DOSY-NMR analysis of ring closing metathesis (RCM) products from β-lactam precursors

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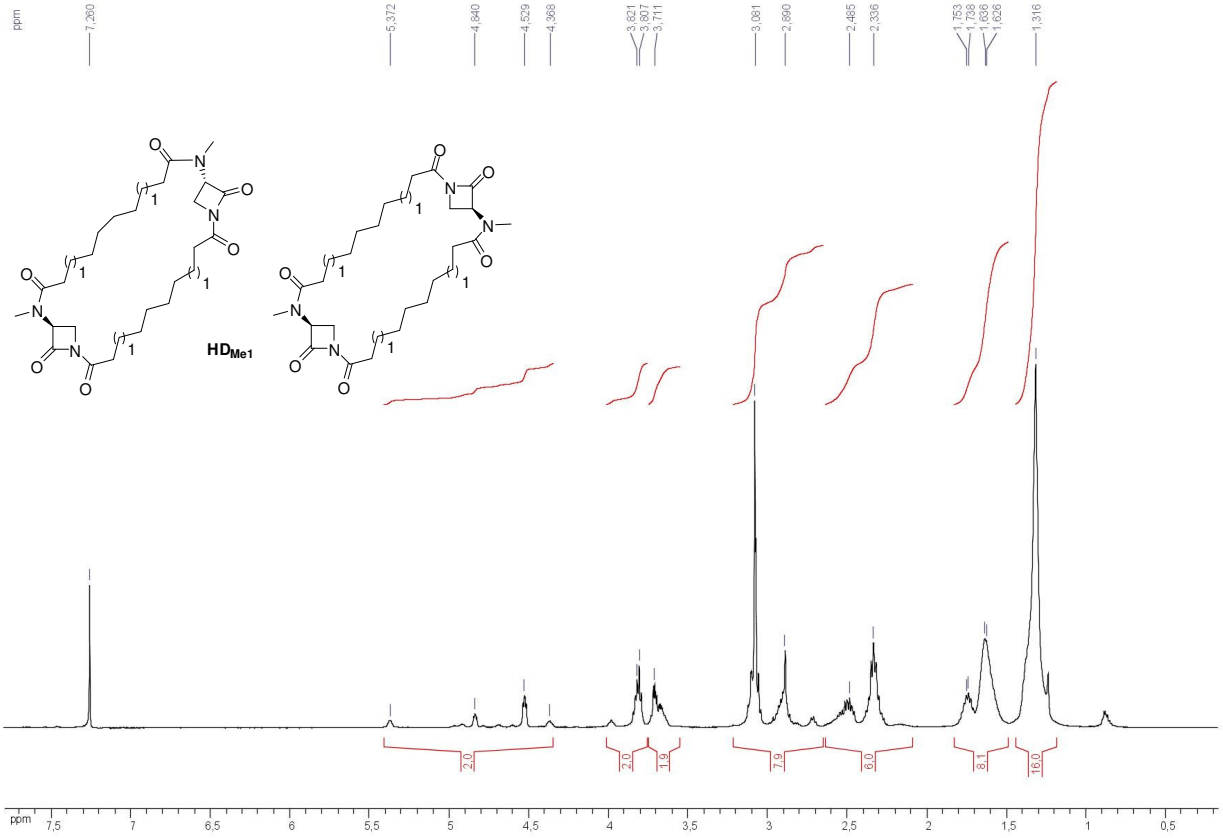
Supplementary Material

^{1}H NMR chemical shifts (δ, ppm) of $\mathbf{M_{Boc2}}$, $\mathbf{D_{Boc2}}$, all precursors and hydrogenated RCM products (multiplicities, J values in Hz and integrations are given in parentheses)

Compound	H-3	H-4a,b	HC=CH$_{(2)}$	R (Boc, H or Me)	CH$_2$ acyl chain
$\mathbf{P_{Boc1}}$	5.71-5.86 (m°, 1H)	3.81-3.85 (m, 1H) 3.61 (dd, 4.2, 7.1 Hz, 1H)	5.71-5.86 (m°, 2H) 