Supporting Information:

Structural Analysis of the End-groups and Substructures of Commercial Poly (ethylene terephthalate) by Multiple-WET ^{1}H/ ^{13}C-NMR

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Table S1. Chemical shifts and spin-lattice relaxation times T_1 of PET solution.

Figure S1. MWET(n) 2D and 3D, IR pulse sequences.

Figure S2. The excitation profile for MWET(n) with n=10 points suppression at 900 MHz.

Figure S3. ^{1}H NMR spectrum of the 3% PET solution at 900 MHz.

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a) ^{1}H-decoupled ^{13}C spectrum with NOE; b) ^{1}H-decoupled ^{13}C spectrum without NOE.

Figure S8. DEPT-135 spectrum of the 10% PET solution at 150 MHz.

Figure S9. MWET(7) HSQC spectrum of the 3% PET solution at 900 MHz.

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Figure S11. MWET(7) HMBC spectrum of the 3% PET solution at 900 MHz.

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Figure S13. Stacked plot of MWET(7)-IR data of the 3% PET solution at 900 MHz.

Table S1. Chemical shifts and spin-lattice relaxation times T_1 of PET solution.

		^{1}H Chemical shift δ_H		^{13}C Chemical shift δ_C		$T_1(^1H)$ / s (3% solution)	
		in this work (3% solution)	in previous work[*1]	in this work (10% solution)	in previous work[*2]	IR	MWET-IR
$CHCl_3$ / $CDCl_3$		7.240		77.00			
TA	*a*	8.076		129.99		3.26	
	b			133.60			
	c			167.37	167.7		
EG	*d*	4.678	4.676	63.50	63.3	1.25	
End-EG	*f*	4.450	4.470	66.90	66.6	1.24	1.23
	g	3.990	3.967	61.22	60.9	1.09	1.11
	e			167.89			
DEG	*i*	4.489	4.492	64.65		0.96	0.99
	j	3.933	3.746	69.00		1.01	1.03
	h			167.57			
End-DEG	*l*	(4.497)[*3]	4.645	(64.14)[*3]			
	m	(3.854)[*3]	3.813	(68.85)[*3]			
	n	3.662	3.645	71.84			
	o	3.764	3.868	61.67			
TEG	*q*	(4.457)[*3]	4.457	(64.44)[*3]			
	r	3.868	3.823	(69.21)[*3]			
	s	3.749	3.700	70.25			
methyl ester	*u*	(3.931)[*3]	(3.931)[*4]	52.96			
	t			168.42			
vinyl	*a*	5.157		100.35			1.89
	b′	4.815					1.60
	c′	7.336		140.84			3.91
IA	*d′*	8.659		131.09			3.86
	f′	8.223		134.80			3.00
	g′	7.550		128.79			3.28

*1 Amiya, S.; Mathumura, K.; Taniguchi, T. Microstructure of Poly (ethylene terephthalate). *Anal. Sci.* **1991**, *7 Suppl.*, 1649–1650.

*2 Fox, B.; Moad, G.; van Diepen, G.; Willing, I.; Cook, W. D. Characterization of Poly(ethylene terephthalate) and Poly(ethylene terephthalate) Blends. *Polymer* **1997**, *38*, 3035–3043.

*3 overlapped.

4 model compound by Amiya, S. et.al.[1]

