

Supplementary Information

Self-Assembly of Structurally Rigid Diamondoid Esters on a HOPG Surface

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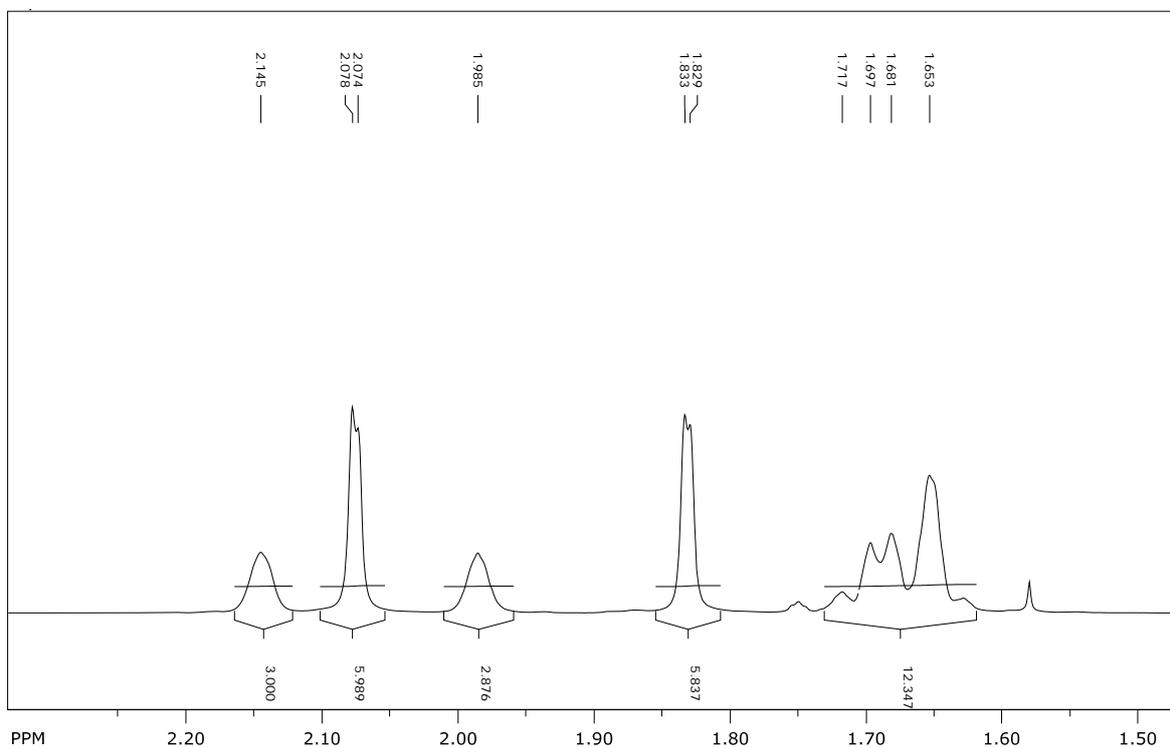