4.96-5.07 (m, 4H)	1.51 (s, 9H)	2.85-2.90 (m, 2H) 2.71-2.76 (m, 2H) 2.06-2.16 (m, 4H) 1.69-1.83 (m, 4H)
$\mathbf{P_{Boc2}}$	5.72-5.86 (m°, 1H)	3.80-3.85 (m, 1H) 3.61 (dd, 4.2, 7.1 Hz, 1H)	5.72-5.86 (m°, 2H) 4.92-5.03 (m, 4H)	1.51 (s, 9H)	2.85-2.90 (m, 2H) 2.71-2.76 (m, 2H) 2.03-2.11 (m, 4H) 1.60-1.74 (m, 4H) 1.38-1.48 (m, 4H)
$\mathbf{P_{Me1}}$	4.91 (dd, 3.9, 6.5 Hz, 1H)	3.79-3.84 (m, 1H) 3.64 (dd, 3.9, 7.4 Hz, 1H)	5.70-5.84 (m, 2H) 4.95-5.05 (m, 4H)	3.04 (s, 3H)	2.71-2.77 (m, 2H) 2.31-2.36 (m, 2H) 2.06-2.15 (m, 4H) 1.69-1.81 (m, 4H)
$\mathbf{P_{Me2}}$	4.89-5.02 (m°, 1H)	3.79-3.83 (m, 1H) 3.63 (dd, 3.9, 7.4 Hz, 1H)	5.71-5.84 (m, 2H) 4.89-5.02 (m°, 4H)	3.05 (s, 3H)	2.70-2.76 (m, 2H) 2.31-2.36 (m, 2H) 2.03-2.10 (m, 4H) 1.59-1.72 (m, 4H) 1.37-1.49 (m, 4H)
$\mathbf{P_{H1}}$	4.71 (td, 3.9, 7.4 Hz, 1H)	3.84-3.88 (m, 1H) 3.65-3.68 (m, 1H)	5.69-5.85 (m, 2H) 4.96-5.05 (m, 4H)	6.18 (d, 7.0 Hz, 1H)	2.73 (td, 3.8, 7.4 Hz, 2H) 2.21-2.29 (m, 2H) 2.05-2.15 (m, 4H) 1.69-1.82 (m, 4H)
$\mathbf{P_{H2}}$	4.70 (td, 3.9, 6.9 Hz, 1H)	3.83-3.88 (m, 1H) 3.66 (dd, 3.9, 7.4 Hz, 1H)	5.70-5.85 (m, 2H) 4.92-5.03 (m, 4H)	6.21 (d, 7.0 Hz, 1H)	2.64-2.81 (m, 2H) 2.24 (t, 7.5 Hz, 2H) 2.02-2.10 (m, 4H) 1.59-1.72 (m, 4H) 1.36-1.50 (m, 4H)
