

## Electronic Supplementary Information

### Synthesis of Diastereometrically Pure Cubane-Like Photodimers from 2,4-Pentanediyyl-bis-2-Naphthoates

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#### Table of Contents

<b>Figure S1:</b> $^1\text{H}$ NMR spectrum of <b>1R</b> in $\text{CDCl}_3$	S2
<b>Figure S2:</b> $^{13}\text{C}$ NMR spectrum of <b>1R</b> in $\text{CDCl}_3$	S2
<b>Figure S3:</b> EI-MS spectrum of <b>1R</b>	S3
<b>Figure S4:</b> $^1\text{H}$ NMR spectrum of <b>1S</b> in $\text{CDCl}_3$	S3
<b>Figure S5:</b> $^{13}\text{C}$ NMR spectrum of <b>1S</b> in $\text{CDCl}_3$	S4
<b>Figure S6:</b> EI-MS spectrum of <b>1S</b>	S4
<b>Figure S7:</b> $^1\text{H}$ NMR spectrum of <b>1M</b> in $\text{CDCl}_3$	S5
<b>Figure S8:</b> $^{13}\text{C}$ NMR spectrum of <b>1M</b> in $\text{CDCl}_3$	S5
<b>Figure S9:</b> EI-MS spectrum of <b>1M</b>	S6
<b>Figure S10:</b> $^1\text{H}$ NMR spectrum of <i>anti</i> <sup>HH</sup> - <b>2-A</b> in $\text{CDCl}_3$	S6
<b>Figure S11:</b> $^{13}\text{C}$ NMR spectrum of <i>anti</i> <sup>HH</sup> - <b>2-A</b> in $\text{CDCl}_3$	S7
<b>Figure S12:</b> MALDI-TOF spectrum of <i>anti</i> <sup>HH</sup> - <b>2-A</b>	S7
<b>Figure S13:</b> $^1\text{H}$ NMR spectrum of <i>anti</i> <sup>HH</sup> - <b>2-B</b> in $\text{CDCl}_3$	S8
<b>Figure S14:</b> $^{13}\text{C}$ NMR spectrum of <i>anti</i> <sup>HH</sup> - <b>2-B</b> in $\text{CDCl}_3$	S8
<b>Figure S15:</b> MALDI-TOF spectrum of <i>anti</i> <sup>HH</sup> - <b>2-B</b>	S9
<b>Figure S16:</b> $^1\text{H}$ NMR spectrum of <i>syn</i> <sup>HH</sup> - <b>2</b> in $\text{CDCl}_3$	S9
<b>Figure S17:</b> $^{13}\text{C}$ NMR spectrum of <i>syn</i> <sup>HH</sup> - <b>2</b> in $\text{CDCl}_3$	S10
<b>Figure S18:</b> MALDI-TOF spectrum of <i>syn</i> <sup>HH</sup> - <b>2</b>	S10
<b>Figure S19:</b> $^1\text{H}$ NMR spectrum of <i>anti</i> <sup>HH</sup> - <b>3A</b> in $\text{CDCl}_3$	S11
<b>Figure S20:</b> $^{13}\text{C}$ NMR spectrum of <i>anti</i> <sup>HH</sup> - <b>3A</b> in $\text{DMSO-}d_6$	S11
<b>Figure S21:</b> ESI-MS spectrum of <i>anti</i> <sup>HH</sup> - <b>3A</b>	S12
<b>Figure S22:</b> $^1\text{H}$ NMR spectrum of <i>syn</i> <sup>HH</sup> - <b>3</b> in $\text{CDCl}_3$	S12
<b>Figure S23:</b> $^{13}\text{C}$ NMR spectrum of <i>syn</i> <sup>HH</sup> - <b>3</b> in $\text{DMSO-}d_6$	S13
<b>Figure S24:</b> ESI-MS spectrum of <i>syn</i> <sup>HH</sup> - <b>3</b>	S13
<b>Figure S25:</b> Perspective drawing of <i>anti</i> <sup>HH</sup> - <b>2-A</b> with atomic numbering	S14
<b>Table S1:</b> Important bond distances and angles for <i>anti</i> <sup>HH</sup> - <b>2-A</b>	S14
<b>Figure S26:</b> Perspective drawing of <i>syn</i> <sup>HH</sup> - <b>2</b> with atomic numbering	S15
<b>Table S2:</b> Important bond distances and angles for <i>syn</i> <sup>HH</sup> - <b>2</b>	S15

