Supporting Information

Constraining Carbon Nanothread Structures by Experimental and Calculated Nuclear Magnetic Resonance Spectra

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Rescaling of the CH-only Spectrum

In this section, we describe how for fitting purposes a "thread-only" 1D NMR spectrum, without aromatic signals, can be obtained by analyzing and rescaling two experimental spectra which also contain signals of other moieties. In the experimental ^{13}C NMR spectrum of alkene (sp^2) adjacent to alkyl CH (sp^3), the alkene component is assumed to originate entirely from isolated C=C double bonds within degree-4 nanothreads that are bonded to alkyl (i.e. sp^3) carbon atoms. Meanwhile, the sp^3 component of the CH-only spectrum is assumed to originate entirely from sp^3 carbon atoms that reside in degree-4 or degree-6 threads, while both degree-4 threads *and* aromatic rings can contribute to the sp^2 carbon component of the CH-only spectrum. Since computationally we consider only ideal degree-4 and degree-6 threads, for fitting purposes we constructed a "thread-only" NMR spectrum by combining the sp^2 part of the alkene-plus-nearby-alkyl spectrum with the sp^3 part of the –CH-only spectrum in appropriate proportions (i.e. excluding residual aromatic rings). The latest NMR measurements indicate that 11% of the carbon atoms are alkene, which leads to 33% degree-4 threads where one-third (11% of 33%) of carbons are sp^2 and the remaining two-thirds (22% of the 33%) are sp^3. The experimental analysis indicates that ~32% carbons reside in degree-6 threads.[1] Thus, the fraction of carbon atoms that are nanothread-resident and sp^3 is 22+32=54%, while 11% are nanothread-resident and sp^2. Therefore, we rescaled the whole CH-only spectrum such that the ratio of the sp^3 carbon (in the frequency range of 10–85 ppm) in the rescaled CH-only spectrum to the sp^2 carbon (in the frequency range of 85–160 ppm) in the spectrum of alkene and nearby alkyl CH is

54:11. Then we combined the sp^2 regime (85–160 ppm) of spectrum of alkene and nearby alkyl and the sp^3 regime (10–85 ppm) of the rescaled CH-only spectrum to obtain the "thread-only" spectrum for fitting analysis.

Methods for Generating Axially Disordered Threads

Fully axially disordered degree-6 nanothreads (with class 2 or class 3 bonding patterns[2]) and degree-4 nanothreads were generated by starting from an ordered configuration within a large crystallographic unit cell and performing Stone-Wales transformations about C-C bonds in the six-fold carbon rings corresponding to those of the benzene precursor (these transformations serve to swap two adjacent polymerization bonds that project from a given ring to neighboring progenitor benzene rings). Since we wish to generate a test case with maximal disorder, such transformations were accepted with a generous acceptance probability depending on the corresponding energy change in accord with the usual Metropolis scheme (using an effective temperature of ~20 eV). With this method, the bonding configurations were sampled ergodically and relaxed into local energy minima, with an energy weighting that only slightly favored configurations of lower energy– all structures so generated a structurally plausible, kinetically stable local minimum. The axial unit cell lengths of these threads were optimized by 0.01 Å step-size scanning using VASP with PBE density functional.

The formation of a nanothread can be viewed as proceeding through a succession of intermediate polymers of increasing degree of saturation (i.e. degree 2, degree 4, degree 6), as discussed in Chen et. al.,[3] where the transformation from one polymer to the next occurs through a series of radical additions or cycloaddition reactions between the benzene rings. Therefore, it is of interest to investigate the NMR spectra of nanothreads disordered but subject to the constraint that they are derivable from [4+2] additions, including $[4_s+2_s]$ and $[4_a+2_a]$. For the degree 4

nanothread we impose the constraint that it can be formed entirely via [4+2] polymerization. Since the transformation from degree 4 to degree 6 polymers occurs through double bond zipping (Chen et. al.[3]), for a fully saturated nanothread we impose the constraint that it is derivable from an intermediate degree-4 polymer that in turn can be formed entirely via [4+2] polymerization.

The generation of disordered nanothreads proceeds as previously described, but in the constrained case the Stone-Wales transformations are restricted to certain C-C bonds within a ring. Specifically, a degree-4 part of the structure is defined that is derivable from [4+2] additions, and the allowed transformations preserve this property of the degree-4 part. The possible bonding structures subject to this constraint are then ergodically sampled, with an energy weighting such that lower energy structures are slightly favored (as described previously).

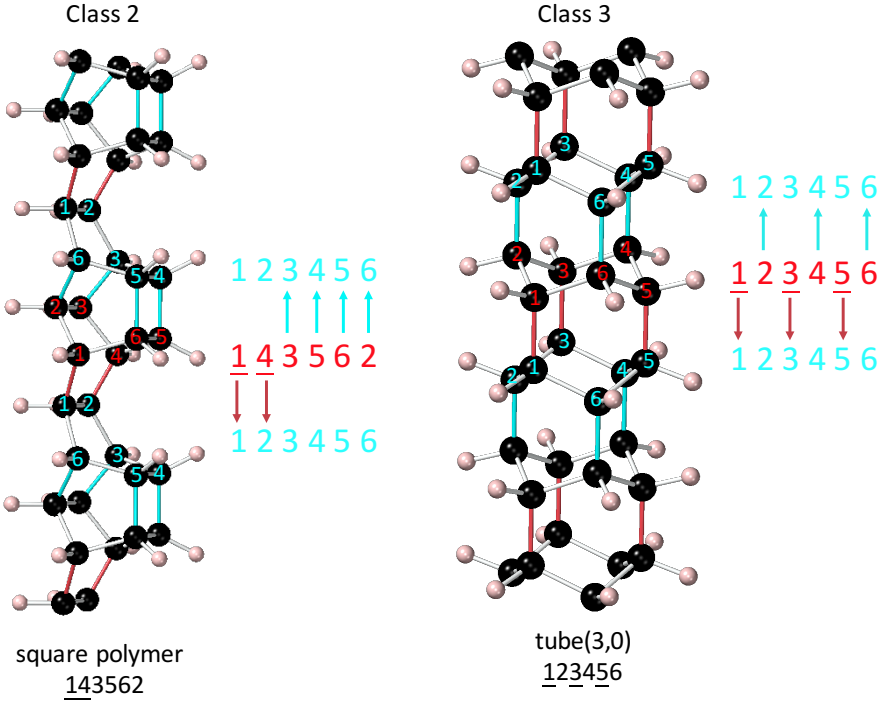

Figure S1: Square polymer and tube (3,0) as examples of degree-6 threads with class 2 and class 3 bonding patterns. The successive progenitor benzene rings share 2(red), 4(cyan), 2, 4…bonds or 3(red), 3(cyan), 3, 3 bonds with each other for class 2 and class 3 threads, respectively. The identifier of a thread gives the atom numbers of the middle benzene ring (red digits) bonded to neighbors as marked with underlines for atoms bonding downward; this is sufficient since the topological unit cell contains only two progenitor benzene rings (i.e., the top benzene ring (cyan digits) is the same as the bottom one (cyan digits)).

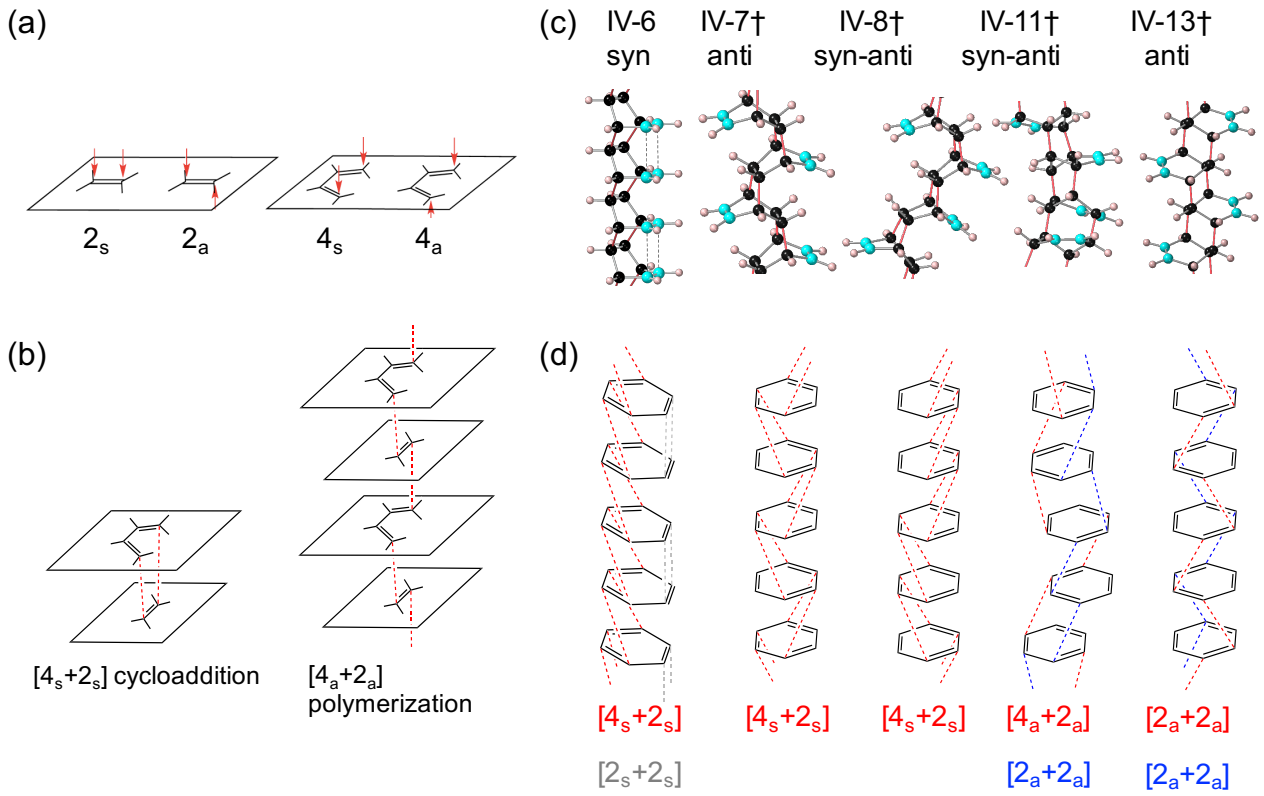

Figure S2: (a) Modes of addition to π bonds. In the nomenclature, the number denotes how many π electrons are involved, and the subscript indicates whether the addition is on the same face (*supra*, subscript s) or on the opposite face (*antara*, subscript a). (b) Two examples of addition to π bonds, $[4_s+2_s]$ cycloaddition and $[4_a+2_a]$ polymerization. (c) DFT-optimized

structures of five degree-4 nanothreads[3] with new formed bonds colored red. The left four structures are the most likely candidates for the sp^2 components of the synthesized products, based on the comparison of calculated and experimental NMR data. The sp^2 carbon atoms are colored cyan. IV-6 collapses upon relaxation to square polymer with new bonds marked by dashed gray lines. (d) Topological relationships of the five threads with respect to the parent benzene molecules, illustrated by color-coded reaction schemes. IV-6, IV-7 and IV-8 can be formed by a series of $[4_s+2_s]$ cycloadditions. IV-11 can be formed by sequential $[4_a+2_a]$ and $[2_a+2_a]$ polymerizations, and IV-13 two sequential $[2_a+2_a]$ polymerizations.

Determining the Reference Isotropic Chemical Shielding

Figure S3 shows the linear regression for the calculated isotropic chemical shielding with respect to the experimental isotropic chemical shifts for several partially and fully saturated hydrocarbon compounds.[4-6] The slope of the best fitting line is 1.05, which is approximately unity and indicates that the adopted approach is reasonable for investigating the chemical shifts for nanothreads. The computed isotropic chemical shift is obtained as

$$\delta_{\mathrm{iso}}^{\mathrm{calc}} = \frac{\sigma_{\mathrm{iso}}^{\mathrm{ref}} - \sigma_{\mathrm{iso}}^{\mathrm{calc}}}{m} \tag{4}$$

where the reference isotropic chemical shielding $\sigma_{\mathrm{iso}}^{\mathrm{ref}}$ is extrapolated to be 169.37 ppm.

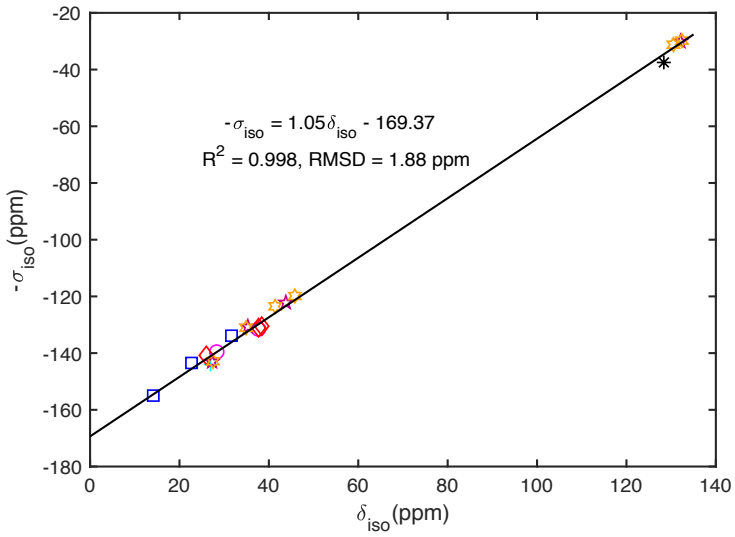

Figure S3: Linear fitting for ^{13}C calculated isotropic chemical shielding for a set of hydrocarbon compounds vs. their experimental isotropic chemical shifts.

Solid-state NMR Experiments

Solid-state NMR experiments were performed on a Bruker Advance DSX400 spectrometer at a ^{13}C resonance frequency of 100 MHz, using a Bruker double-resonance 4-mm magic-angle spinning (MAS) probe head. The ~1-mg of carbon nanothreads, made from uniformly ^{13}C-enriched benzene in a Paris-Edinburgh pressure cell,[7] was center-packed between cylindrical glass and hollow KelF spacers in a 4-mm rotor. Uniformly ^{13}C-enriched valine, unlabeled valine, and polystyrene (PS) 20% ^{13}C-labeled in the backbone CH were center-packed with one glass spacer. It is a favorable coincidence that the PS CH isotropic shift near 40 ppm is a good match for the carbon nanothread signal maximum. ^{1}H and ^{13}C 90° pulse lengths were 4.2 μs, and ^{1}H TPPM decoupling[8] was applied at $|\gamma B_1|/2\pi = 60$ kHz. The detection time was 5 ms. The recycle delay was 1.5 s unless otherwise stated.

