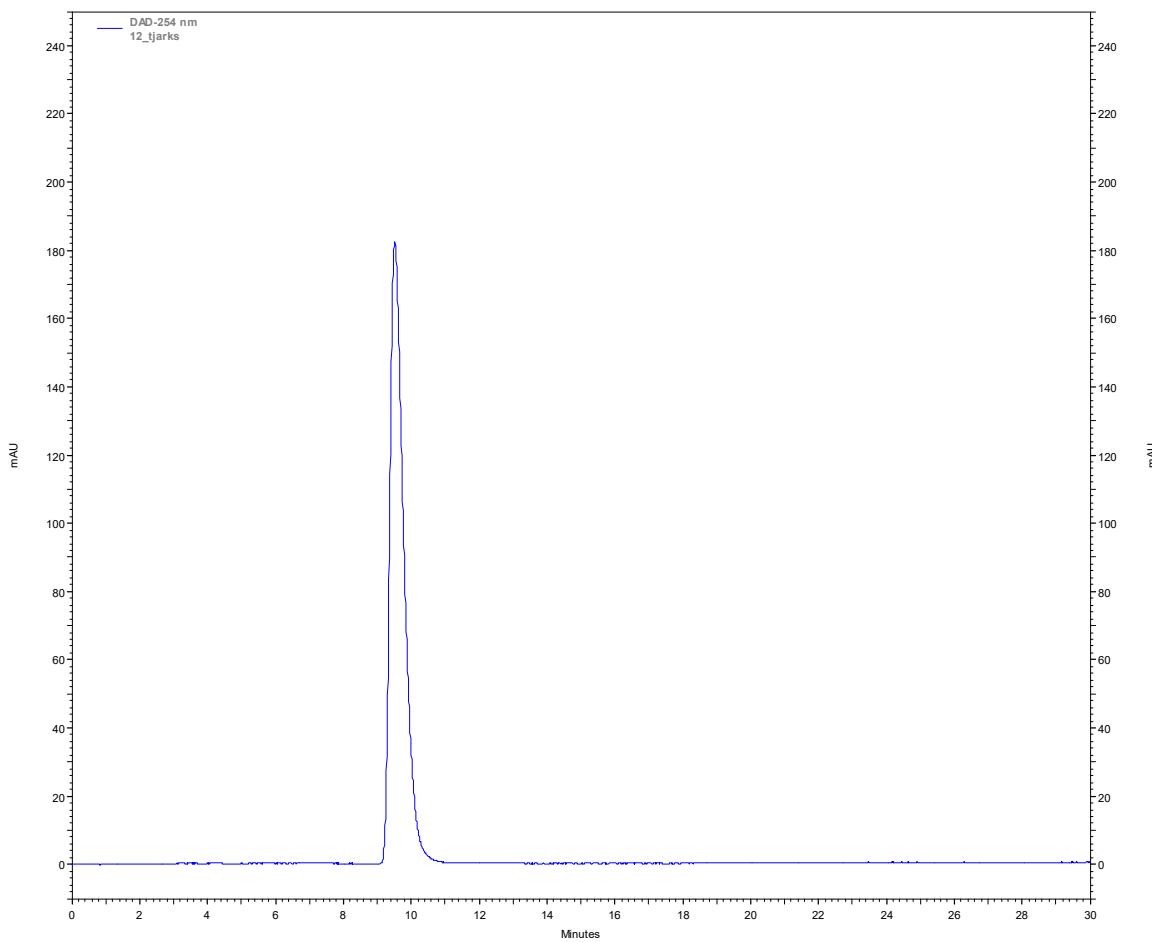


<sup>1</sup>H NMR of Compound (R)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]heptan-1-ol



<sup>13</sup>C NMR of Compound (R)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]heptan-1-ol

MS of Compound (*R*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol



#### DAD-254 nm Results

| Retention Time | Area     | Area % |
|----------------|----------|--------|
| 7.200          | 33816    | 0.16   |
| 9.520          | 20939229 | 99.84  |
| Totals         | 20973045 | 100.00 |

HPLC of Compound (*R*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol

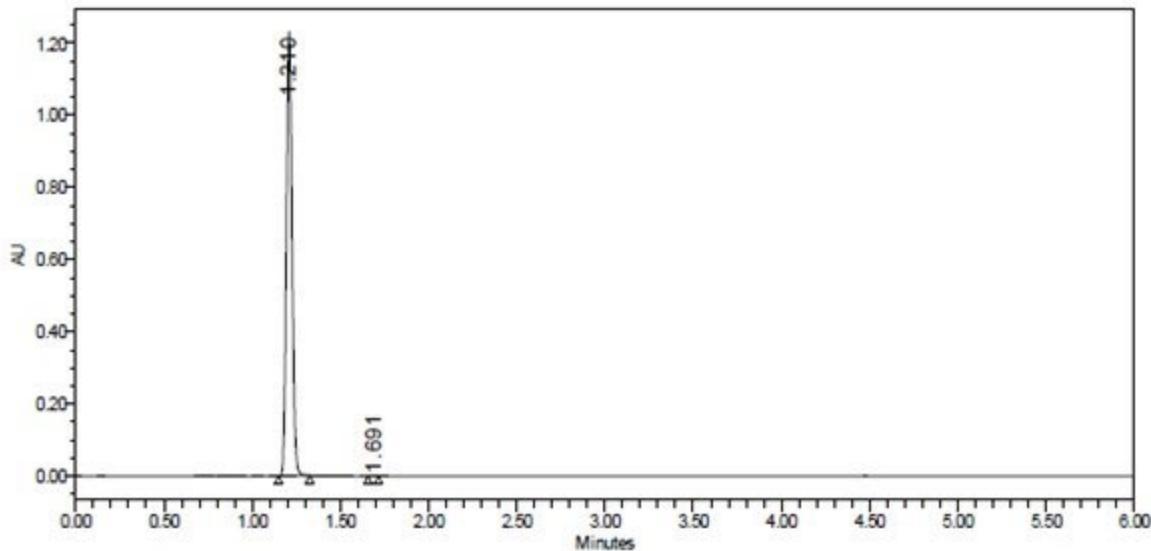
A portion of (*R*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol (**8**) was purified by chiral SFC using the conditions outlined below (ChiralPak AD column with IPA / CO<sub>2</sub> as eluting solvents) to afford a sample of (*R*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol (**8**) that was 99.9%ee.

## Chiral SFC Report

Anal-SFC-3

### S A M P L E I N F O R M A T I O N

|                     |   |                    |             |
|---------------------|---|--------------------|-------------|
| Sample ID:          | et24777-26-plal                                 | Acquired By:       | System      |
| Compound ID:        | WT-VI-12  | Sample Set Name:   | 20190117_18 |
| Acq Method :        | AD_IPA_IPAm_40_25_35                            | Processing Method: | 15          |
| Date Acquired:      | 17/1/2019 PM 9:07:16 CST                        | Injection Volume:  | 1.00 ul     |
| Date Processed:     | 18/1/2019 PM 7:25:25 CST                        | Vial:              | 2:C,8       |
| Proc. Chnl. Descr.: | PDA Ch1 220nm@4.8nm                             | Run Time:          | 6.0 Minutes |
| Channel Name:       | PDA Ch1 220nm@4.8nm                             |                    |             |
| Instrument:         | CAS-TJ-ANA-SFC-3(Waters UPCC with PDA detector) |                    |             |

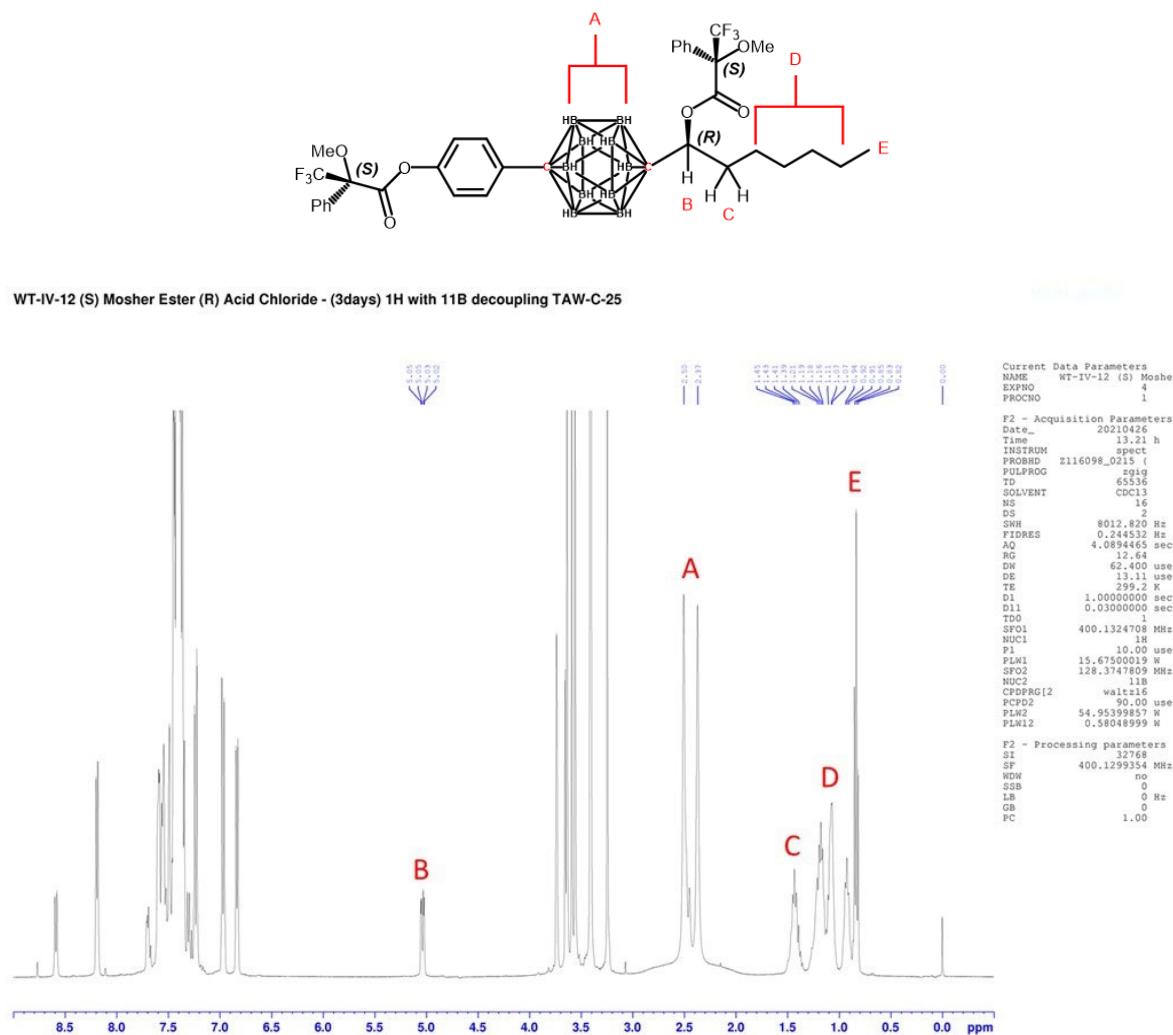


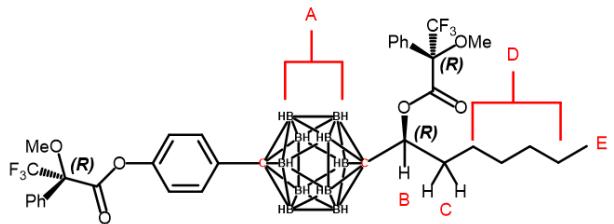
|   | RT    | Area    | % Area | Height  |
|---|-------|---------|--------|---------|
| 1 | 1.210 | 2749915 | 99.99  | 1230353 |
| 2 | 1.691 | 356     | 0.01   | 184     |

Chiral SFC of Compound (*R*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol

