Supporting Information

Hypervalent iodine initiated intramolecular alkene dimerisation: a stereodivergent entry to cyclobutanes

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Compound 2l: 3l: $^1$H NMR and $^{13}$C NMR

Compound 2m: $^1$H NMR and $^{13}$C NMR

Compound 3m: $^1$H NMR and $^{13}$C NMR

Compounds 2n: 3n: $^1$H NMR and $^{13}$C NMR

Compound 5c: $^1$H NMR and $^{13}$C NMR

Compound 5d: $^1$H NMR and $^{13}$C NMR

Compound 5e: $^1$H NMR and $^{13}$C NMR

Compound 5g: $^1$H NMR and $^{13}$C NMR, COSY, HSQC and NOESY-2D

Compound 6a: $^1$H NMR and $^{13}$C NMR

Compound 6b: $^1$H NMR and $^{13}$C NMR

Compound 7a: $^1$H NMR and $^{13}$C NMR

Compound 8a: $^1$H NMR and $^{13}$C NMR, COSY, HSQC and NOESY-2D

Compound 8b: $^1$H NMR and $^{13}$C NMR

Compound 8c: $^1$H NMR and $^{13}$C NMR, COSY, HSQC and NOESY-2D

Compound 8d: $^1$H NMR and $^{13}$C NMR
$\text{^{1}H NMR (400 MHz, CDCl}_3$)
$^{13}\text{C} \text{NMR} \ (100 \text{ MHz}, \text{CDCl}_3)$

Me

S1c

$\begin{align*}
\text{Me} & \quad 4 \\
\text{OAc} & \quad 3 \\
\text{H} & \quad 2 \\
\text{H} & \quad 1
\end{align*}$
$^{1}$H NMR (400 MHz, CDCl$_3$)

1a

MeO

H NMR (400 MHz, CDCl$_3$)
$^{13}$C NMR (100 MHz, CDCl$_3$)

![Chemical structure with NMR spectrum](image)

- 158.86 ppm
- 131.18 ppm
- 130.64 ppm
- 129.21 ppm
- 124.39 ppm
- 114.02 ppm
- 77.48 ppm (CDCl$_3$)
- 77.16 ppm (CDCl$_3$)
- 76.84 ppm (CDCl$_3$)
- 62.95 ppm
- 55.40 ppm
- 38.74 ppm
- 17.53 ppm
- 12.28 ppm
$^1$H NMR (400 MHz, CDCl$_3$)
$^{13}$C NMR (100 MHz, CDCl$_3$)

$^{13}$C NMR (100 MHz, CDCl$_3$)
$^1$H NMR (400 MHz, CDCl$_3$)
$^{13}$C NMR (100 MHz, CDCl$_3$)
$^1$H NMR (400 MHz, CDCl$_3$)

MeO-\(\text{C}_6\text{H}_4\)\(\text{O-Si-O}\)\(\text{C}_6\text{H}_4\)-OMe

1j
$^13$C NMR (100 MHz, CDCl$_3$)
$^{1}$H NMR (400 MHz, CDCl$_3$)

1k
$^{13}$C NMR (100 MHz, CDCl$_3$)

1k

MeO-$\text{Ph}$-$\text{Ph}$-$\text{Ph}$-$\text{Ph}$-$\text{Me}$

$^{13}$C NMR (100 MHz, CDCl$_3$)

1k

MeO-$\text{Ph}$-$\text{Ph}$-$\text{Ph}$-$\text{Ph}$-$\text{Me}$
$^1$H NMR (400 MHz, CDCl$_3$)

MeO

Ph

O-Si-O

11

1.98

2.06

3.73

4.13

4.54

6.37

7.46

7.44

7.43

7.75

7.74

7.73

8.19

8.17

8.16

8.15

8.14
$^{13}$C NMR (100 MHz, CDCl$_3$)

![Compound Structure]

1 I

Peak Frequencies (ppm):
- 198.25
- 135.11
- 132.79
- 130.52
- 130.31
- 128.81
- 128.05
- 127.58
- 127.29
- 126.01
- 114.04
- 84.31
- 55.41

Chemical Shift Values:
- 77.48 CDCl$_3$
- 77.18 CDCl$_3$
- 76.34 CDCl$_3$
$^1$H NMR (400 MHz, CDCl$_3$)
$^{13}$C NMR (100 MHz, CDCl$_3$)