A CH-only spectrum was obtained by dipolar DEPT[9], see Figure S4 (a), at 5.787 kHz MAS, with 6144 scans. ^{1}H magnetization is converted to heteronuclear multiple quantum (MQ)

coherence with ^{13}C. Subsequently, under the influence of the ^{13}C-^{1}H dipolar coupling, ^{13}C-^{1}H spin-pair MQ coherence is invariant and therefore retained, while ^{13}C-^{1}H MQ coherence in a CH_2 group is dephased by the second ^{1}H. The surviving MQ coherence is converted to ^{13}C magnetization, which is detected after total suppression of spinning sidebands (TOSS).[10]

A two-dimensional (2D) ^{1}H-^{13}C heteronuclear correlation (HetCor) spectrum[11] was measured at 7.5 kHz MAS with frequency-switched Lee-Goldburg homonuclear decoupling[12] and TOSS before detection, see Figure S4 (b). The Hartmann-Hahn cross polarization (HHCP) time was 0.1 ms. 100 transients in the ^{1}H dimension and 64 transients in the ^{13}C dimension were collected. The alkene plus nearby alkyl CH subspectrum in Figure 1(a) was obtained as a slice taken at a ^{1}H chemical shift of 5.4 ppm.

Static spectra were measured using multiCP without sample spinning. A Hahn spin echo generated by an 180° pulse[13] with EXORCYCLE phase cycling[14] was used to avoid pulse dead-time effects. A minimal echo delay of $2\tau = 33$ μs was used to minimize loss from T_2 relaxation. 4096 scans were collected for nanothreads, and 64 scans for $^{13}C\alpha$-polystyrene. Static spectra with homonuclear decoupling were first measured as 2D ^{13}C-^{13}C, with t_1 evolution under moderately strong MREV-8 homonuclear decoupling[15] and strong ^{1}H decoupling, see Figure S4 (c), while during detection (t_2 dimension) only ^{1}H decoupling was applied. 48 increments of 52.4 μs were collected in the t_1 dimension and 128 t_2 transients were averaged. After the 2D spectrum had been obtained by double Fourier transformation, the homonuclear decoupled 1D spectra were obtained by projecting the 2D spectrum onto the F_1 dimension.

The 5-pulse CSA recoupling data shown in Figure 2 were measured using 1-ms CP followed by a "five-pulse" chemical shift anisotropy (CSA) filter,[16] see Figure S4 (d), at 5 kHz MAS. Each of the three 180° pulses was fully EXORCYCLE phase cycled[14]. The recoupling time t_{csa}

was incremented from 0 to 100 μs, in 10-μs steps. 2016 scans were collected for each recoupling time for nanothreads, 504 scans for natural-abundance valine, and 168 scans for ^{13}C-valine and ^{13}Cα-polystyrene.

A two-dimensional double quantum/single quantum (DQ/SQ, solid-state INADEQUATE) NMR spectrum was measured at 14 kHz MAS after long CP with ~0.5 ms of ^{13}C-^{13}C dipolar recoupling by SPC5[17] without ^{1}H irradiation, relying on ^{13}C irradiation at $|\gamma B_1|/2\pi$ = 70 kHz for heteronuclear decoupling,[18] see Figure S4 (e). A corner of such a spectrum of carbon nanothreads after 45° vertical shearing[18] is shown in Figure S11.

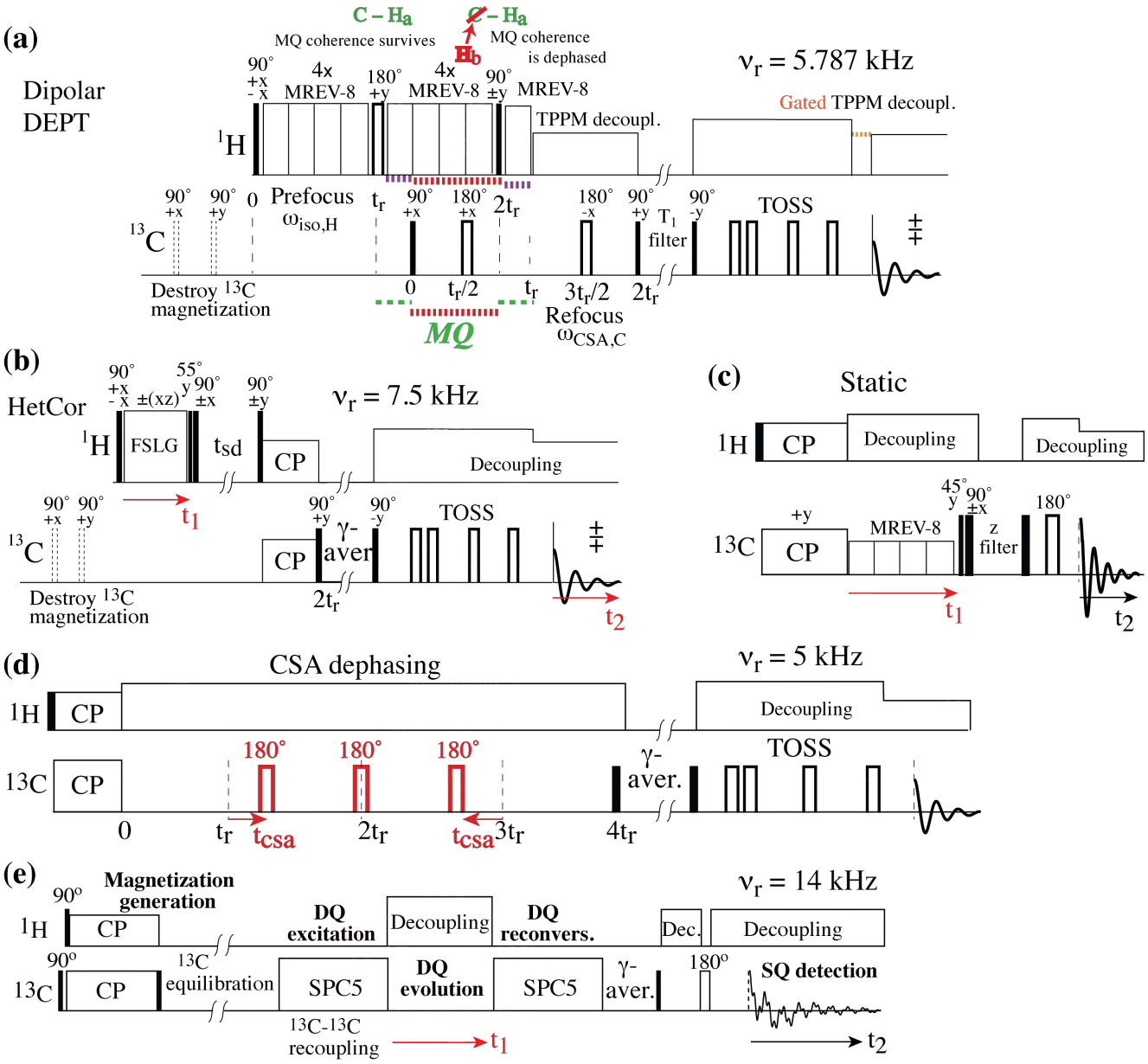

Figure S4: Solid-state NMR pulse sequences used to obtain the experimental spectra shown in this work. (a) Dipolar DEPT for selection of CH (methine) ^{13}C NMR signals with MREV-8[15] ^{1}H homonuclear decoupling, at 5787 Hz MAS.[9] A second spectrum recorded after dipolar dephasing by gating decoupling off can be used to remove the residual signals of nonprotonated carbons, but was not needed for nanothreads. (b) ^{1}H-^{13}C heteronuclear correlation (HetCor)[11] with frequency-switched Lee-Goldburg (FSLG) homonuclear ^{1}H decoupling,[12]spin diffusion during t_{sd}, short cross polarization (CP) to ^{13}C, and ^{13}C detection. Spinning frequency: 7.5 kHz. (c)

Measurement of the ^{13}C chemical-shift anisotropy powder spectrum without sample rotation under homonuclear decoupling by MREV-8 in the first dimension of a 2D experiment. (d) ^{13}C chemical-shift anisotropy dephasing by three 180° recoupling pulses and two 90° pulses flanking a γ-averaging period incremented in 4 steps of $t_r/4$.[16] Total suppression of spinning sidebands (TOSS)[10] is applied before detection. (e) Two-dimensional double-quantum/single-quantum experiment for obtaining cross peaks of bonded ^{13}C-^{13}C spin pairs.

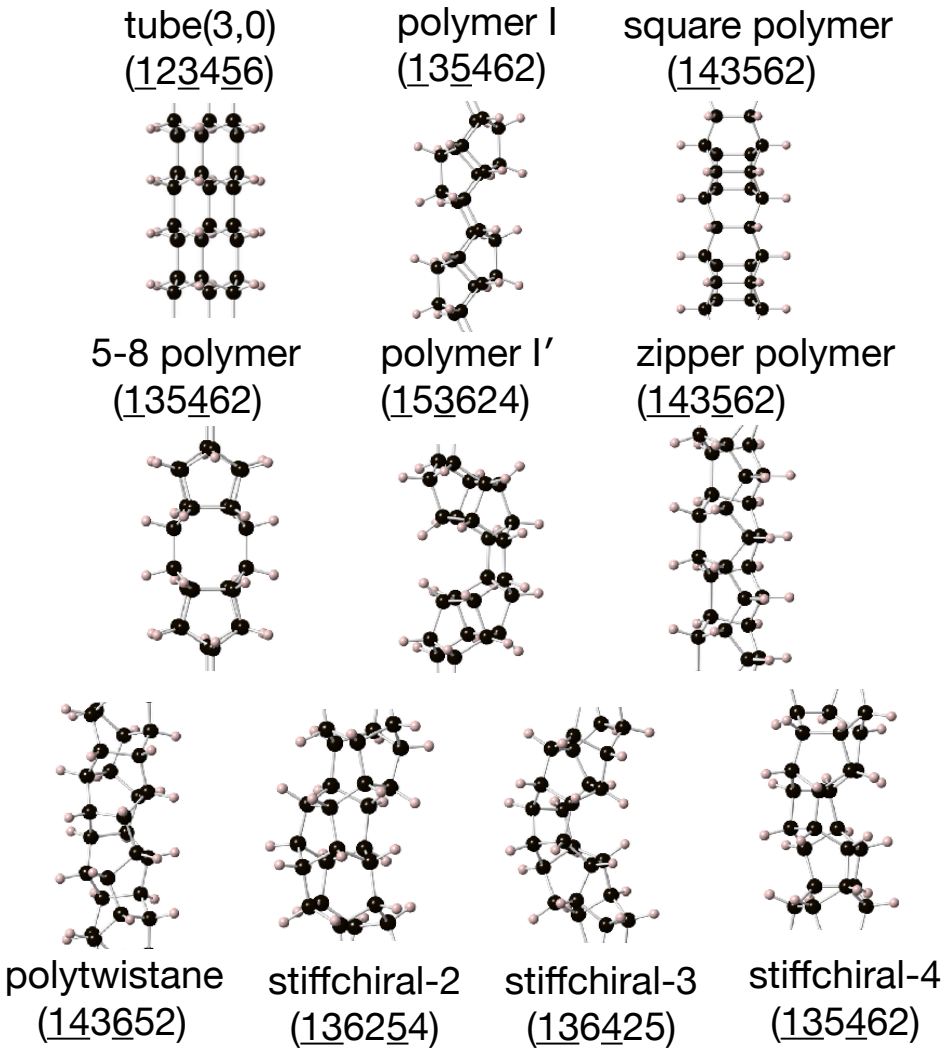

Figure S5: DFT-optimized structures of degree-6 nanothreads with both informal (top) and formal (bottom) names based on the structural and bonding features.[2]

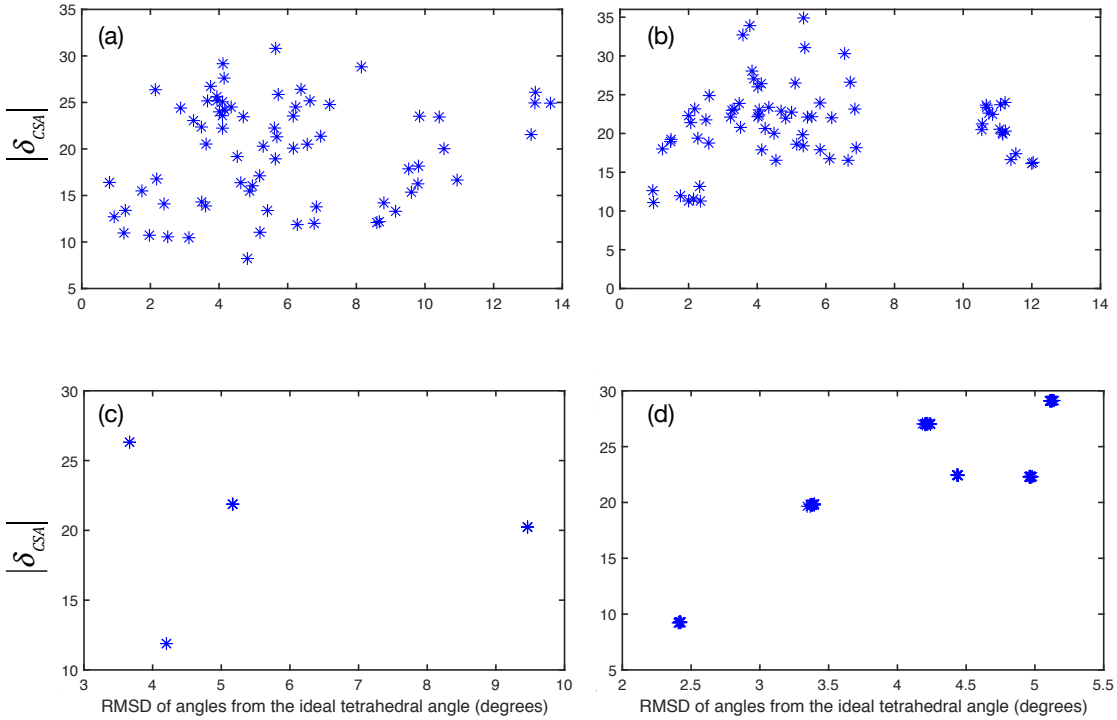

Figure S6: Calculated correlation between the root mean square deviation (RMSD) of the six angles from the ideal tetrahedral angle for a given carbon site and the magnitude of the site's chemical shift anisotropy (CSA) for (a) axially fully disordered degree-6 class-3 thread; (b) axially disordered degree-6 class-3 thread generated with a [4+2] constraint; (c) degree-6 square polymer and (d) degree-6 stiffchiral-3 thread.