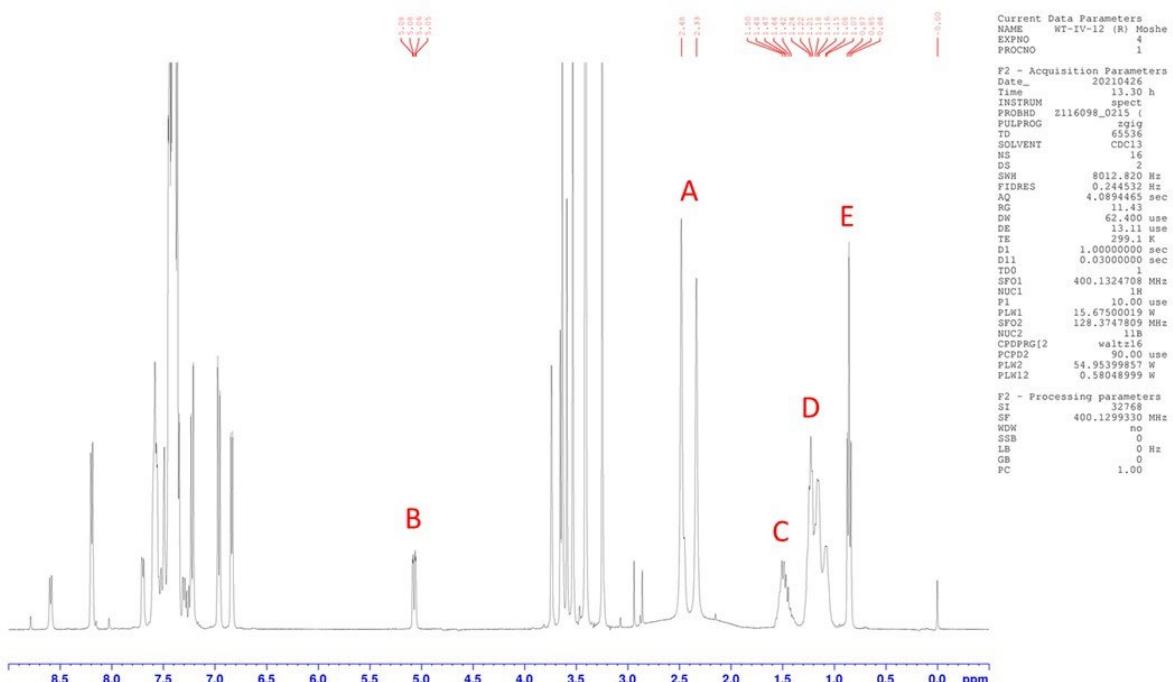
The absolute configuration of (*R*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol (**8**) was determined by HNMR analysis of both the *bis*-(*R*)-Mosher ester and *bis*-(*S*)-Mosher ester of (*R*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol (**8**). Synthesis of the *bis*-(*R*)-Mosher ester and *bis*-(*S*)-Mosher ester of (*R*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol (**8**) were synthesized using the procedure below for the *bis*-(*S*)-Mosher ester:

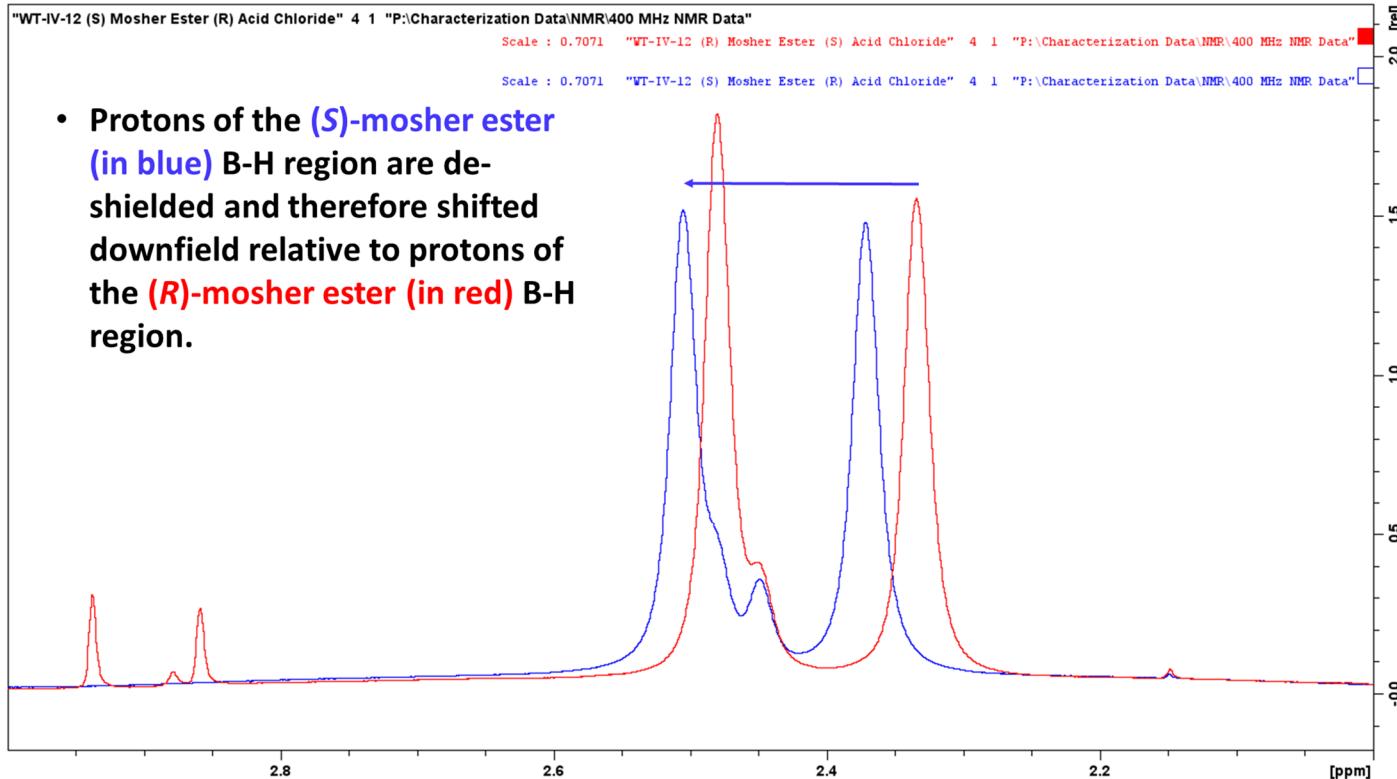
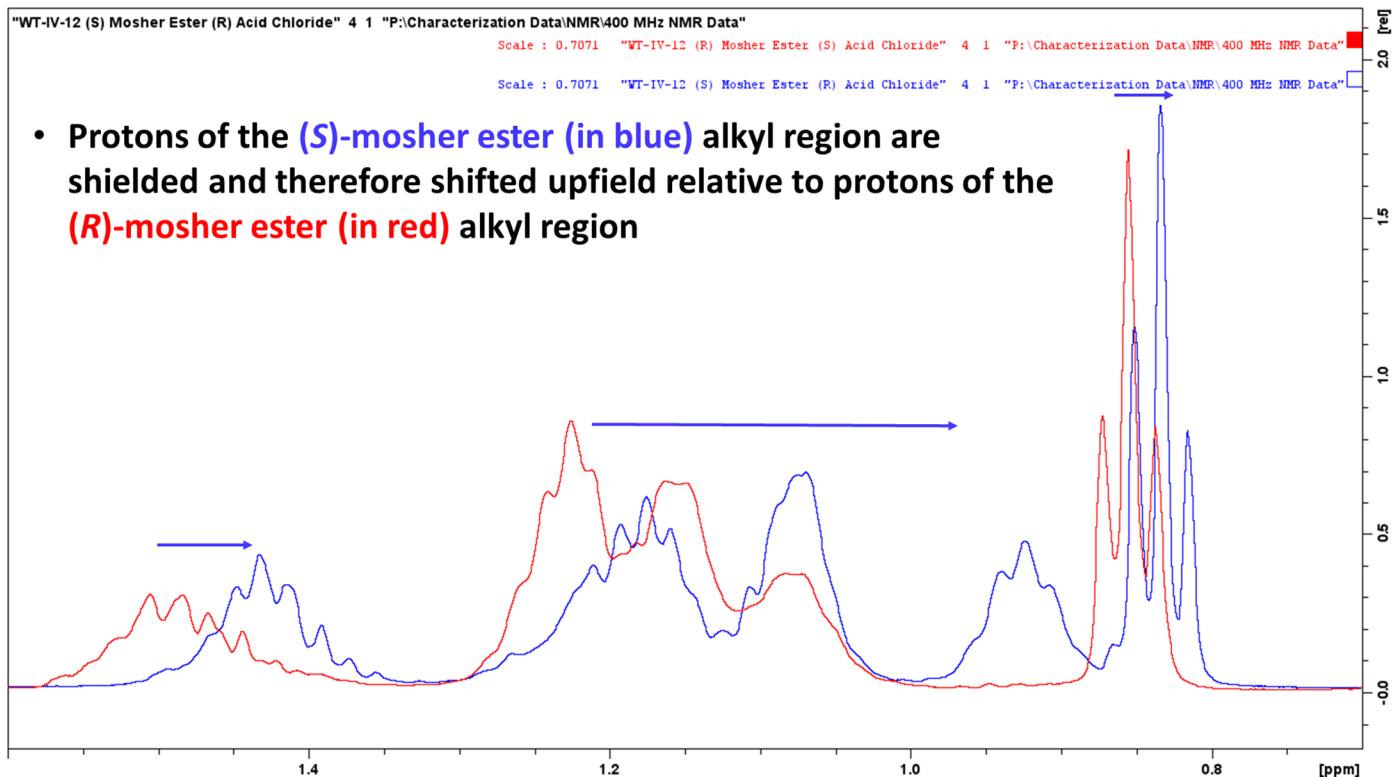
In a 1.5 dram vial equipped with a stir bar, (*R*)-1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol (**8**) (20.0 mg, 57.1  $\mu$ mol), Pyridine-*d*<sub>5</sub> (48.9  $\mu$ L, 10 eq., 571  $\mu$ mol), and DMAP (6.97 mg, 57.1  $\mu$ mol) were dissolved in CDCl<sub>3</sub> (1.00 mL), and (*R*)-(−)-MTPA-Cl (74.1  $\mu$ L, 6.9 eq., 396  $\mu$ mol) was added thereto and stirred for 3 days at RT. Aliquots were removed periodically to monitor the reaction progress by <sup>1</sup>H NMR.