S2

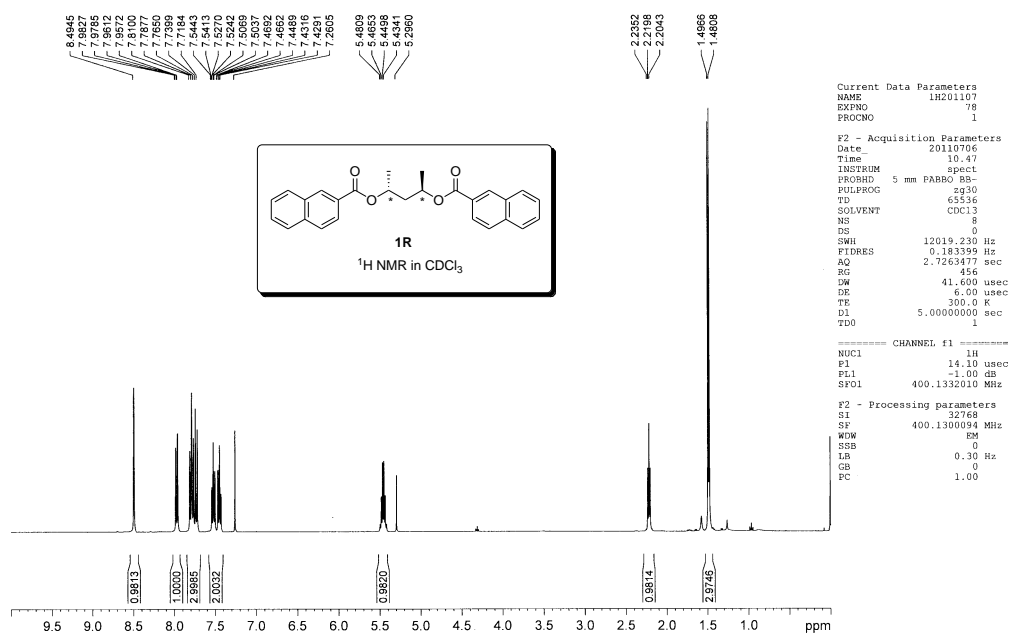


Figure S1. <sup>1</sup>H NMR spectrum of **1R** in CDCl<sub>3</sub>

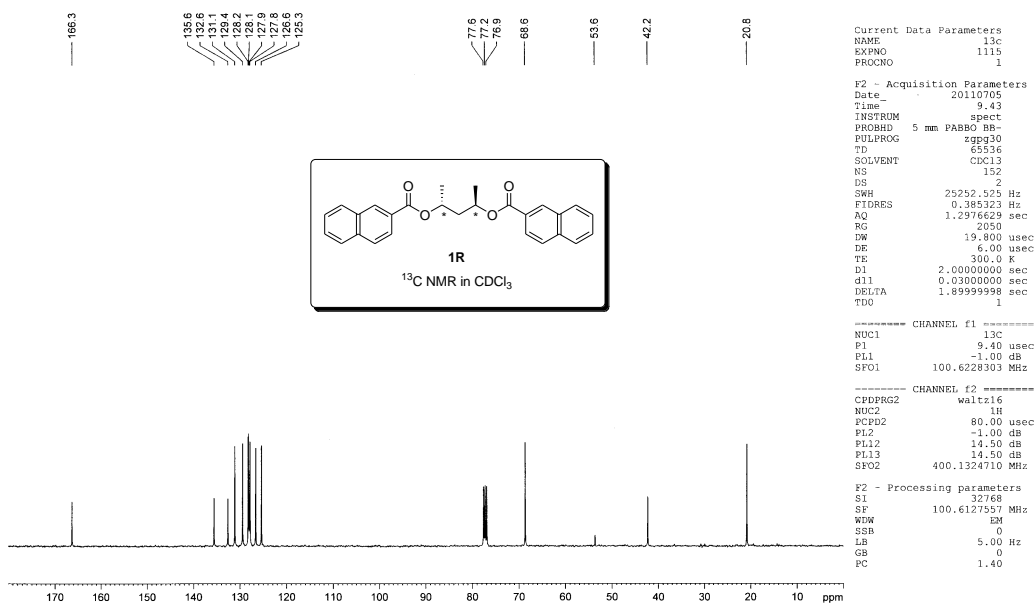


Figure S2. <sup>13</sup>C NMR spectrum of **1R** in CDCl<sub>3</sub>

S3

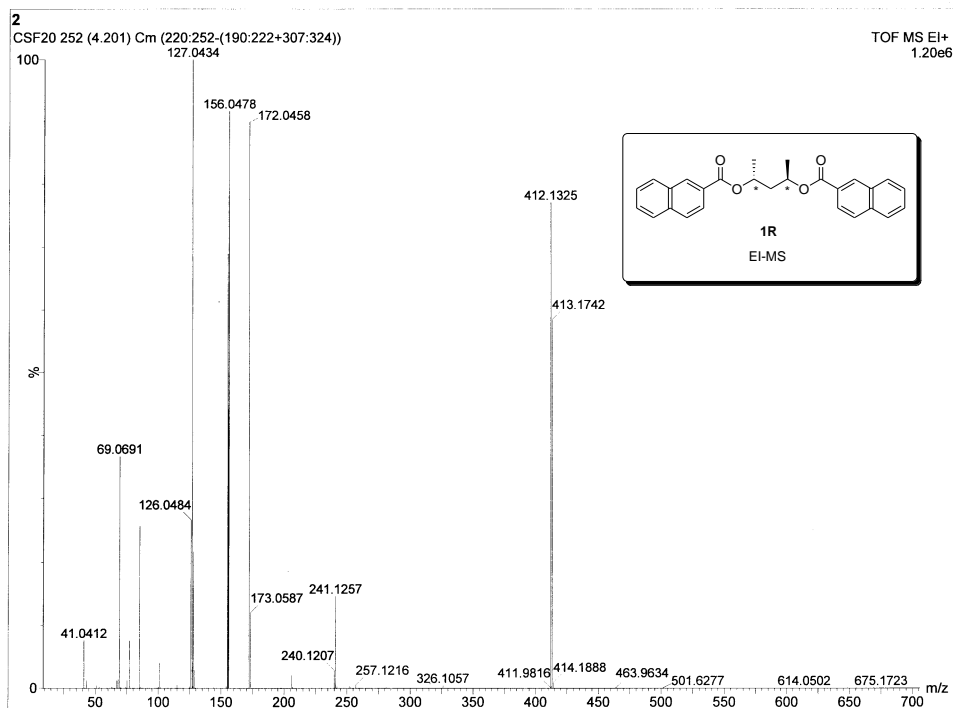


Figure S3. EI-MS spectrum of **1R**

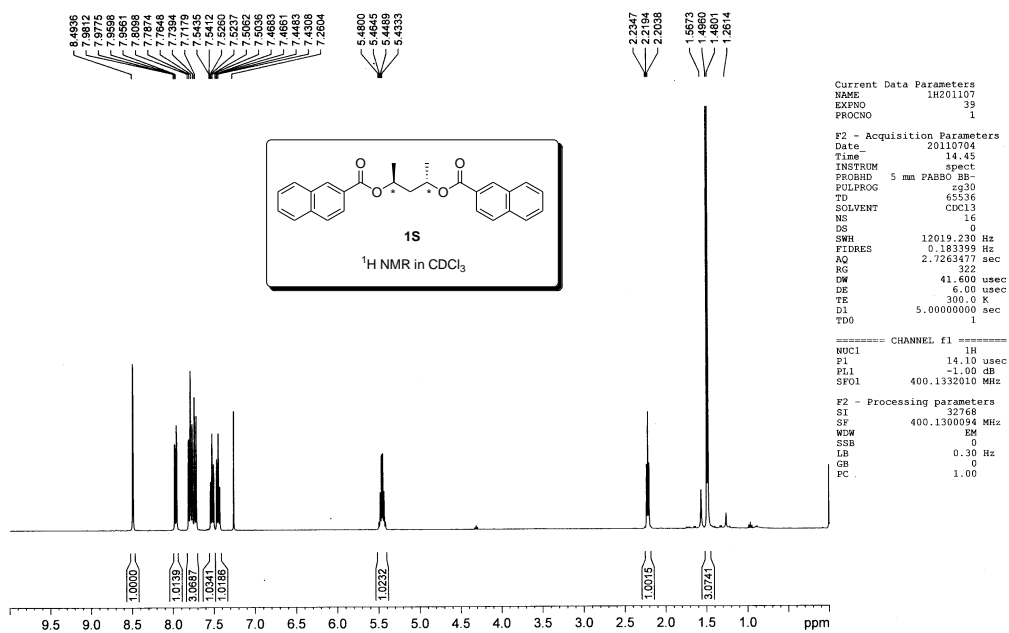


Figure S4. <sup>1</sup>H NMR spectrum of **1S** in CDCl<sub>3</sub>

S4