Figure S6 indicates that deviation from tetrahedral bonding symmetry around C-H carbons in nanothreads correlates only weakly with the magnitude of the site's chemical shift anisotropy (CSA).

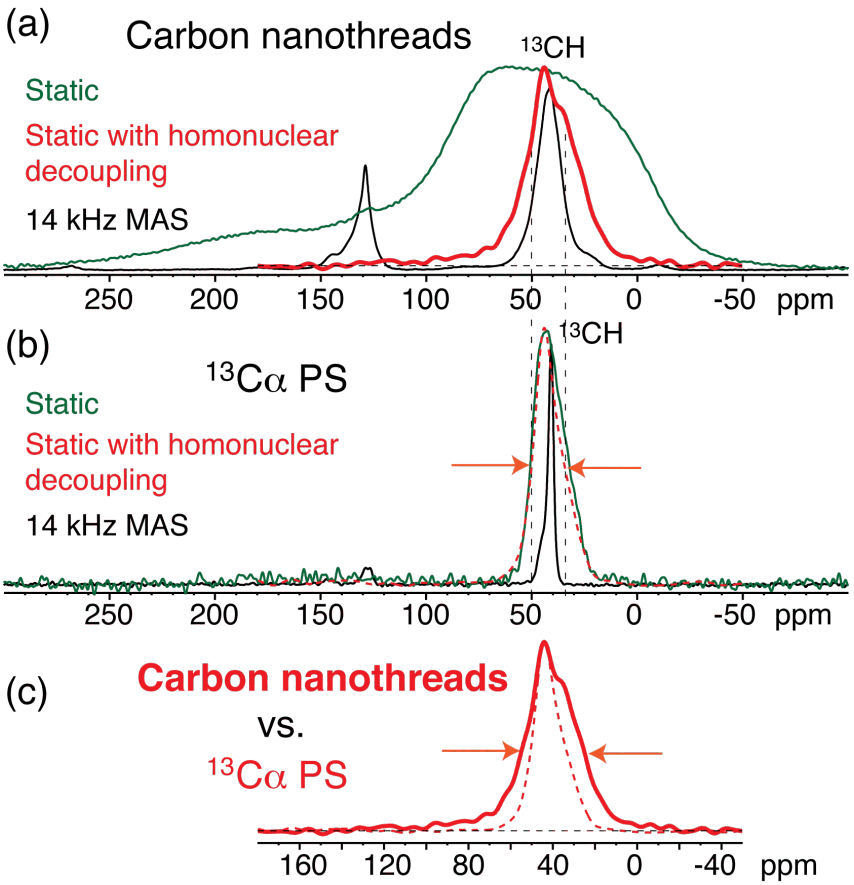

Figure S7: Chemical-shift anisotropy in carbon nanothreads and in ^{13}Cα-enriched polystyrene (PS). (a-c) Comparison of anisotropic broadening of the 41 ppm CH peak in static spectra of carbon nanothreads and in ^{13}C-enriched PS. (a) Carbon nanothreads; (b) backbone ^{13}CH-enriched PS. (c) Comparison of homonuclear decoupled anisotropy pattern of PS (dashed) and carbon nanothreads (solid line).

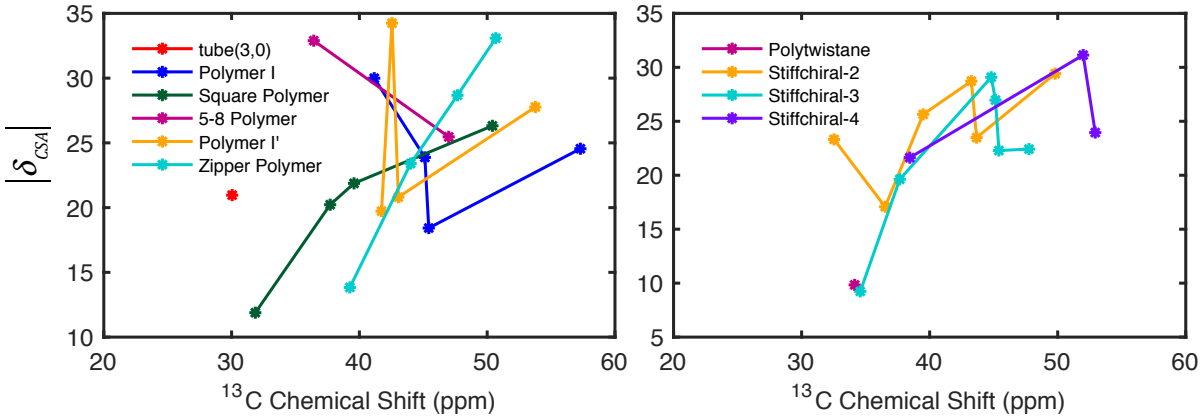

Figure S8: The calculated magnitude of the chemical shift anisotropy ($|\delta_{CSA}|$) tends to increase when the chemical shift (CS) value increases for square polymer, zipper polymer and stiffchiral-3.

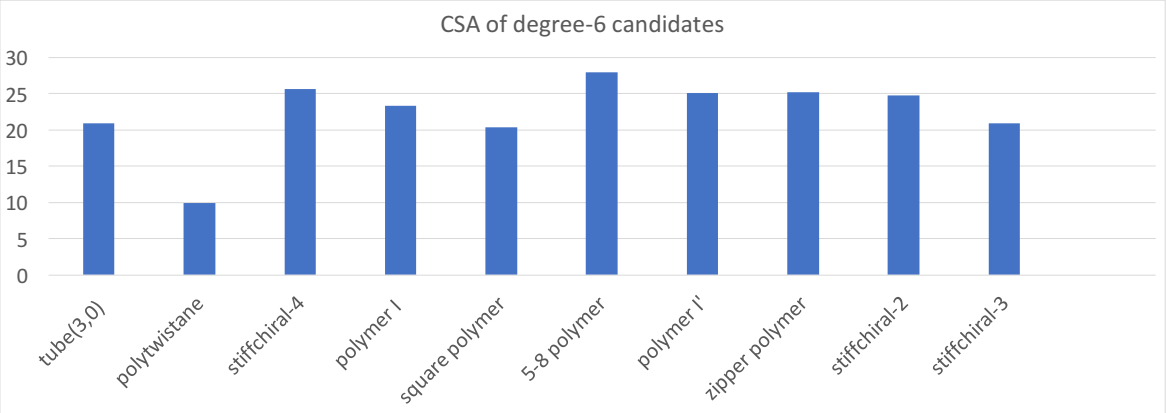

Figure S9: The calculated average magnitude of the CSA of the six degree-6 candidates. Tube (3,0), square polymer and stiffchiral-3 have a magnitude of CSA around 20 ppm, consistent with the experimental measurement. Polymer I cannot be ruled out only based on the magnitude of its CSA, which is slightly larger than 20 ppm. The others have too-large or too-small magnitudes and can thus likely be ruled out as dominant components.

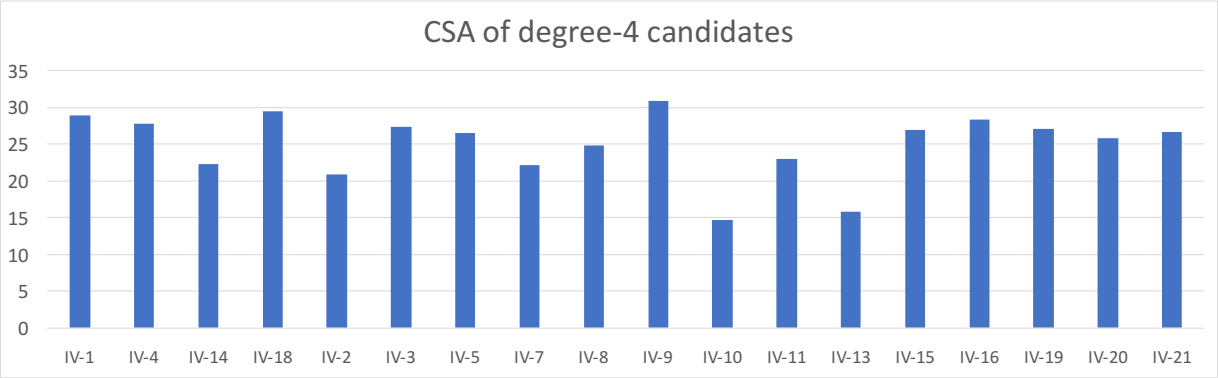

Figure S10: The calculated average magnitude of CSA of the 14 degree-4 candidates. The average magnitude of the CSA for IV-2, IV-7, IV-8, IV-11, IV-13 and IV-14 are in the range of 15–25 ppm, which is acceptable compared with experimental measurement (20 ppm). The sign of the CSA should also be considered when assessing the plausibility of a candidate thread. The other degree-4 threads have much smaller or larger magnitudes of the CSA and are thus less likely to be the candidates of the sample.

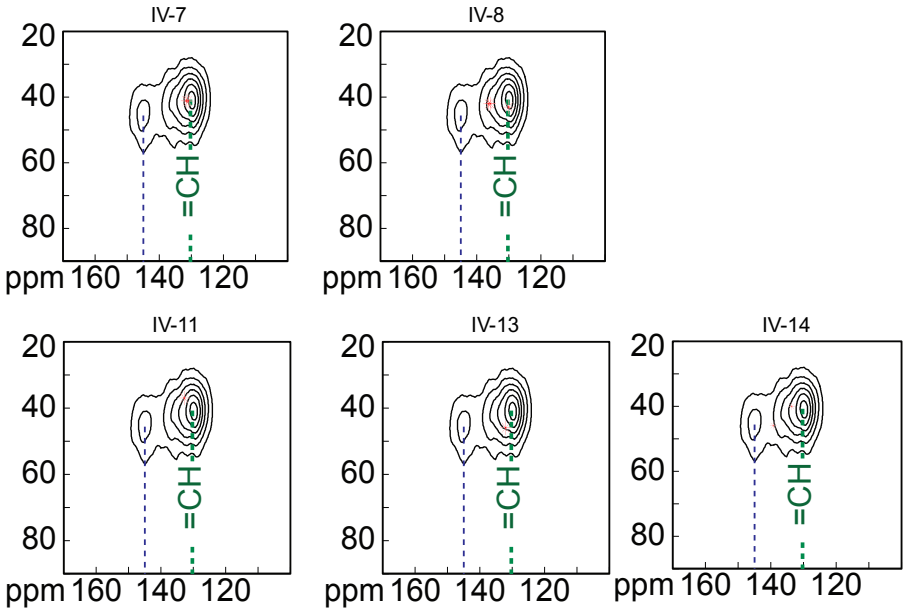

Figure S11: Comparison between calculated and measured isotropic chemical shifts for bonded sp^2 and sp^3 carbon sites for degree-4 IV-7, IV-8, IV-11, IV-13 and IV-14.

Table S1: Calculated isotropic chemical shifts and chemical shielding anisotropy parameters for degree-6 and degree-4 threads.

Degree-6 polymer	δ_{iso}(ppm)	symmetry-equivalent C atoms in a unit cell	symmetry-equivalent C atoms (%)	σ_{iso}(ppm)	σ_{xx}(ppm)	σ_{yy}(ppm)	σ_{zz}(ppm)	anisotropy parameter δ(ppm)	asymmetry parameter η
tube(3,0)	30.07	12	100	137.80	124.10	130.50	158.80	-20.97	0.3052
polymer I	41.16	2	16.67	126.20	106.10	116.30	156.10	-30.00	0.3401
	45.12	2	16.67	122.00	101.30	118.80	145.90	-23.88	0.7312
	45.43	4	33.33	121.70	106.80	118.10	140.10	-18.42	0.6157
	57.30	4	33.33	109.20	85.70	108.20	133.80	-24.55	0.9154
square	31.86	2	16.67	135.90	147.40	136.40	124.00	11.89	0.9251
	37.73	4	33.33	129.80	109.70	129.60	150.00	-20.25	0.9819
	39.59	4	33.33	127.80	113.10	120.60	149.70	-21.88	0.3458
	50.41	2	16.67	116.40	134.30	124.90	90.12	26.32	0.3549
5-8 polymer	36.44	8	33.33	131.10	110.40	118.90	164.00	-32.91	0.2560
	47.04	16	66.67	120.00	104.30	110.10	145.50	-25.53	0.2292
polymer I'	41.78	8	33.33	125.50	142.80	128.00	105.70	19.80	0.7443
	42.60	4	16.67	124.60	102.20	112.80	158.90	-34.30	0.3084
	43.08	4	16.67	124.10	105.30	122.10	145.00	-20.83	0.8083
	53.83	8	33.33	112.80	89.72	108.10	140.70	-27.85	0.6610
ipper polyme	39.27	6	16.67	128.10	116.90	125.60	142.00	-13.84	0.6289
	44.01	12	33.33	123.20	105.50	117.40	146.60	-23.40	0.5059
	47.69	12	33.33	119.30	92.92	117.00	148.00	-28.67	0.8397
	50.69	6	16.67	116.20	133.60	131.80	83.06	33.09	0.0522
polytwistane	34.31	72	100	133.30	124.90	131.90	143.30	-9.94	0.6995
stiffchiral-2	32.68	18	16.67	135.10	121.10	125.60	158.50	-23.39	0.1937
	36.70	18	16.67	130.80	116.30	128.10	148.00	-17.22	0.6867
	39.90	18	16.67	127.50	142.90	138.00	101.50	26.00	0.1889
	43.41	18	16.67	123.80	148.50	128.00	94.90	28.89	0.7095
	43.76	18	16.67	123.40	146.10	124.20	100.00	23.42	0.9359
	49.92	18	16.67	117.00	139.80	123.80	87.22	29.73	0.5380
stiffchiral-3	34.65	22	16.67	133.00	124.10	132.60	142.30	-9.29	0.9208
	37.74	22	16.67	129.70	143.80	135.50	109.90	19.80	0.4172
	44.90	22	16.67	122.20	99.11	116.20	151.40	-29.15	0.5859
	45.44	44	33.33	121.70	143.70	121.90	99.40	22.26	0.9800
	47.80	22	16.67	119.20	97.85	118.00	141.60	-22.46	0.8995
stiffchiral-4	38.53	16	33.33	128.90	107.60	128.50	150.60	-21.68	0.9615
	52.08	16	33.33	114.70	144.20	116.20	83.66	31.03	0.9046
	53.07	16	33.33	113.70	91.71	111.50	137.80	-24.12	0.8190