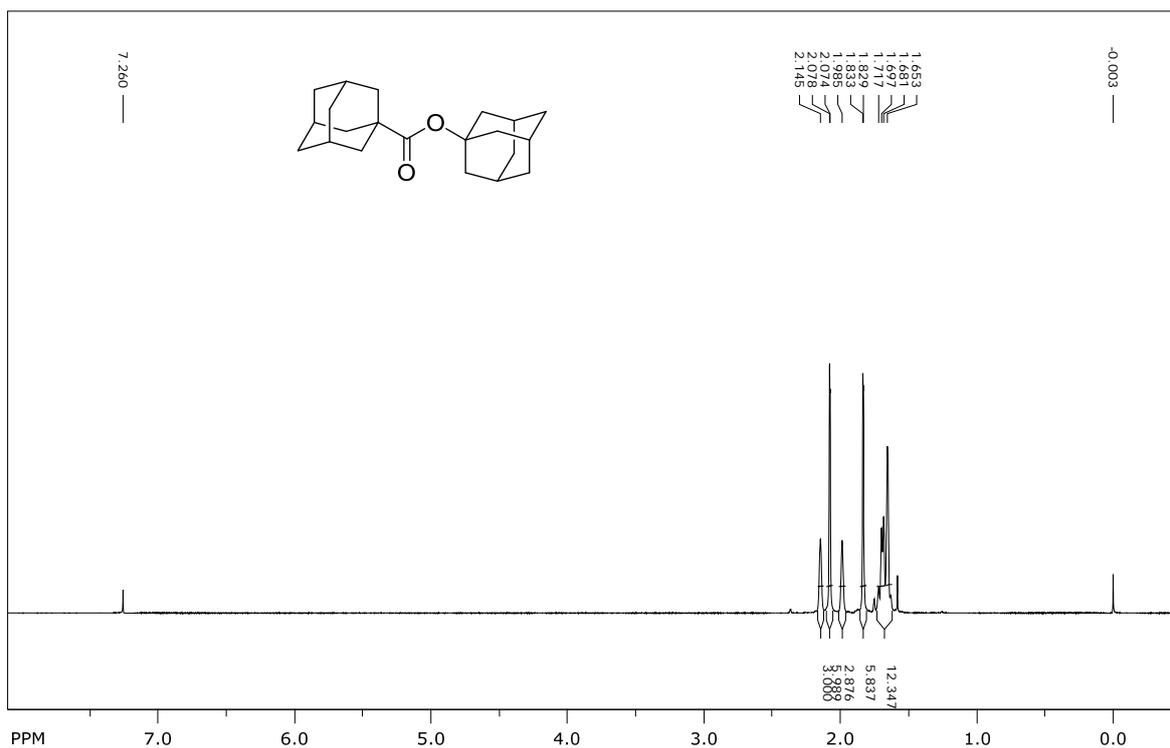
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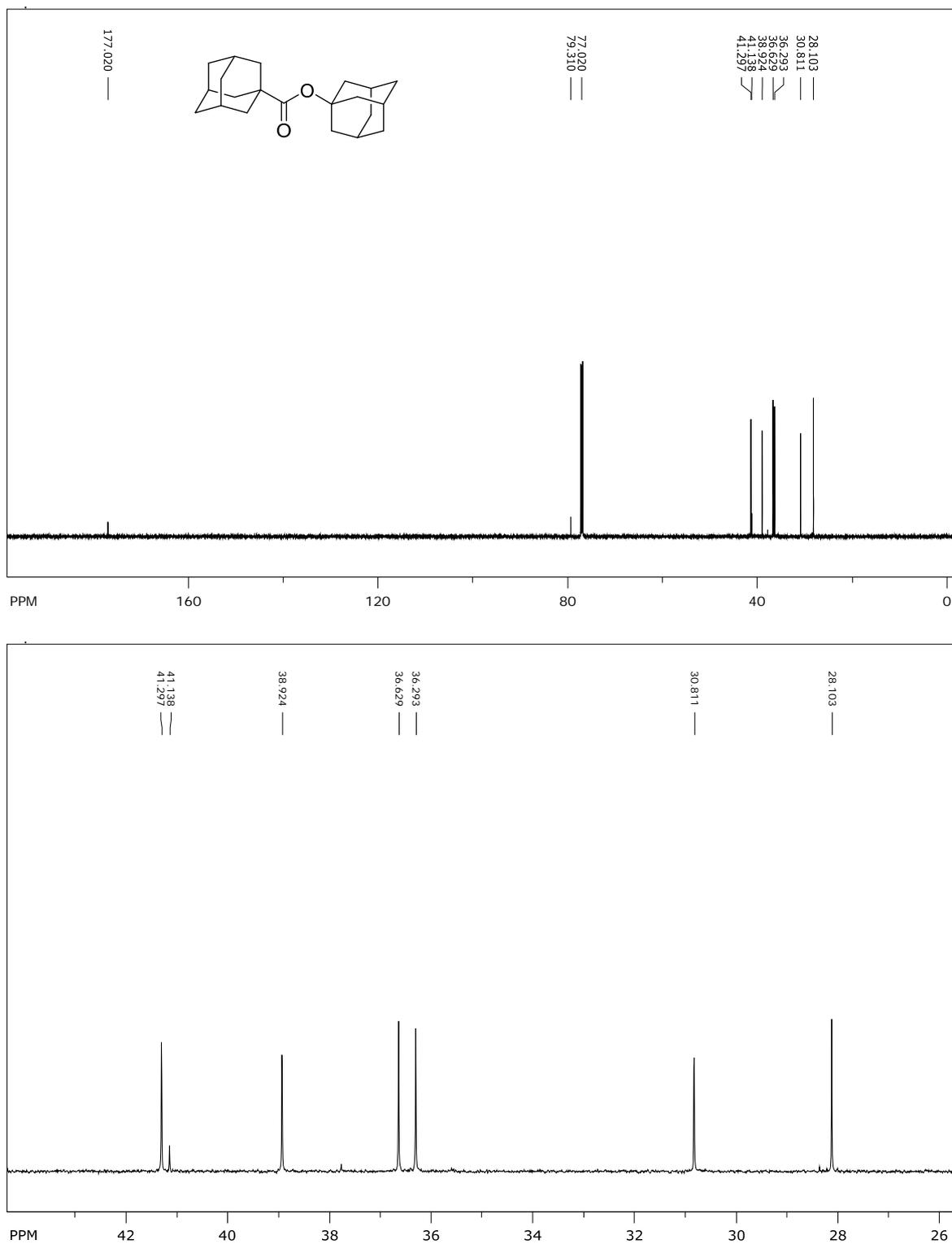
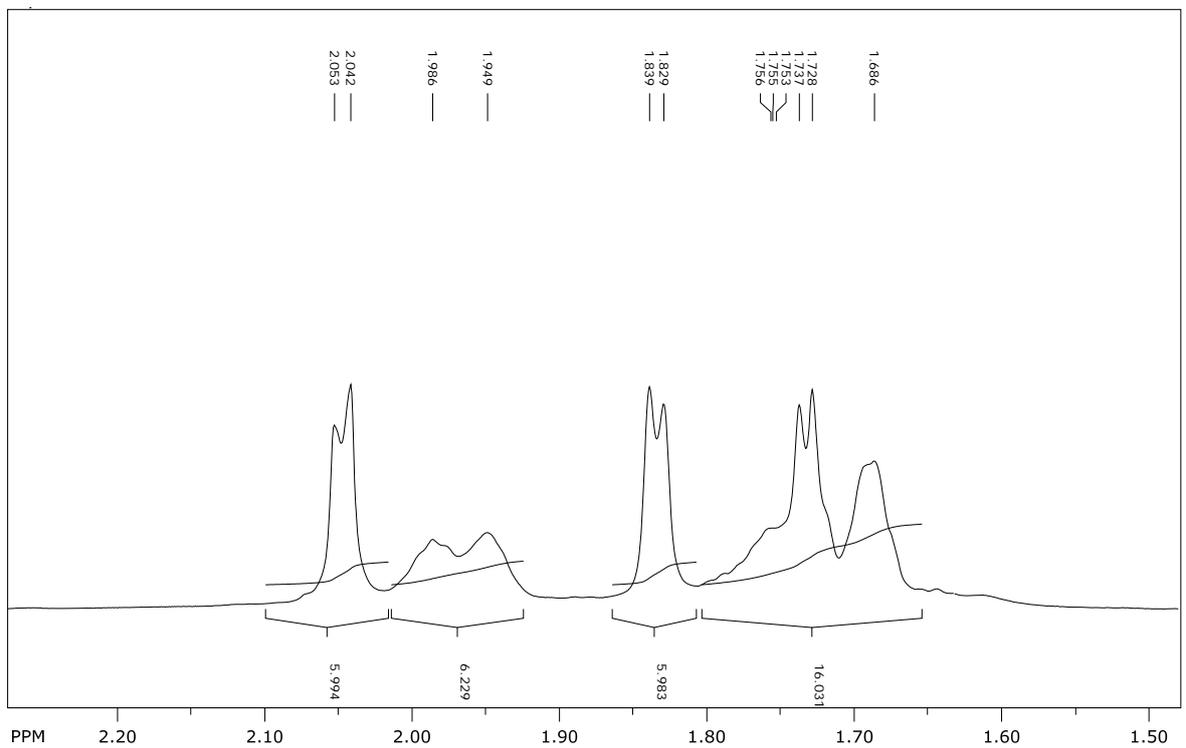
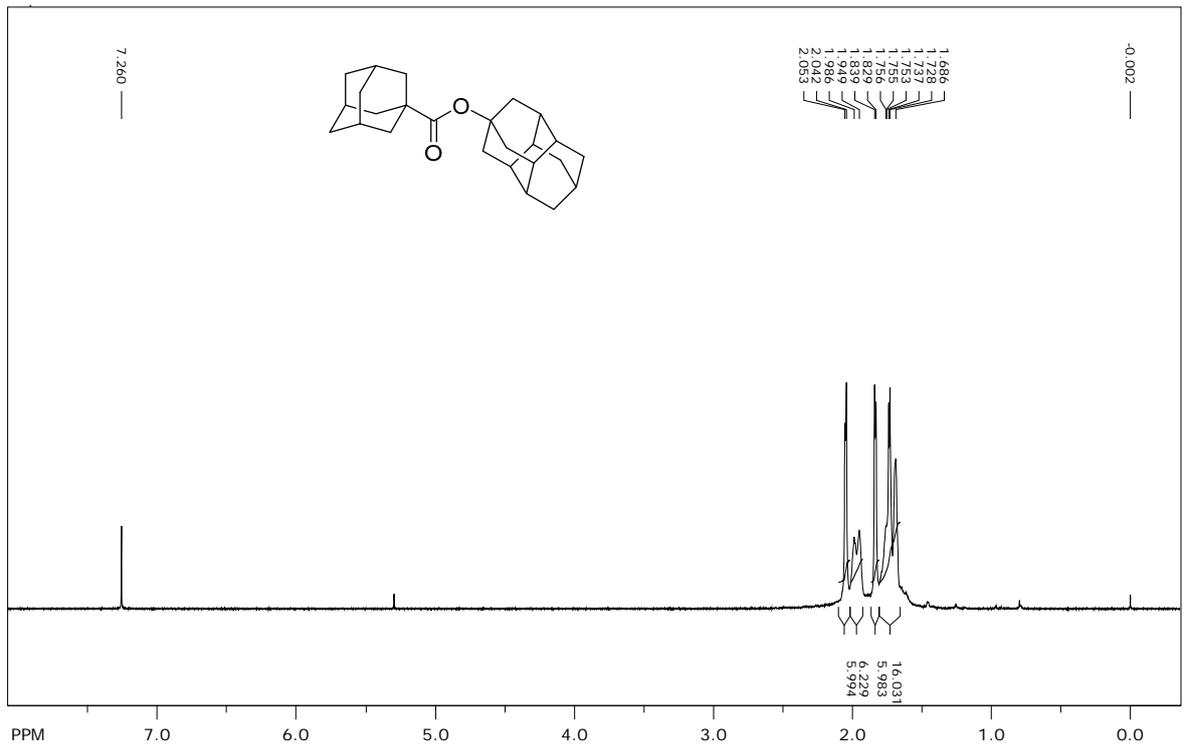