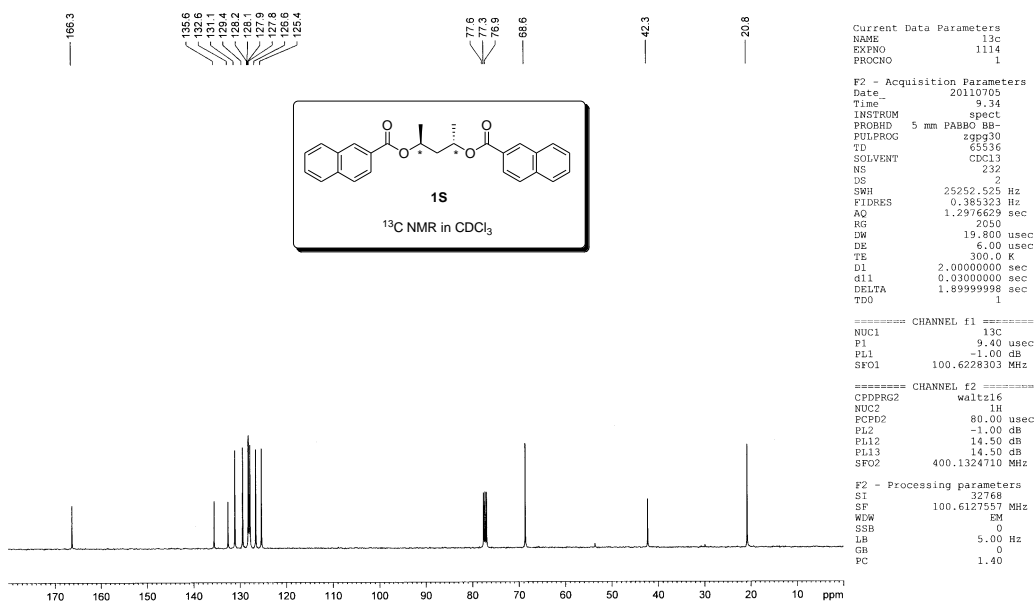


Figure S5.  $^{13}\text{C}$  NMR spectrum of **1S** in  $\text{CDCl}_3$

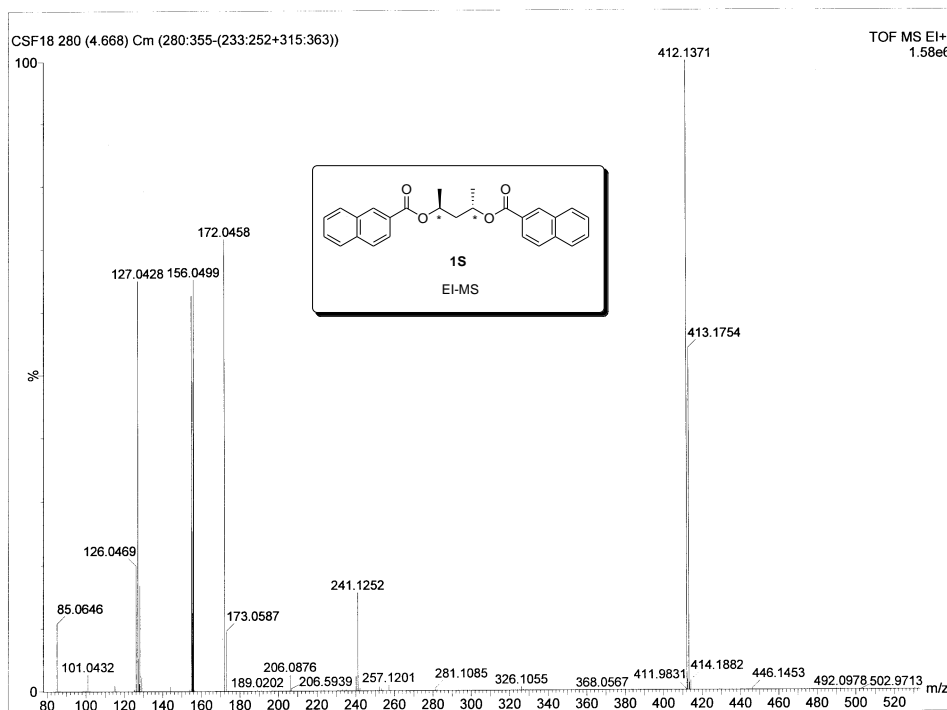


Figure S6. EI-MS spectrum of **1S**

S5

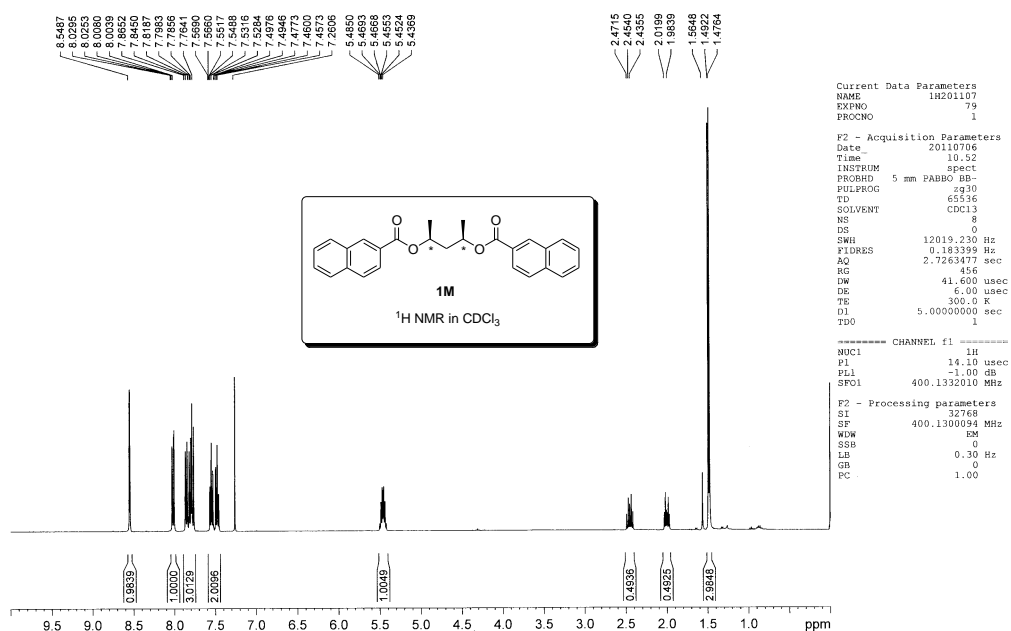


Figure S7. <sup>1</sup>H NMR spectrum of **1M** in CDCl<sub>3</sub>

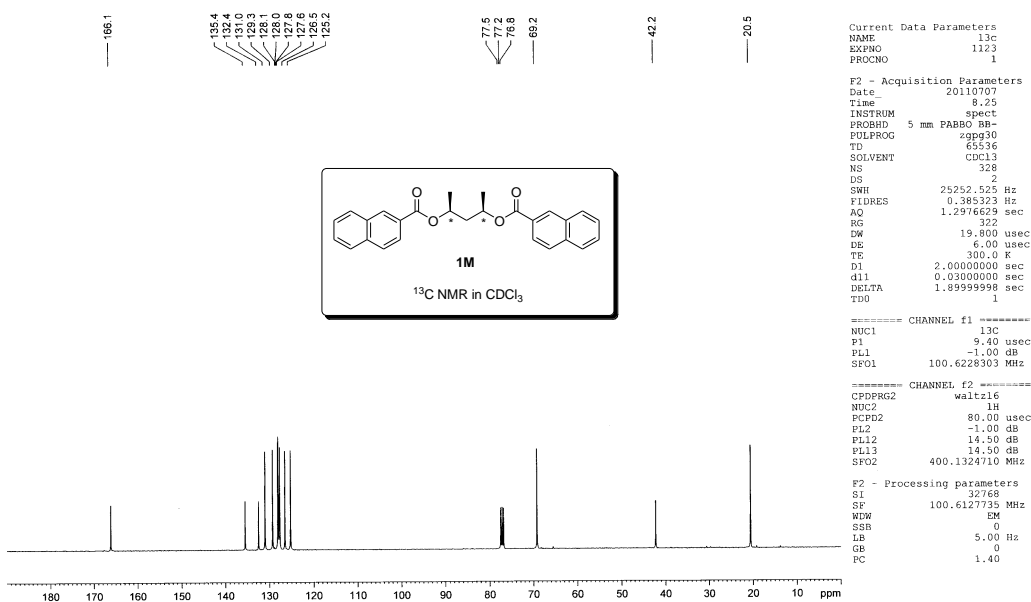


Figure S8. <sup>13</sup>C NMR spectrum of **1M** in CDCl<sub>3</sub>

S6