$\mathbf{M_{Boc2}}$	5.34-5.45 (m°, 1H)	3.81-3.85 (m, 1H) 3.71-3.74 (m, 1H)	5.60-5.70 (m, 1H) 5.34-5.45 (m°, 1H)	1.25-2.33 (m°, 9H)	2.90-2.99 (m, 1H) 2.66-2.82 (m,2H) 1.25-2.33 (m°, 13H)
$\mathbf{D_{Boc2}}$	5.71-5.76 (m, 2H)	3.80-3.86 (m, 2H) 3.59-3.65 (m, 2H)	5.33-5.41 (m, 4H)	1.51-1.68 (m°, 18H)	2.63-2.91 (m, 8H) 1.96-2.10 (m, 8H) 1.51-1.68 (m°, 8H) 1.35-1.47 (m, 8H)
$\mathbf{HM_{Boc2}}$	5.36-5.38 (m,1H)	3.82-3.86 (m, 1H) 3.70-3.72 (m, 1H)		1.55 (s, 9H)	3.08-3.13 (m, 1H) 2.72-2.84 (m, 2H) 2.30-2.36 (m, 1H) 1.67-1.95 (m, 4H) 1.21-1.45 (m, 12H)
$\mathbf{HD_{Boc1}}$	5.48-5.77 (m, 2H)	3.80-3.85 (m, 2H) 3.61-3.65 (m, 2H)		1.50-1.77 (m°, 18H)	2.52-2.92 (m, 8H) 1.50-1.77 (m°, 8H) 1.24-1.43 (m, 16H)
$\mathbf{HD_{Boc2}}$	5.53-5.77 (m, 2H)	3.82-3.85 (m, 2H) 3.62-3.64 (m, 2H)		1.51-1.75 (m°, 18H)	2.57-2.86 (m, 8H) 1.51-1.75 (m°, 10H) 1.24-1.39 (m, 22H)
$\mathbf{HD_{Me1}}$	4.37-5.37 (m, 2H)	3.79-3.99 (m, 2H) 3.63-3.73 (m, 2H)		2.70-3.12 (m°, 6H)	2.70-3.12 (m°, 2H) 2.27-2.38 (m, 6H) 1.55-1.80 (m, 8H) 1.24-1.40 (m, 16H)