Degree-4 polymer	δ_{iso}(ppm)	symmetry-equivalent C atoms in a unit cell	symmetry-equivalent C atoms (%)	σ_{iso}(ppm)	σ_{xx}(ppm)	σ_{yy}(ppm)	σ_{zz}(ppm)	anisotropy parameter δ(ppm)	asymmetry parameter η
IV-1	44.50	2	16.67	122.70	97.55	118.50	151.90	-29.27	0.7154
	50.12	2	16.67	116.70	89.47	114.40	146.40	-29.63	0.8405
	52.10	2	16.67	114.70	91.98	104.90	147.20	-32.49	0.3964
	53.69	2	16.67	113.00	133.90	116.40	88.77	24.23	0.7214
	126.10	2	16.67	37.00	-68.06	24.34	154.70	-117.70	0.7849
	141.40	2	16.67	20.95	-96.52	13.04	146.30	-125.40	0.8740
IV-2	32.64	4	16.67	135.10	125.10	132.60	147.60	-12.53	0.5986
	41.47	2	8.333	125.80	148.30	127.10	102.10	23.78	0.8918
	41.95	2	8.333	125.30	147.00	127.50	101.40	23.91	0.8160
	43.32	4	16.67	123.90	106.20	122.20	143.30	-19.42	0.8226
	46.86	2	8.333	120.20	143.40	124.80	92.26	27.91	0.6670
	47.26	2	8.333	119.80	142.70	124.00	92.57	27.18	0.6893
	128.40	2	8.333	34.57	149.40	36.30	-82.04	116.60	0.9703
	129.20	2	8.333	33.69	-81.42	32.93	149.60	-115.90	0.9868
	135.30	2	8.333	27.26	145.50	30.27	-94.04	121.30	0.9504
	135.90	2	8.333	26.64	145.50	26.83	-92.43	119.10	0.9969
IV-3	34.23	1	8.333	133.40	103.90	133.30	163.10	-29.66	0.9908
	45.78	1	8.333	121.30	105.30	116.50	142.10	-20.79	0.5421
	46.30	1	8.333	120.80	101.70	117.30	143.40	-22.60	0.6898
	46.61	1	8.333	120.40	95.46	114.80	151.00	-30.55	0.6345
	46.99	1	8.333	120.00	106.20	108.20	145.60	-25.60	0.0794
	52.87	1	8.333	113.90	142.50	118.60	80.47	33.39	0.7142
	54.99	1	8.333	111.60	87.32	109.20	138.40	-26.77	0.8163
	59.30	1	8.333	107.10	78.59	106.00	136.70	-29.57	0.9280
	125.60	1	8.333	37.52	-73.96	36.11	150.40	-112.90	0.9749
	128.70	1	8.333	34.23	143.70	45.05	-86.11	120.30	0.8202
	131.00	1	8.333	31.81	136.00	44.01	-84.60	116.40	0.7904
	138.30	1	8.333	24.14	-95.90	22.04	146.30	-122.10	0.9657
IV-4	36.40	2	8.333	131.20	107.10	130.60	155.70	-24.58	0.9568
	38.23	2	8.333	129.20	113.40	128.60	145.70	-16.48	0.9211
	42.30	2	8.333	125.00	106.80	120.40	147.70	-22.72	0.5995
	46.59	2	8.333	120.50	141.80	124.60	95.01	25.44	0.6758
	51.78	2	8.333	115.00	87.83	113.40	143.80	-28.77	0.8889
	52.74	2	8.333	114.00	147.90	115.20	78.84	35.15	0.9294
	54.18	2	8.333	112.50	140.60	115.40	81.45	31.03	0.8133
	59.81	2	8.333	106.60	76.13	99.25	144.30	-37.76	0.6122
	127.50	2	8.333	35.46	138.60	41.07	-73.23	108.70	0.8969
	128.60	2	8.333	34.31	-77.84	33.11	147.70	-113.40	0.9788
	132.40	2	8.333	30.30	141.90	37.97	-88.93	119.20	0.8714
	143.10	2	8.333	19.15	-102.70	14.54	145.60	-126.50	0.9271

Degree-4 polymer	δ_{iso}(ppm)	symmetry-equivalent C atoms in a unit cell	symmetry-equivalent C atoms (%)	σ_{iso}(ppm)	σ_{xx}(ppm)	σ_{yy}(ppm)	σ_{zz}(ppm)	anisotropy parameter δ(ppm)	asymmetry parameter η
IV-5	37.91	1	2.778	129.60	112.30	126.60	149.80	-20.20	0.7073
	38.45	1	2.778	129.00	117.30	128.60	141.10	-12.06	0.9331
	38.79	1	2.778	128.60	116.30	127.10	142.40	-13.80	0.7829
	39.10	1	2.778	128.30	110.30	124.40	150.20	-21.93	0.6445
	39.70	1	2.778	127.70	153.00	141.80	88.23	39.45	0.2829
	40.71	2	5.556	126.60	111.20	121.90	146.80	-20.18	0.5304
	42.21	1	2.778	125.00	109.80	123.80	141.60	-16.52	0.8447
	44.49	1	2.778	122.70	98.14	118.40	151.50	-28.80	0.7027
	45.03	2	5.556	122.10	148.40	127.90	89.98	32.11	0.6356
	45.48	2	5.556	121.60	94.50	120.00	150.30	-28.73	0.8888
	46.19	3	8.333	120.90	99.43	114.70	148.50	-27.63	0.5521
	46.86	1	2.778	120.20	95.56	116.20	148.70	-28.57	0.7227
	47.33	1	2.778	119.70	142.60	127.00	89.34	30.33	0.5140
	48.58	2	5.556	118.40	149.30	119.70	86.13	32.23	0.9181
	49.25	1	2.778	117.70	131.60	125.40	95.94	21.72	0.2853
	52.97	1	2.778	113.80	90.15	107.00	144.10	-30.39	0.5536
	53.22	1	2.778	113.50	87.50	107.60	145.30	-31.82	0.6332
	54.97	1	2.778	111.70	89.74	101.50	143.70	-32.02	0.3681
	126.20	1	2.778	36.89	144.70	42.78	-76.77	113.70	0.8964
	129.00	2	5.556	33.88	142.30	37.71	-78.35	112.20	0.9317
	129.70	1	2.778	33.18	142.80	39.10	-82.37	115.60	0.8975
	131.70	1	2.778	31.10	-81.44	24.38	150.30	-119.20	0.8875
	133.50	4	11.11	29.24	145.80	31.18	-89.27	118.50	0.9673
	135.30	2	5.556	27.32	-86.58	22.92	145.60	-118.30	0.9256
	136.00	1	2.778	26.56	-89.31	20.27	148.70	-122.20	0.8969
IV-7	41.18	4	33.33	126.10	112.60	121.40	144.50	-18.36	0.4795
	44.87	4	33.33	122.30	146.00	124.50	96.34	25.92	0.8280
	131.00	2	16.67	31.83	-80.64	26.57	149.60	-117.70	0.9107
	131.60	2	16.67	31.14	-79.40	24.21	148.60	-117.50	0.8820
IV-8	42.12	4	16.67	125.10	110.80	119.60	145.00	-19.82	0.4453
	42.80	2	8.333	124.40	109.20	117.80	146.30	-21.85	0.3904
	43.10	2	8.333	124.10	108.40	118.00	145.90	-21.81	0.4381
	45.48	4	16.67	121.60	98.06	118.60	148.20	-26.60	0.7710
	52.00	4	16.67	114.80	143.30	117.10	83.98	30.79	0.8513
	130.10	4	16.67	32.76	-85.66	31.37	152.60	-119.80	0.9770
	135.70	2	8.333	26.86	-90.66	22.19	149.10	-122.20	0.9235
	136.30	2	8.333	26.25	-90.39	20.83	148.30	-122.10	0.9112
IV-9	33.50	8	22.22	134.20	105.90	125.30	171.30	-37.14	0.5228
	33.81	4	11.11	133.90	106.40	124.40	170.80	-36.98	0.4861
	35.05	8	22.22	132.60	117.40	123.10	157.20	-24.58	0.2312
	35.48	4	11.11	132.10	116.90	122.80	156.70	-24.56	0.2391
	124.90	4	11.11	38.26	151.10	40.40	-76.70	115.00	0.9628
	125.40	4	11.11	37.67	151.60	38.78	-77.41	115.10	0.9808
	126.00	4	11.11	37.11	151.90	37.74	-78.35	115.50	0.9890

Degree-4 polymer	δ_{iso}(ppm)	symmetry-equivalent C atoms in a unit cell	symmetry-equivalent C atoms (%)	σ_{iso}(ppm)	σ_{xx}(ppm)	σ_{yy}(ppm)	σ_{zz}(ppm)	anisotropy parameter δ(ppm)	asymmetry parameter η
IV-10	32.63	4	22.22	135.10	150.60	136.80	117.90	17.23	0.7994
	33.22	2	11.11	134.50	149.30	136.50	117.70	16.80	0.7591
	44.12	1	5.556	123.00	112.30	121.30	135.60	-12.52	0.7188
	44.37	1	5.556	122.80	112.20	120.60	135.50	-12.77	0.6555
	44.81	1	5.556	122.30	112.30	121.00	133.70	-11.37	0.7657
	45.29	3	16.67	121.80	112.40	119.10	134.00	-12.14	0.5486
	132.80	1	5.556	29.95	140.00	32.96	-83.15	113.10	0.9466
	133.20	2	11.11	29.55	138.60	31.34	-81.30	110.90	0.9677
	133.70	3	16.67	29.03	138.30	30.47	-81.65	110.70	0.9739
IV-11	32.97	18	16.67	134.80	108.30	134.30	161.60	-26.82	0.9691
	34.51	18	16.67	133.10	117.90	122.50	159.00	-25.89	0.1795
	37.26	18	16.67	130.20	146.70	132.40	111.70	18.59	0.7638
	41.24	18	16.67	126.10	112.40	118.90	146.90	-20.81	0.3092
	132.80	36	33.33	29.94	143.90	32.04	-86.14	116.10	0.9639
IV-13	32.00	4	33.33	135.80	120.90	134.00	152.40	-16.60	0.7909
	46.19	4	33.33	120.90	109.30	117.30	135.90	-15.08	0.5304
	132.10	4	33.33	30.64	139.70	33.71	-81.52	112.20	0.9453
IV-14	30.36	4	16.67	137.50	117.50	135.10	159.80	-22.33	0.7868
	31.06	4	16.67	136.80	120.30	127.50	162.40	-25.68	0.2819
	40.16	4	16.67	127.20	111.40	125.00	145.20	-17.98	0.7586
	46.03	4	16.67	121.00	98.36	120.50	144.30	-23.26	0.9500
	133.80	4	16.67	28.90	141.00	29.77	-84.11	113.00	0.9845
	139.40	4	16.67	22.99	-94.54	15.13	148.40	-125.40	0.8746
IV-15	36.24	6	16.67	131.30	119.10	123.80	151.00	-19.70	0.2382
	47.17	6	16.67	119.80	106.50	114.50	138.50	-18.66	0.4308
	52.66	2	5.556	114.10	152.00	114.20	76.07	38.01	0.9942
	53.18	4	11.11	113.50	151.00	113.60	75.93	37.60	0.9950
	56.87	2	5.556	109.70	77.92	109.40	141.70	-32.02	0.9825
	57.37	4	11.11	109.10	78.19	108.80	140.40	-31.29	0.9777
	131.90	6	16.67	30.90	-80.37	30.35	142.70	-111.80	0.9903
	133.80	2	5.556	28.85	142.10	29.38	-84.95	113.80	0.9908
	134.50	4	11.11	28.16	-84.82	27.99	141.30	-113.20	0.9968
IV-16	41.18	14	16.67	126.10	104.80	121.10	152.50	-26.36	0.6186
	45.31	2	2.381	121.80	96.89	117.50	151.00	-29.18	0.7058
	45.97	12	14.29	121.10	96.15	117.70	149.40	-28.33	0.7610
	51.36	10	11.9	115.40	83.54	109.20	153.50	-38.09	0.6750
	51.83	1	1.19	115.00	83.94	108.30	152.60	-37.70	0.6452
	53.21	15	17.86	113.50	91.95	111.40	137.20	-23.67	0.8213
	53.92	2	2.381	112.80	90.54	109.90	137.80	-25.03	0.7743
	130.00	3	3.571	32.85	138.80	39.01	-79.27	112.10	0.8901
	130.50	3	3.571	32.32	139.20	37.98	-80.25	112.60	0.8995
	131.00	8	9.524	31.78	139.40	38.22	-82.25	114.00	0.8870
	131.30	3	3.571	31.53	138.60	37.28	-81.32	112.90	0.8981
	131.70	2	2.381	31.06	135.80	36.02	-78.67	109.70	0.9098
	132.20	1	1.19	30.60	136.10	35.48	-79.72	110.30	0.9116
	133.10	6	7.143	29.66	135.60	34.45	-81.03	110.70	0.9135
	133.40	2	2.381	29.34	136.10	33.61	-81.73	111.10	0.9230