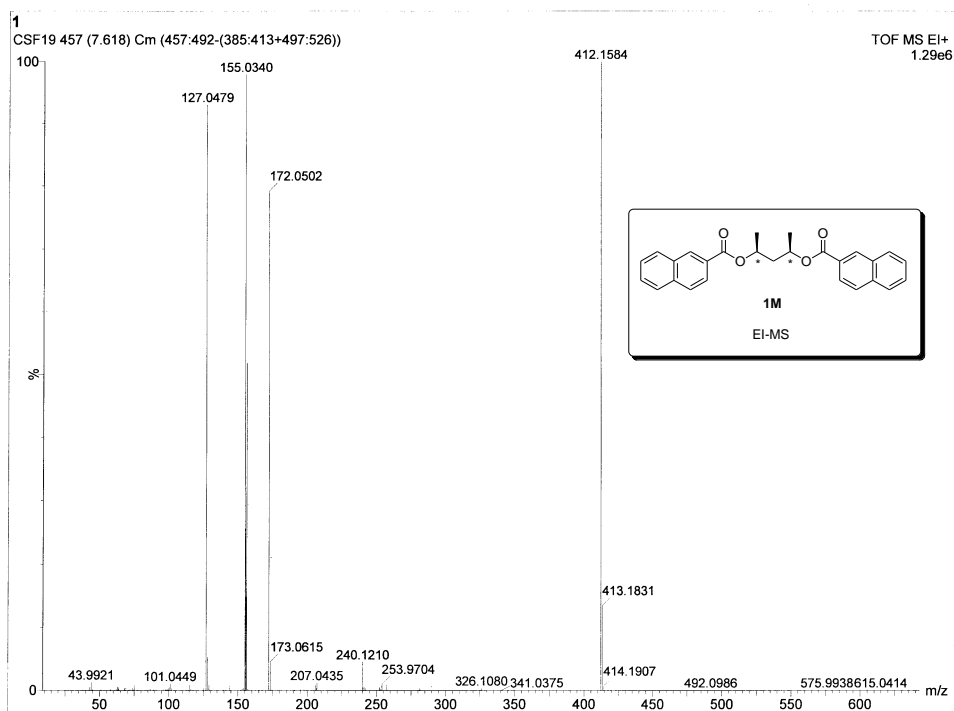


Figure S9. EI-MS spectrum of **1M**

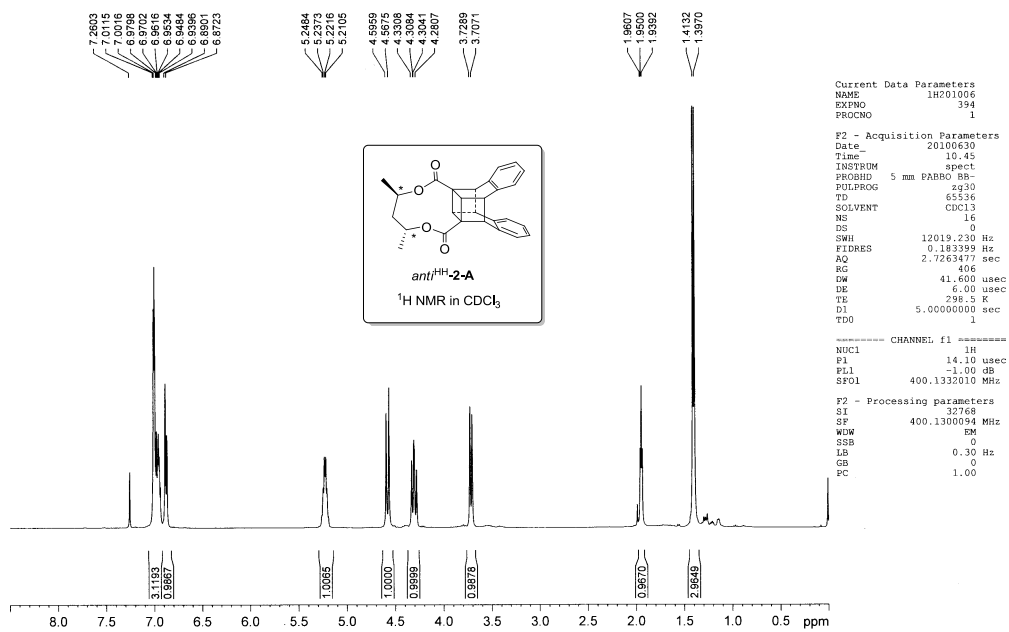


Figure S10.  $^1\text{H}$  NMR spectrum of *anti*<sup>HH</sup>-**2-A** in  $\text{CDCl}_3$

S7

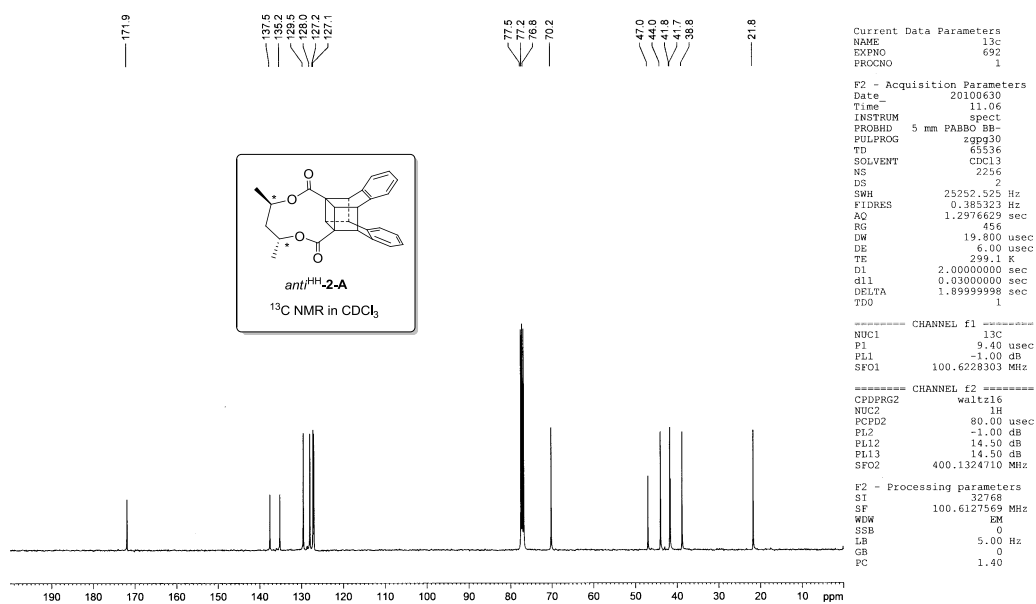


Figure S11.  $^{13}\text{C}$  NMR spectrum of *anti*<sup>HH</sup>-2-A in  $\text{CDCl}_3$

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MALDI-TOF, CCA, 1, 2011, 08, 25