$\mathbf{HD_{Me2}}$	4.70-5.31 (m, 2H)	3.80-3.99 (m, 2H) 3.64-3.74 (m, 2H)		2.51-3.08 (m°, 6H)	2.51-3.08 (m°, 4H) 2.32-2.37 (m, 4H) 1.56-1.77 (m, 8H) 1.22-1.40 (m, 24H)
$\mathbf{HD_{H1}}$	4.78-4.96 (m, 2H)	3.86-3.95 (m, 2H) 3.62-3.69 (m, 2H)		6.45-6.52 (m, 2H)	2.21-2.29 (m, 4H) 1.61-1.77 (m, 8H) 1.25-1.41 (m, 16H)
$\mathbf{HD_{H2}}$	4.72-4.79 (m, 2H)	3.85-3.89 (m, 2H) 3.64-3.71 (m, 2H)		6.49-6.67 (m, 2H)	2.57-2.84 (m, 4H) 2.18-2.32 (m, 4H) 1.56-1.81 (m, 10H) 1.22-1.44 (m, 22H)

° overlapping signals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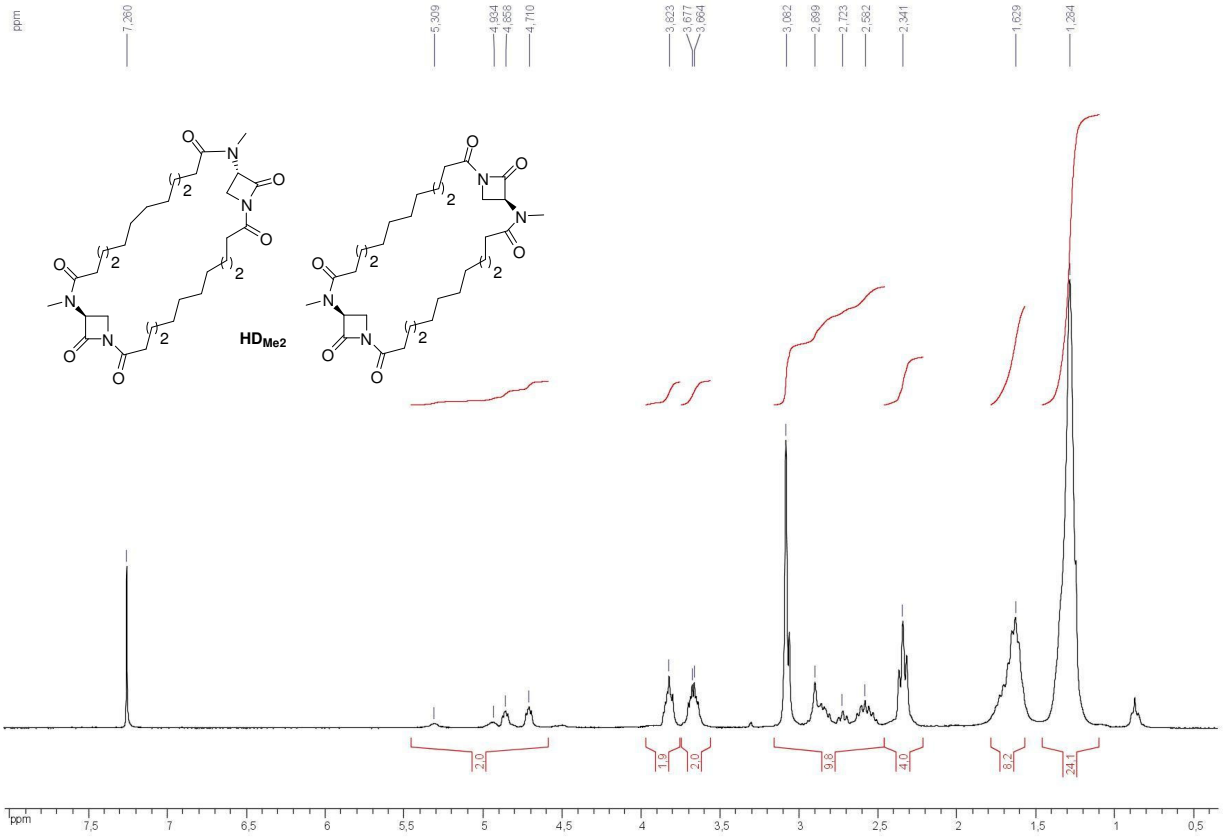