Degree-4 polymer	δ_{iso}(ppm)	symmetry-equivalent C atoms in a unit cell	symmetry-equivalent C atoms (%)	σ_{iso}(ppm)	σ_{xx}(ppm)	σ_{yy}(ppm)	σ_{zz}(ppm)	anisotropy parameter δ(ppm)	asymmetry parameter η
IV-18	45.30	1	8.333	121.80	98.68	116.90	149.80	-27.97	0.6530
	45.86	1	8.333	121.20	96.05	118.20	149.50	-28.25	0.7825
	52.50	1	8.333	114.20	139.10	116.30	87.45	26.80	0.8506
	53.02	1	8.333	113.70	138.10	115.50	87.50	26.20	0.8653
	53.86	1	8.333	112.80	145.90	113.00	79.59	33.23	0.9892
	55.37	2	16.67	111.20	81.43	110.10	142.20	-30.93	0.9261
	56.50	1	8.333	110.00	80.25	108.10	141.70	-31.70	0.8792
	129.40	1	8.333	33.48	-73.41	29.42	144.40	-110.90	0.9269
	132.00	1	8.333	30.77	-77.66	25.01	144.90	-114.20	0.8993
	145.00	1	8.333	17.12	-97.06	-5.12	153.50	-136.40	0.6740
	147.10	1	8.333	14.91	-101.10	-5.60	151.40	-136.50	0.6994
IV-19	38.16	6	16.67	129.30	106.90	127.10	153.90	-24.63	0.8236
	46.34	6	16.67	120.70	142.70	121.20	98.20	22.51	0.9570
	48.17	6	16.67	118.80	152.80	122.90	80.63	38.16	0.7849
	50.86	6	16.67	116.00	98.81	110.10	139.00	-23.06	0.4885
	128.40	6	16.67	34.52	143.90	40.29	-80.63	115.20	0.8998
	130.50	6	16.67	32.39	137.80	40.51	-81.14	113.50	0.8569
IV-20	39.89	8	16.67	127.50	140.20	131.80	110.60	16.93	0.4947
	46.03	4	8.333	121.00	152.80	132.80	77.49	43.55	0.4609
	46.89	4	8.333	120.10	103.30	113.20	143.90	-23.78	0.4134
	48.78	4	8.333	118.20	105.10	109.90	139.40	-21.27	0.2243
	49.74	8	16.67	117.10	140.80	119.50	91.19	25.95	0.8201
	53.30	4	8.333	113.40	84.76	110.20	145.20	-31.79	0.8012
	125.80	4	8.333	37.33	144.40	40.88	-73.32	110.60	0.9360
	129.90	4	8.333	33.00	140.50	40.87	-82.35	115.30	0.8636
	130.30	4	8.333	32.60	142.90	39.28	-84.42	117.00	0.8857
	131.40	4	8.333	31.45	143.10	34.28	-83.01	114.50	0.9505
IV-21	39.05	6	8.333	128.40	110.10	127.60	147.40	-19.04	0.9240
	40.67	6	8.333	126.70	107.10	121.30	151.60	-24.90	0.5711
	42.82	6	8.333	124.40	150.10	126.10	97.00	27.41	0.8774
	44.07	6	8.333	123.10	92.63	123.10	153.60	-30.53	0.9964
	45.78	6	8.333	121.30	102.90	120.50	140.50	-19.19	0.9166
	46.59	6	8.333	120.50	147.80	120.70	92.93	27.52	0.9849
	51.15	6	8.333	115.70	145.60	116.90	84.47	31.19	0.9228
	54.19	6	8.333	112.50	137.80	120.00	79.54	32.93	0.5399
	127.20	6	8.333	35.78	144.80	40.74	-78.17	113.90	0.9130
	135.10	6	8.333	27.48	146.90	34.33	-98.81	126.30	0.8915
	136.50	6	8.333	26.02	-98.35	25.50	150.90	-124.90	0.9915
	138.80	6	8.333	23.60	-98.02	19.68	149.10	-125.50	0.9375

Table S2: The calculated isotropic chemical shifts and chemical shielding anisotropy parameter for bonded sp^2 and sp^3 carbon sites for 18 degree-4 nanothreads. Note that the data in column 2 is repeated in column 5 to facilitate sight reading.

Degree-4 polymer	δ_{iso}(ppm) (sp^2)	symmetry-equivalent sp^2 C atoms in a unit cell	symmetry-equivalent sp^2 C atoms (%)	δ_{iso}(ppm) (sp^2)	anisotropy parameter δ(ppm)	asymmetry parameter η	δ_{iso}(ppm) (sp^3 C bonded to sp^2 C)	anisotropy parameter δ(ppm)	asymmetry parameter η
IV-1	126.10	2	50	126.10	-117.70	0.7849	50.07	-29.62	0.8434
	141.40	2	50	141.40	-125.40	0.8740	52.06	-32.55	0.3964
IV-2	128.40	2	25	128.40	116.60	0.9703	43.32	-19.42	0.8226
	129.20	2	25	129.20	-115.90	0.9868	43.21	-19.89	0.8509
	135.30	2	25	135.30	121.30	0.9504	47.26	27.18	0.6902
	135.90	2	25	135.90	119.10	0.9969	46.86	27.91	0.6670
IV-3	125.60	1	25	125.60	-112.90	0.9749	45.78	-20.79	0.5421
	128.70	1	25	128.70	120.30	0.8202	46.30	-22.60	0.6898
	131.00	1	25	131.00	116.40	0.7904	46.61	-30.55	0.6345
	138.30	1	25	138.30	-122.10	0.9657	46.99	-25.60	0.0794
IV-4	127.50	2	25	127.50	108.70	0.8969	54.18	31.03	0.8133
	128.60	2	25	128.60	-113.40	0.9788	42.28	-22.71	0.6038
	132.40	2	25	132.40	119.20	0.8714	38.21	-16.47	0.9290
	143.10	2	25	143.10	-126.50	0.9271	51.78	-28.78	0.8858
IV-5	126.20	1	8.333	126.20	113.70	0.8964	38.79	-13.80	0.7829
	129.00	2	16.67	129.00	112.20	0.9317	40.68	-15.57	0.7969
	129.70	1	8.333	129.70	115.60	0.8975	38.45	-12.06	0.9331
	131.70	1	8.333	131.70	-119.20	0.8875	46.19	-27.63	0.5521
	133.50	4	33.33	133.50	118.50	0.9673	44.49	-28.80	0.7027
	135.30	2	16.67	135.30	-118.30	0.9256	46.86	-28.57	0.7227
	136.00	1	8.333	136.00	-122.20	0.8969	39.10	-21.93	0.6445
IV-7	131.00	2	50	131.00	-117.70	0.9107	41.18	-18.36	0.4800
	131.60	2	50	131.60	-117.50	0.8820	41.14	-18.08	0.5519
IV-8	130.10	4	50	130.10	-119.80	0.9770	43.10	-21.81	0.4381
	135.70	2	25	135.70	-122.20	0.9235	42.10	-19.86	0.4626
	136.30	2	25	136.30	-122.10	0.9112	42.12	-19.82	0.4453
IV-9	124.90	4	33.33	124.90	115.00	0.9628	35.05	-24.58	0.2312
	125.40	4	33.33	125.40	115.10	0.9808	34.94	-23.35	0.2685
	126.00	4	33.33	126.00	115.50	0.9890	35.46	-24.54	0.2392
IV-10	132.80	1	16.67	132.80	113.10	0.9466	45.24	-12.21	0.5169
	133.20	2	33.33	133.20	110.90	0.9677	44.37	-12.77	0.6555
	133.70	3	50	133.70	110.70	0.9739	44.81	-11.37	0.7657

Degree-4 polymer	δ_{iso}(ppm) (sp^2)	symmetry-equivalent sp^2 C atoms in a unit cell	symmetry-equivalent sp^2 C atoms (%)	δ_{iso}(ppm) (sp^2)	anisotropy parameter δ(ppm)	asymmetry parameter η	δ_{iso}(ppm) (sp^3 C bonded to sp^2 C)	anisotropy parameter δ(ppm)	asymmetry parameter η
IV-11	132.80	36	100	132.80	116.10	0.9639	37.05	19.18	0.7066
IV-13	132.10	4	100	132.10	112.20	0.9453	46.02	-15.02	0.6009
IV-14	133.80	4	50	133.80	113.00	0.9845	40.16	-17.99	0.7586
	139.40	4	50	139.40	-125.40	0.8746	46.03	-23.26	0.9500
IV-15	131.90	6	50	131.90	-111.80	0.9903	36.24	-19.70	0.2382
	133.80	2	16.67	133.80	113.80	0.9908	47.17	-18.66	0.4308
	134.50	4	33.33	134.50	-113.20	0.9968	46.85	-18.09	0.4629
IV-16	130.00	3	10.71	130.00	112.10	0.8901	52.98	-23.32	0.7912
	130.50	3	10.71	130.50	112.60	0.8995	52.99	-24.66	0.8277
	131.00	8	28.57	131.00	114.00	0.8870	52.59	-24.14	0.7908
	131.30	3	10.71	131.30	112.90	0.8981	52.91	-24.78	0.8215
	131.70	2	7.143	131.70	109.70	0.9098	45.52	-28.83	0.7146
	132.20	1	3.571	132.20	110.30	0.9116	45.97	-28.33	0.7610
	133.10	6	21.43	133.10	110.70	0.9135	45.83	-28.54	0.8068
	133.40	2	7.143	133.40	111.10	0.9230	45.92	-29.17	0.7430
IV-18	129.40	1	25	129.40	-110.90	0.9269	55.37	-30.93	0.9261
	132.00	1	25	132.00	-114.20	0.8993	56.50	-31.70	0.8792
	145.00	1	25	145.00	-136.40	0.6740	45.86	-28.25	0.7825
	147.10	1	25	147.10	-136.50	0.6994	45.30	-27.97	0.6530
IV-19	128.40	6	50	128.40	115.20	0.8998	38.16	-24.63	0.8236
	130.50	6	50	130.50	113.50	0.8569	50.82	-23.03	0.4905
IV-20	125.80	4	25	125.80	110.60	0.9360	49.73	25.94	0.8205
	129.90	4	25	129.90	115.30	0.8636	48.78	-21.27	0.2243
	130.30	4	25	130.30	117.00	0.8857	49.70	20.24	0.9295
	131.40	4	25	131.40	114.50	0.9505	39.65	28.09	0.9195
IV-21	127.20	6	25	127.20	113.90	0.9130	45.74	-19.15	0.8985
	135.10	6	25	135.10	126.30	0.8915	39.02	-18.99	0.9127
	136.50	6	25	136.50	-124.90	0.9915	40.64	-24.88	0.5786
	138.80	6	25	138.80	-125.50	0.9375	42.82	27.41	0.8774

Table S3: Calculated isotropic chemical shifts and chemical shielding anisotropy parameters for fully axially disordered and [4+2]-constrained axially disordered degree-6 and degree-4 threads.

degree6_class2 thread

δ_{iso}(ppm)	symmetry-equivalent C atoms in a unit cell	symmetry-equivalent C atoms (%)	σ_{iso}(ppm)	σ_{xx}(ppm)	σ_{yy}(ppm)	σ_{zz}(ppm)	anisotropy parameter δ(ppm)	asymmetry parameter η
55.914	1	1.3889	110.66	87.029	103.65	141.3	-30.635	0.5427
46.019	2	2.7778	121.05	91.428	120.6	151.13	-30.079	0.96984
40.41	2	2.7778	126.94	104.63	121.07	155.13	-28.186	0.58324
40.838	2	2.7778	126.49	105.49	121.32	152.67	-26.177	0.60485
58.219	2	2.7778	108.24	93.128	97.387	134.21	-25.965	0.16404
32.41	1	1.3889	135.34	118.42	126.4	161.21	-25.867	0.30834
33.41	2	2.7778	134.29	113.43	130.55	158.88	-24.587	0.69614
32.019	2	2.7778	135.75	116.52	130.68	160.05	-24.3	0.58261
46.429	1	1.3889	120.62	102.76	114.28	144.82	-24.204	0.47605
44.571	1	1.3889	122.57	100.53	120.42	146.74	-24.175	0.82299
39.495	2	2.7778	127.9	106.88	125.19	151.64	-23.739	0.7711
47.933	4	5.5556	119.04	103.68	111.06	142.38	-23.337	0.31615
48.905	1	1.3889	118.02	102.65	110.79	140.62	-22.598	0.36036
57.848	2	2.7778	108.63	92.721	102.03	131.14	-22.506	0.41361
44.981	1	1.3889	122.14	102	119.82	144.59	-22.454	0.79371
29.19	1	1.3889	138.72	124.33	130.81	161.01	-22.291	0.29088
44.133	1	1.3889	123.03	102.59	121.3	145.2	-22.173	0.84376
48.295	2	2.7778	118.66	101.11	114.26	140.63	-21.968	0.59844
48.667	2	2.7778	118.27	99.38	115.25	140.18	-21.913	0.72422
37.981	1	1.3889	129.49	110.5	127.14	150.83	-21.338	0.7798
49.19	1	1.3889	117.72	105.58	109.16	138.42	-20.703	0.17295
32.286	1	1.3889	135.47	121.93	128.71	155.78	-20.314	0.33387
46.657	1	1.3889	120.38	108.33	112.15	140.66	-20.282	0.18834
42.781	1	1.3889	124.45	107.06	121.92	144.38	-19.928	0.74587
36.533	1	1.3889	131.01	112.6	129.76	150.67	-19.659	0.87294
46.952	1	1.3889	120.07	101.2	119.4	139.61	-19.54	0.93153
45.114	2	2.7778	122	107.51	117.76	140.73	-18.729	0.5475
43.419	2	2.7778	123.78	108.33	120.52	142.49	-18.706	0.65181
34.838	1	1.3889	132.79	117.26	130.09	151.02	-18.228	0.70374
47.105	1	1.3889	119.91	106.93	115.3	137.52	-17.606	0.47536
31.571	1	1.3889	136.22	125.35	129.93	153.38	-17.165	0.26658
33.981	1	1.3889	133.69	124.36	127.28	149.44	-15.747	0.18532
34.314	1	1.3889	133.34	122.6	130.29	147.13	-13.792	0.55769
34.467	1	1.3889	133.18	119.87	132.74	146.93	-13.746	0.93635
35.514	1	1.3889	132.08	120.11	131.24	144.87	-12.792	0.86978
31.905	1	1.3889	135.87	126.43	134.38	146.8	-10.931	0.72773
36.038	1	1.3889	131.53	140.49	134.17	119.92	11.605	0.54487
35.324	1	1.3889	132.28	143.78	133.96	119.1	13.185	0.74428
37.152	1	1.3889	130.36	140.19	135.37	115.51	14.846	0.32483
37.533	1	1.3889	129.96	139.75	135.05	115.07	14.889	0.31561
37.4	1	1.3889	130.1	142.07	136.5	111.75	18.349	0.30351
36.81	1	1.3889	130.72	142.07	138.04	112.05	18.666	0.21579
36.695	1	1.3889	130.84	149.58	131.78	111.17	19.672	0.90463
39.343	2	2.7778	128.06	147.15	129.03	107.98	20.08	0.9025
41.429	1	1.3889	125.87	145.74	127.24	104.64	21.229	0.87156
51.267	1	1.3889	115.54	131.35	121.47	93.796	21.744	0.45409
57.562	1	1.3889	108.93	128.27	111.91	86.604	22.326	0.73309
43.257	1	1.3889	123.95	137.91	133.07	100.88	23.072	0.20982
43.914	1	1.3889	123.26	145.04	124.7	100.05	23.209	0.87632
45.505	1	1.3889	121.59	141.14	126.61	97.015	24.575	0.59136
44.295	1	1.3889	122.86	150.25	123.29	95.047	27.813	0.96925
56.324	1	1.3889	110.23	138.76	111.73	80.196	30.034	0.90007
50.848	1	1.3889	115.98	141.9	122.46	83.581	32.4	0.60002
53.648	1	1.3889	113.04	137.78	122.22	79.118	33.922	0.45856
53.4	1	1.3889	113.3	146.3	117.79	75.819	37.481	0.76057
55.105	1	1.3889	111.51	143.85	117.37	73.301	38.209	0.69306