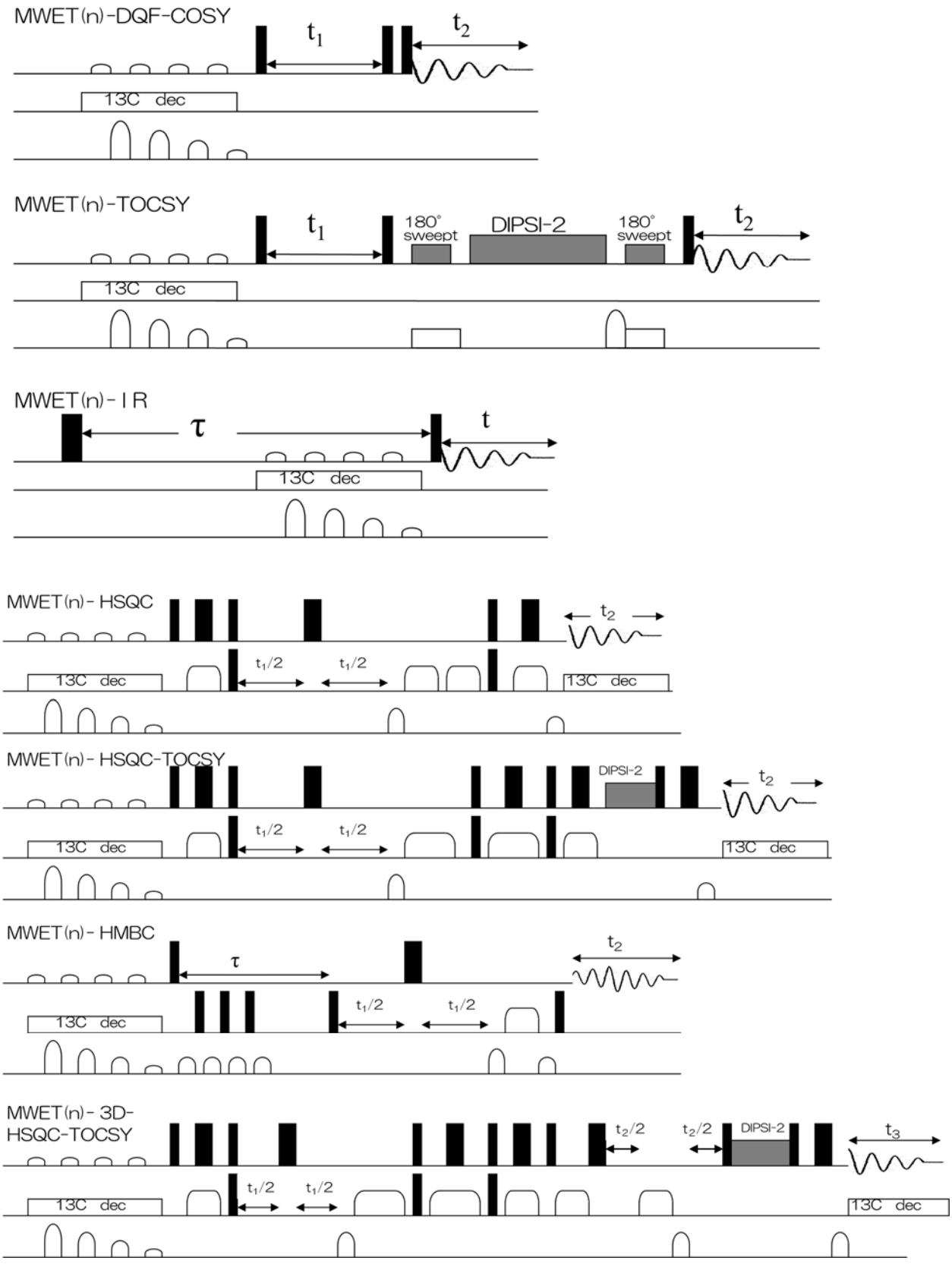

Figure S1. MWET(n) 2D and 3D, IR pulse sequences.