![Chemical Structure](image)

$1m$
^H NMR (400 MHz, CDCl\textsubscript{3})

\[\text{Structure Image}\]
$^{13}$C NMR (100 MHz, CDCl$_3$)
$^1$H NMR (400 MHz, CDCl$_3$)

$\text{MeO}$

1g

$\text{i-Pr}$

$\text{O}$

$\text{Si}$

$\text{O}$

$\text{i-Pr}$

$3$

$2$

$1$

$4$

$5$

$6$

$7$

$\text{Me}$
$^{13}$C NMR (100 MHz, CDCl$_3$)
$^1$H NMR (400 MHz, CDCl$_3$)
$^{13}$C NMR (100 MHz, CDCl$_3$)

![Chemical Structure](image)

1h
$^{13}$C NMR (100 MHz, CDCl$_3$)

![Chemical Structure Image]

S31
$^{13}$C NMR (100 MHz, CDCl$_3$)

1n
$^1$H NMR (400 MHz, CDCl$_3$)
$^{13}$C NMR (100 MHz, CDCl$_3$)

$4c$
$^1$H NMR (500 MHz, CDCl$_3$)
$^{13}$C NMR (125 MHz, CDCl$_3$)

MeO

$^{4d}$
$\text{MeO}$

$\text{Me}$

1H NMR (500 MHz, CDCl$_3$)
$^1$H NMR (500 MHz, CDCl$_3$)

MeO

[Diagram of a chemical structure with peaks labeled]
$^{13}$C NMR (125 MHz, CDCl$_3$)
$^1$H NMR (400 MHz, CDCl$_3$)
$\text{Si} - i$-Pr $i$-Pr MeO OMe

$^{13}$C NMR (100 MHz, CDCl$_3$)
$^{1}$H NMR (500 MHz, CDCl$_3$)
$^{13}$C NMR (125 MHz, CDCl$_3$)

$t$-Bu, $t$-Bu

MeO

OMe

2b
$^{13}$C NMR (100 MHz, CDCl$_3$)

\[
\text{MeO} \quad 2c \quad \text{OMe}
\]
$^1$H NMR (400 MHz, CDCl$_3$)

![NMR Spectrogram]
\(^{13}\)C NMR (100 MHz, CDCl\(_3\))

![Chemical Structure](image)

**Chemical Shifts (ppm):**
- 77.41 (CDCl\(_3\))
- 76.31 (CDCl\(_3\))
- 62.58
- 62.55
- 55.41
- 52.12
- 44.39
- 44.16
- 38.13
- 38.79
- 21.17
- 17.17
- 17.81
- 12.07

**Assignments:**
- 1: 158.17
- 2: 140.81
- 3: 139.05
- 4: 135.60
- 5: 129.18
- 6: 127.99
- 7: 126.93
- 8: 113.89
$^{13}$C NMR (125 MHz, CDCl$_3$)

dr 4:1
$^1$H NMR (500 MHz, CDCl$_3$)

![NMR spectrum]
$^{13}$C NMR (125 MHz, CDCl$_3$)
$^{13}$C NMR (125 MHz, CDCl$_3$)
$^1$H NMR (500 MHz, CDCl$_3$)

dr 1:1.7
$^{13}$C NMR (125 MHz, CDCl$_3$)

\[ \text{dr 1:1.7} \]

[Chemical structures and spectral data]
$^{1}{\text{H}}$ (500 MHz) - $^{13}\text{C}$ (125 MHz) HSQC (CDCl$_3$)

dr 1:1.7
NOESY-2D (500 MHz, CDCl₃)

2h

3h
de 1:1.7
$^{1}$H NMR (500 MHz, CDCl$_3$)

$\text{MeO}^\text{2i}$ + $\text{MeO}^\text{3i}$

$\text{dr} \ 1:1.5$
$^{13}$C NMR (125 MHz, CDCl$_3$)

2i + 3i

dr 1:1.5

MeO

Me
$^1$H NMR (400 MHz, CDCl$_3$)

2j
$^{13}$C NMR (100 MHz, CDCl$_3$)
$^1$H NMR (400 MHz, CDCl$_3$)