degree6_class3 thread

δ_{iso}(ppm)	symmetry-equivalent C atoms in a unit cell	symmetry-equivalent C atoms (%)	σ_{iso}(ppm)	σ_{xx}(ppm)	σ_{yy}(ppm)	σ_{zz}(ppm)	anisotropy parameter δ(ppm)	asymmetry parameter η
55.914	1	1.3889	110.66	87.029	103.65	141.3	-30.635	0.5427
46.019	2	2.7778	121.05	91.428	120.6	151.13	-30.079	0.96984
40.41	2	2.7778	126.94	104.63	121.07	155.13	-28.186	0.58324
40.838	2	2.7778	126.49	105.49	121.32	152.67	-26.177	0.60485
58.219	2	2.7778	108.24	93.128	97.387	134.21	-25.965	0.16404
32.41	1	1.3889	135.34	118.42	126.4	161.21	-25.867	0.30834
33.41	2	2.7778	134.29	113.43	130.55	158.88	-24.587	0.69614
32.019	2	2.7778	135.75	116.52	130.68	160.05	-24.3	0.58261
46.429	1	1.3889	120.62	102.76	114.28	144.82	-24.204	0.47605
44.571	1	1.3889	122.57	100.53	120.42	146.74	-24.175	0.82299
39.495	2	2.7778	127.9	106.88	125.19	151.64	-23.739	0.7711
47.933	4	5.5556	119.04	103.68	111.06	142.38	-23.337	0.31615
48.905	1	1.3889	118.02	102.65	110.79	140.62	-22.598	0.36036
57.848	2	2.7778	108.63	92.721	102.03	131.14	-22.506	0.41361
44.981	1	1.3889	122.14	102	119.82	144.59	-22.454	0.79371
29.19	1	1.3889	138.72	124.33	130.81	161.01	-22.291	0.29088
44.133	1	1.3889	123.03	102.59	121.3	145.2	-22.173	0.84376
48.295	2	2.7778	118.66	101.11	114.26	140.63	-21.968	0.59844
48.667	2	2.7778	118.27	99.38	115.25	140.18	-21.913	0.72422
37.981	1	1.3889	129.49	110.5	127.14	150.83	-21.338	0.7798
49.19	1	1.3889	117.72	105.58	109.16	138.42	-20.703	0.17295
32.286	1	1.3889	135.47	121.93	128.71	155.78	-20.314	0.33387
46.657	1	1.3889	120.38	108.33	112.15	140.66	-20.282	0.18834
42.781	1	1.3889	124.45	107.06	121.92	144.38	-19.928	0.74587
36.533	1	1.3889	131.01	112.6	129.76	150.67	-19.659	0.87294
46.952	1	1.3889	120.07	101.2	119.4	139.61	-19.54	0.93153
45.114	2	2.7778	122	107.51	117.76	140.73	-18.729	0.5475
43.419	2	2.7778	123.78	108.33	120.52	142.49	-18.706	0.65181
34.838	1	1.3889	132.79	117.26	130.09	151.02	-18.228	0.70374
47.105	1	1.3889	119.91	106.93	115.3	137.52	-17.606	0.47536
31.571	1	1.3889	136.22	125.35	129.93	153.38	-17.165	0.26658
33.981	1	1.3889	133.69	124.36	127.28	149.44	-15.747	0.18532
34.314	1	1.3889	133.34	122.6	130.29	147.13	-13.792	0.55769
34.467	1	1.3889	133.18	119.87	132.74	146.93	-13.746	0.93635
35.514	1	1.3889	132.08	120.11	131.24	144.87	-12.792	0.86978
31.905	1	1.3889	135.87	126.43	134.38	146.8	-10.931	0.72773
36.038	1	1.3889	131.53	140.49	134.17	119.92	11.605	0.54487
35.324	1	1.3889	132.28	143.78	133.96	119.1	13.185	0.74428
37.152	1	1.3889	130.36	140.19	135.37	115.51	14.846	0.32483
37.533	1	1.3889	129.96	139.75	135.05	115.07	14.889	0.31561
37.4	1	1.3889	130.1	142.07	136.5	111.75	18.349	0.30351
36.81	1	1.3889	130.72	142.07	138.04	112.05	18.666	0.21579
36.695	1	1.3889	130.84	149.58	131.78	111.17	19.672	0.90463
39.343	2	2.7778	128.06	147.15	129.03	107.98	20.08	0.9025
41.429	1	1.3889	125.87	145.74	127.24	104.64	21.229	0.87156
51.267	1	1.3889	115.54	131.35	121.47	93.796	21.744	0.45409
57.562	1	1.3889	108.93	128.27	111.91	86.604	22.326	0.73309
43.257	1	1.3889	123.95	137.91	133.07	100.88	23.072	0.20982
43.914	1	1.3889	123.26	145.04	124.7	100.05	23.209	0.87632
45.505	1	1.3889	121.59	141.14	126.61	97.015	24.575	0.59136
44.295	1	1.3889	122.86	150.25	123.29	95.047	27.813	0.96925
56.324	1	1.3889	110.23	138.76	111.73	80.196	30.034	0.90007
50.848	1	1.3889	115.98	141.9	122.46	83.581	32.4	0.60002
53.648	1	1.3889	113.04	137.78	122.22	79.118	33.922	0.45856
53.4	1	1.3889	113.3	146.3	117.79	75.819	37.481	0.76057
55.105	1	1.3889	111.51	143.85	117.37	73.301	38.209	0.69306

degree 4 thread

δ_{iso}(ppm)	symmetry-equivalent C atoms in a unit cell	symmetry-equivalent C atoms (%)	σ_{iso}(ppm)	σ_{xx}(ppm)	σ_{yy}(ppm)	σ_{zz}(ppm)	anisotropy parameter δ(ppm)	asymmetry parameter η
55.914	1	1.3889	110.66	87.029	103.65	141.3	-30.635	0.5427
46.019	2	2.7778	121.05	91.428	120.6	151.13	-30.079	0.96984
40.41	2	2.7778	126.94	104.63	121.07	155.13	-28.186	0.58324
40.838	2	2.7778	126.49	105.49	121.32	152.67	-26.177	0.60485
58.219	2	2.7778	108.24	93.128	97.387	134.21	-25.965	0.16404
32.41	1	1.3889	135.34	118.42	126.4	161.21	-25.867	0.30834
33.41	2	2.7778	134.29	113.43	130.55	158.88	-24.587	0.69614
32.019	2	2.7778	135.75	116.52	130.68	160.05	-24.3	0.58261
46.429	1	1.3889	120.62	102.76	114.28	144.82	-24.204	0.47605
44.571	1	1.3889	122.57	100.53	120.42	146.74	-24.175	0.82299
39.495	2	2.7778	127.9	106.88	125.19	151.64	-23.739	0.7711
47.933	4	5.5556	119.04	103.68	111.06	142.38	-23.337	0.31615
48.905	1	1.3889	118.02	102.65	110.79	140.62	-22.598	0.36036
57.848	2	2.7778	108.63	92.721	102.03	131.14	-22.506	0.41361
44.981	1	1.3889	122.14	102	119.82	144.59	-22.454	0.79371
29.19	1	1.3889	138.72	124.33	130.81	161.01	-22.291	0.29088
44.133	1	1.3889	123.03	102.59	121.3	145.2	-22.173	0.84376
48.295	2	2.7778	118.66	101.11	114.26	140.63	-21.968	0.59844
48.667	2	2.7778	118.27	99.38	115.25	140.18	-21.913	0.72422
37.981	1	1.3889	129.49	110.5	127.14	150.83	-21.338	0.7798
49.19	1	1.3889	117.72	105.58	109.16	138.42	-20.703	0.17295
32.286	1	1.3889	135.47	121.93	128.71	155.78	-20.314	0.33387
46.657	1	1.3889	120.38	108.33	112.15	140.66	-20.282	0.18834
42.781	1	1.3889	124.45	107.06	121.92	144.38	-19.928	0.74587
36.533	1	1.3889	131.01	112.6	129.76	150.67	-19.659	0.87294
46.952	1	1.3889	120.07	101.2	119.4	139.61	-19.54	0.93153
45.114	2	2.7778	122	107.51	117.76	140.73	-18.729	0.5475
43.419	2	2.7778	123.78	108.33	120.52	142.49	-18.706	0.65181
34.838	1	1.3889	132.79	117.26	130.09	151.02	-18.228	0.70374
47.105	1	1.3889	119.91	106.93	115.3	137.52	-17.606	0.47536
31.571	1	1.3889	136.22	125.35	129.93	153.38	-17.165	0.26658
33.981	1	1.3889	133.69	124.36	127.28	149.44	-15.747	0.18532
34.314	1	1.3889	133.34	122.6	130.29	147.13	-13.792	0.55769
34.467	1	1.3889	133.18	119.87	132.74	146.93	-13.746	0.93635
35.514	1	1.3889	132.08	120.11	131.24	144.87	-12.792	0.86978
31.905	1	1.3889	135.87	126.43	134.38	146.8	-10.931	0.72773
36.038	1	1.3889	131.53	140.49	134.17	119.92	11.605	0.54487
35.324	1	1.3889	132.28	143.78	133.96	119.1	13.185	0.74428
37.152	1	1.3889	130.36	140.19	135.37	115.51	14.846	0.32483
37.533	1	1.3889	129.96	139.75	135.05	115.07	14.889	0.31561
37.4	1	1.3889	130.1	142.07	136.5	111.75	18.349	0.30351
36.81	1	1.3889	130.72	142.07	138.04	112.05	18.666	0.21579
36.695	1	1.3889	130.84	149.58	131.78	111.17	19.672	0.90463
39.343	2	2.7778	128.06	147.15	129.03	107.98	20.08	0.9025
41.429	1	1.3889	125.87	145.74	127.24	104.64	21.229	0.87156
51.267	1	1.3889	115.54	131.35	121.47	93.796	21.744	0.45409
57.562	1	1.3889	108.93	128.27	111.91	86.604	22.326	0.73309
43.257	1	1.3889	123.95	137.91	133.07	100.88	23.072	0.20982
43.914	1	1.3889	123.26	145.04	124.7	100.05	23.209	0.87632
45.505	1	1.3889	121.59	141.14	126.61	97.015	24.575	0.59136
44.295	1	1.3889	122.86	150.25	123.29	95.047	27.813	0.96925
56.324	1	1.3889	110.23	138.76	111.73	80.196	30.034	0.90007
50.848	1	1.3889	115.98	141.9	122.46	83.581	32.4	0.60002
53.648	1	1.3889	113.04	137.78	122.22	79.118	33.922	0.45856
53.4	1	1.3889	113.3	146.3	117.79	75.819	37.481	0.76057
55.105	1	1.3889	111.51	143.85	117.37	73.301	38.209	0.69306

