

## Supplementary Material

### Practical method for the synthesis of $\beta$ -[(trimethylsilyl)ethynyl]- $\lambda^3$ -iodane

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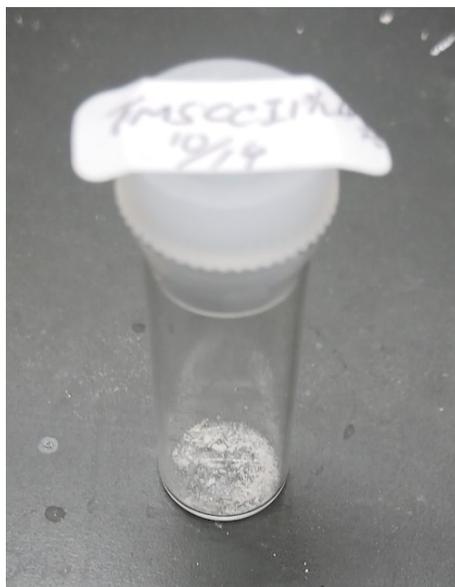
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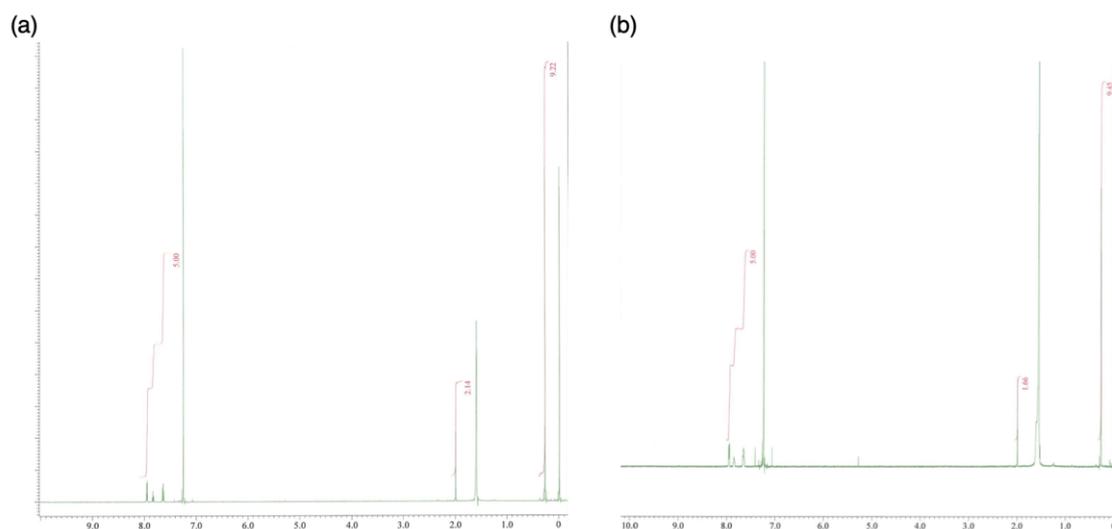
#### Table of Contents

1.	Stability study of the <b>3a</b> -B(C <sub>6</sub> F <sub>5</sub> ) <sub>4</sub> ·MeCN complex on the benchtop	S2
2.	Spectra	S3

1. Stability study of the **3a**-B(C<sub>6</sub>F<sub>5</sub>)<sub>4</sub>·MeCN complex on the benchtop

**Figure S1.** Appearance of **3a**-B(C<sub>6</sub>F<sub>5</sub>)<sub>4</sub>·MeCN after standing on the benchtop for two weeks.

No detectable color change was observed, while melting point slightly increased (mp. 104–109 °C, day 0; mp. 120–128 °C, day 14).



**Figure S2.** <sup>1</sup>H NMR spectra of **3a**-B(C<sub>6</sub>F<sub>5</sub>)<sub>4</sub>·MeCN (a) after 2 weeks and (b) after 1 month.

A slight decrease in the MeCN peak intensity was observed during storage, while no decomposition of the I(III) unit was detected.

## 2. Spectra