3j

MeO

OMe
$^{13}$C NMR (100 MHz, CDCl$_3$)

3j
$^{13}$C NMR (100 MHz, CDCl$_3$)
$^1$H NMR (400 MHz, CDCl$_3$)

[Chemical structure image with peaks labeled]
$^{13}$C NMR (100 MHz, CDCl$_3$)
$^{1}H - ^{1}H$ COSY - 90 (400 MHz, CDCl$_3$)
$\text{H (400 MHz) - } ^{13}\text{C (100 MHz) HSQC (CDCl}_3\text{)}$
NOESY-2D (400 MHz, CDCl₃)

3k
$^1$H NMR (500 MHz, CDCl$_3$)

2I

3I

dr 1:2

S74
$^{13}$C NMR (125 MHz, CDCl$_3$)

![Chemical structure diagram]

- Ph$_5$Si$^-$O$_3$
- Ph$_5$Si$^-$O$_3$
- MeO
- OMe

**2I** + **3I**
dr 1:2

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<td>7</td>
<td>170</td>
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<tr>
<td>8</td>
<td>180</td>
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$^1$H NMR (500 MHz, CDCl$_3$)
$^{13}$C NMR (125 MHz, CDCl$_3$)
$^{13}$C NMR (125 MHz, CDCl$_3$)
$^1$H NMR (500 MHz, CDCl$_3$)

dr 1:4
$^1$H NMR (400 MHz, CDCl$_3$)
$^{13}$C NMR (100 MHz, CDCl$_3$)

![Chemical structure](image)

**Resonance Frequencies (ppm)**

- 157.73
- 140.14
- 132.67
- 130.90
- 128.91
- 123.14
- 119.52
- 113.45
- 77.48 (dec)
- 77.16 (dec)
- 76.84 (dec)
- 74.08
- 73.99
- 52.28
- 46.73
- 46.48
- 42.62
- 42.27

**Chemical Groups**

- MeO
- Br
- 5c
$^1$H NMR (400 MHz, CDCl$_3$)
$^1$C NMR (100 MHz, CDCl$_3$)
$^1$H NMR (400 MHz, CDCl$_3$)
$^{13}$C NMR (100 MHz, CDCl$_3$)

MeO

Me

13C NMR (100 MHz, CDCl$_3$)
$^{13}$C NMR (125 MHz, CDCl$_3$)

MeO

5g
$^1\text{H} - ^1\text{H COSY} - 90$ (500 MHz, CDCl$_3$)
$^1$H (500 MHz) - $^{13}$C (125 MHz) HSQC (CDCl$_3$)
$^1$H NMR (500 MHz, CDCl$_3$)
$^{13}$C NMR (125 MHz, CDCl$_3$)
$^{1}H$ NMR (500 MHz, CDCl$_3$)
$^{13}$C NMR (125 MHz, CDCl$_3$)

[Chemical structure diagram]

Chemical shifts:
- 158.41
- 134.63
- 127.91
- 114.02
- 77.48 (CDCl$_3$)
- 77.16 (CDCl$_3$
- 76.34 (CDCl$_3$
- 65.80
- 55.42
- 47.84
- 47.28
$^1$H NMR (500 MHz, CDCl$_3$)
$^{13}$C NMR (125 MHz, CDCl$_3$)
$^1$H NMR (500 MHz, CDCl$_3$)

![NMR spectrum of compound 8a]
$^{13}$C NMR (125 MHz, CDCl$_3$)
$^1$H - $^1$H COSY - 90 (500 MHz, CDCl$_3$)
NOESY-2D (500 MHz, CDCl\textsubscript{3})

\text{MeO}  

8a
$^{1}$H NMR (500 MHz, CDCl$_3$)

8b
$^{13}$C NMR (125 MHz, CDCl$_3$)
$^{13}$C NMR (125 MHz, CDCl$_3$)
$^1H - ^1H \text{ COSY - 90 (500 MHz, CDCl}_3\text{)}$
$^1$H (500 MHz) · $^{13}$C (125 MHz) HSQC (CDCl$_3$)
$^1$H NMR (500 MHz, CDCl$_3$)
$^{13}$C NMR (125 MHz, CDCl$_3$)

![Chemical Structure](image)