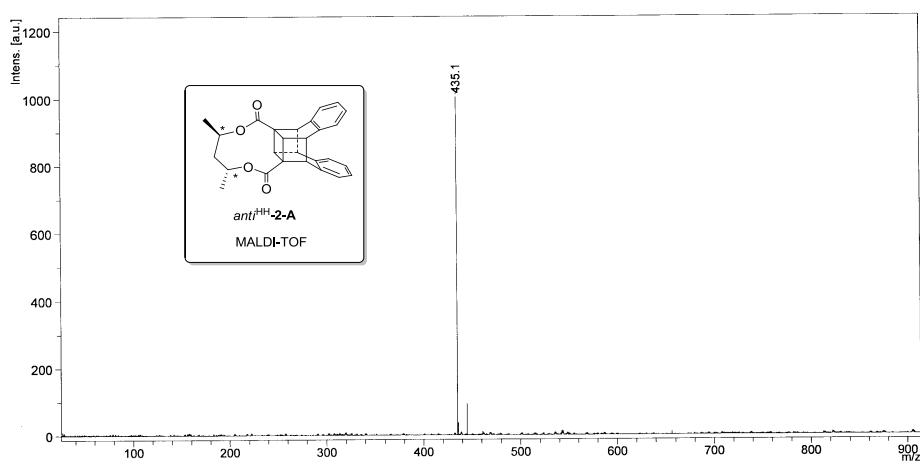


Figure S12. MALDI-TOF spectrum of *anti*<sup>HH</sup>-2-A

S8

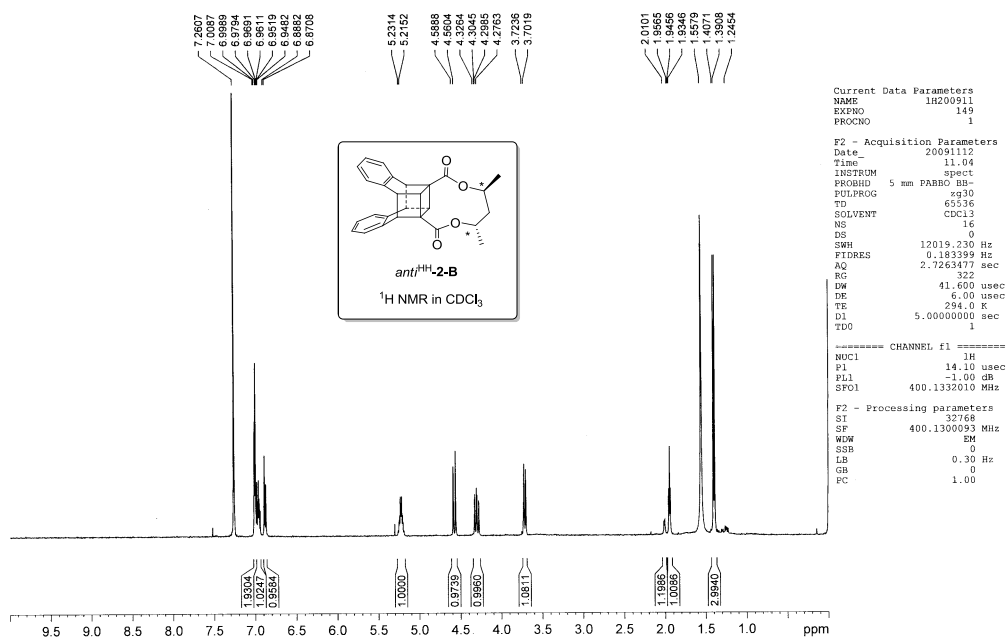


Figure S13. <sup>1</sup>H NMR spectrum of *anti*<sup>HH</sup>-2-B in CDCl<sub>3</sub>

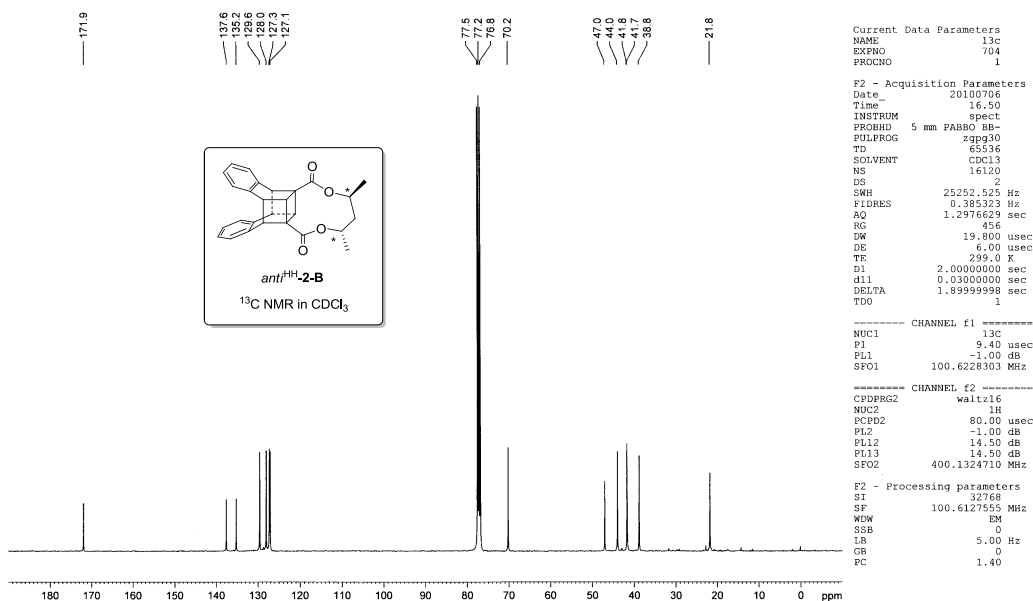


Figure S14. <sup>13</sup>C NMR spectrum of *anti*<sup>HH</sup>-2-B in CDCl<sub>3</sub>



S9

D:\Data\_IC\2011\11-08\201110830\201110830260\_C3V1

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### MALDI-TOF, CCA, 2, 2011, 08, 30