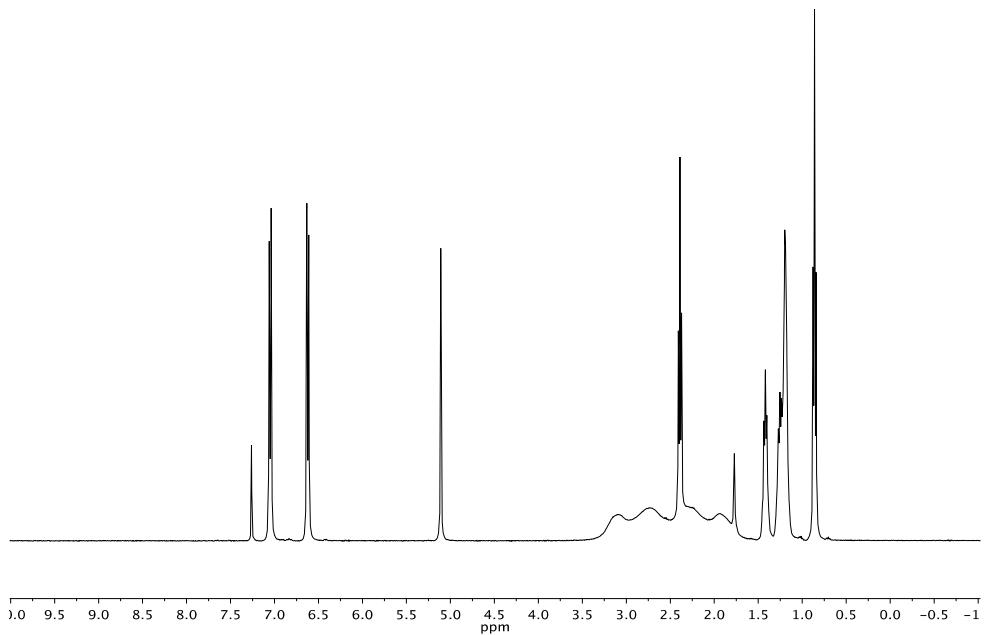
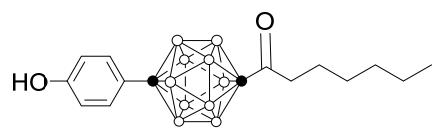




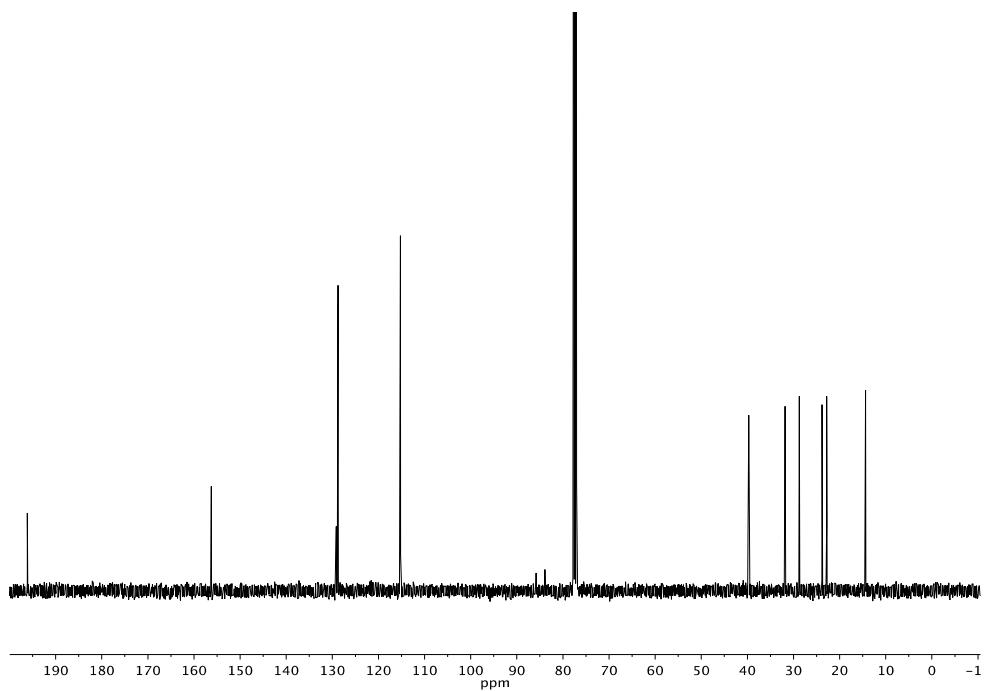
WT-IV-12 (R) Mosher Ester (S) Acid Chloride - (3 days) 1H with 11B decoupling TAW-C-26



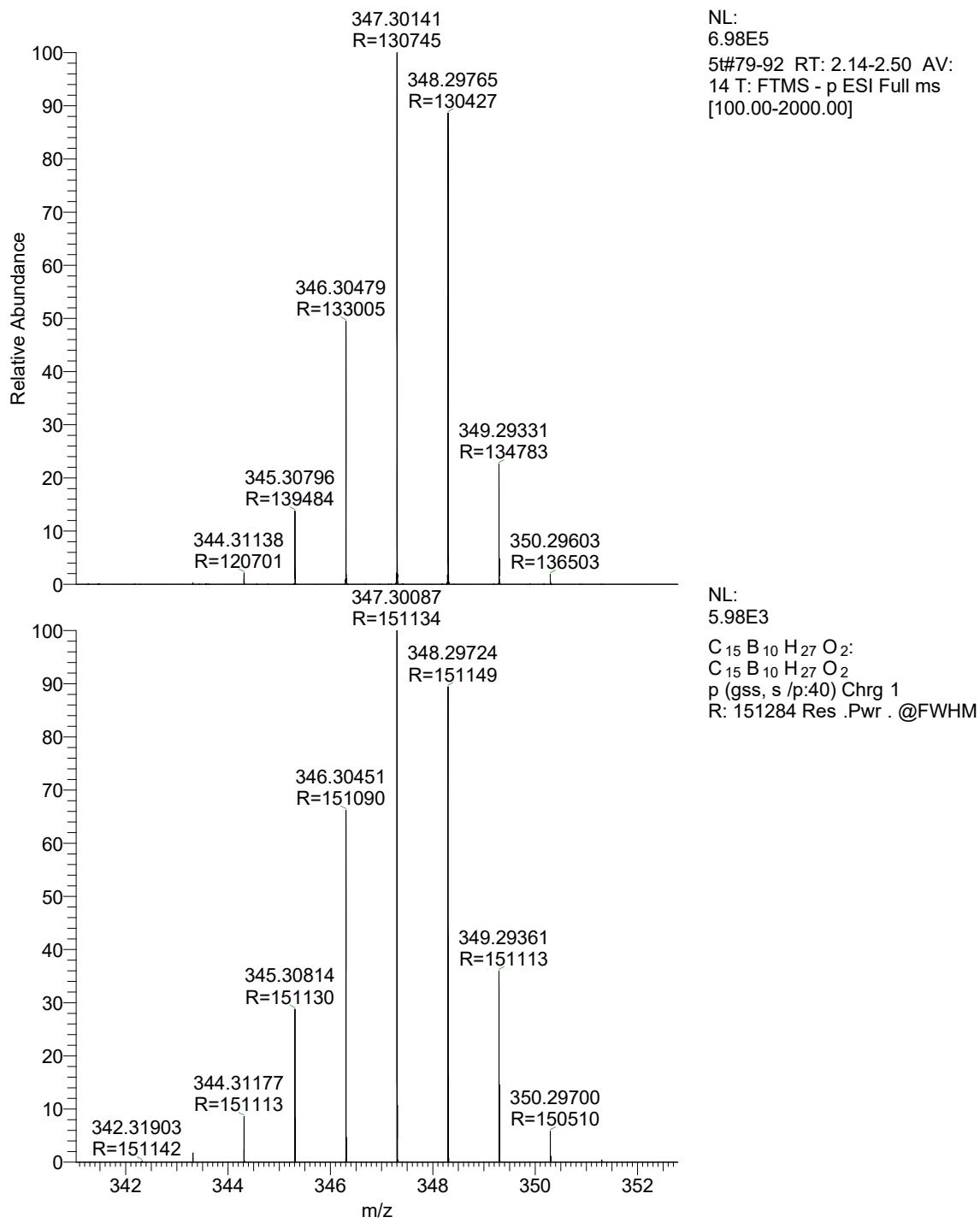


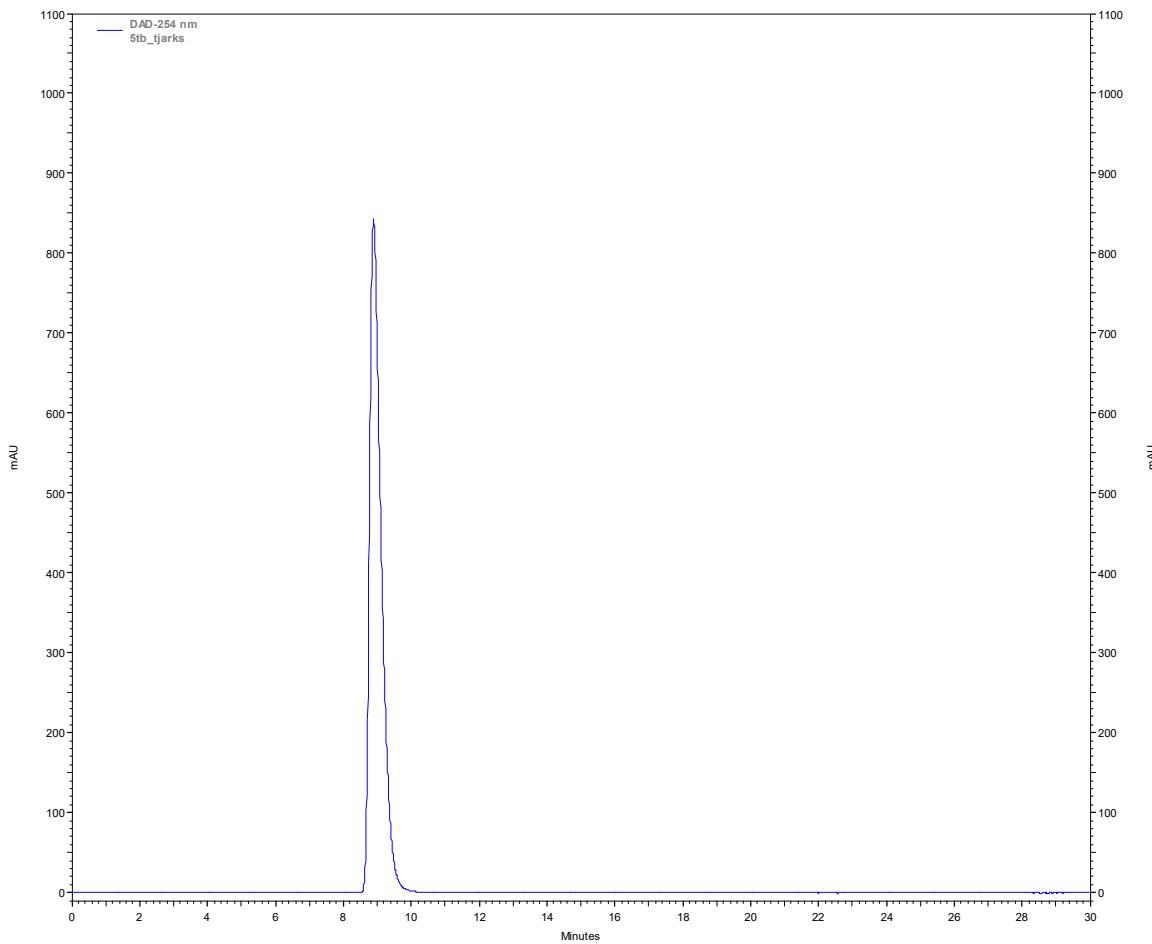


<sup>1</sup>H NMR of Compound 1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-one



$^{13}\text{C}$  NMR of Compound 1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*-o-dodecaborane-12-yl]heptan-1-one