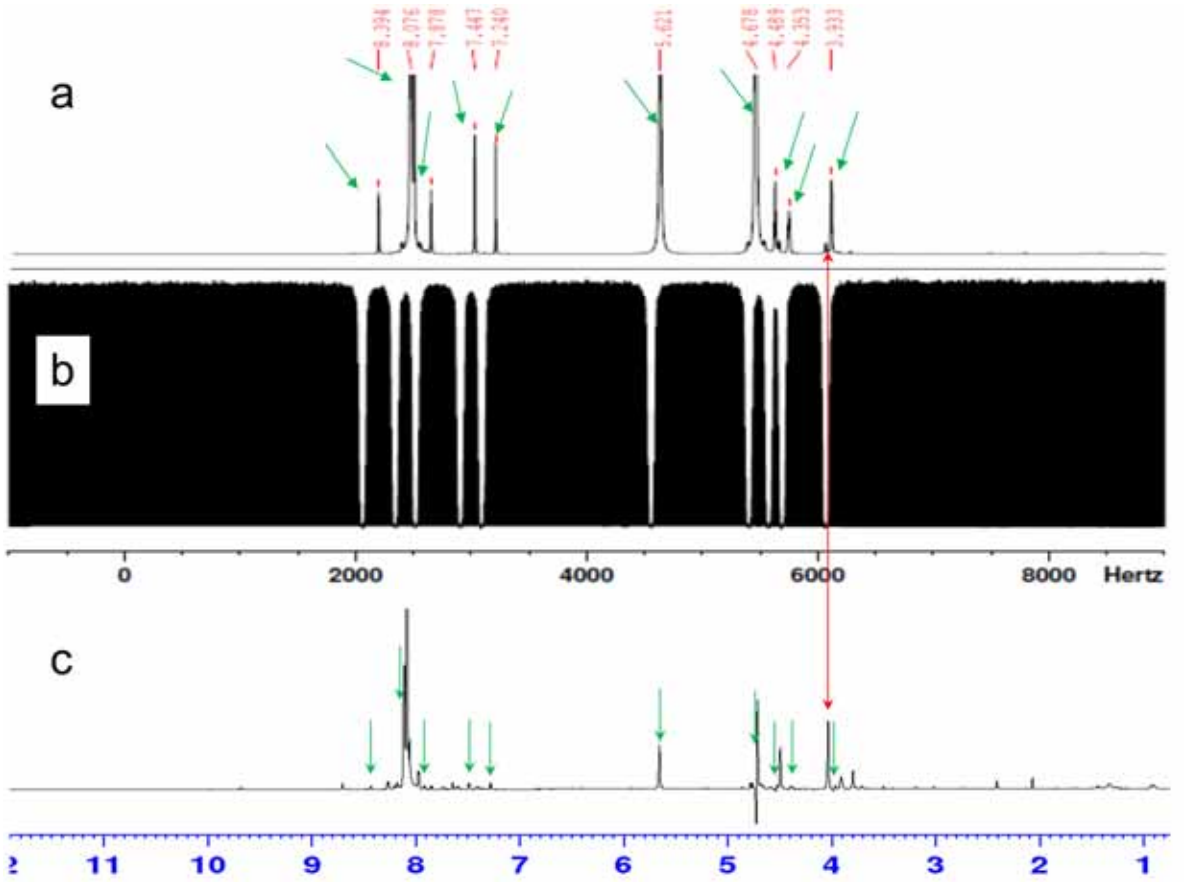

Figure S2. The excitation profile for MWET(n) with n=10 points suppression (marked with green arrows (→)) of the 3% PET solution at 900 MHz. Suppression points were taken at δ_H 8.08 (TA); 4.68 (EG); 5.62 (OH); 7.24 (chloroform); 8.39, 7.88 and 7.45 (pyridine); 4.50 and 3.93 (DEG); and 4.35 (HFIP-d_2). a) ^{1}H NMR spectrum; b) the excitation profile for MWET(10) recorded by 5 Hz step; c) spectrum of MWET(10).

a) 16 scans, total experiment time: 7 min 30 sec.