[4+2]_degree6_class2 thread

δ_{iso}(ppm)	symmetry-equivalent C atoms in a unit cell	symmetry-equivalent C atoms (%)	σ_{iso}(ppm)	σ_{xx}(ppm)	σ_{yy}(ppm)	σ_{zz}(ppm)	anisotropy parameter δ(ppm)	asymmetry parameter η
44.981	1	1.3889	122.14	97.498	113.4	155.53	-33.387	0.47636
45.362	1	1.3889	121.74	97.669	112.55	155.01	-33.267	0.44738
49.714	1	1.3889	117.17	98.071	109.28	144.15	-26.984	0.41547
49.524	1	1.3889	117.37	98.14	109.64	144.32	-26.95	0.42661
52.581	2	2.7778	114.16	89.54	111.91	141.03	-26.873	0.83241
40.514	3	4.1667	126.83	105.43	121.89	153.16	-26.329	0.62494
52.971	1	1.3889	113.75	89.589	111.7	139.97	-26.217	0.84332
33.143	1	1.3889	134.57	112.38	131.45	159.89	-25.316	0.75318
46.486	1	1.3889	120.56	102.56	113.33	145.8	-25.236	0.42674
33.286	1	1.3889	134.42	112.39	131.25	159.62	-25.2	0.74805
33.924	2	2.7778	133.75	110.91	131.82	158.53	-24.784	0.84349
43.467	1	1.3889	123.73	101.95	121.29	147.94	-24.212	0.79879
43.638	1	1.3889	123.55	101.83	121.59	147.24	-23.689	0.83428
42.476	1	1.3889	124.77	105.02	121.62	147.66	-22.886	0.72522
43.095	1	1.3889	124.12	104.16	121.66	146.54	-22.419	0.78017
50.81	2	2.7778	116.02	95.827	114.55	137.69	-21.673	0.86368
51	1	1.3889	115.82	95.786	114.25	137.41	-21.592	0.85531
37.41	1	1.3889	130.09	109.61	129.96	150.7	-20.614	0.98733
30.029	1	1.3889	137.84	125.59	129.65	158.29	-20.445	0.19884
37.524	1	1.3889	129.97	110.19	129.3	150.41	-20.444	0.93477
30.229	1	1.3889	137.63	125.85	129.55	157.49	-19.861	0.18624
42.067	1	1.3889	125.2	105.36	125.18	145.06	-19.86	0.99792
40.81	1	1.3889	126.52	107.38	126	146.17	-19.652	0.9476
40.038	5	6.9444	127.33	110.01	125.01	146.96	-19.628	0.76408
38.99	9	12.5	128.43	141.77	128.65	114.87	13.565	0.96702
46.8	1	1.3889	120.23	133.35	121.24	106.09	14.141	0.85663
39.733	1	1.3889	127.65	141.72	127.85	113.39	14.262	0.97207
40.41	1	1.3889	126.94	137.65	133.29	109.88	17.055	0.2554
39.914	3	4.1667	127.46	138.34	133.71	110.34	17.125	0.27037
41.219	3	4.1667	126.09	146.65	126.5	105.1	20.985	0.96015
48.619	1	1.3889	118.32	130.15	128.88	95.93	22.39	0.056754
48.057	1	1.3889	118.91	131.62	128.79	96.331	22.579	0.12506
41.705	2	2.7778	125.58	142.15	132.52	102.08	23.504	0.40969
41.533	1	1.3889	125.76	143.23	132.23	101.83	23.925	0.45997
46.581	3	4.1667	120.46	137.41	128.05	95.93	24.53	0.38136
50.714	1	1.3889	116.12	136.24	120.64	91.482	24.638	0.63339
46.238	1	1.3889	120.82	137.71	129.07	95.692	25.127	0.34361
51.114	1	1.3889	115.7	138.58	118.24	90.271	25.429	0.79962
38.829	1	1.3889	128.6	152.27	130.44	103.09	25.507	0.85556
50.295	1	1.3889	116.56	139.84	119.58	90.26	26.3	0.7704
39.438	3	4.1667	127.96	153.75	129.23	100.89	27.067	0.90598
53.314	1	1.3889	113.39	139.23	117	83.941	29.449	0.75468
54.019	2	2.7778	112.65	139.96	117.13	80.854	31.796	0.71787
58.829	2	2.7778	107.6	142.4	111.22	69.162	38.438	0.81102

[4+2]_degree6_class3 thread

δ_{iso}(ppm)	symmetry-equivalent C atoms in a unit cell	symmetry-equivalent C atoms (%)	σ_{iso}(ppm)	σ_{xx}(ppm)	σ_{yy}(ppm)	σ_{zz}(ppm)	anisotropy parameter δ(ppm)	asymmetry parameter η
52.01	1	1.3889	114.76	88.675	110.54	145.06	-30.298	0.72157
41.086	1	1.3889	126.23	105.07	119.35	154.28	-28.051	0.50894
48.124	1	1.3889	118.84	94.899	116.18	145.45	-26.609	0.79973
38.581	1	1.3889	128.86	108.37	122.94	155.28	-26.419	0.55159
30.638	1	1.3889	137.2	117.49	132.03	162.08	-24.884	0.58434
48.238	1	1.3889	118.72	98.785	114.72	142.66	-23.939	0.66565
44.238	1	1.3889	122.92	99.886	122.1	146.79	-23.867	0.9307
37.867	1	1.3889	129.61	116.17	119.68	152.98	-23.373	0.15006
35.219	1	1.3889	132.39	114.95	126.45	155.76	-23.366	0.49213
37.19	2	2.7778	130.32	114.03	123.56	153.36	-23.038	0.41381
44.733	4	5.5556	122.4	102.39	119.52	145.29	-22.891	0.74832
36.771	1	1.3889	130.76	113.71	125.43	153.14	-22.384	0.52362
31.914	1	1.3889	135.86	122.33	127.09	158.16	-22.297	0.21321
40.867	3	4.1667	126.46	107.42	123.33	148.63	-22.165	0.71771
38.219	1	1.3889	129.24	109.49	126.86	151.35	-22.111	0.7857
46.257	1	1.3889	120.8	104.59	114.98	142.81	-22.014	0.47194
33.048	1	1.3889	134.67	119.83	127.76	156.42	-21.752	0.36471
36.876	1	1.3889	130.65	118.71	121.37	151.88	-21.23	0.12495
36.59	4	5.5556	130.95	119.24	122.17	151.44	-20.489	0.1428
31.61	1	1.3889	136.18	125.51	127.63	155.42	-19.238	0.11007
30.086	1	1.3889	137.78	128.24	128.36	156.74	-18.956	0.0064624
28.962	1	1.3889	138.96	126.41	132.76	157.72	-18.763	0.33851
29.305	1	1.3889	138.6	128.51	130.69	156.6	-18	0.12103
54.286	1	1.3889	112.37	97.813	110.18	129.11	-16.738	0.7386
54.505	1	1.3889	112.14	97.397	110.34	128.68	-16.535	0.78251
32.2	2	2.7778	135.56	126.27	131.7	148.73	-13.168	0.41248
33.962	1	1.3889	133.71	125.33	129.46	146.33	-12.623	0.3266
43.029	4	5.5556	124.19	112.33	124.09	136.16	-11.969	0.98274
41.619	1	1.3889	125.67	115	124.79	137.22	-11.551	0.8482
41.21	1	1.3889	126.1	115.64	125.26	137.39	-11.289	0.85161
40.61	2	2.7778	126.73	116.32	125.85	138.01	-11.283	0.8447
33.657	1	1.3889	134.03	126.73	130.25	145.12	-11.093	0.31747
47.676	1	1.3889	119.31	133.95	121.22	102.77	16.54	0.77008
36.19	1	1.3889	131.37	142.38	136.99	114.74	16.626	0.32447
47.429	1	1.3889	119.57	135.67	121.34	101.68	17.886	0.8015
42.457	1	1.3889	124.79	141.58	126.61	106.18	18.612	0.8041
39.21	1	1.3889	128.2	139.41	136.38	108.8	19.397	0.15645
48.781	1	1.3889	118.15	133.76	122.56	98.123	20.027	0.55902
35.124	1	1.3889	132.49	151.03	134.04	112.39	20.103	0.84466
35.533	1	1.3889	132.06	150.77	133.9	111.52	20.542	0.82155
42.114	1	1.3889	125.15	145.56	125.39	104.52	20.632	0.97757
47.181	1	1.3889	119.83	136.53	123.9	99.056	20.774	0.60774
46.895	1	1.3889	120.13	140.79	121.63	97.974	22.156	0.86472
49.771	1	1.3889	117.11	136.51	120.16	94.649	22.461	0.72768
49.638	1	1.3889	117.25	136.27	120.85	94.624	22.626	0.68127
45.086	3	4.1667	122.03	142.76	124.06	99.284	22.746	0.82212
43.305	1	1.3889	123.9	140.17	130.62	100.92	22.985	0.41547
48.581	1	1.3889	118.36	138.09	121.79	95.209	23.151	0.70398
51.838	1	1.3889	114.94	136.08	117.51	91.23	23.71	0.78297
51.524	1	1.3889	115.27	136.84	117.71	91.274	23.996	0.79706
51.39	1	1.3889	115.41	140.61	116.73	88.904	26.506	0.90096
53.924	1	1.3889	112.75	141.45	115.13	81.684	31.066	0.84708
45.571	1	1.3889	121.52	139.96	135.79	88.807	32.713	0.12745
46.59	1	1.3889	120.45	139.86	134.95	86.544	33.906	0.14484
54.79	2	2.7778	111.84	141.64	116.93	76.949	34.891	0.70818

[4+2]_degree4 thread

δ_{iso}(ppm)	symmetry-equivalent C atoms in a unit cell	symmetry-equivalent C atoms (%)	σ_{iso}(ppm)	σ_{xx}(ppm)	σ_{yy}(ppm)	σ_{zz}(ppm)	anisotropy parameter δ(ppm)	asymmetry parameter η
136.28	1	1.3889	26.28	138.88	33.114	-93.164	119.44	0.88548
135.88	1	1.3889	26.7	140.08	34.359	-94.327	121.03	0.87351
135.41	1	1.3889	27.19	136.45	32.988	-87.869	115.06	0.89923
134.69	1	1.3889	27.95	144.8	30.477	-91.429	119.38	0.95768
133.92	1	1.3889	28.75	141.24	33.354	-88.336	117.09	0.92139
133.08	1	1.3889	29.64	146.7	32.413	-90.203	119.84	0.95363
132.74	1	1.3889	29.99	143.68	35.318	-89.027	119.02	0.91048
131.99	1	1.3889	30.78	139.4	38.05	-85.112	115.89	0.87449
131.68	2	2.7778	31.11	149.5	31.153	-87.331	118.44	0.99918
130.95	2	2.7778	31.87	136.41	38.489	-79.287	111.16	0.88091
130.18	1	1.3889	32.68	135.69	39.318	-76.958	109.64	0.87902
129.75	1	1.3889	33.13	146.29	38.624	-85.535	118.67	0.90734
129.64	1	1.3889	33.25	140.96	38.508	-79.713	112.96	0.90691
129.11	1	1.3889	33.8	142.26	38.372	-79.221	113.02	0.91916
128.9	1	1.3889	34.03	143.53	39.573	-81.001	115.03	0.90369
128.22	1	1.3889	34.74	143.13	42.163	-81.078	115.82	0.87182
128.07	1	1.3889	34.9	147.51	36.352	-79.158	114.06	0.97455
127.08	1	1.3889	35.94	150.76	39.823	-82.78	118.72	0.93447
126.37	1	1.3889	36.68	148.24	36.891	-75.086	111.77	0.9963
124.62	1	1.3889	38.52	-70.259	38.328	147.49	-108.97	0.99648
124.22	1	1.3889	38.94	-70.835	38.635	149.02	-110.08	0.99447
122.78	1	1.3889	40.45	145.85	44.015	-68.513	108.96	0.93456
54.324	1	1.3889	112.33	84.026	110.26	142.71	-30.377	0.86363
49.962	1	1.3889	116.91	89.351	110.73	150.65	-33.744	0.63358
49.848	1	1.3889	117.03	98.342	111.18	141.58	-24.552	0.52277
49.543	1	1.3889	117.35	105.47	106.93	139.63	-22.285	0.065722
49.038	1	1.3889	117.88	101.73	111.19	140.72	-22.84	0.41451
48.714	1	1.3889	118.22	109.14	110.78	134.73	-16.509	0.099454
48.314	1	1.3889	118.64	141.47	118.67	95.787	22.853	0.99751
47.533	1	1.3889	119.46	107.82	116.76	133.79	-14.332	0.62361
47.276	1	1.3889	119.73	107	109.92	142.26	-22.533	0.12998
46.6	2	2.7778	120.44	105.05	115.29	140.98	-20.541	0.49871
46.371	1	1.3889	120.68	101.85	120.05	140.15	-19.466	0.93483
45.238	1	1.3889	121.87	98.177	117.04	150.39	-28.517	0.66137
44.714	1	1.3889	122.42	108.79	120.11	138.38	-15.959	0.70933
43.943	2	2.7778	123.23	102.82	120.87	146	-22.767	0.79295
43.067	1	1.3889	124.15	107.69	119.01	145.76	-21.606	0.52393
41.905	1	1.3889	125.37	119.21	123.63	133.26	-7.8874	0.56135
41.781	1	1.3889	125.5	146.18	126.26	104.07	21.43	0.92991
41.438	2	2.7778	125.86	104.3	114.74	158.53	-32.671	0.31951
40.648	2	2.7778	126.69	112.46	126.56	141.04	-14.351	0.98277
40.305	1	1.3889	127.05	105.87	120.53	154.74	-27.689	0.5295
40.105	1	1.3889	127.26	148.83	128.52	104.42	22.838	0.88919
39.819	1	1.3889	127.56	142.02	128.46	112.19	15.366	0.88248
39.429	1	1.3889	127.97	152.14	131.84	99.926	28.044	0.72406
39.286	1	1.3889	128.12	114.71	123.88	145.76	-17.643	0.52004
39.095	3	4.1667	128.32	152.38	129.08	103.51	24.813	0.93884
38.686	1	1.3889	128.75	149.43	129.79	107.02	21.728	0.90401
38.581	1	1.3889	128.86	159.11	134.47	93	35.86	0.68703
38.429	1	1.3889	129.02	151.45	129.44	106.17	22.852	0.96323
38.171	1	1.3889	129.29	116.65	125.14	146.09	-16.8	0.50517
38.029	1	1.3889	129.44	153.71	129.96	104.65	24.793	0.958
35.21	2	2.7778	132.4	106.59	124.83	165.78	-33.379	0.54641
34.81	1	1.3889	132.82	116.2	128.38	153.87	-21.046	0.57872
34.238	4	5.5556	133.42	151.12	133.48	115.66	17.756	0.99372
33.505	1	1.3889	134.19	118.23	131.7	152.62	-18.432	0.73097
32.581	1	1.3889	135.16	117.91	127.9	159.66	-24.496	0.40772
32.41	2	2.7778	135.34	125.94	129.03	151.04	-15.703	0.19626
31.8	1	1.3889	135.98	113.91	135.61	158.4	-22.421	0.96794

Fitting Quality Analysis

The calculated isotropic chemical shift signals were broadened using Gaussian function for fitting the experimental 1D NMR spectrum. The standard deviation σ is constrained to be less or equal to 2.5 ppm, which is acceptable due to packing effects. We fit the spectrum by linear regression with σ varying from 0.1 to 2.5 ppm with a step of 0.1 ppm and the relative fraction from each component is constrained to be positive. Here we choose coefficient of determination (denoted R^2) instead of root-mean-square deviation (RMSD) as a parameter to measure the goodness of fit since the intensity of the spectrum is of magnitude ~10^8, so that RMSDs for different fits are less easily directly compared. The dataset has more than 1000 data points, thus the difference between R^2 and adjusted R^2 is negligible when number of predictors (i.e., number of types of threads for fitting) is less than 6. Thus, we simply compare R^2 for different fits.