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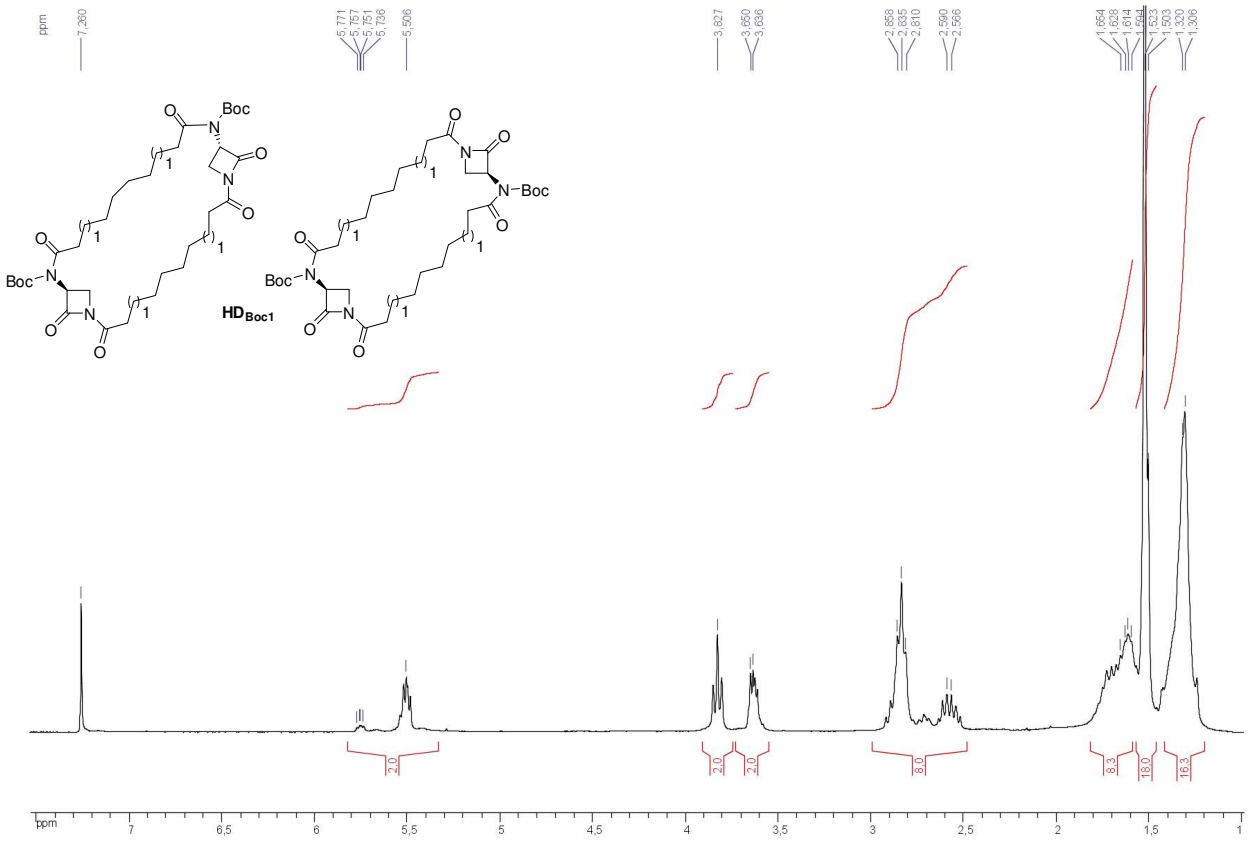
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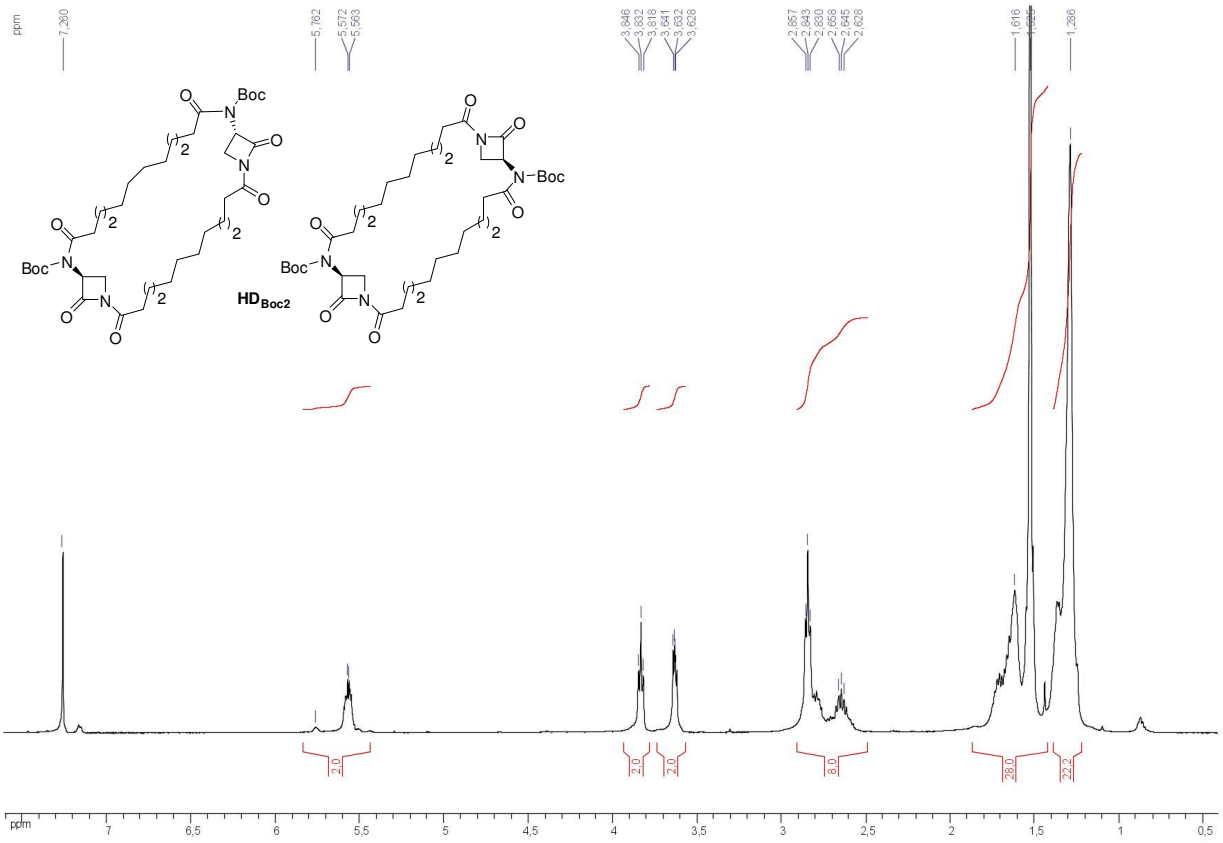
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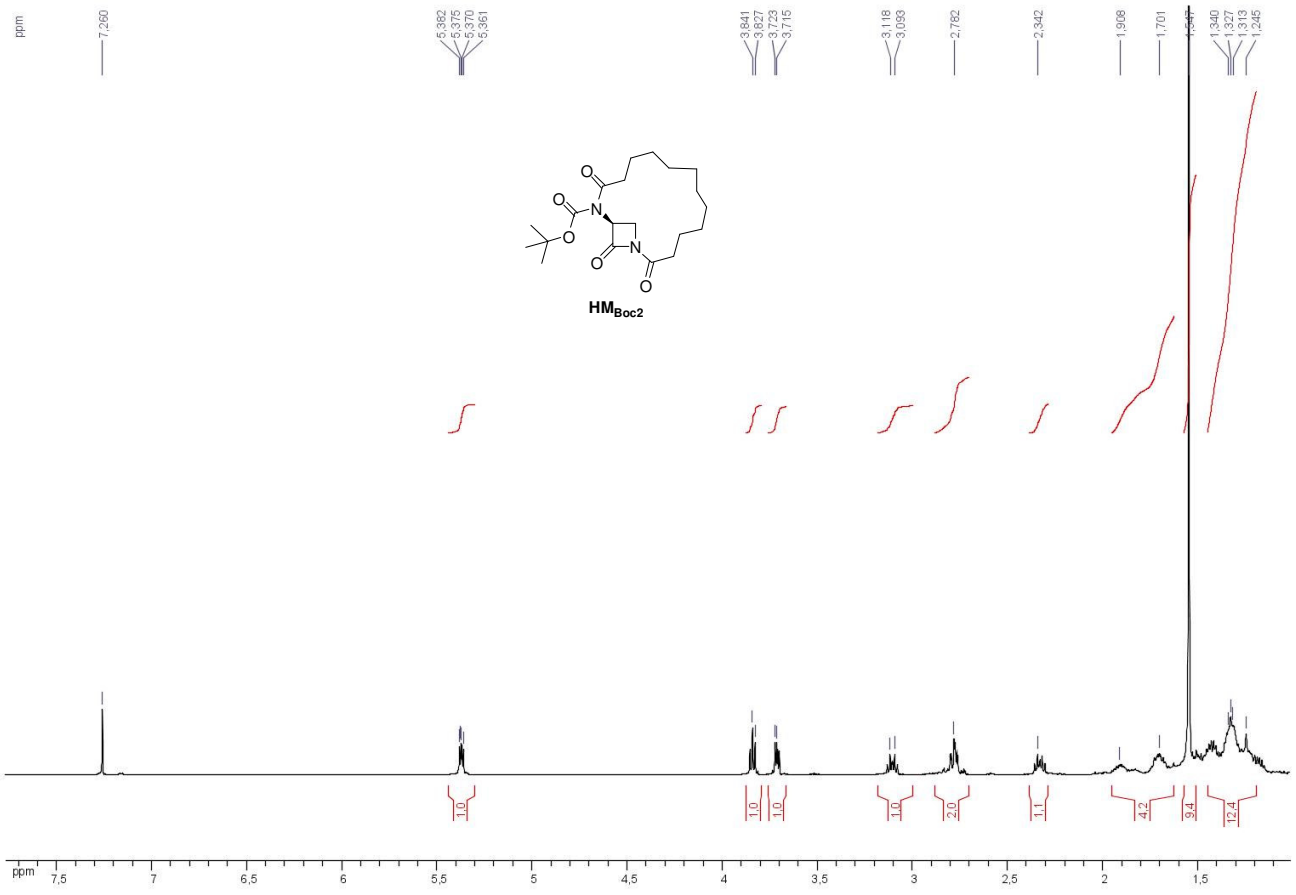
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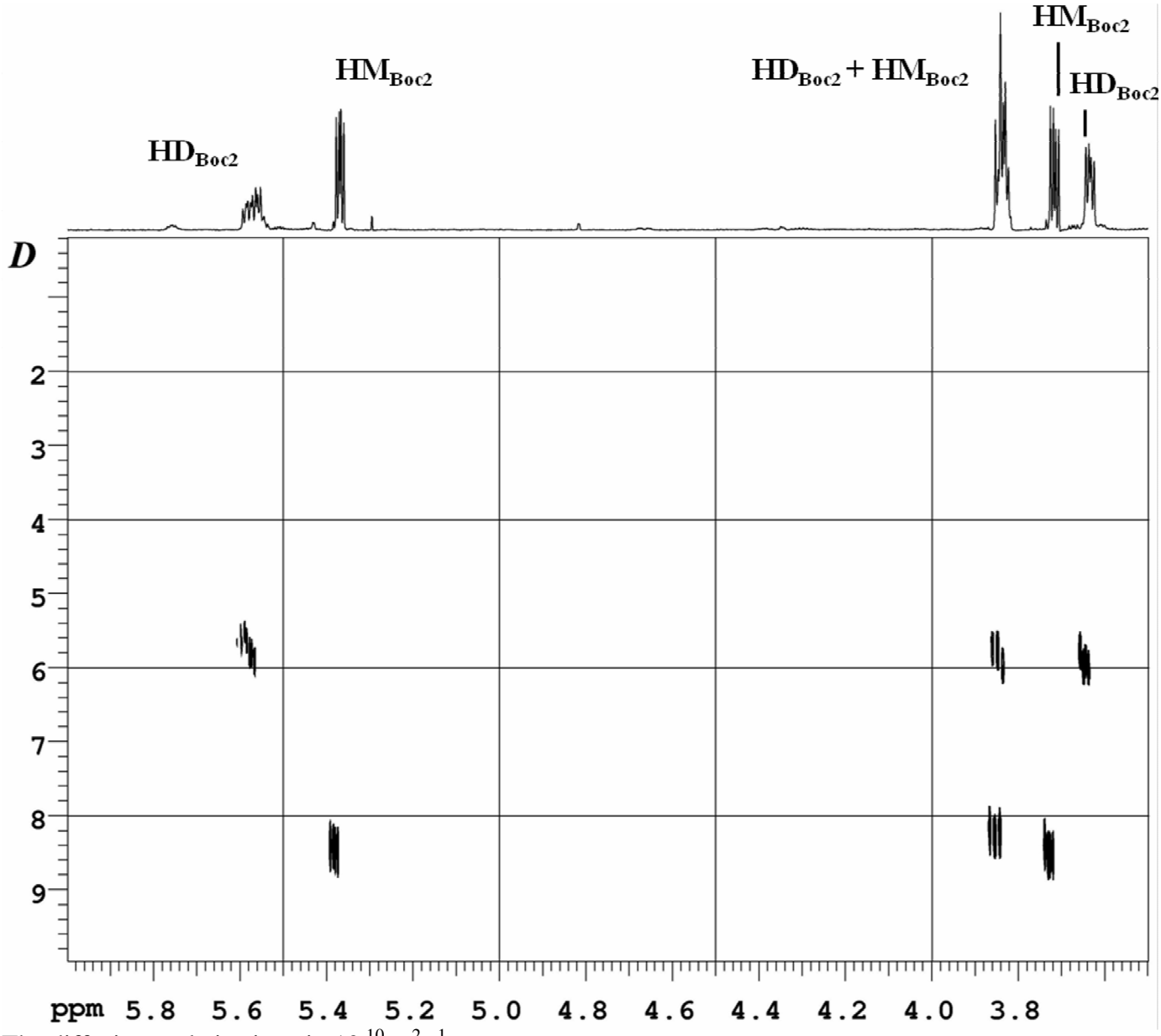
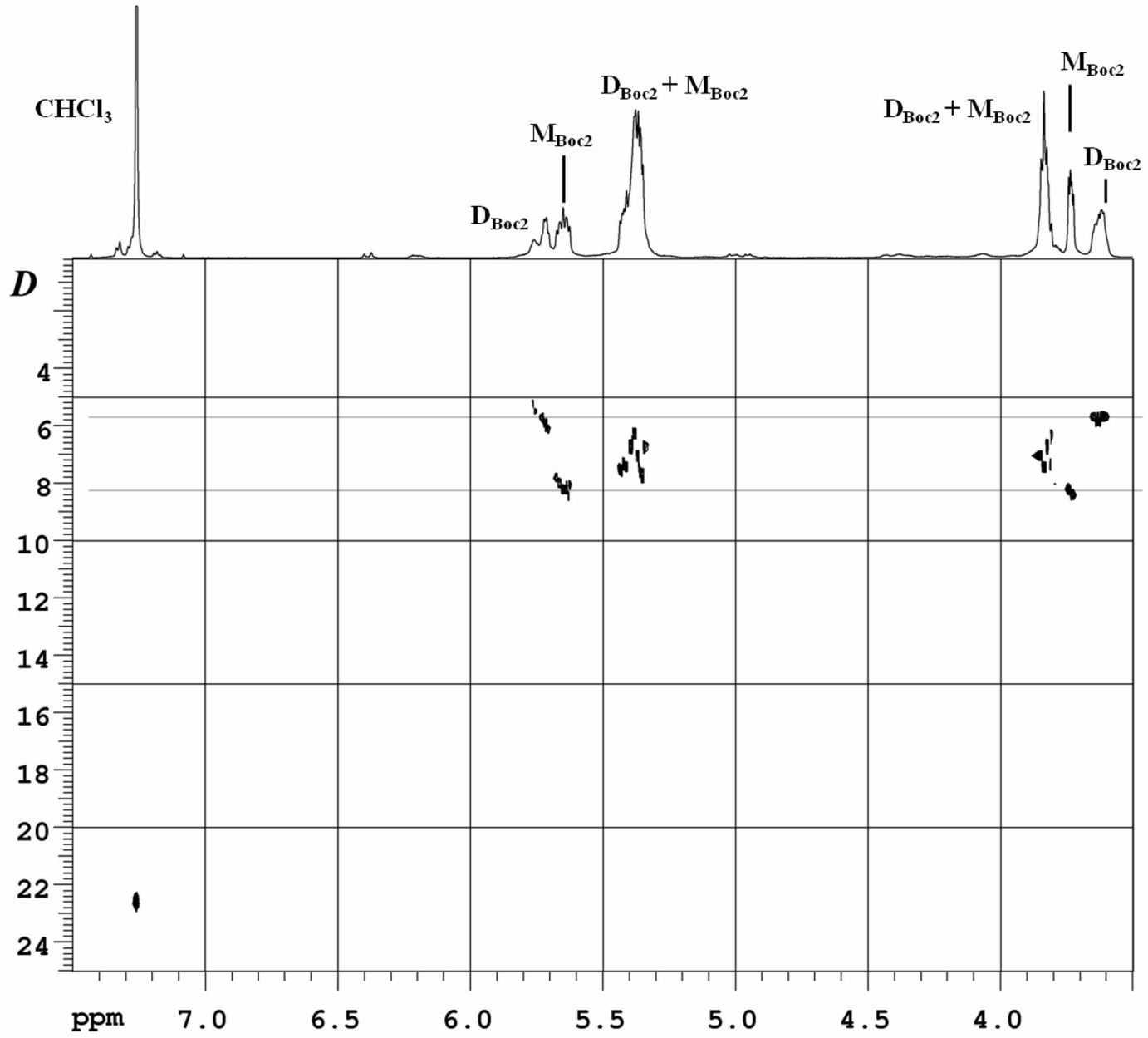
Region of the ^{1}H DOSY-NMR spectrum of a 1:1 mixture of **HM_{Boc2}** and **HD_{Boc2}** in $CDCl_3$ (600 MHz, 298 K) with resolution enhancement



The diffusion scale is given in 10^{-10} m^2s^{-1}.