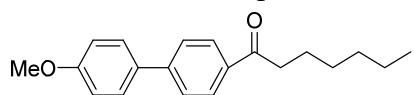


#### DAD-254 nm Results

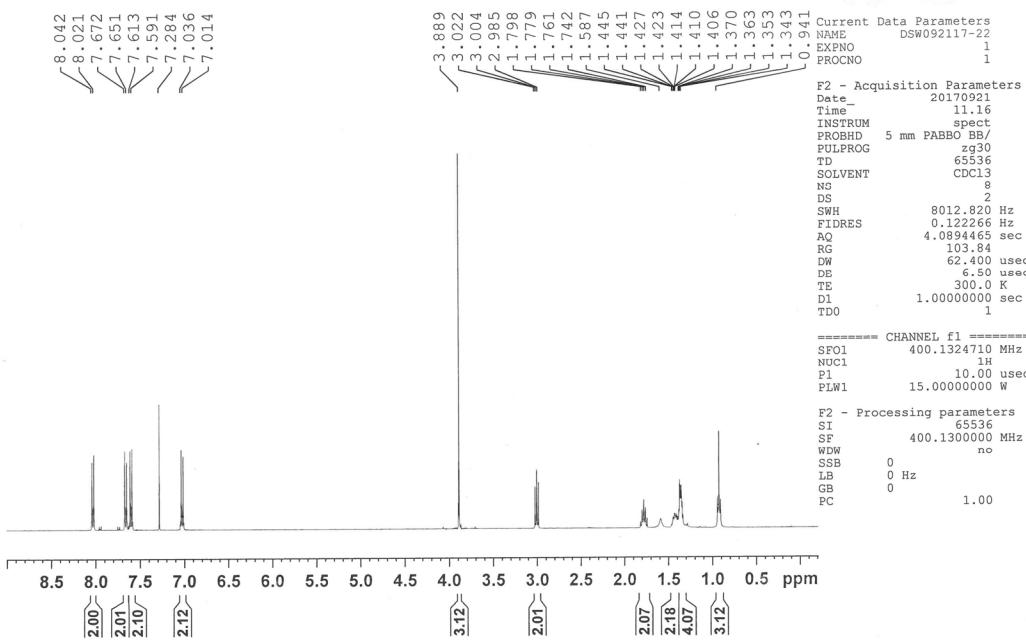
| Retention Time | Area     | Area % |
|----------------|----------|--------|
| 8.893          | 80566168 | 100.00 |
| Totals         | 80566168 | 100.00 |

HPLC of Compound 1-[1-(4-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-one

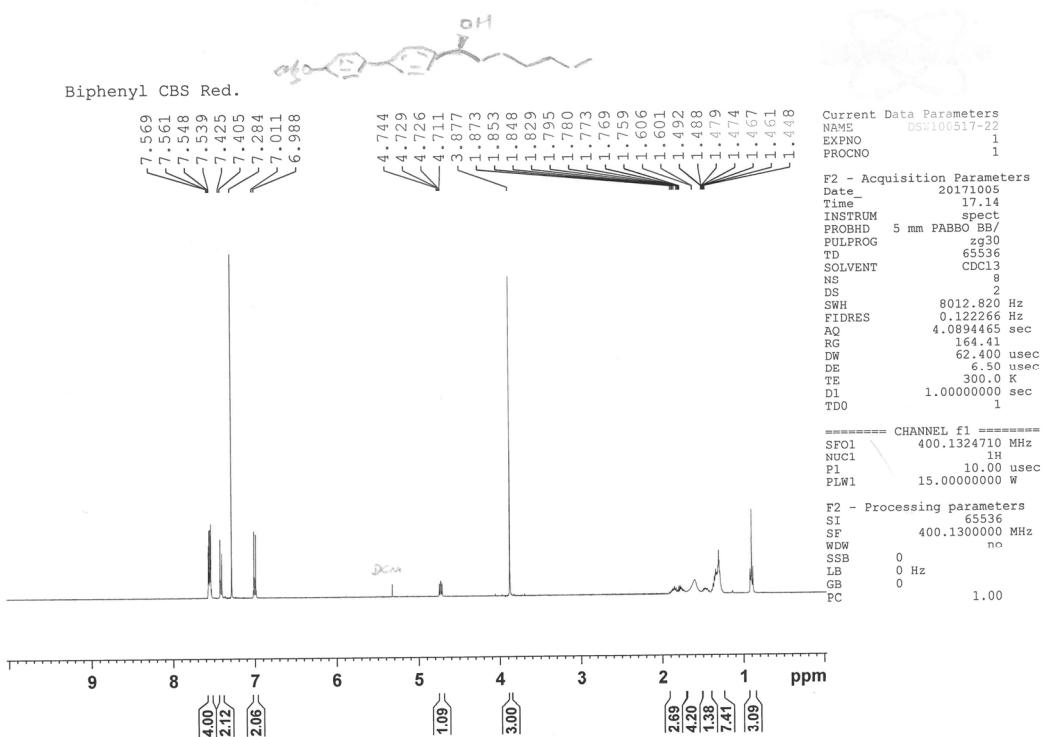
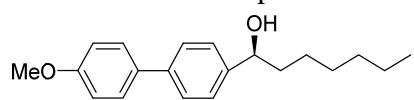
## <sup>1</sup>H NMR of Compound 11



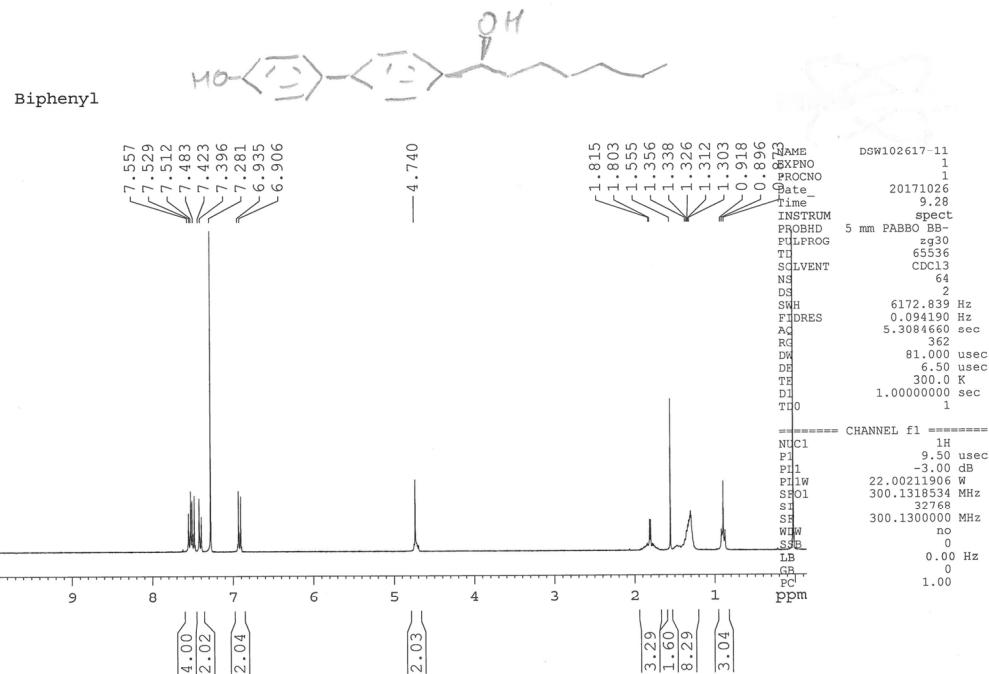
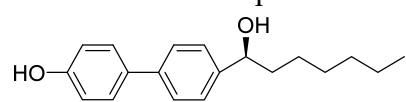
### Biphenyl ketone



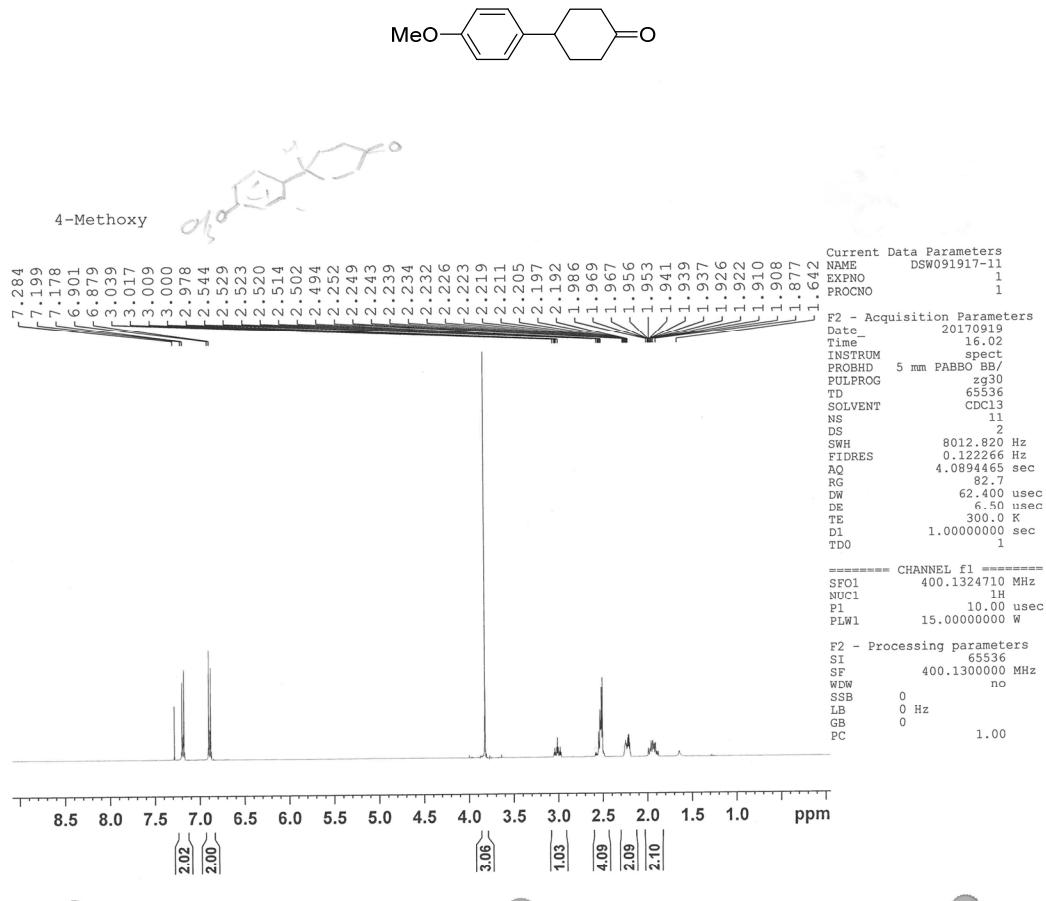
<sup>1</sup>H NMR of Compound 12



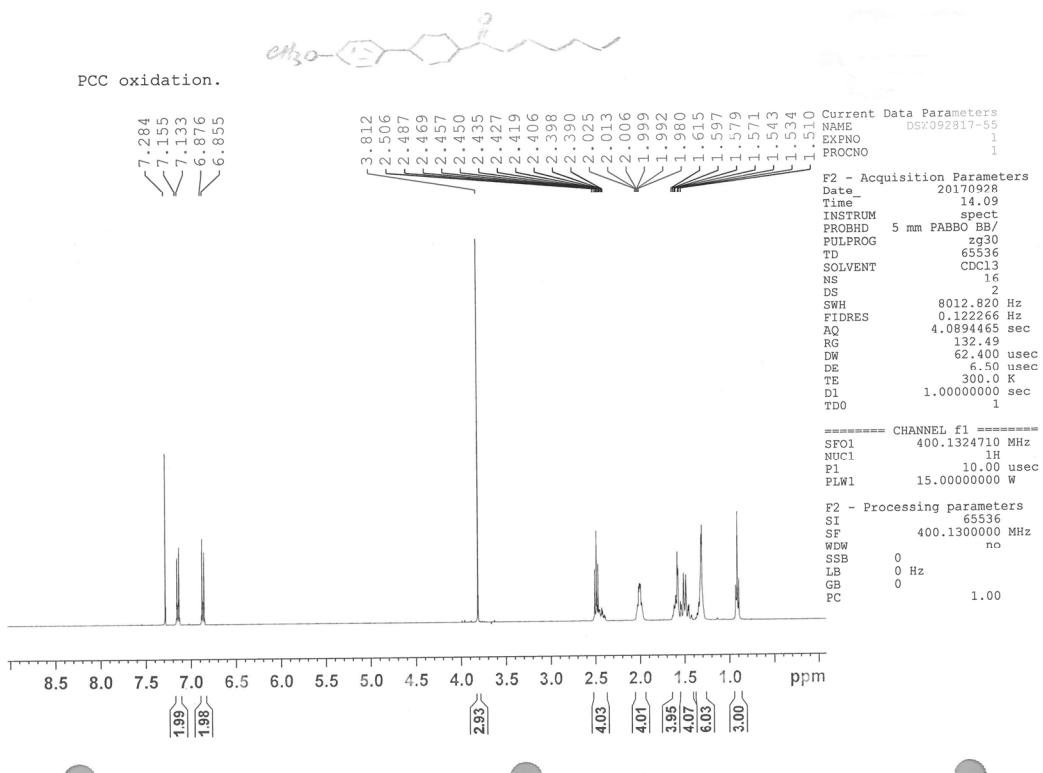
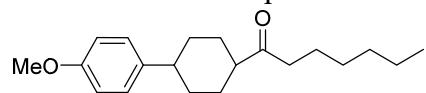
<sup>1</sup>H NMR of Compound 13