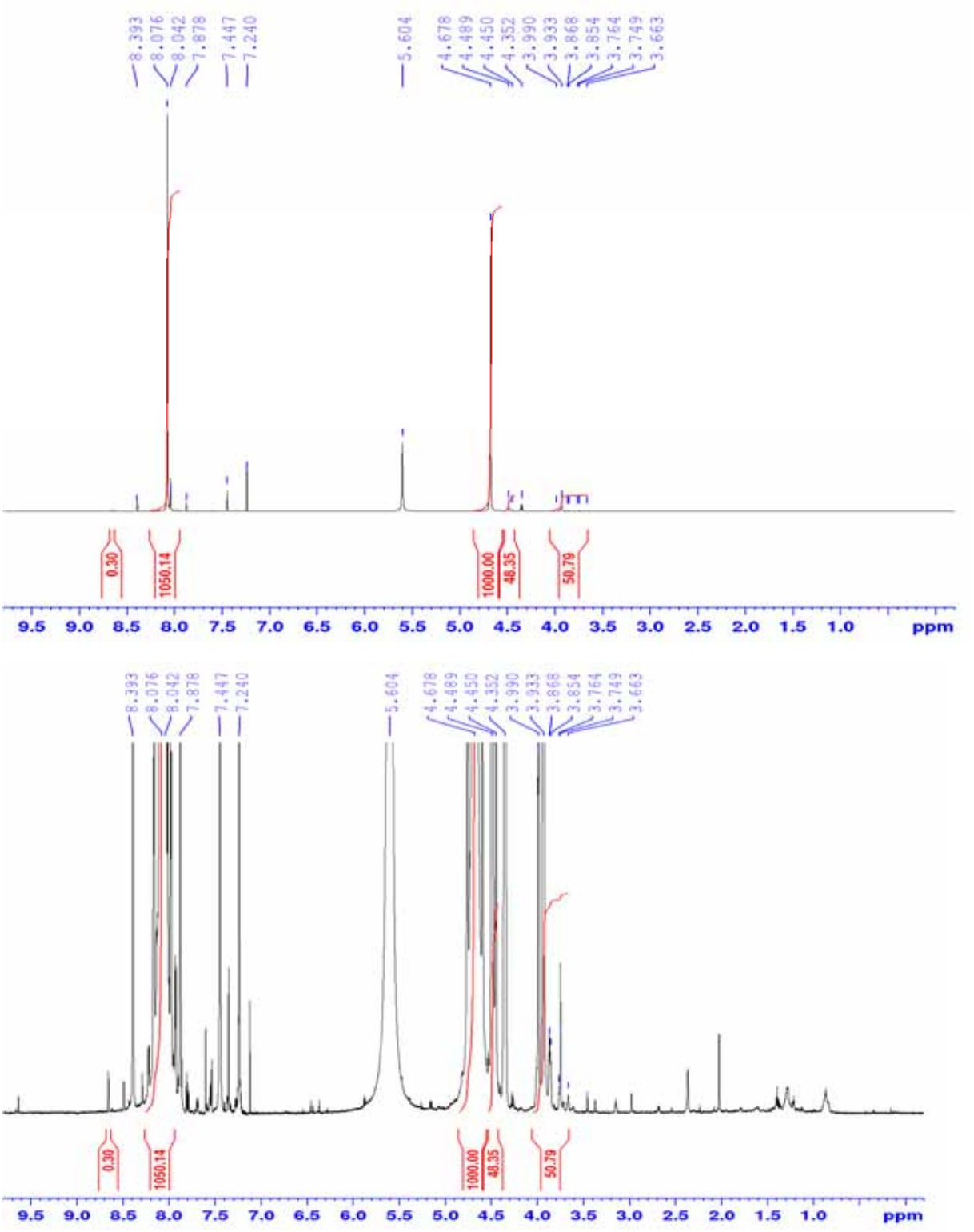

Figure S3. ^{1}H NMR spectrum of the 3% PET solution at 900 MHz with a cryogenic probe. 90° pulse, repetition time (AQ+D1): 25 s, DIGMOD: baseopt and DE: 55 μs.

b) 128 scans, total experiment time: 54 min 14 sec.