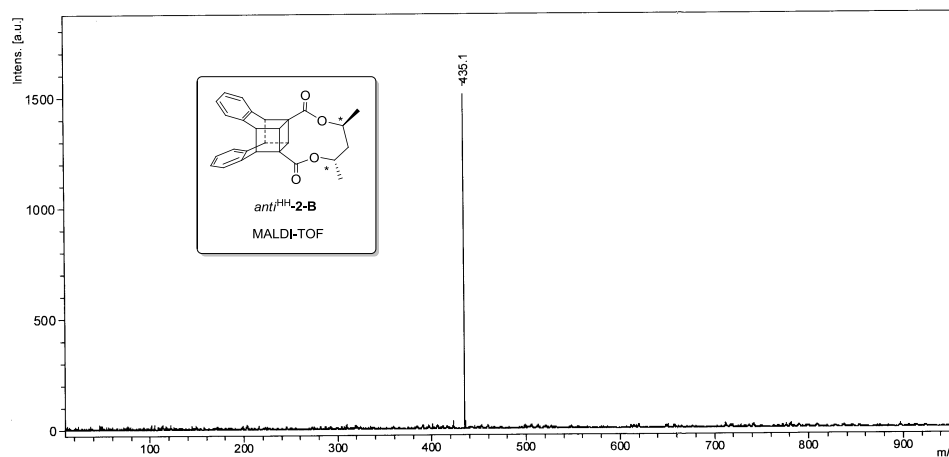


Figure S15. MALDI-TOF spectrum of anti<sup>HH</sup>-2-B

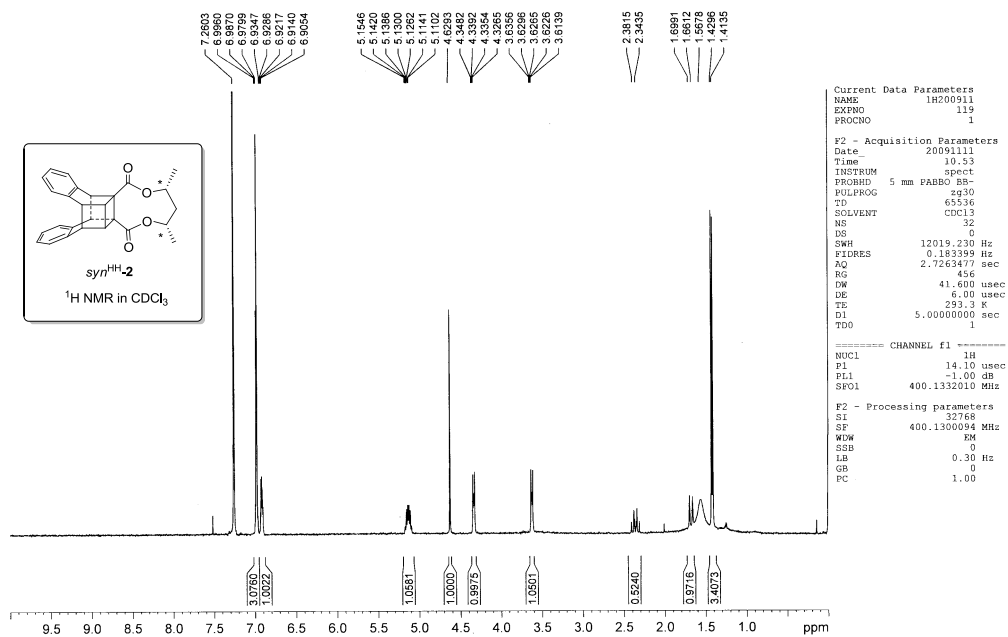


Figure S16. <sup>1</sup>H NMR spectrum of syn<sup>HH</sup>-2 in CDCl<sub>3</sub>

S10

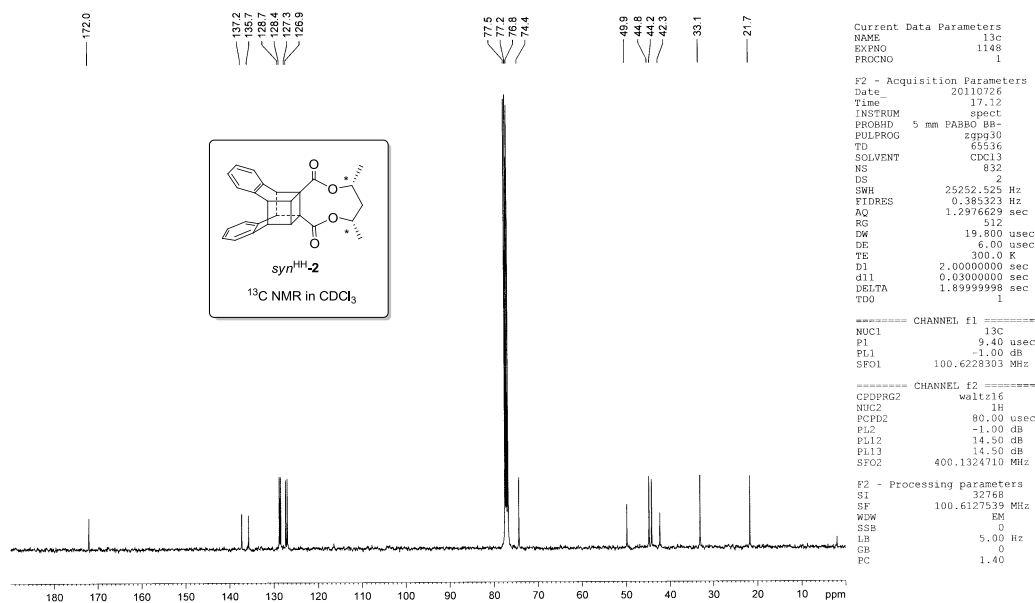


Figure S17. <sup>13</sup>C NMR spectrum of *syn*<sup>HH</sup>-2 in CDCl<sub>3</sub>

D:\Data\_IC\2011\11-08\20110826\20110826140\_K18\1

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MALDI-TOF, CCA, 3, 2011, 08, 25

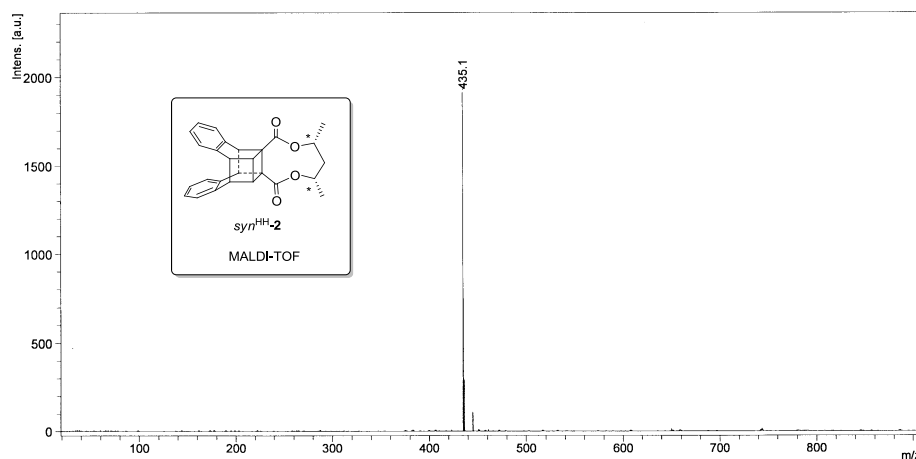


Figure S18. MALDI-TOF spectrum of *syn*<sup>HH</sup>-2

S11

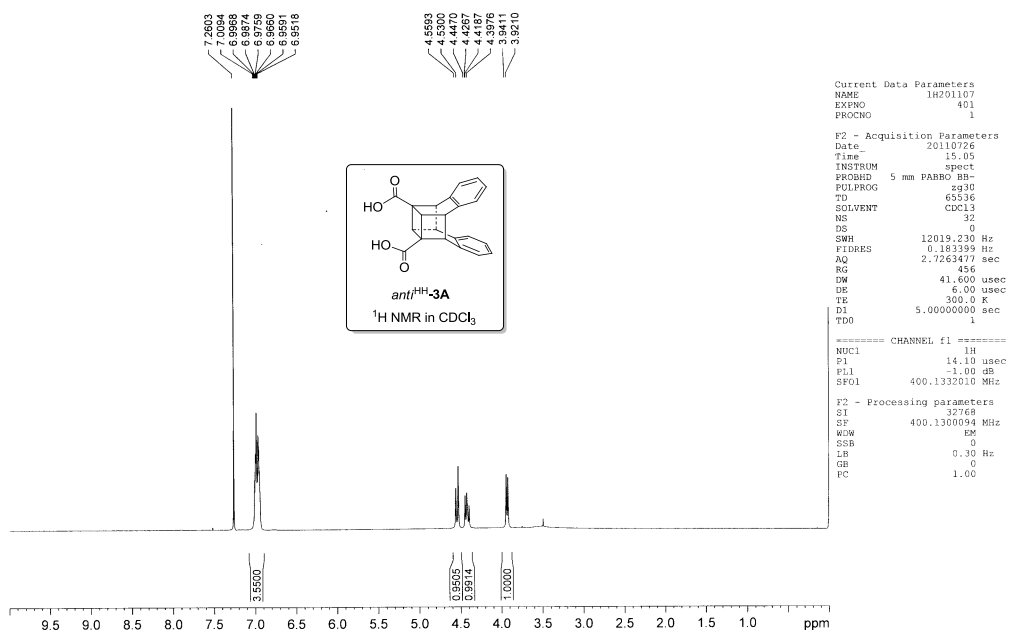


Figure S19. <sup>1</sup>H NMR spectrum of *anti*<sup>HH</sup>-3A in CDCl<sub>3</sub>

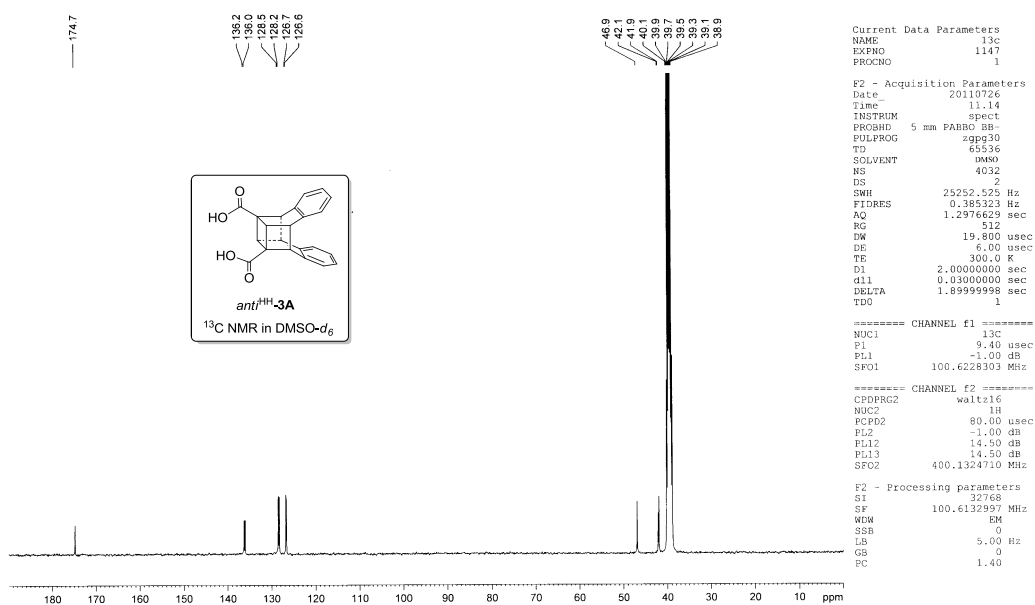


Figure S20. <sup>13</sup>C NMR spectrum of *anti*<sup>HH</sup>-3A in DMSO-*d*<sub>6</sub>