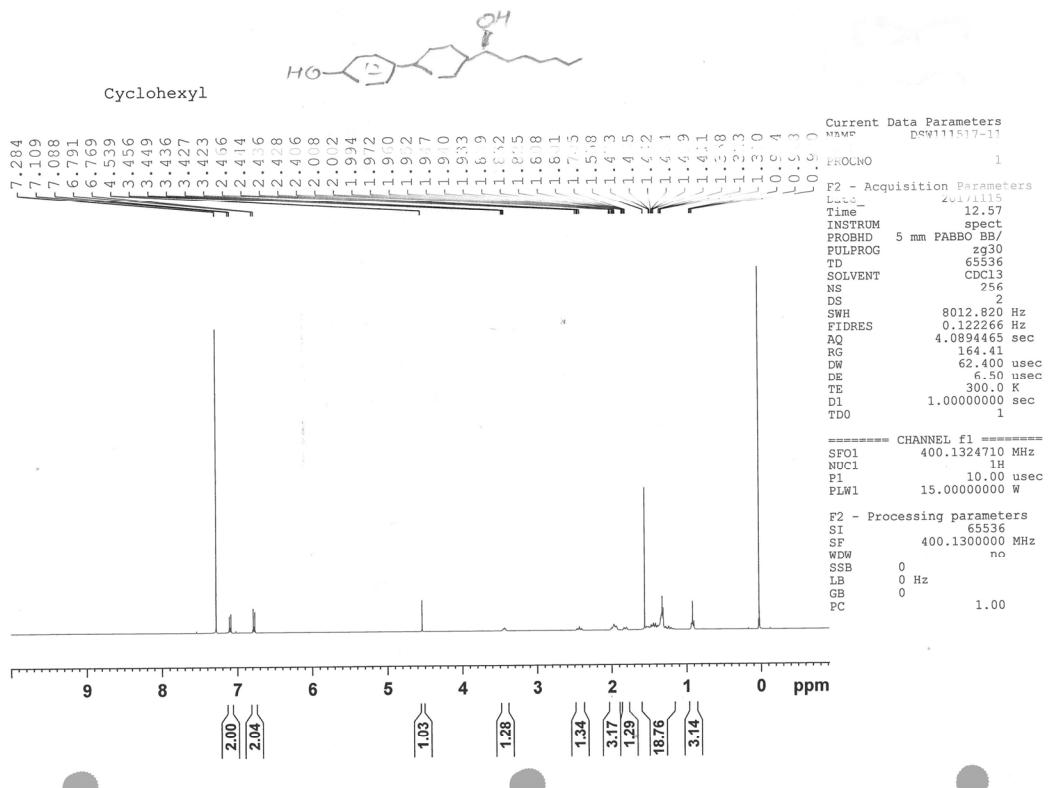
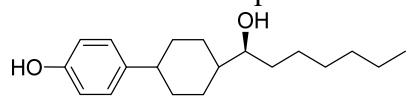
<sup>1</sup>H NMR of Compound 15



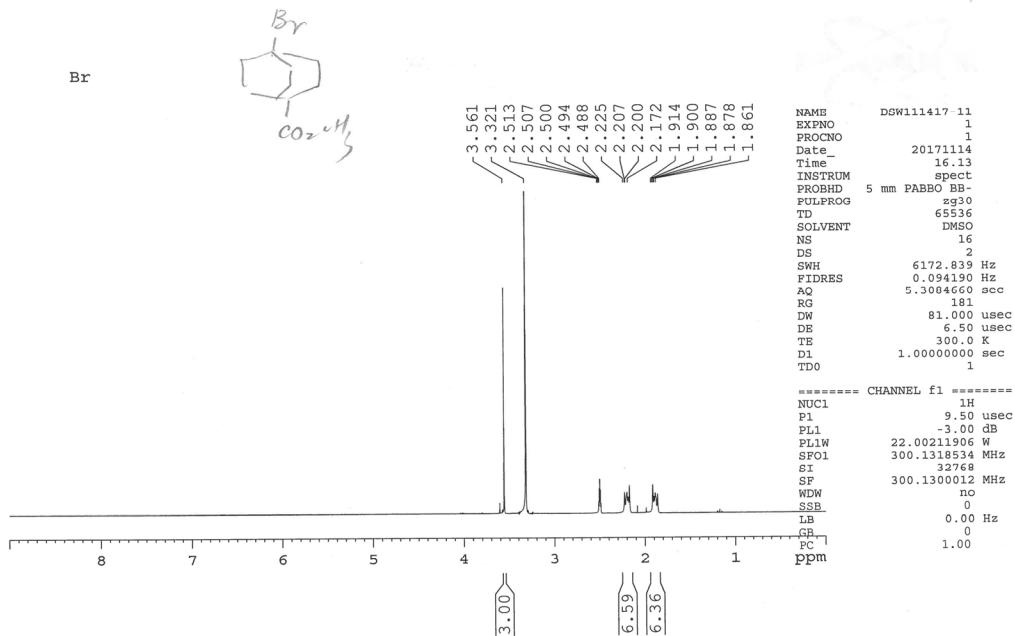
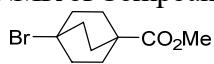
<sup>1</sup>H NMR of Compound 16