The best fitting results for different model mixtures (shown with the combinations of the first column and row in Table S4-6) are shown in Table S4-6. The highest fitting quality in term of R^2 (highlighted yellow in Table S4) is achieved with a combination of the degree-4 threads IV-7 and IV-8, and the degree-6 threads square polymer and stiffchiral-3. Six other combinations (highlighted red) have relatively large values of R^2, however, the corresponding fitting curves fail to reproduce the shape of experimental spectrum, as shown in Figure S12 (b)-(g). The fit to the mixture of fully axially disordered degree 6 class 3 and degree 4 threads performs as well as the fit with degree 6 class 2 included as well (Table S5). The best fit to the [4+2]-constrained ensemble is obtained with all three types of threads as shown in Table S6, while the other combinations cannot produce acceptable fitting quality.

Table S4: Coefficients of determination (R^2) of fits with different combinations of axially ordered candidate threads. A given entry includes the threads from both row and column labels.

R^2	square polymer	stiffchiral-3	square polymer stiffchiral-3
IV-7	0.9300	0.8423	0.9758
IV-8	0.9457	0.8139	0.9775
IV-11	0.6394	0.8272	0.8896
IV-13	0.7429	0.7347	0.8876
IV-7,8	0.9561	0.8429	0.9856
IV-7,11	0.8179	0.8844	0.9196
IV-7,13	0.9313	0.4281	0.5038
IV-8,11	0.9213	0.8844	0.9764
IV-8,13	0.9475	0.5822	0.8967
IV-11,13	0.5913	0.3707	0.884
IV-7,8,11	0.9088	0.8899	0.9696
IV-7,8,13	0.9565	0.5125	0.6202
IV-7,11,13	0.7984	0.3713	0.4412
IV-8,11,13	0.8775	0.4551	0.6091
IV-7,8,11,13	0.8897	0.4784	0.5645

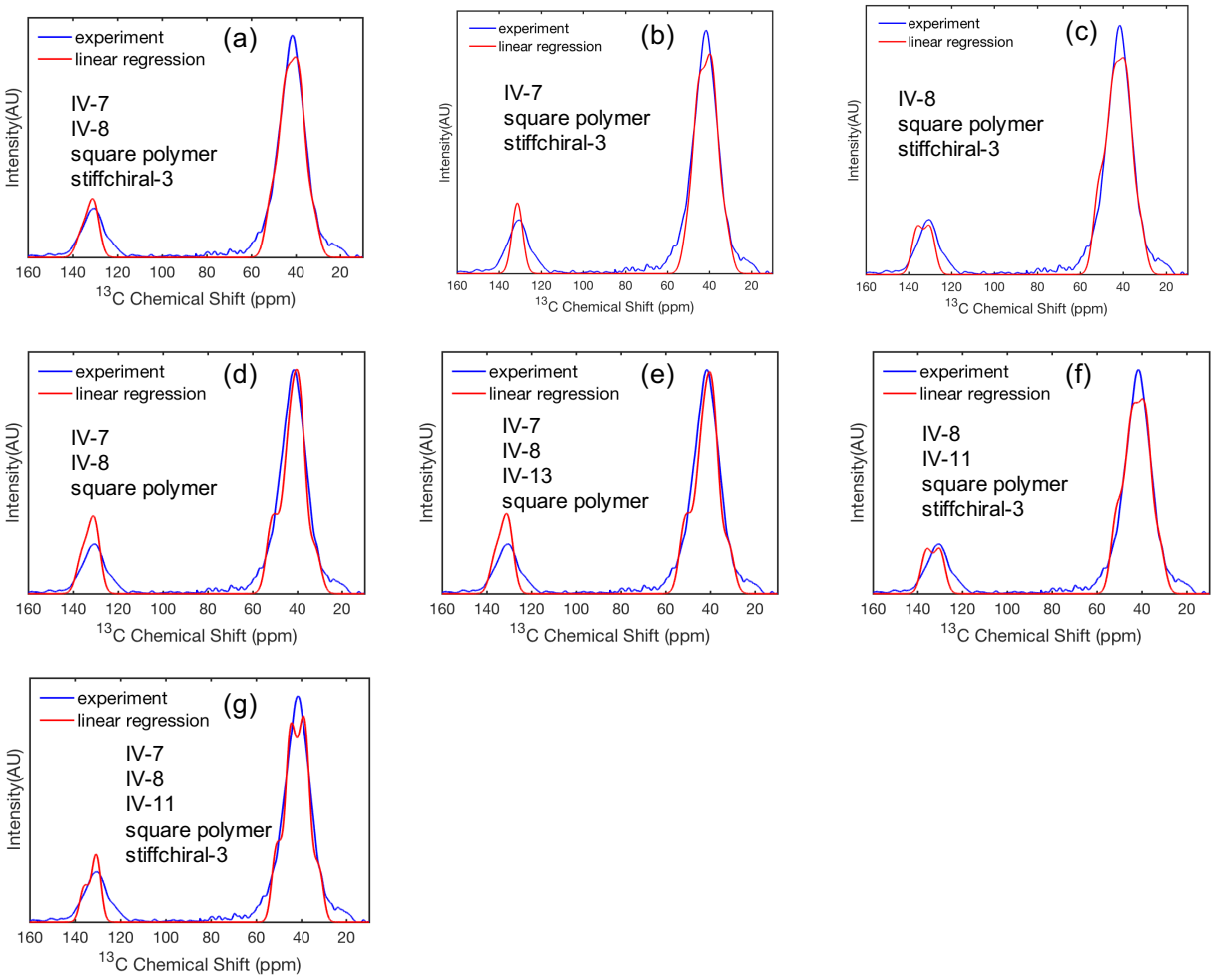

Figure S12: Fits of the experimental data with different mixtures of axially ordered degree-4 and degree-6 candidate nanothreads by multi-linear regression. (a) shows the best fitting result with specific mixture of candidate threads. Fits in (b)-(g) cannot reproduce the shape of main peaks in the spectrum as well as that in (a). The simulated spectra were broadened by convolution with a Gaussian with $\sigma \leq 2.5$ ppm.

Table S5: Coefficients of determination (R^2) of fits with different combinations of fully axially disordered threads.

R^2	degree-6 class2	degree-6 class3	degree-6 class2 degree-6 class3
degree 4	0.916	0.978	0.9781

Table S6: Coefficients of determination (R^2) of fits with different combinations of [4+2] axially disordered threads.

R^2	[4+2] degree-6 class2	[4+2] degree-6 class3	[4+2] degree-6 class2 [4+2] degree-6 class3
[4+2] degree-4	0.9626	0.9449	0.971

We characterized the sensitivity of variations on fraction of each thread included in fits shown in the main text, Figure 3 (18% IV-7, 25% IV-8, 36% square polymer and 21% stiffchiral-3) and Figure 5 (b) (49% degree 6-class3 and 51% degree 4 for fully axially disordered threads; 36% [4+2] degree 6 class 2, 24% [4+2] degree 6 class 3, and 40% [4+2] degree 4 threads). Starting with the fractions for each component in the best fits mentioned above, we explored the variations within ± 30% for each component as a fixed predictor (for those with less than 30% fraction, the lower bound is set to be 0%) and performed fitting by linear regression with the remaining predictors, and obtained the variation of the relative fraction in which the coefficient of determination only changes within 0.5% from that of the best fit to ensure the fitting quality to be acceptable. For example, in Table S7, the "fix IV-7" columns show that the fraction of IV-7 in

the fit with mixture of IV-7, IV-8, square polymer and stiffchiral-3, can vary in a range of 4–33% while the corresponding R^2 is still larger than 99.5% of the R^2 achieved by the best fit (where R^2=0.9856). Tables S7-9 provide the variations for each predictor (highlighted red) and the fractions for other threads in each scenario. As shown in Figure S7, the fractions of IV-7 and IV-8 have a relatively large range of variations compared with square polymer and stiffchiral-3. However, those two components are strongly anticorrelated, and the error bar for the fraction of total degree 4 threads is ± 6%. The fractions of the degree-6 class 3 and degree-4 threads can change in a range of around ± 9% from those of the best fit (49% and 51%, respectively). The fit to [4+2] constrained ensembles is less sensitive to the variations of the fractions for each component, which can vary by ± 10% while the R^2 only changes within 0.5%.

Table S7: The lower and upper limits (highlighted red) for the relative fractions of components and the corresponding contributions from the remaining threads in the mixture of IV-7, IV-8, square polymer and stiffchiral-3.

percentage (%)	fix IV-7		fix IV-8		fix square polymer		fix stiffchiral-3	
IV-7	4	33	31	6	22	15	19	17
IV-8	38	11	8	40	22	27	30	20
square polymer	37	35	36	36	30	41	38	33
stiffchiral-3	21	21	25	18	26	17	13	30

Table S8: The lower and upper limits (highlighted red) of fractions for degree-6 class 3 and degree-4 for the fit to unconstrained ensemble.

percentage (%)	fix degree-6 class3		fix degree-4	
degree 6-class3	40	58	58	41

degree 4	60	42	42	59

Table S9: The lower and upper limits (highlighted red) of fractions for degree-6 class2, degree-6 class 3 and degree 4 for the fit to [4+2]-constrained ensemble.

percentage (%)	fix [4+2] degree-6 class2		fix [4+2] degree-6 class2		fix [4+2] degree-4	
[4+2] degree 6-class2	21	52	49	23	41	32
[4+2] degree 6-class3	37	9	6	41	31	17
[4+2] degree 4	42	39	45	36	28	51

References

1. Duan, P.; Li, X.; Wang, T.; Chen, B.; Juhl, S. J.; Koeplinger, D.; Crespi, V. H.; Badding, J. V.; Schmidt-Rohr, K. The Chemical Structure of Carbon Nanothreads Analyzed by Advanced Solid-State NMR. *J. Am. Chem. Soc.* **2018**, 140, (24), 7658–7666.
2. Xu, E.-s.; Lammert, P. E.; Crespi, V. H. Systematic Enumeration of sp^3 Nanothreads. *Nano Lett.* **2015,** 15, (8), 5124-5130.
3. Chen, B.; Hoffmann, R.; Ashcroft, N. W.; Badding, J.; Xu, E.; Crespi, V. Linearly Polymerized Benzene Arrays As Intermediates, Tracing Pathways to Carbon Nanothreads. *J. Am. Chem. Soc.* **2015,** 137, (45), 14373-14386.
4. Gottlieb, H. E.; Kotlyar, V.; Nudelman, A. NMR chemical shifts of common laboratory solvents as trace impurities. *J. Org. Chem.* **1997,** 62, (21), 7512-7515.
5. Yoneda, J. D., Leal, K. Z. & Seidl, P. R. ^{13}C Chemical Shifts of Di-and Triamantanes: A Similar Effect of a Methyl Substituent on Adamantanes. *Ann. Magn. Reson.* **2005,** 4, (1-2), 9-16.
6. Maryasin, B.; Olbrich, M.; Trauner, D.; Ochsenfeld, C. Calculated Nuclear Magnetic Resonance Spectra of Polytwistane and Related Hydrocarbon Nanorods. *J. Chem. Theory Comput.* **2015,** 11, (3), 1020-1026.
7. Fitzgibbons, T. C.; Guthrie, M.; Xu, E.-S.; Crespi, V. H.; Davidowski, S. K.; Cody, G. D.; Alem, N.; Badding, J. V. Benzene-derived carbon nanothreads. *Nat. Mater.* **2014,** 14, 43.
8. Bennett, A. E.; Rienstra, C. M.; Auger, M.; Lakshmi, K. V.; Griffin, R. G. Heteronuclear decoupling in rotating solids. *J. Chem. Phys.* **1995,** 103, (16), 6951-6958.
9. Schmidt-Rohr, K.; Mao, J. D. Efficient CH-Group Selection and Identification in ^{13}C Solid-State NMR by Dipolar DEPT and ^{1}H Chemical-Shift Filtering. *J. Am. Chem. Soc.* **2002,** 124, (46), 13938-13948.
10. Dixon, W. T.; Schaefer, J.; Sefcik, M. D.; Stejskal, E. O.; McKay, R. A. Total suppression of sidebands in CPMAS C-13 NMR. *J. Magn. Reson.* **1982,** 49, (2), 341-345.

11. Caravatti, P.; Braunschweiler, L.; Ernst, R. R. Heteronuclear correlation spectroscopy in rotating solids. *Chem. Phys. Lett.* **1983,** 100, (4), 305-310.
12. Bielecki, A.; Kolbert, A. C.; De Groot, H. J. M.; Griffin, R. G.; Levitt, M. H., Frequency-Switched Lee—Goldburg Sequences in Solids. In *Advances in Magnetic and Optical Resonance*, Warren, W. S., Ed. Academic Press: 1990; Vol. 14, pp 111-124.
13. Hahn, E. L. Spin Echoes. *Phys. Rev.* **1950,** 80, (4), 580-594.
14. Bodenhausen, G.; Freeman, R.; Turner, D. L. Suppression of artifacts in two-dimensional J spectroscopy. *J. Magn. Reson.* **1977,** 27, (3), 511-514.
15. Rhim, W. K.; Elleman, D. D.; Vaughan, R. W. Analysis of multiple pulse NMR in solids. *J. Chem. Phys.* **1973,** 59, (7), 3740-3749.
16. Mao, J. D.; Schmidt-Rohr, K. Separation of aromatic-carbon ^{13}C NMR signals from di-oxygenated alkyl bands by a chemical-shift-anisotropy filter. *Solid State Nucl. Magn. Reson.* **2004,** 26, (1), 36-45.
17. Hohwy, M.; Rienstra, C. M.; Jaroniec, C. P.; Griffin, R. G. Fivefold symmetric homonuclear dipolar recoupling in rotating solids: Application to double quantum spectroscopy. *J. Chem. Phys.* **1999,** 110, (16), 7983-7992.
18. Johnson, R. L.; Anderson, J. M.; Shanks, B. H.; Fang, X.; Hong, M.; Schmidt-Rohr, K. Spectrally edited 2D ^{13}C – ^{13}C NMR spectra without diagonal ridge for characterizing ^{13}C-enriched low-temperature carbon materials. *J. Magn. Reson.* **2013,** 234, 112-124.