## S12

### ESI-MS Spectrum, 4

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Intensity

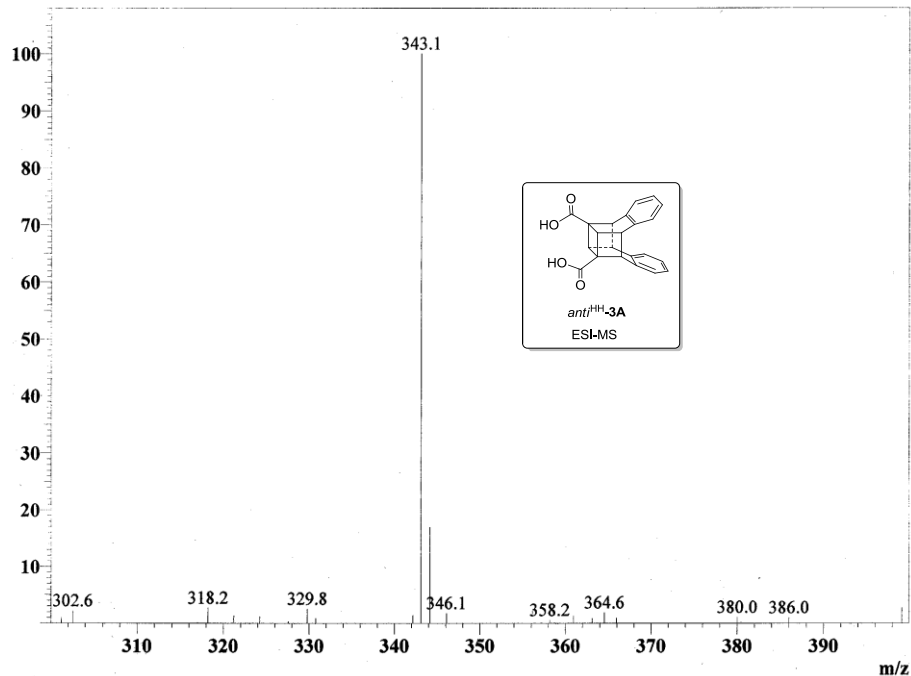


Figure S21. ESI-MS spectrum of anti<sup>HH</sup>-3A

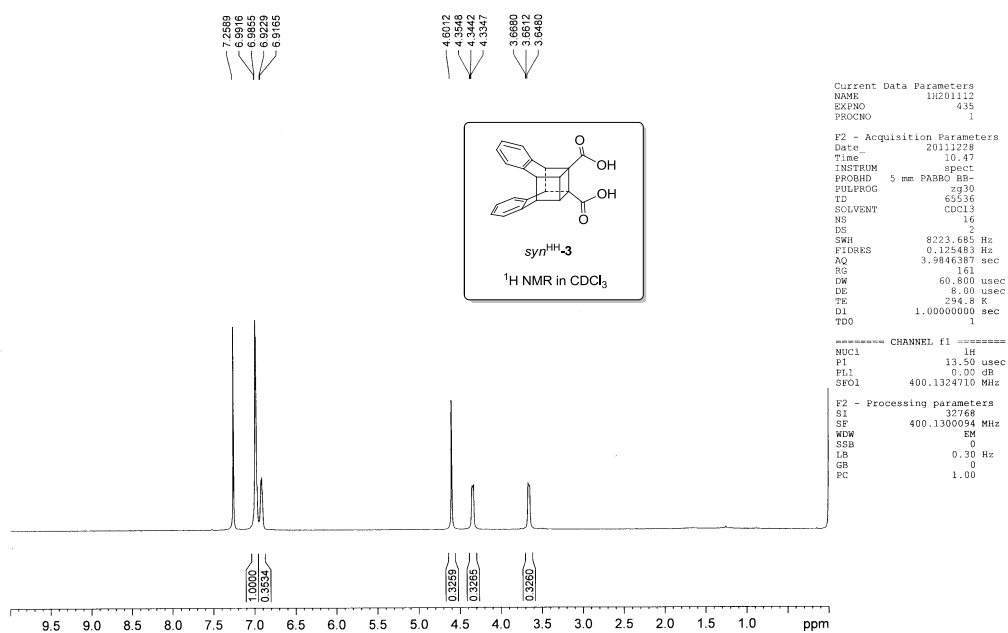


Figure S22. <sup>1</sup>H NMR spectrum of syn<sup>HH</sup>-3 in CDCl<sub>3</sub>

S13

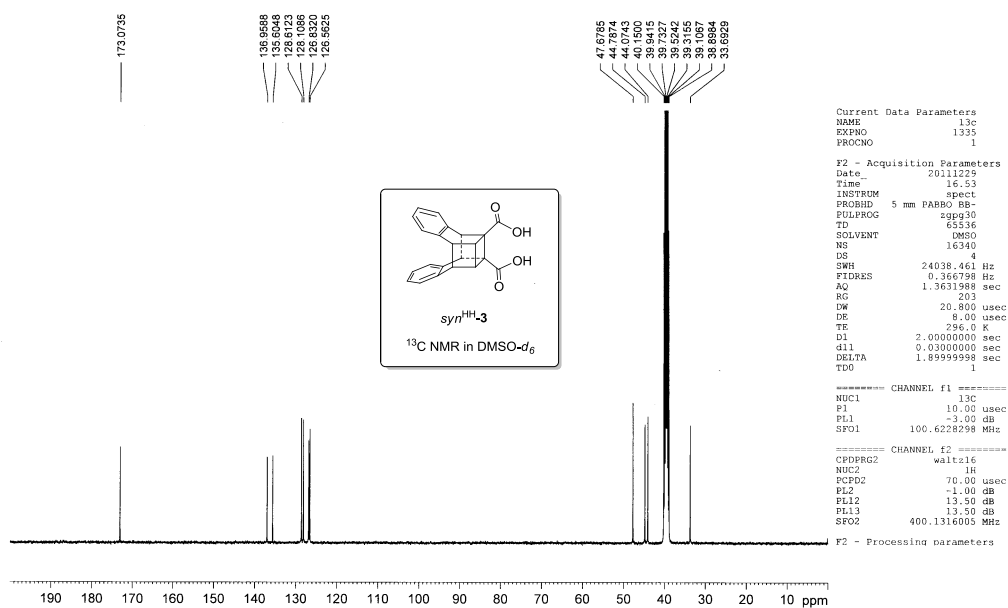


Figure S23:  $^{13}\text{C}$  NMR spectrum of *syn*<sup>HH</sup>-3 in  $\text{DMSO-}d_6$

ESI-MS Spectrum, 1

#:1 Ret.Time:Single 2.817(Scan#:170)