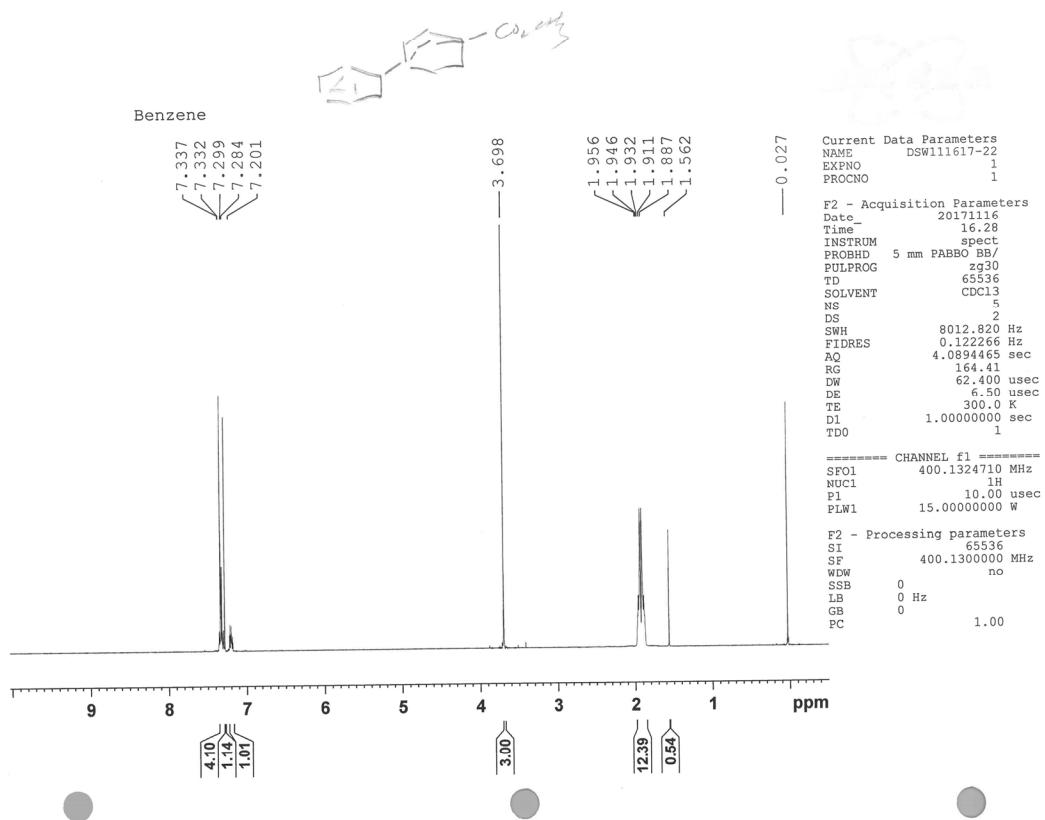
<sup>1</sup>H NMR of Compound 17



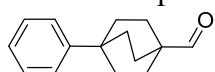
<sup>1</sup>H NMR of Compound 19



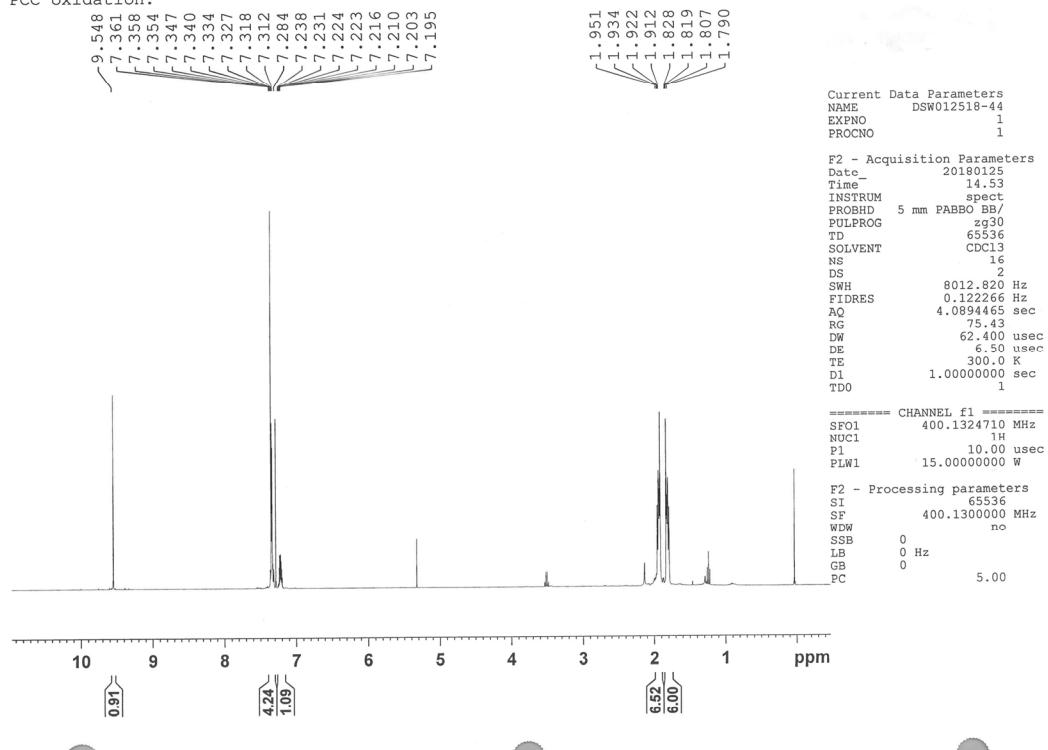
<sup>1</sup>H NMR of Compound 20



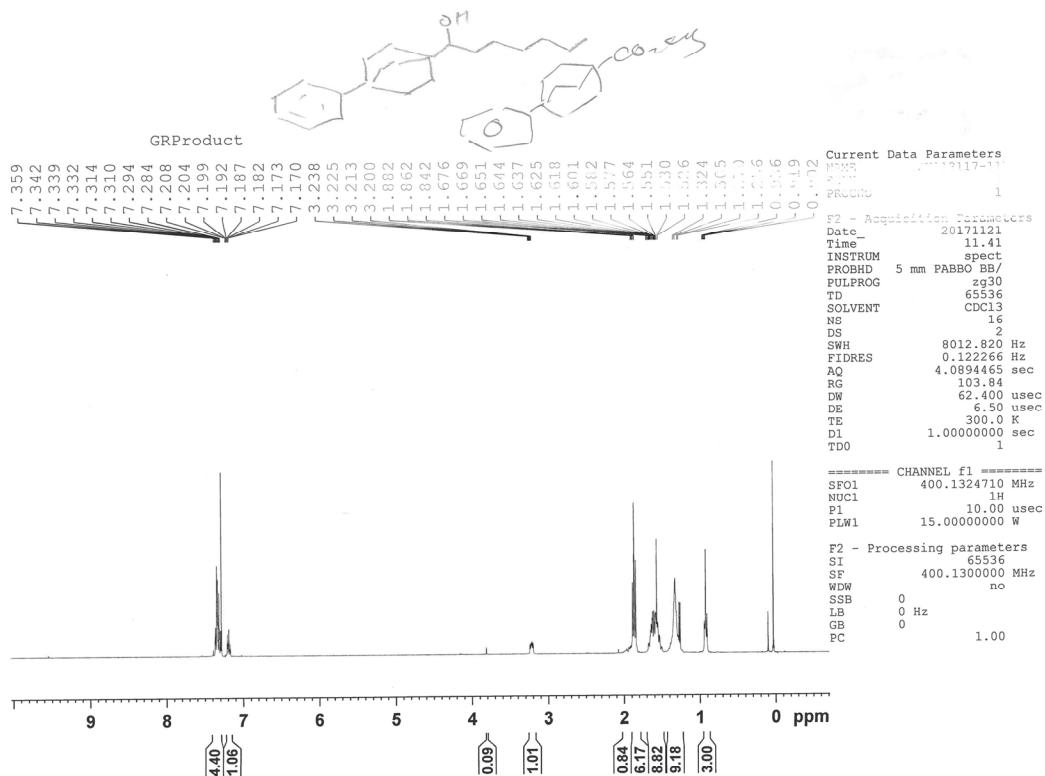
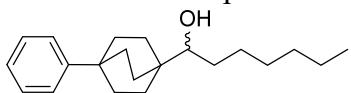
<sup>1</sup>H NMR of Compound 21



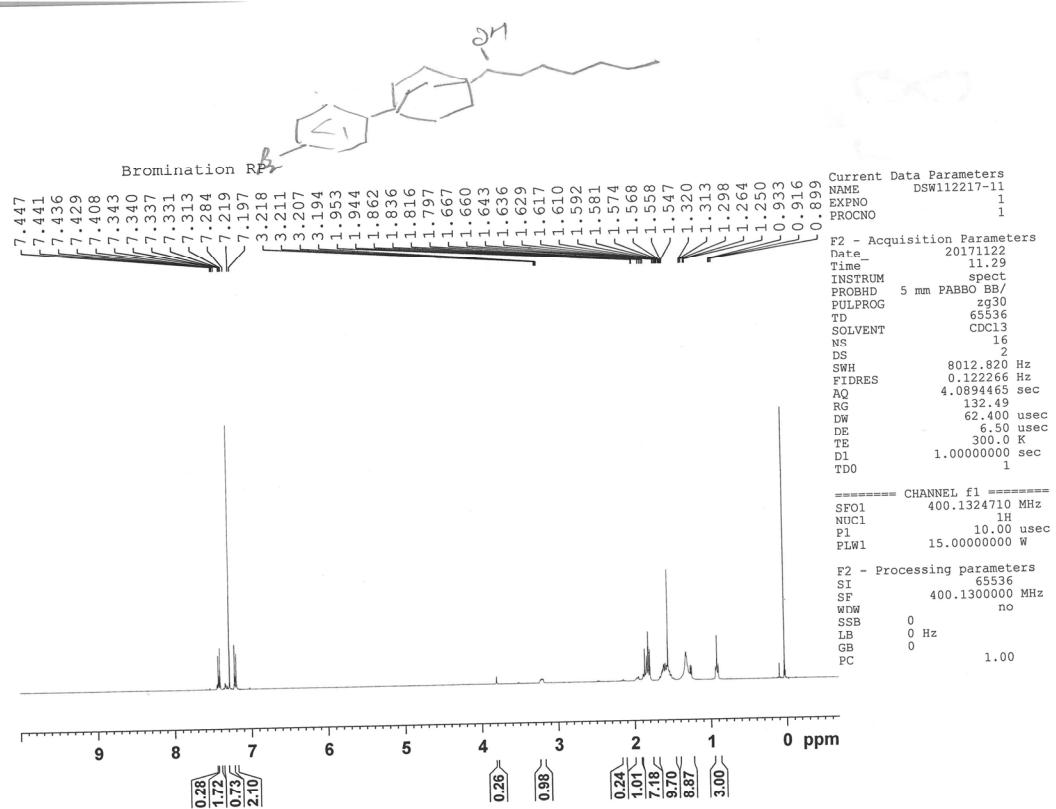
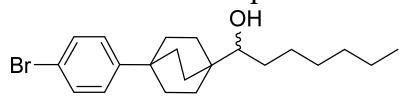
PCC oxidation.



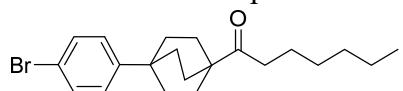
<sup>1</sup>H NMR of Compound 22



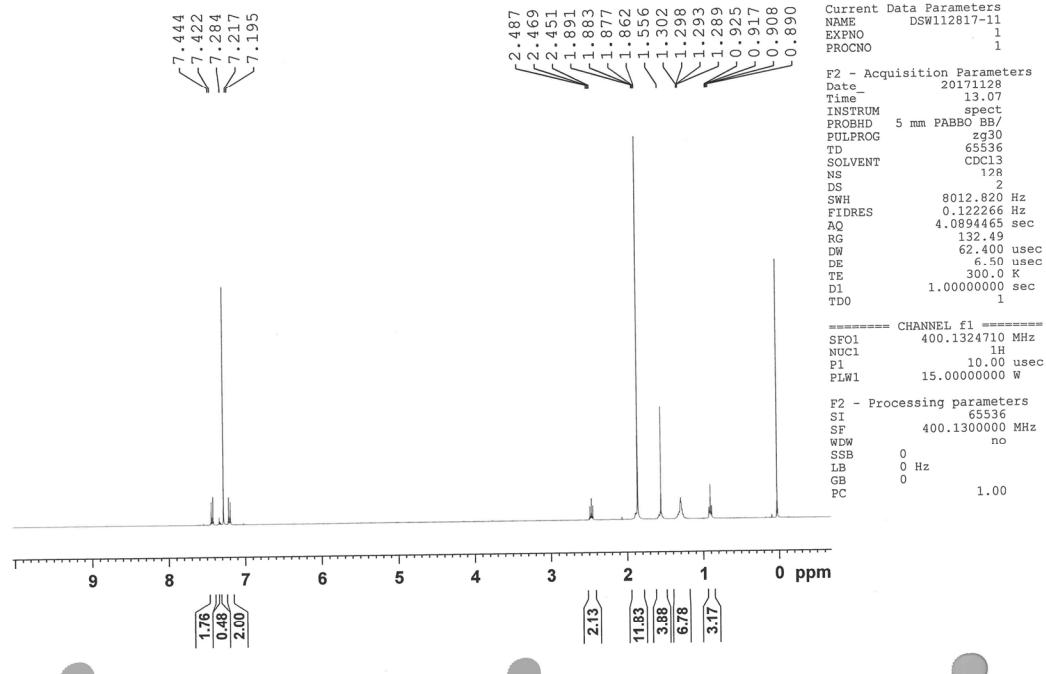
<sup>1</sup>H NMR of Compound 23



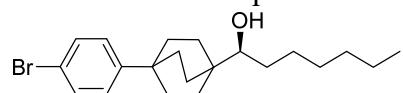
<sup>1</sup>H NMR of Compound 24



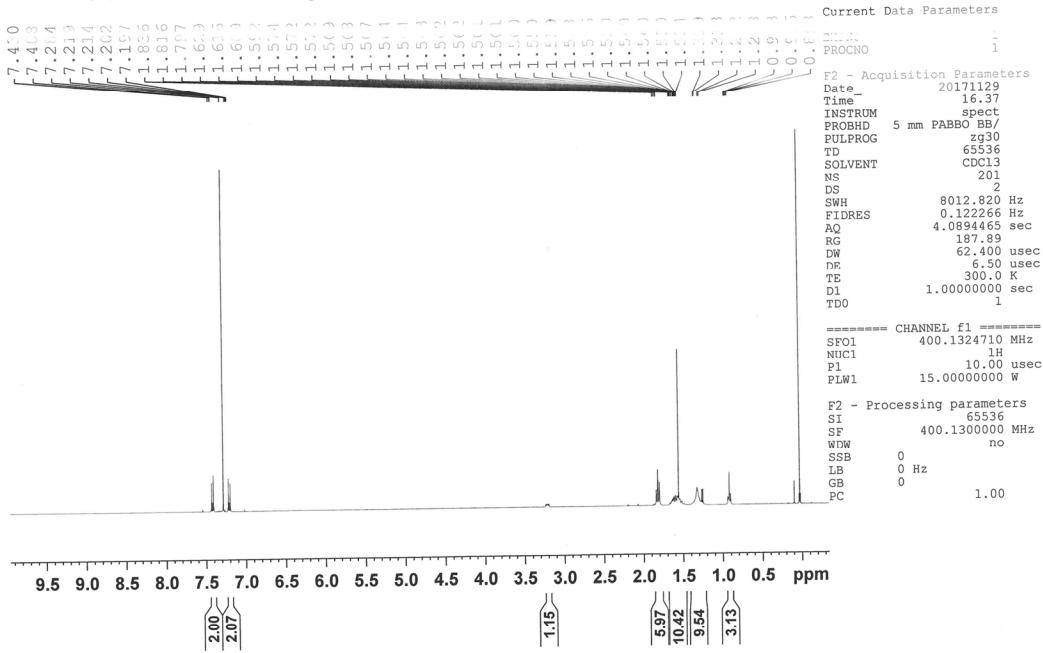
PCC reaction Product.