Figure S1. ¹H (600 MHz, CDCl₃) and ¹³C NMR (150 MHz, CDCl₃) spectra of 1-adamantyl-1-adamantanecarboxylate (**1**).



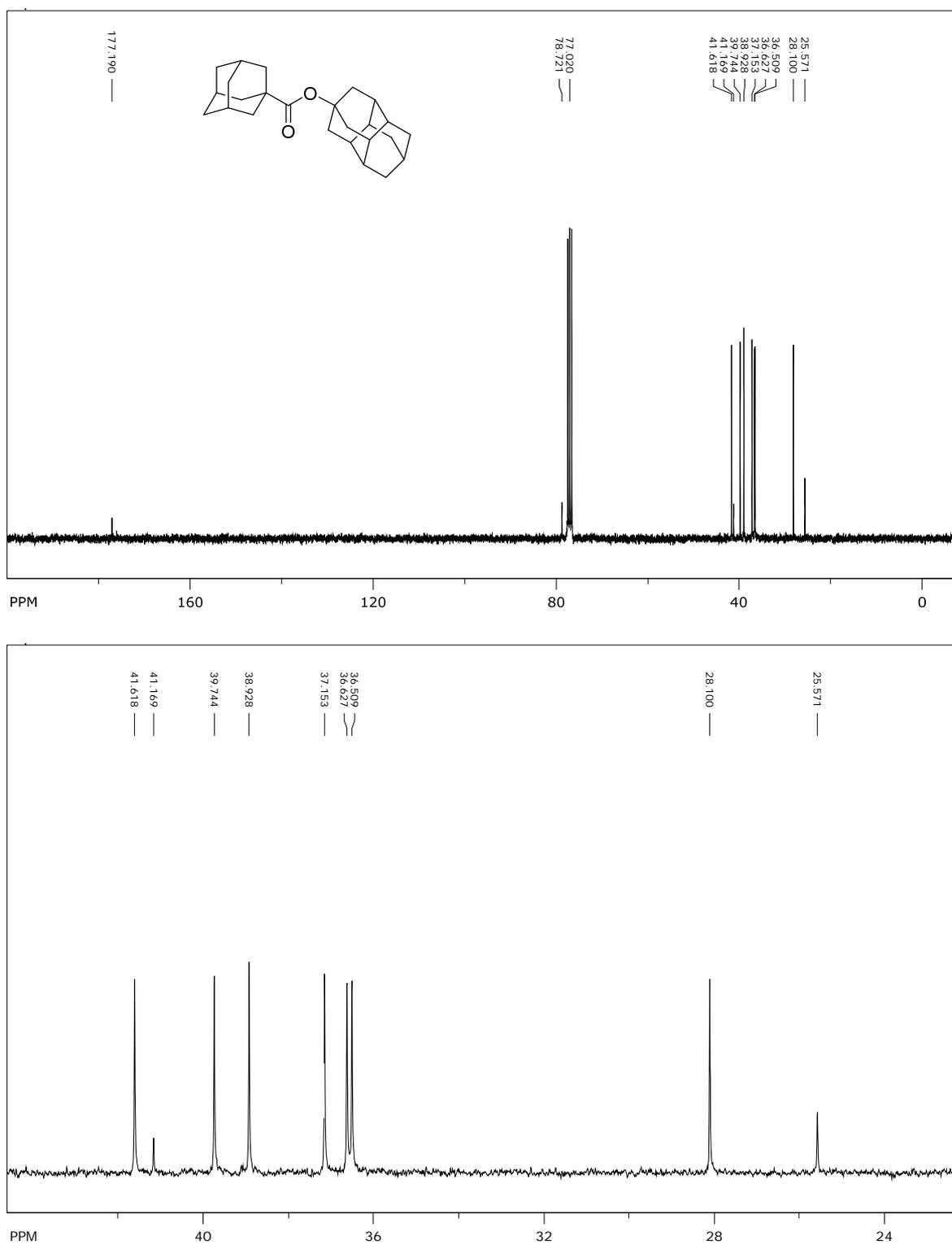
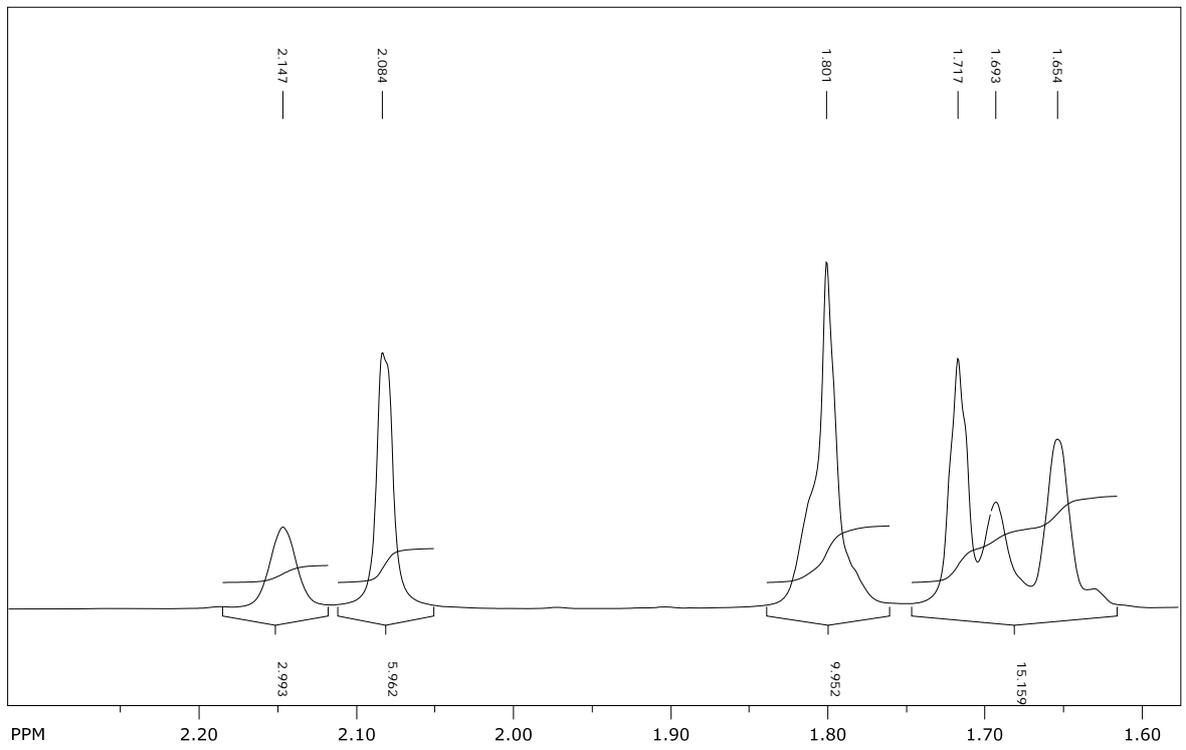
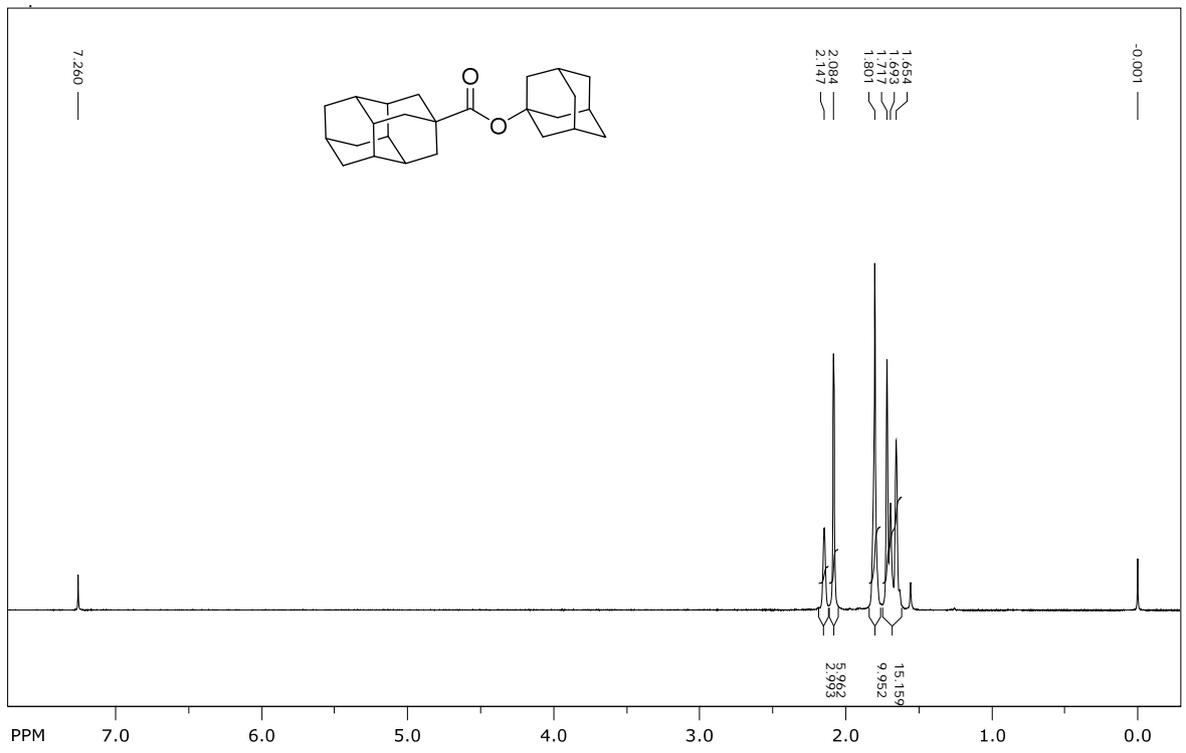


Figure S2. ^1H (300 MHz, CDCl_3) and ^{13}C NMR (75 MHz, CDCl_3) spectra of 4-diamantyl-1-adamantanecarboxylate (**2**).