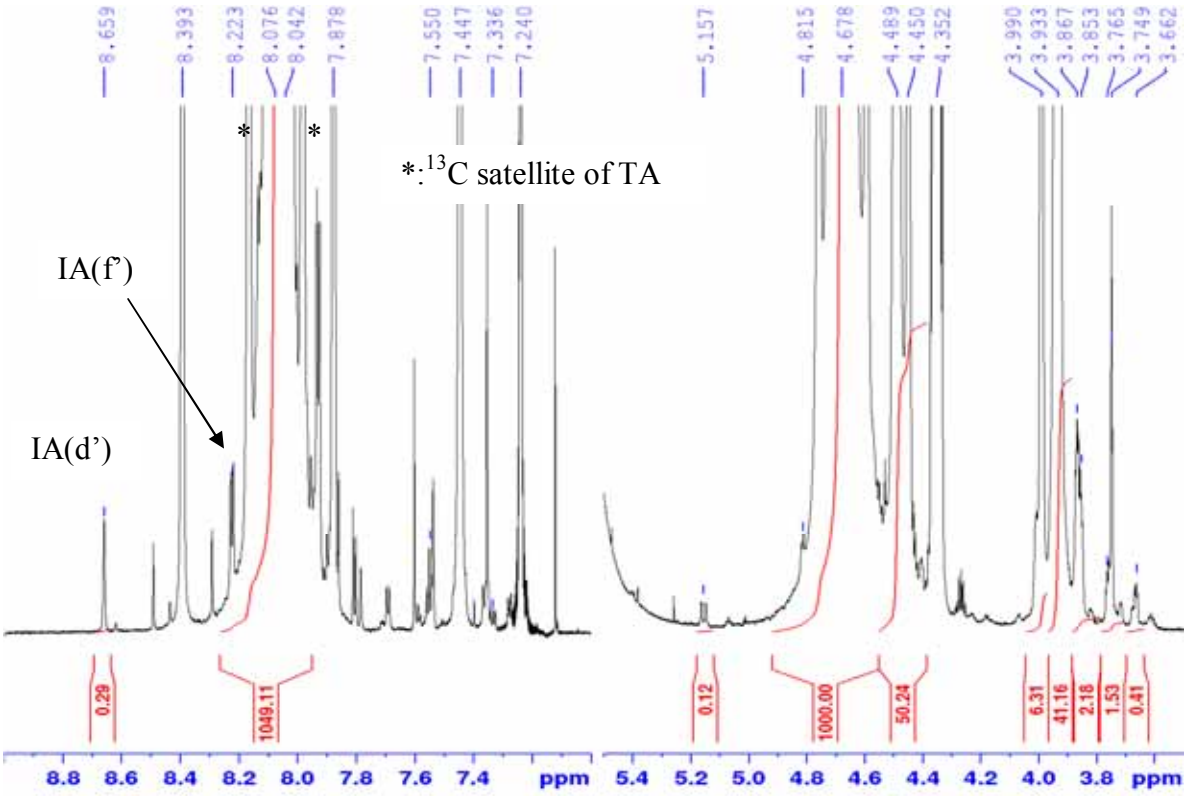

Figure S3. (Continued)

a) 16 scans, total experiment time: 7 min 33 sec.