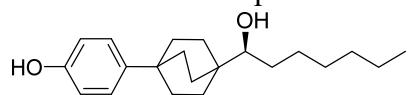
### <sup>1</sup>H NMR of Compound 25



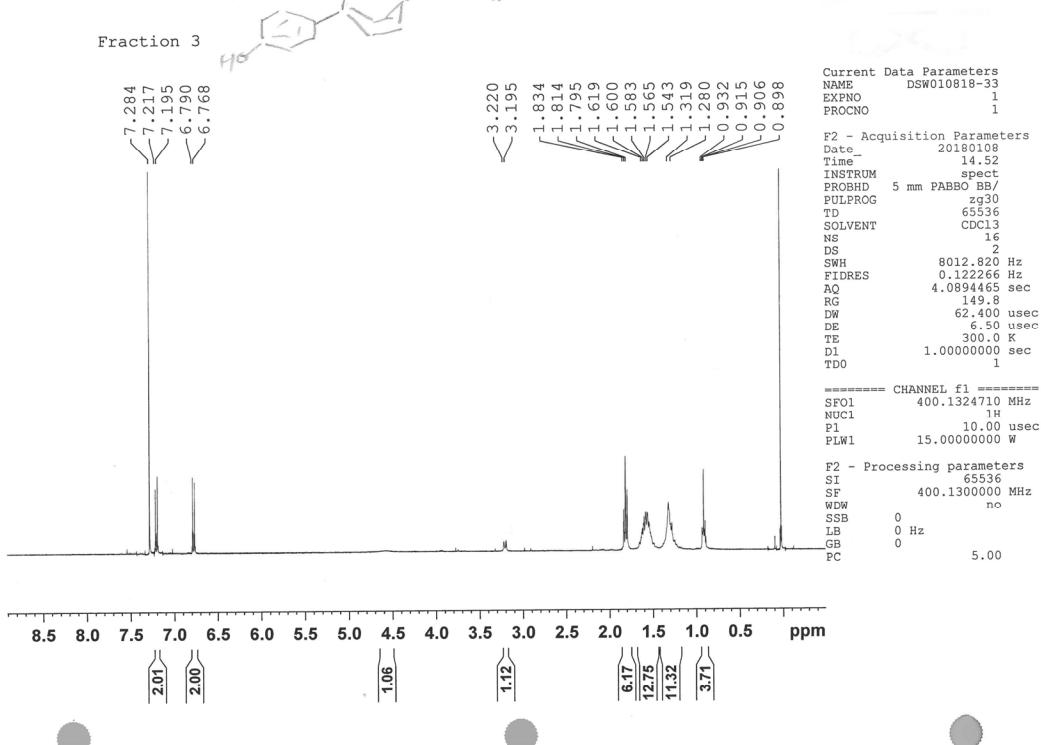
(R)-CBS rec

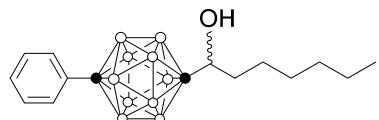


<sup>1</sup>H NMR of Compound 26

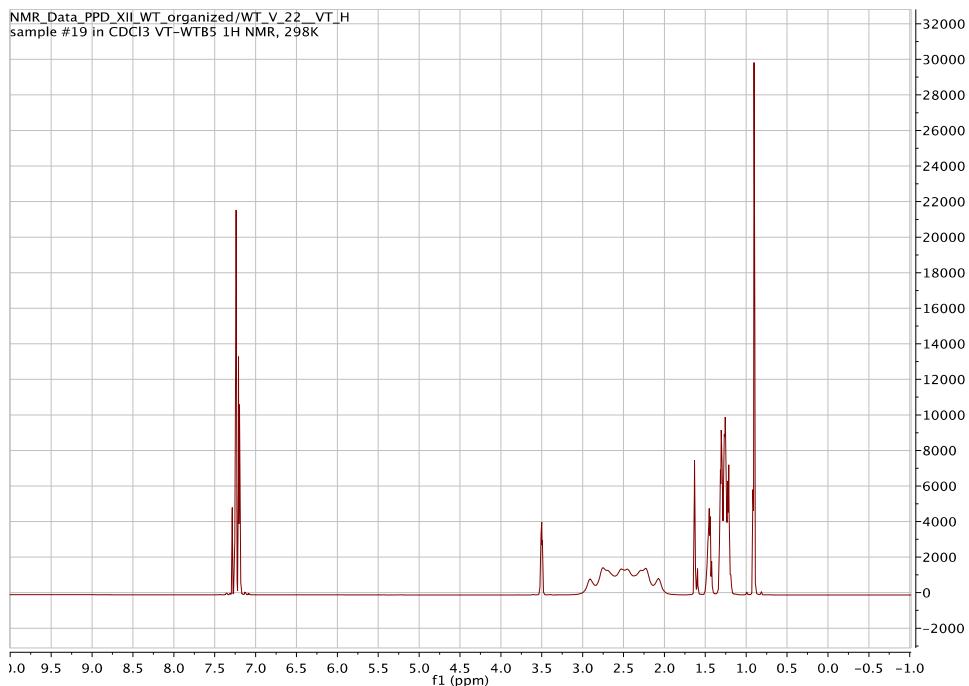


Fraction 3



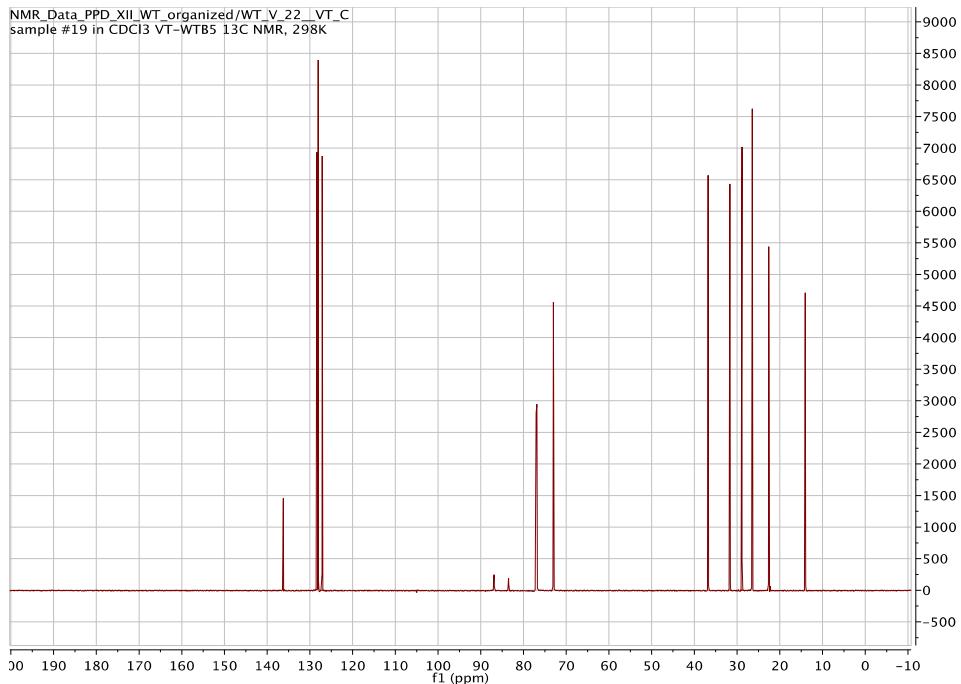


NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_22\_VT\_H  
sample #19 in CDCl<sub>3</sub> VT-WTB5 1H NMR, 298K



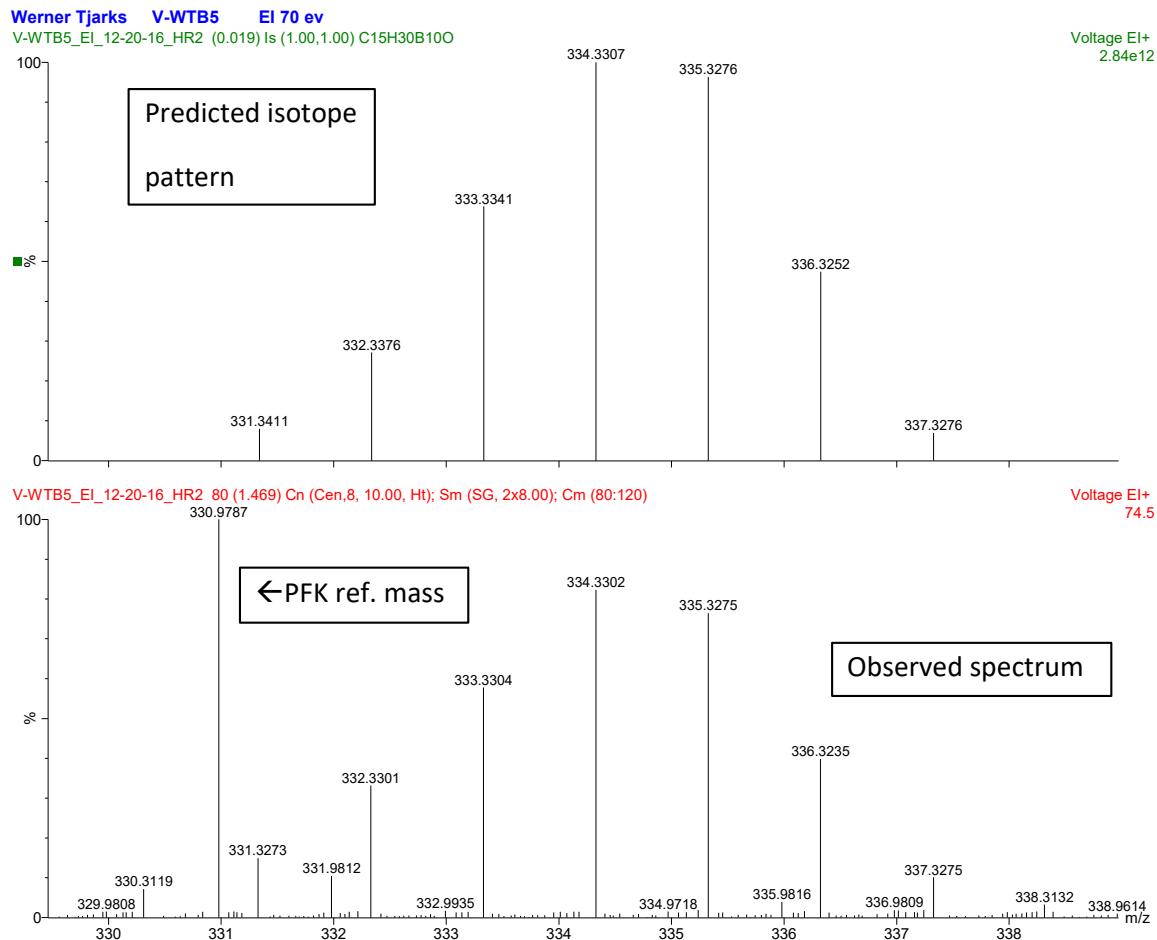
<sup>1</sup>H NMR of Compound 1-[1-(phenyl)-1,12-dicarba-closo-dodecaborane-12-yl]heptan-1-ol

NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_22\_VT\_C  
sample #19 in CDCl<sub>3</sub> VT-WTB5 <sup>13</sup>C NMR, 298K

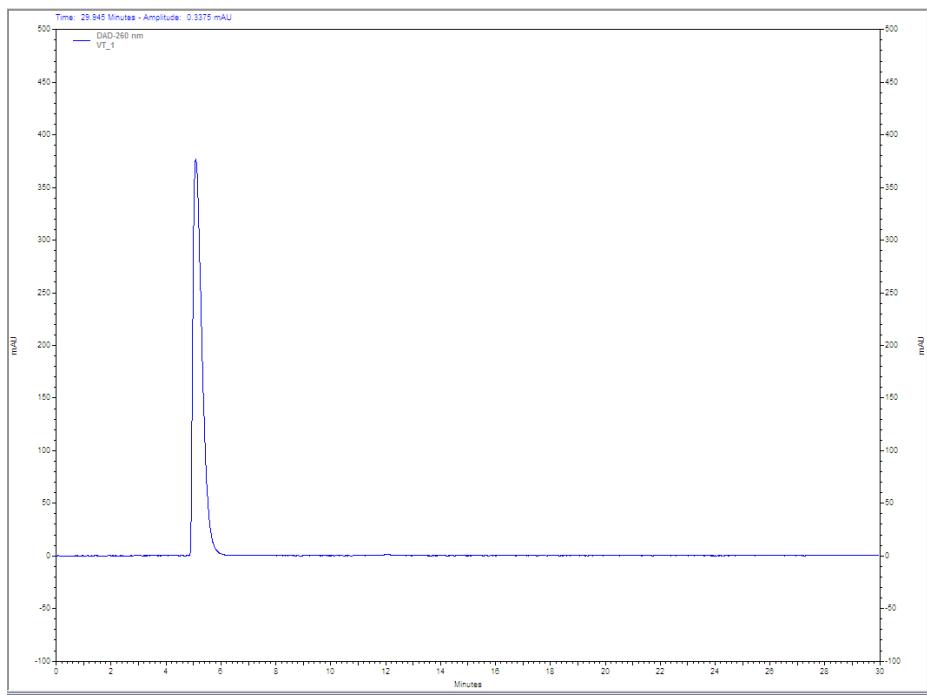


<sup>13</sup>CNMR of Compound 1-[1-(phenyl)-1,12-dicarba-closo-dodecaborane-12-yl]heptan-1-ol

Sample V-WTB5, by Electron Impact Ionization:



MS of Compound 1-[1-(phenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol

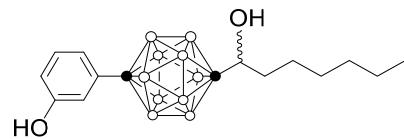


### DAD-260 nm

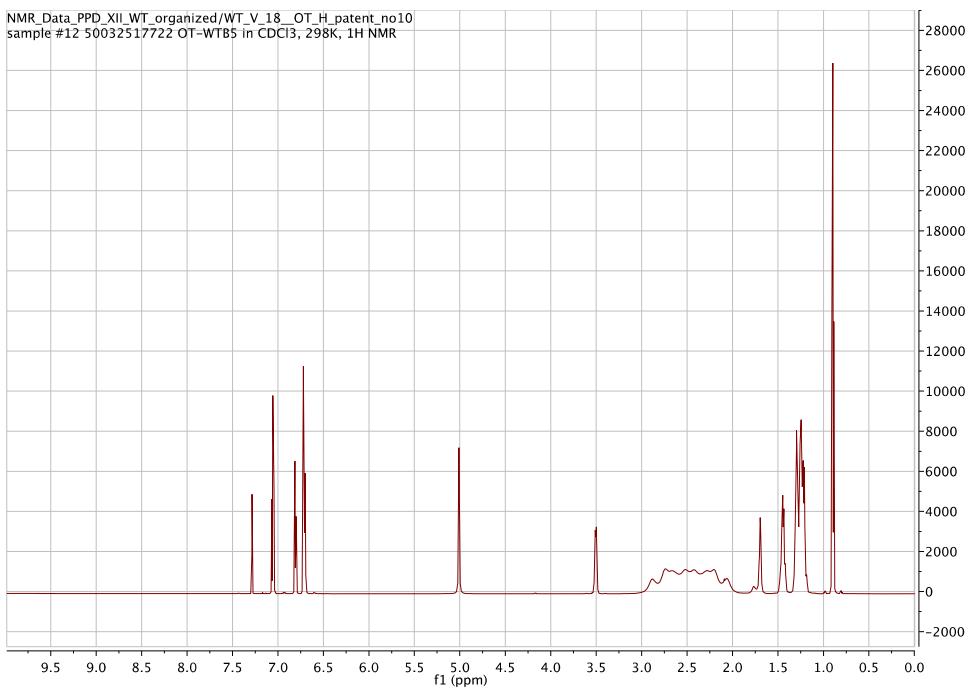
#### Results

| Retention Time | Area     | Area % | Height  | Height % |
|----------------|----------|--------|---------|----------|
| 5.087          | 34881795 | 99.61  | 1508766 | 99.71    |
| 12.087         | 136958   | 0.39   | 4456    | 0.29     |
| Totals         | 35018753 | 100.00 | 1513222 | 100.00   |

HPLC of Compound 1-[1-(phenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol

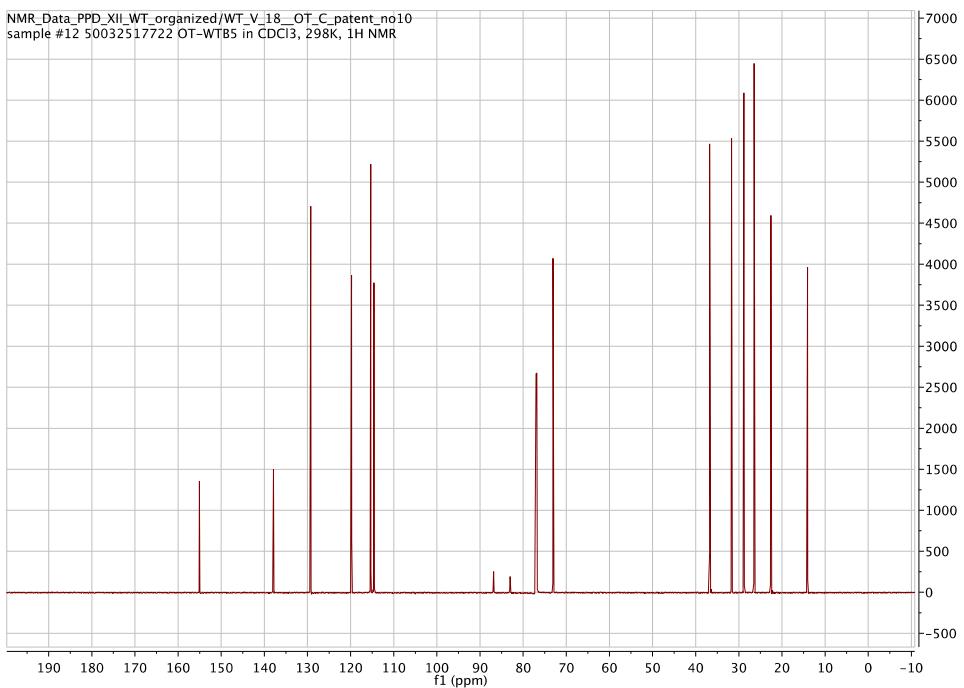


NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_18\_OT\_H\_patent\_no10  
sample #12 50032517722 OT-WTB5 in CDCl<sub>3</sub>, 298K, 1H NMR



<sup>1</sup>HNMR of Compound 1-[1-(3-hydroxyphenyl)-1,12-dicarba-closo-dodecaborane-12-yl]heptan-1-ol

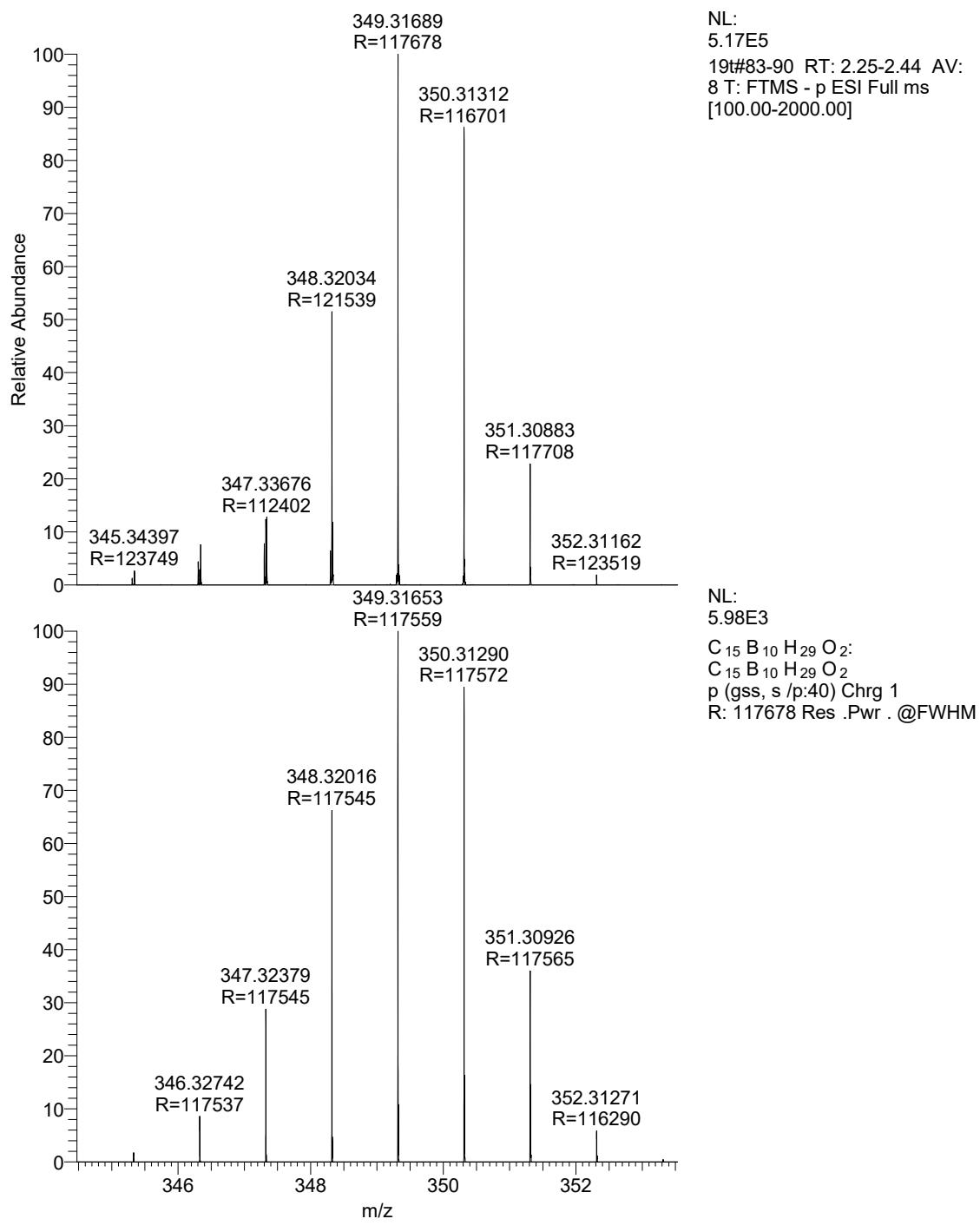
NMR\_Data\_PPD\_XII\_WT\_organized/WT\_V\_18\_OT\_C\_patent\_no10  
sample #12 50032517722 OT-WTB5 in CDCl<sub>3</sub>, 298K, 1H NMR

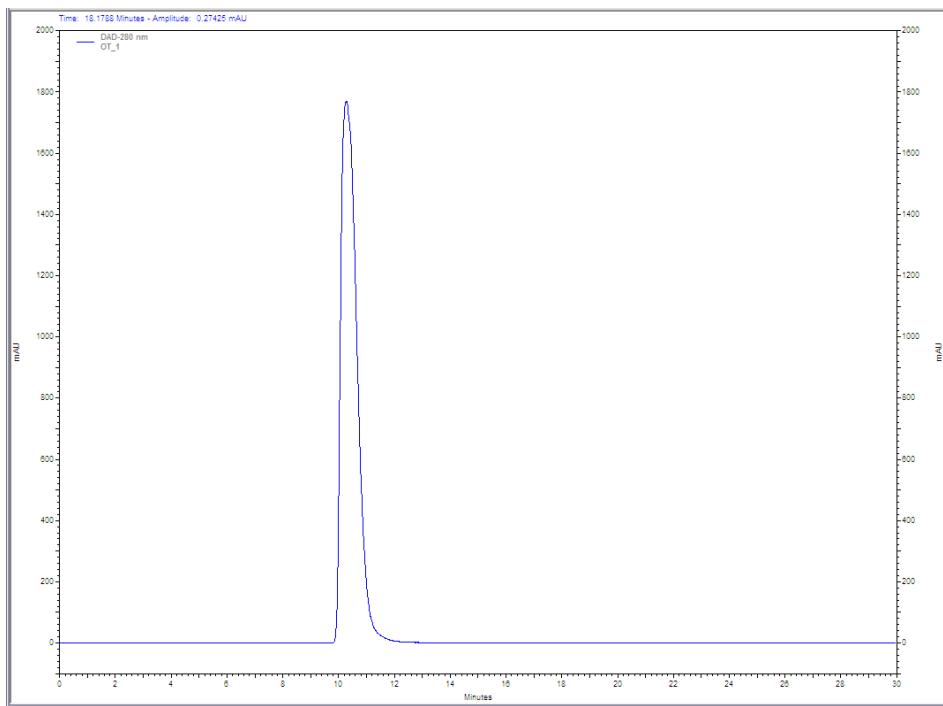


<sup>13</sup>CNMR of Compound 1-[1-(3-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol

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### DAD-280 nm

#### Results

| Retention Time | Area      | Area % | Height  | Height % |
|----------------|-----------|--------|---------|----------|
| 10.267         | 286539401 | 100.00 | 7080067 | 100.00   |
| Totals         | 286539401 | 100.00 | 7080067 | 100.00   |

HPLC of Compound 1-[1-(3-hydroxyphenyl)-1,12-dicarba-*clos*o-dodecaborane-12-yl]heptan-1-ol