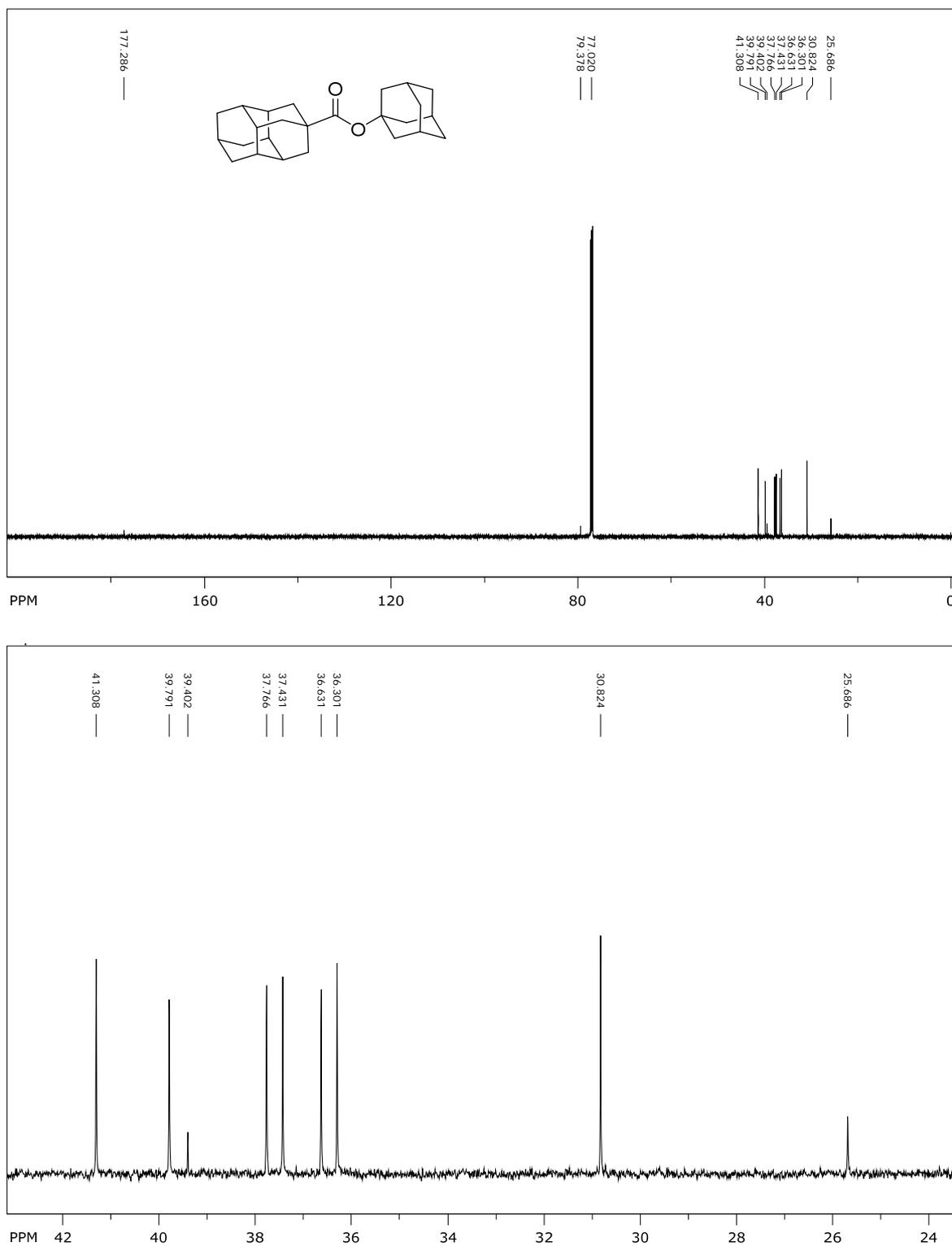
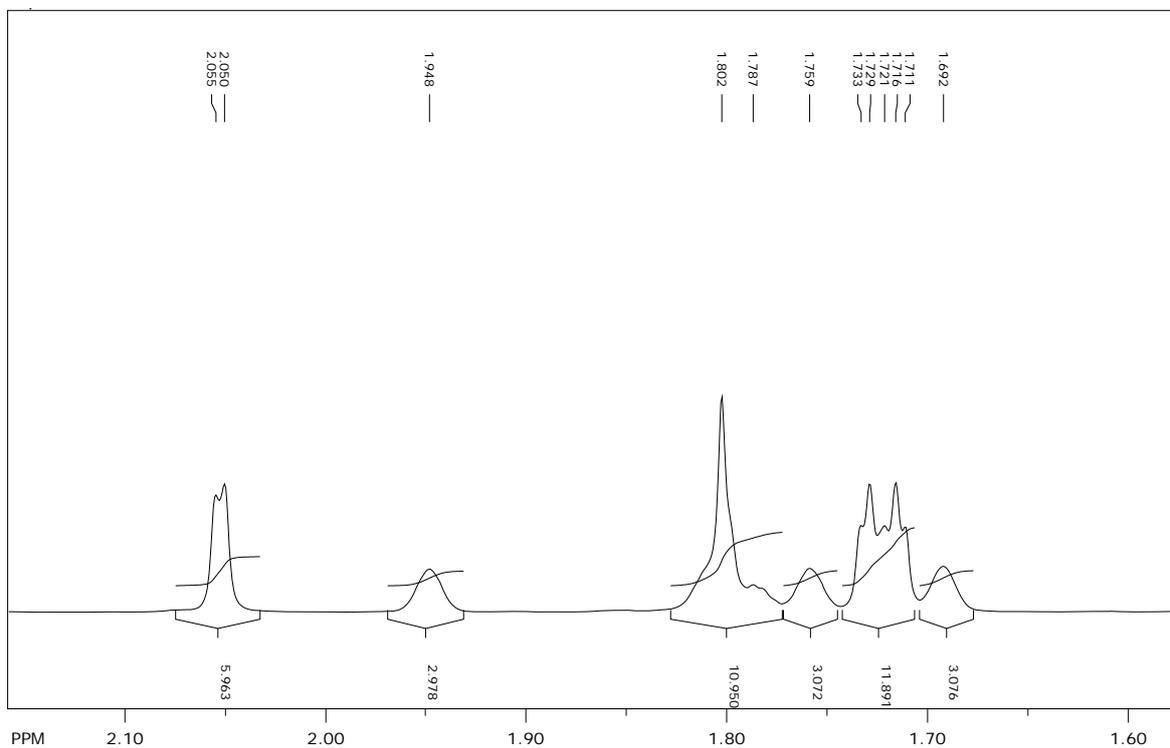
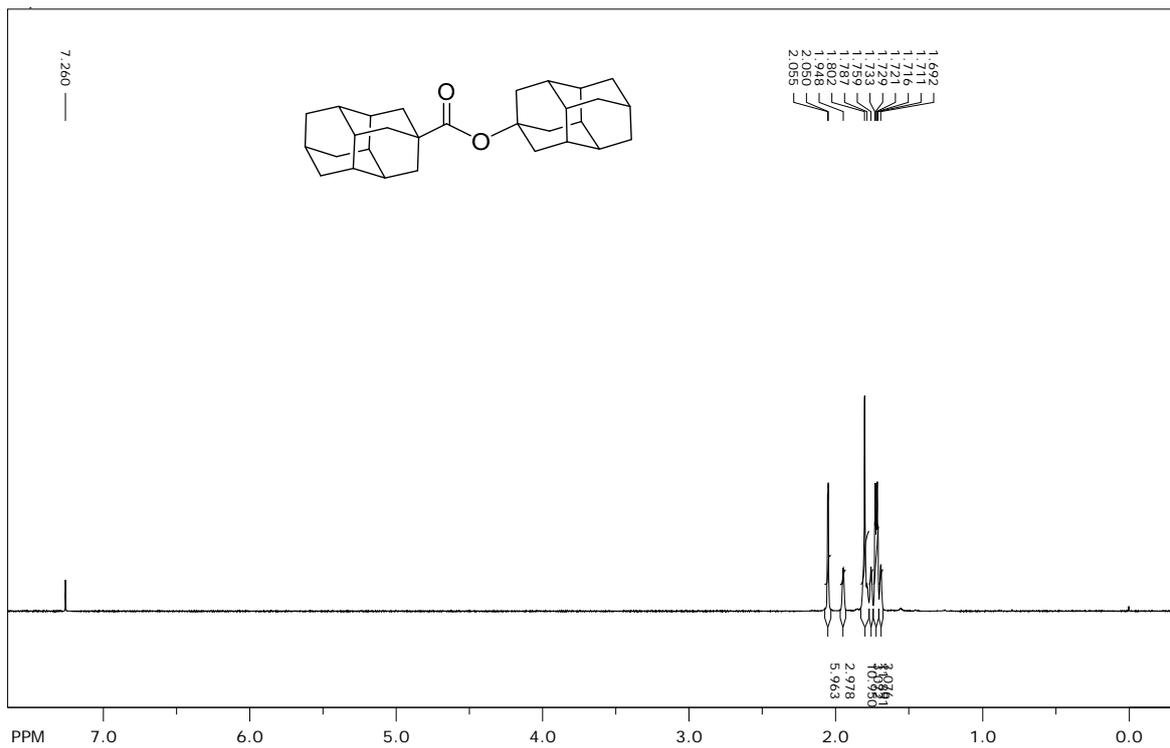