Region of the ^{1}H DOSY-NMR spectrum of a 1:1 mixture of **M_{Boc2}** and **D_{Boc2}** in $CDCl_3$



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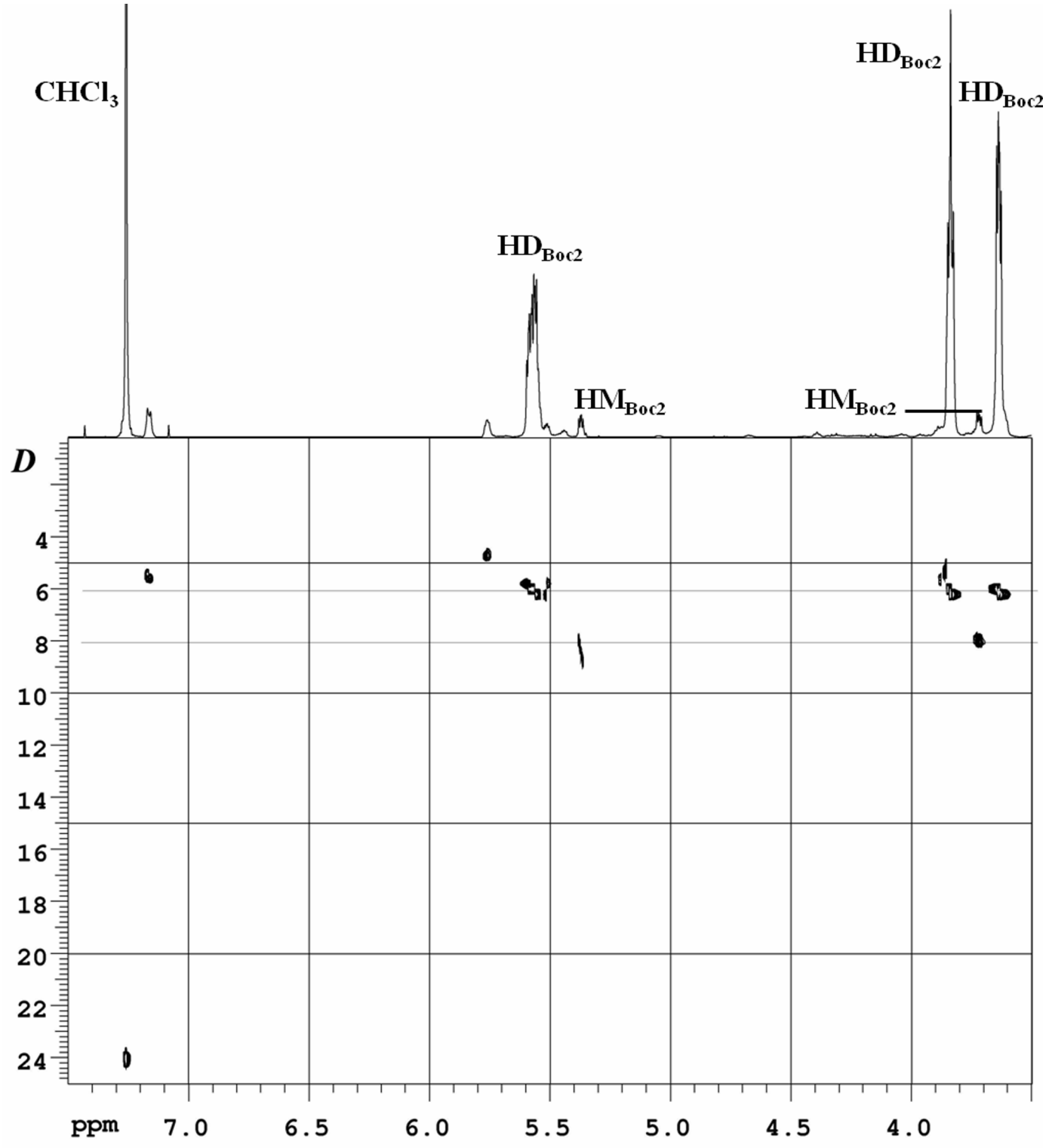
Region of the ^{1}H DOSY-NMR spectrum of a 5:95 mixture of **HM_{Boc2}** and **HD_{Boc2}**



The diffusion scale is given in 10^{-10} m^2s^{-1}.

Despite the fact that the NMR signals of the cyclomonomer **HM_{Boc2}** are weak, they are easily detected in the DOSY spectrum.

The signal next to the residual peak of $CHCl_3$ is a residue of Grubb's catalyst.