Mass Peaks:420 Base Peak:343.10(21301) Polarity:Neg Segment1 - Event1

Intensity

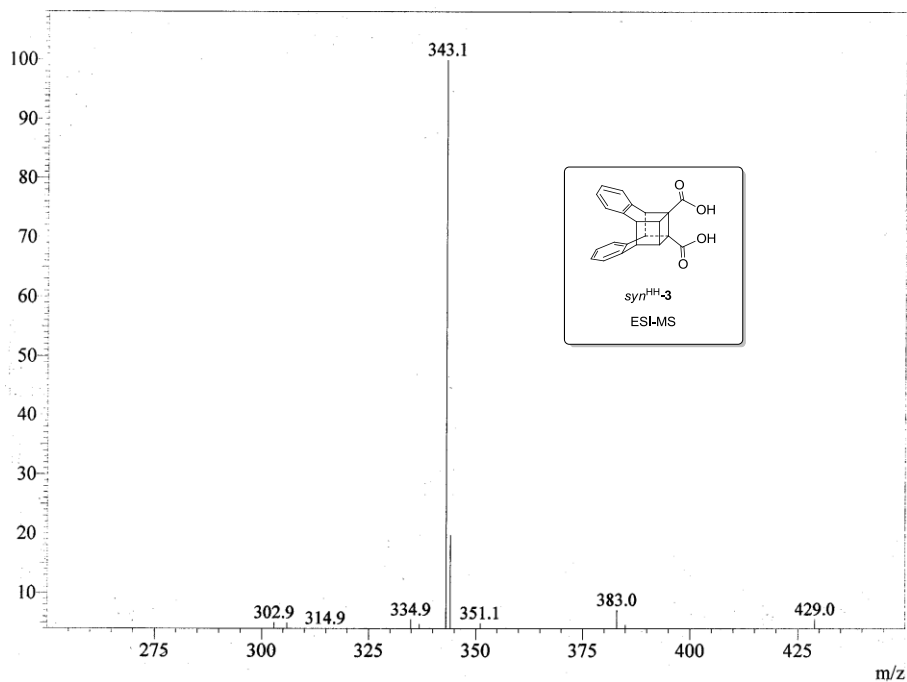
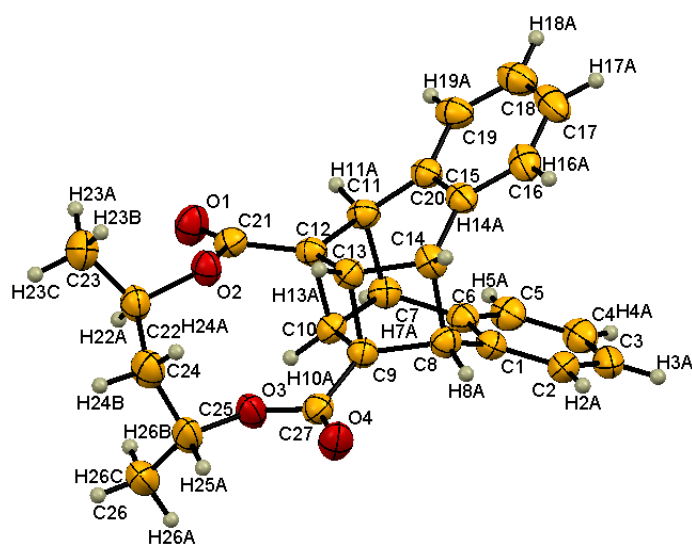


Figure S24. ESI-MS spectrum of *syn*<sup>HH</sup>-3

S14

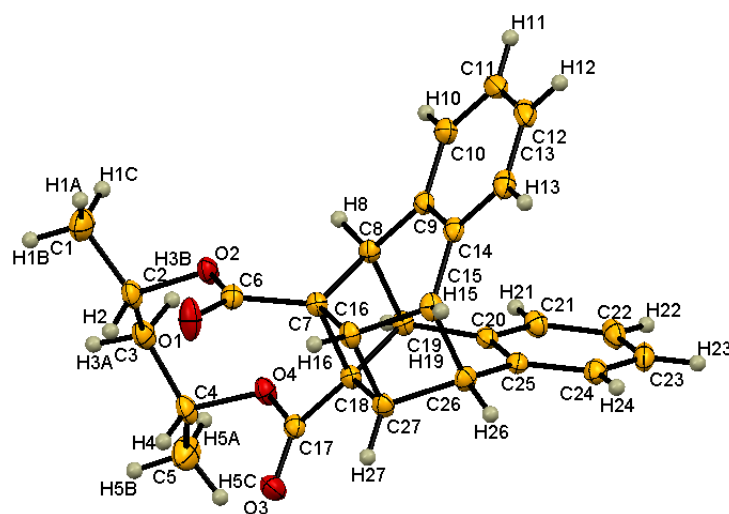


**Figure S25.** Perspective drawing of *anti*<sup>HH</sup>-2-A with atomic numbering

**Table S1.** Important bond distances and angles for *anti*<sup>HH</sup>-2-A

<i>Bond lengths</i> (Å)		<i>Bond angles</i> (deg.)		<i>Dihedral angles</i> (deg.)	
C(9)-C(10)	1.553(3)	C(10)-C(9)-C(13)	89.50(15)	C(27)-C(9)-C(10)-C(7)	-159.73(19)
C(9)-C(13)	1.584(3)	C(9)-C(10)-C(12)	90.12(16)	C(27)-C(9)-C(10)-C(12)	109.23(19)
C(10)-C(12)	1.580(3)	C(13)-C(12)-C(10)	89.84(16)	C(9)-C(10)-C(12)-C(13)	4.68(15)
C(12)-C(13)	1.548(3)	C(12)-C(13)-C(9)	90.16(16)	C(21)-C(12)-C(13)-C(9)	107.98(18)
C(7)-C(10)	1.542(3)	C(21)-C(12)-C(13)	116.08(18)	C(21)-C(12)-C(13)-C(14)	-161.78(19)
C(8)-C(9)	1.546(3)	C(21)-C(12)-C(10)	110.24(17)	C(10)-C(12)-C(13)-C(9)	-4.59(15)
C(11)-C(12)	1.560(3)	C(27)-C(9)-C(13)	111.68(18)	C(8)-C(9)-C(13)-C(14)	1.85(17)
C(13)-C(14)	1.535(3)	C(27)-C(9)-C(10)	115.00(18)	C(11)-C(12)-C(13)-C(14)	-4.0(3)

S15



**Figure S26.** Perspective drawing of  $\text{syn}^{\text{HH}}\text{-2}$  with atomic numbering

**Table S2.** Important bond distances and angles for  $\text{syn}^{\text{HH}}\text{-2}$

<i>Bond lengths (Å)</i>		<i>Bond angles (deg.)</i>		<i>Dihedral angles (deg.)</i>	
C(7)-C(16)	1.5400(15)	C(6)-C(7)-C(16)	115.64(9)	C(6)-C(7)-C(16)-C(27)	117.65(9)
C(7)-C(18)	1.6061(15)	C(6)-C(7)-C(18)	114.91(8)	C(6)-C(7)-C(16)-C(15)	-151.19(10)
C(16)-C(27)	1.5590(15)	C(17)-C(18)-C(27)	117.56(9)	C(17)-C(18)-C(27)-C(26)	149.68(9)
C(18)-C(27)	1.5463(15)	C(17)-C(18)-C(7)	116.38(8)	C(17)-C(18)-C(27)-C(16)	-119.28(9)
C(7)-C(8)	1.5553(15)	C(7)-C(16)-C(27)	90.89(8)	C(7)-C(18)-C(27)-C(16)	0.16(7)
C(15)-C(16)	1.5511(14)	C(27)-C(18)-C(7)	88.91(8)	C(7)-C(16)-C(27)-C(18)	-0.16(8)
C(18)-C(19)	1.5591(14)	C(18)-C(27)-C(16)	90.86(8)	C(15)-C(16)-C(27)-C(26)	-0.38(8)
C(26)-C(27)	1.5555(14)	C(16)-C(7)-C(18)	89.34(8)	C(8)-C(7)-C(16)-C(15)	1.73(13)