Figure S3. ¹H (600 MHz, CDCl₃) and ¹³C NMR (150 MHz, CDCl₃) spectra of 1-adamantyl-4-diamantanecarboxylate (**3**).



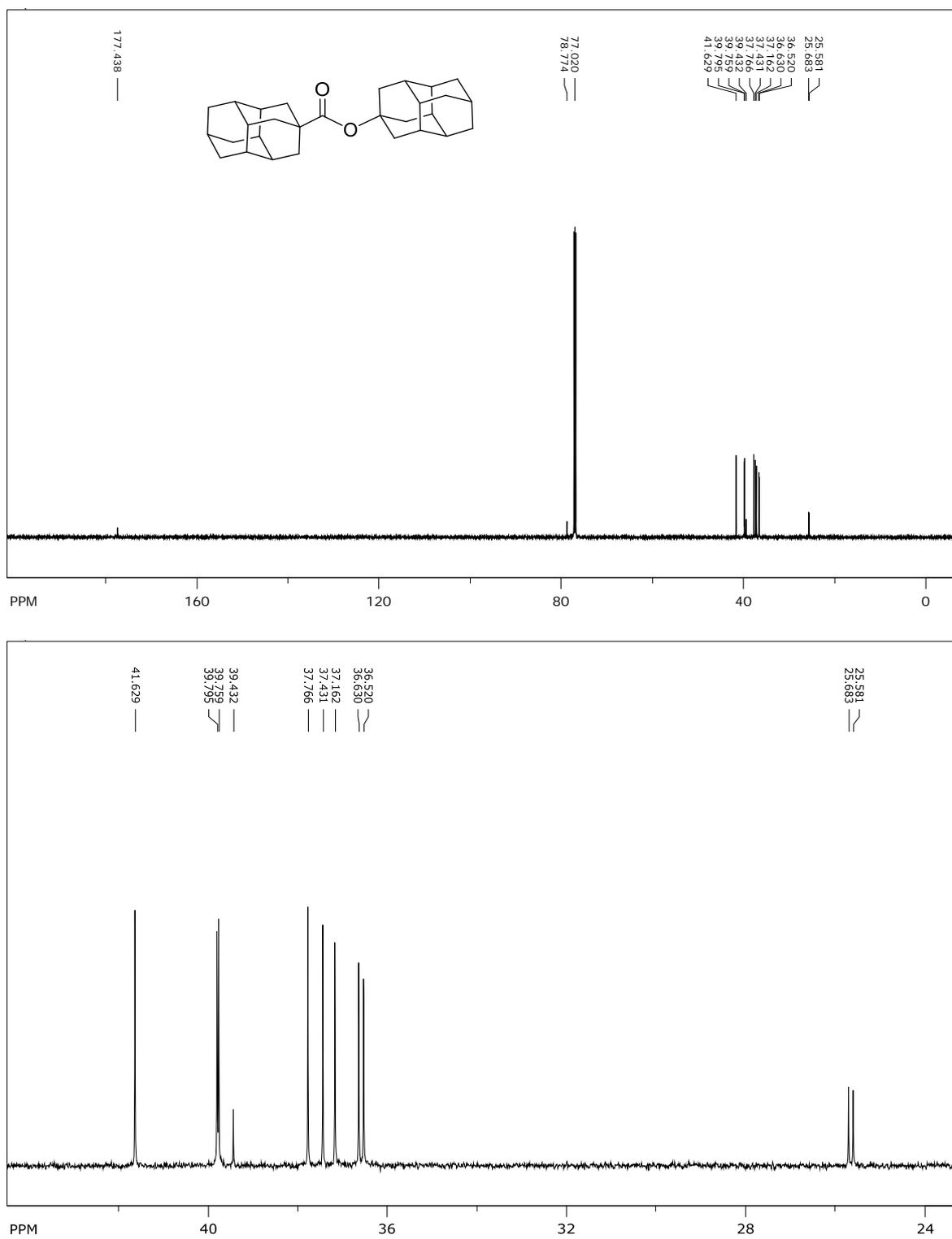


Figure S4. ¹H (600 MHz, CDCl₃) and ¹³C NMR (150 MHz, CDCl₃) spectra of 4-diamantyl-4-diamantanecarboxylate (**4**).

Table S1. Crystallographic data collection and refinement data for compound **2**.

	2 (CCDC: 2518060)
Formula	C ₂₅ H ₃₄ O ₂
Formula Weight	366.52
Crystal System	triclinic
Space group	<i>P1</i>
<i>a, b, c</i> [Å]	6.5364(3) 6.5788(3) 13.1739(5)
<i>α, β, γ</i> [°]	81.826(4) 81.116(4) 62.112(5)
<i>V</i> [Å ³]	493.11(4)
<i>Z</i>	1
D(calc) [g/cm ³]	1.234
Mu(CuKα) [mm ⁻¹]	0.584
F(000)	200.0
Crystal Size [mm]	0.2x0.4x0.5
Data Collection	
Temperature (K)	293(2)
Radiation CuKα	1.54184
Theta Min-Max [°]	6.814 to 158.068°
<i>hkl</i> indices	-8 ≤ <i>h</i> ≤ 6, -8 ≤ <i>k</i> ≤ 8, -16 ≤ <i>l</i> ≤ 16
Tot., Uniq. Data, <i>R</i> _{int}	5965, 2757, 0.0333
Refinement	
<i>N</i> _{ref} , <i>N</i> _{par}	2757, 245
<i>R</i> , <i>wR</i> ² , <i>S</i>	0.040, 0.116, 1.095
Max. and Av. Shift/Error	
Min. and Max. Resd. Dens. [e/Å ³]	0.19/-0.20

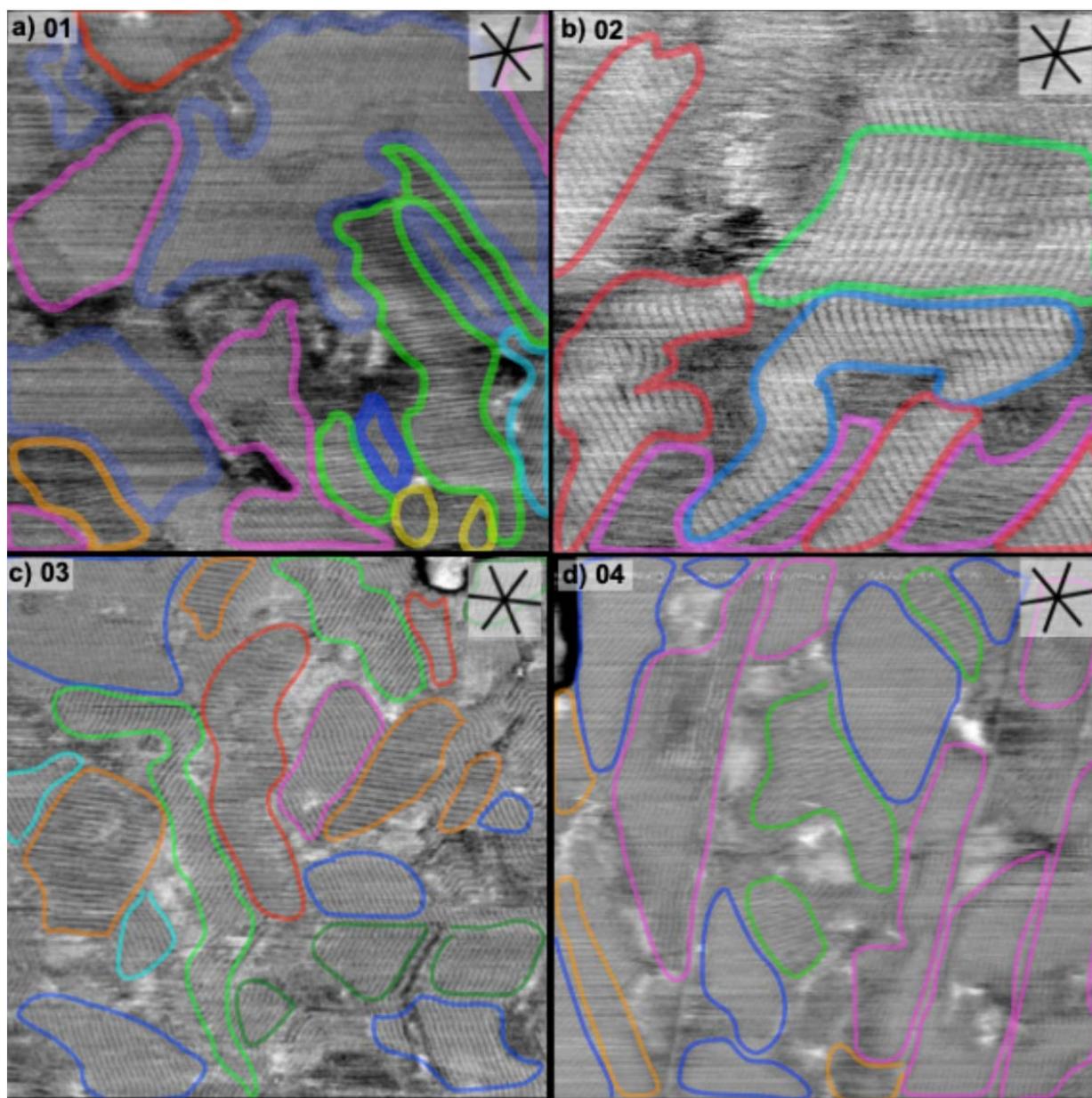
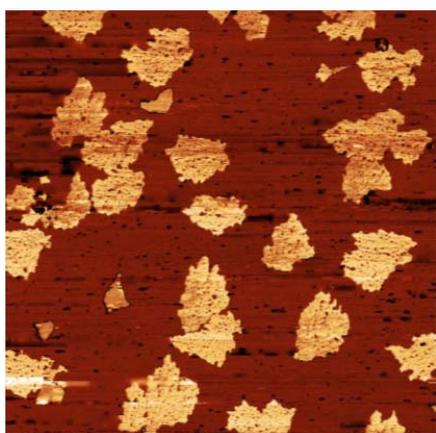


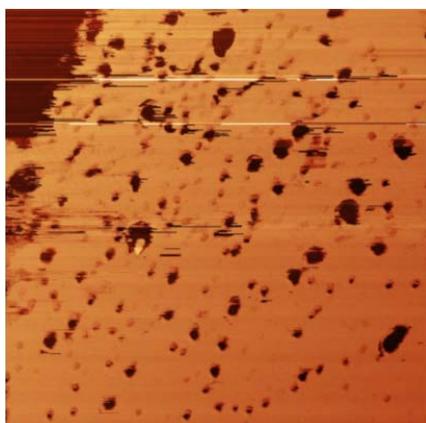
Figure S5. Self-assembly of esters **1–4** on the HOPG surface. The images correspond to the same lock-in phase channel data shown in **Figure 2** of the main manuscript. Distinct molecular domains exhibiting different lattice orientations are highlighted with colored contours for clarity. Panels (**a–d**) correspond to esters **1–4**, respectively.



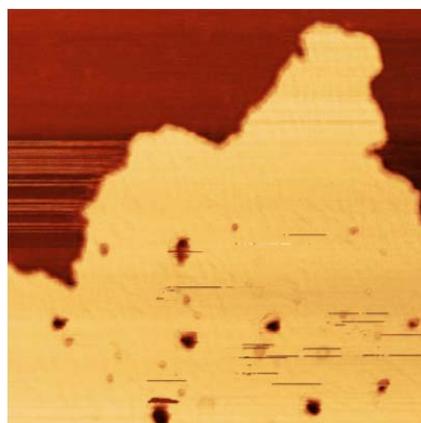
10.0 $\mu\text{m} \times 10.0 \mu\text{m}$



2.0 $\mu\text{m} \times 2.0 \mu\text{m}$



1.0 $\mu\text{m} \times 1.0 \mu\text{m}$



0.5 $\mu\text{m} \times 0.5 \mu\text{m}$

Figure S6. AFM phase images of ester **4** on the mica surface.

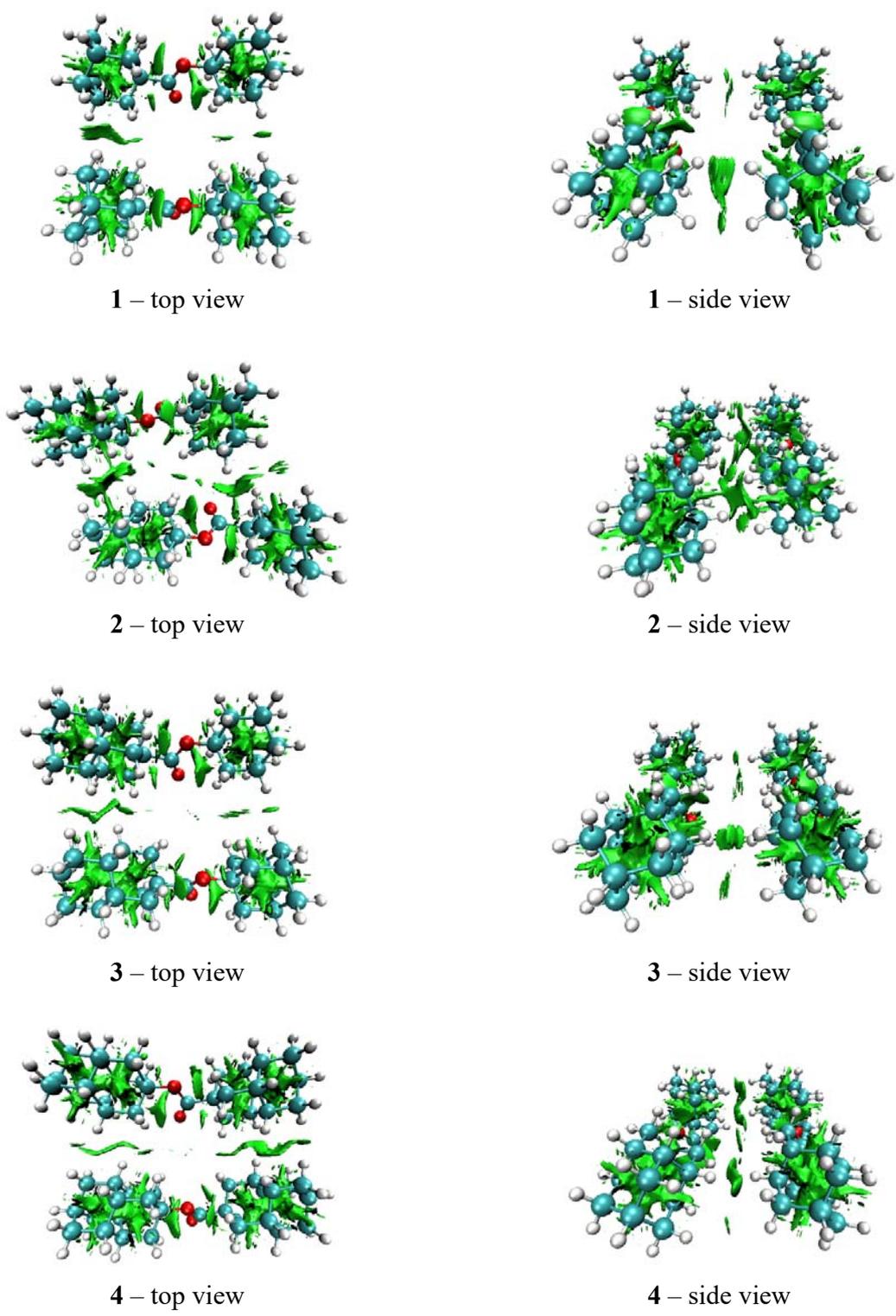
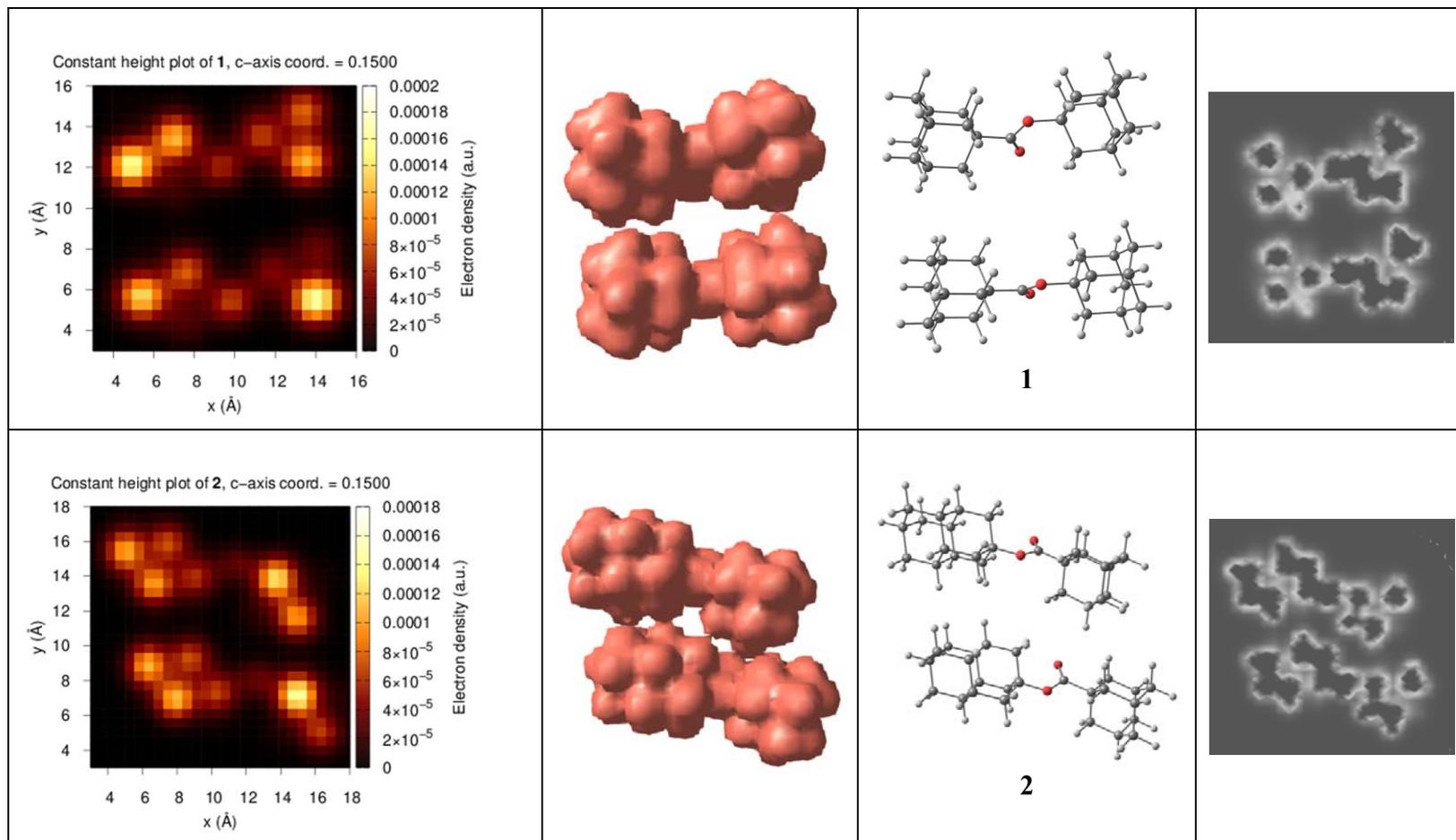


Figure S7. NCI plots of two ester **1–4** molecules positioned in orientation **a_s**. Non-covalent interactions are depicted in green.



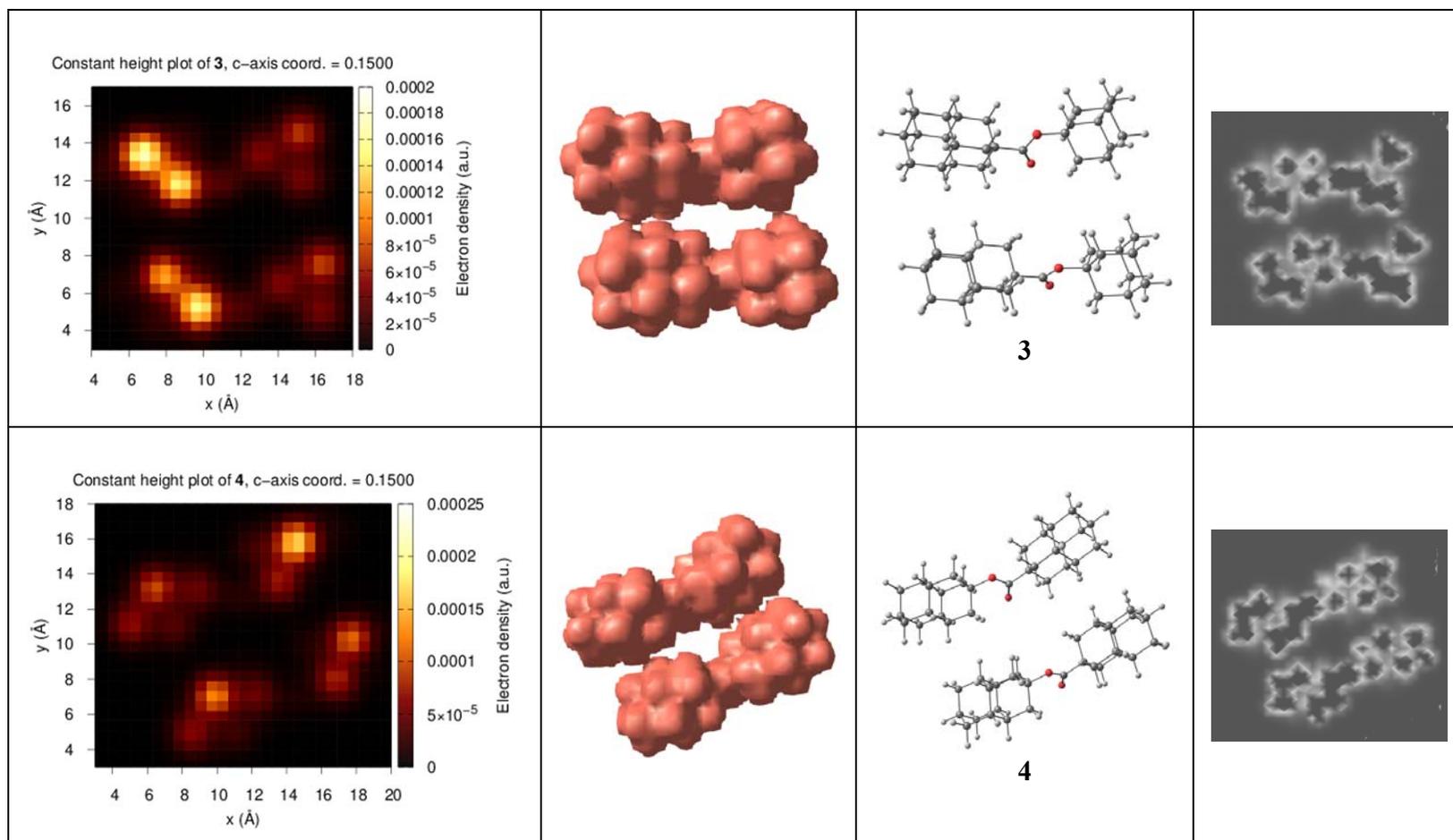


Figure S8. Simulated STM images, electron density distribution, on-surface structures, and 2D mapped plane for electron density (cut from top) of two ester **1–4** molecules. Electron densities obtained from computations at the B3LYP-D3(BJ)/def2-TZVPP level of theory.

Table S2. Electronic energies, zero-point vibrational energies, enthalpies and Gibbs energies of diamondoid esters **1–4** and the hydrogen-terminated graphite cut-out ($C_{216}H_{36}$) in Hartree computed using the GFN2-xTB method.

structure	E	$ZPVE$	H	G
1	-68.569795	0.473935	-68.077301	-68.139748
2	-79.224222	0.545406	-78.658343	-78.724145
3	-79.224250	0.545391	-78.658385	-78.724197
4	-89.877457	0.616916	-89.238180	-89.307681
$C_{216}H_{36}$	-483.030356	1.690411	-481.226080	-481.456984

Table S3. Electronic energies of two ester molecule orientations on the graphite surface (a hydrogen-terminated graphite cut-out, $C_{216}H_{36}$) in Hartree and assembly binding energies in kcal mol⁻¹ computed using the GFN2-xTB method.

structure	E	ΔE^a	structure	E	ΔE^a
1a_s	-620.2164758	-29.2	2a_s	-641.5340741	-34.7
1b_s	-620.2119061	-26.3	2b_s	-641.5259452	-29.6
1c_s	-620.2134259	-27.3	2c_s	-641.5262974	-29.8
1a_a	-620.2154721	-28.6	2a_a	-641.5335672	-34.4
1b_a	-620.2106984	-25.6	2b_a	-641.5265697	-30.0
1c_a	-620.2150176	-28.3	2c_a	-641.5260759	-29.7
3a_s	-641.5296347	-31.9	4a_s	-662.8454416	-37.8
3b_s	-641.5260694	-29.6	4b_s	-662.8374749	-32.8
3c_s	-641.5273804	-30.4	4c_s	-662.840055	-34.4
3a_a	-641.530166	-32.2	4a_a	-662.8447593	-37.3
3b_a	-641.5260405	-29.6	4b_a	-662.8396774	-34.1
3c_a	-641.5260869	-29.6	4c_a	-662.8406419	-34.7

^a Binding energies are defined as a difference between the electronic energy of the assembly and the energies of the corresponding ester molecules and the hydrogen-terminated graphite cut-out.

Table S4. Geometries of structures in Cartesian coordinates in Å computed using the GFN2-xTB method.

C₂₁₆H₃₆			
6	13.531200000	-0.683000000	-0.003200000
6	13.531300000	0.679100000	-0.002100000
6	12.309000000	1.429900000	-0.000200000
6	12.298200000	2.819300000	0.000500000
6	11.081800000	3.554200000	0.001800000
6	11.065900000	4.956600000	0.002000000
6	9.854000000	5.680800000	0.003400000
6	9.832900000	7.092600000	0.003000000
6	8.626400000	7.807700000	0.004800000
6	8.598300000	9.228600000	0.003400000
6	7.400500000	9.932700000	0.005200000
6	7.361600000	11.366700000	0.002100000
6	6.182000000	12.047800000	0.003500000
6	12.297900000	-2.823100000	-0.003900000
6	12.308800000	-1.433700000	-0.002500000
6	11.076600000	-0.707700000	-0.000700000
6	11.076700000	0.704000000	0.000400000
6	9.849200000	1.419600000	0.001500000
6	9.846900000	2.836500000	0.002400000
6	8.620600000	3.549100000	0.003400000
6	8.617300000	4.967000000	0.004600000
6	7.391200000	5.678900000	0.006400000
6	7.387300000	7.097200000	0.007300000
6	6.161500000	7.807700000	0.009500000
6	6.155700000	9.228500000	0.009200000
6	4.933100000	9.934500000	0.010900000
6	4.920700000	11.364600000	0.008300000
6	3.712000000	12.050000000	0.009000000
6	11.065400000	-4.960200000	-0.004900000
6	11.081400000	-3.557800000	-0.003500000
6	9.846600000	-2.840000000	-0.002000000
6	9.849000000	-1.423100000	-0.000800000
6	8.618900000	-0.710400000	-0.000400000
6	8.619000000	0.707000000	0.000800000
6	7.389600000	1.419400000	0.000400000
6	7.388800000	2.837200000	0.002300000
6	6.160300000	3.548600000	0.002700000
6	6.158700000	4.968200000	0.006000000
6	4.931300000	5.677900000	0.008100000
6	4.929200000	7.098800000	0.010800000
6	3.701700000	7.807600000	0.013000000
6	3.699700000	9.229200000	0.013400000
6	2.471500000	9.935700000	0.014500000

6	2.467400000	11.364000000	0.012100000
6	1.245000000	12.051400000	0.011700000
6	9.832100000	-7.096100000	-0.005500000
6	9.853300000	-5.684400000	-0.004300000
6	8.616800000	-4.970400000	-0.002800000
6	8.620200000	-3.552500000	-0.002200000
6	7.388500000	-2.840500000	-0.002400000
6	7.389500000	-1.422700000	-0.002000000
6	6.159400000	-0.711300000	-0.004800000
6	6.159500000	0.708200000	-0.003700000
6	4.930400000	1.418600000	-0.008400000
6	4.930100000	2.838400000	-0.002800000
6	3.700800000	3.548800000	-0.003100000
6	3.700200000	4.968400000	0.005400000
6	2.471000000	5.678200000	0.008400000
6	2.470100000	7.099100000	0.013200000
6	1.241800000	7.807300000	0.015500000
6	1.241200000	9.230000000	0.016100000
6	0.011700000	9.936100000	0.016500000
6	0.011700000	11.364000000	0.013700000
6	-1.221400000	12.051600000	0.011900000
6	8.597300000	-9.232000000	-0.005400000
6	8.625500000	-7.811100000	-0.004300000
6	7.386500000	-7.100500000	-0.002400000
6	7.390500000	-5.682100000	-0.002200000
6	6.158100000	-4.971300000	-0.002100000
6	6.159900000	-3.551700000	-0.003200000
6	4.929800000	-2.841400000	-0.006900000
6	4.930300000	-1.421600000	-0.010000000
6	3.700300000	-0.711000000	-0.021500000
6	3.700300000	0.708300000	-0.021400000
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6	2.470400000	2.838700000	-0.015600000
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6	0.011400000	5.678600000	0.009700000
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6	-1.218000000	9.230200000	0.017900000
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6	4.928400000	-7.101800000	-0.000300000

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6	1.240700000	0.708800000	-0.047600000
6	0.011100000	1.418700000	-0.045100000
6	0.011200000	2.838300000	-0.022300000
6	-1.218800000	3.548400000	-0.008400000
6	-1.218500000	4.969000000	0.006900000
6	-2.448200000	5.678500000	0.014100000
6	-2.447100000	7.099400000	0.018300000
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6	2.470300000	-5.680900000	-0.001600000
6	1.240600000	-4.971500000	-0.004700000
6	1.240800000	-3.550900000	-0.016400000
6	0.010900000	-2.840700000	-0.027600000
6	0.010900000	-1.421100000	-0.047400000
6	-1.218700000	-0.710900000	-0.052700000
6	-1.218800000	0.708800000	-0.050400000
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6	2.466100000	-11.366700000	0.000800000
6	2.470400000	-9.938400000	0.002200000
6	1.240100000	-9.232600000	0.003900000
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6	0.010700000	-7.101500000	0.004500000

6	0.010800000	-5.681000000	0.000000000
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6	-1.219000000	-3.550800000	-0.015600000
6	-2.448100000	-2.841000000	-0.017700000
6	-2.448200000	-1.420600000	-0.035300000
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6	-3.678000000	0.708800000	-0.020200000
6	-4.908000000	1.418900000	0.001800000
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6	-6.137700000	3.549200000	0.018800000
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6	-7.368300000	5.679700000	0.021900000
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6	-7.366300000	2.838000000	0.021500000
6	-8.598000000	3.550100000	0.023100000
6	-8.594600000	4.967900000	0.022000000
6	-9.831100000	5.681900000	0.019200000
6	-9.809900000	7.093700000	0.016400000
6	-1.222800000	-12.053900000	0.002900000
6	-2.445200000	-11.366500000	0.005600000
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6	-3.677500000	-9.231700000	0.011500000
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3b a

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