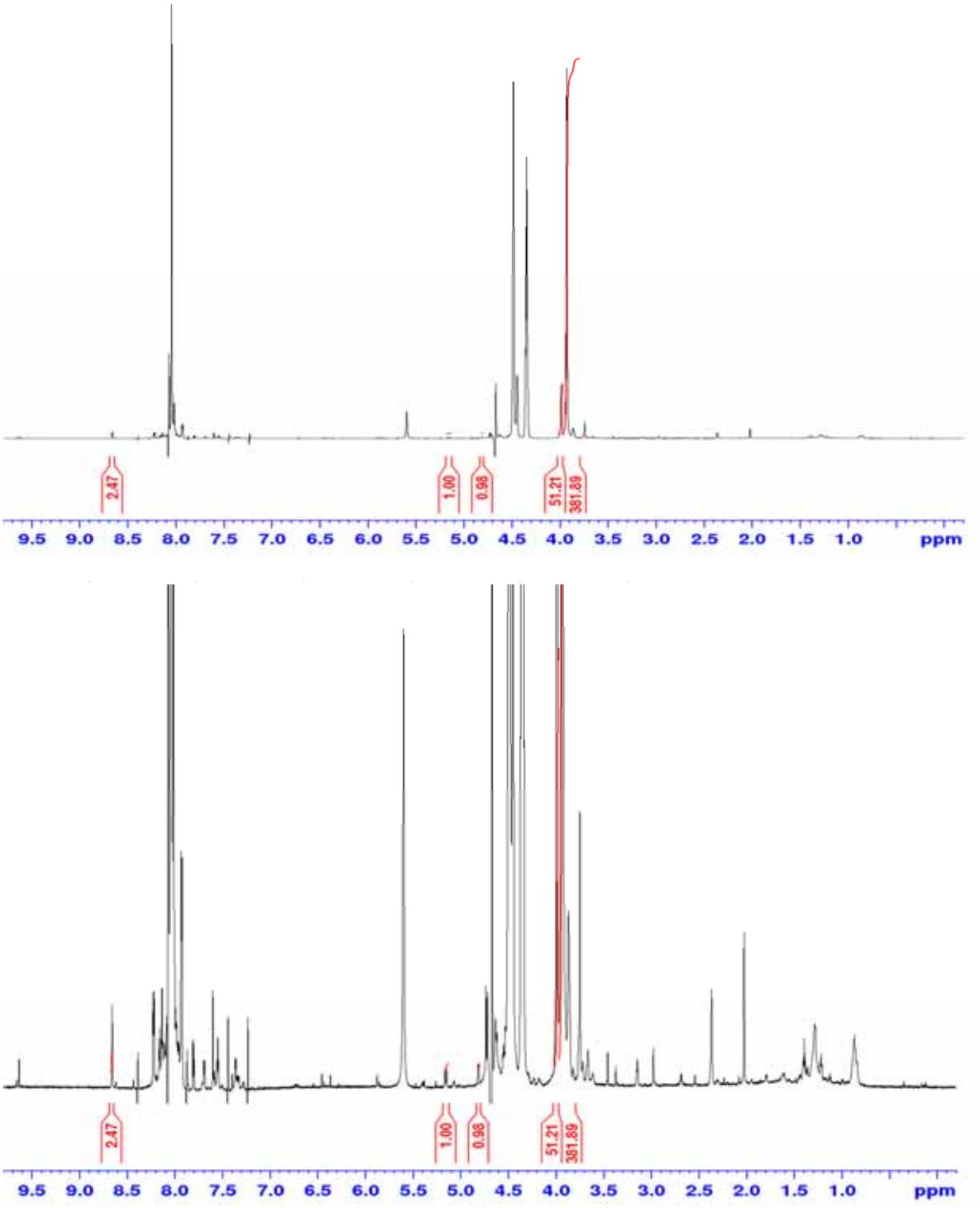

Figure S4. MWET(7) ^{1}H NMR spectrum of the 3% PET solution with 7 points suppression at 900 MHz with a cryogenic probe. Suppression points were set at δ_H 8.08 (TA); 4.68 (EG); 5.62 (OH); 7.24 (chloroform); and 8.39, 7.88, and 7.45 (pyridine). 90° pulse, repetition time: 25 s, DIGMOD: baseopt, DE: 55 μs

b) 128 scans, total experiment time: 54 min 31 sec.

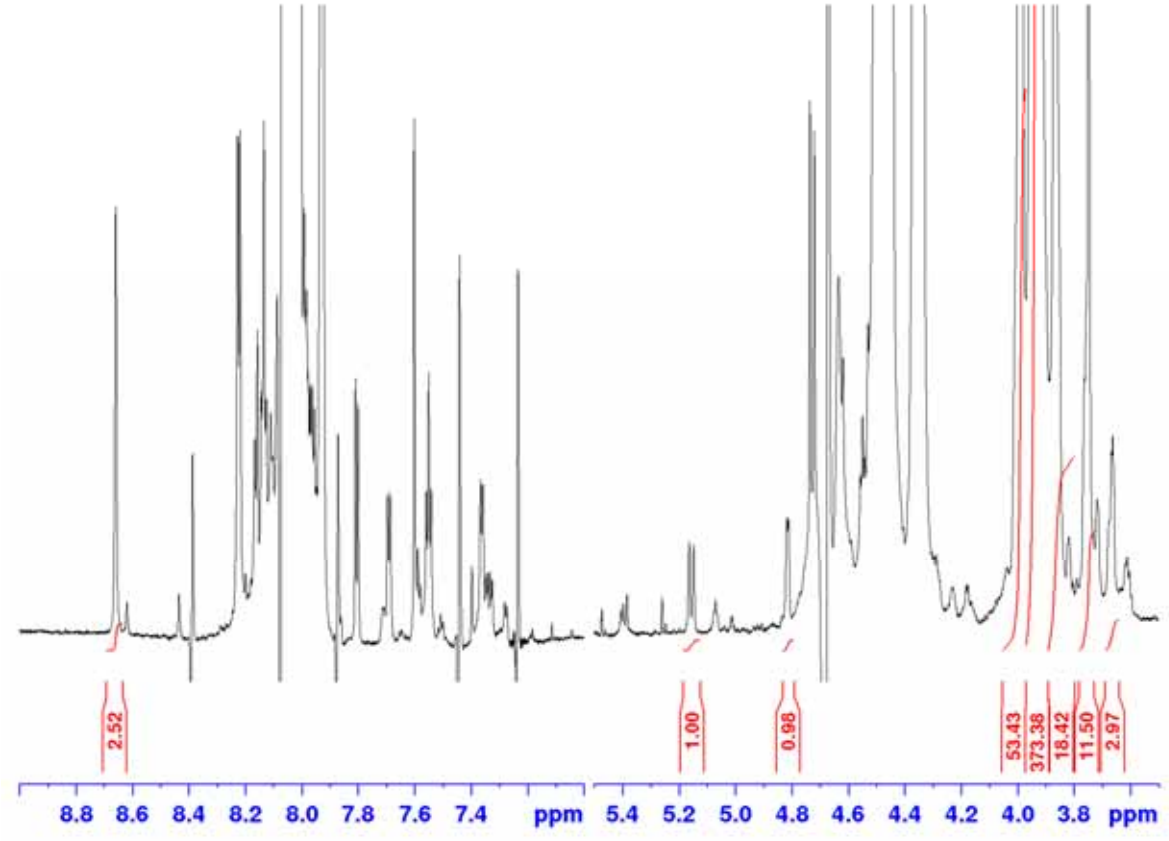

Figure S4. (Continued)

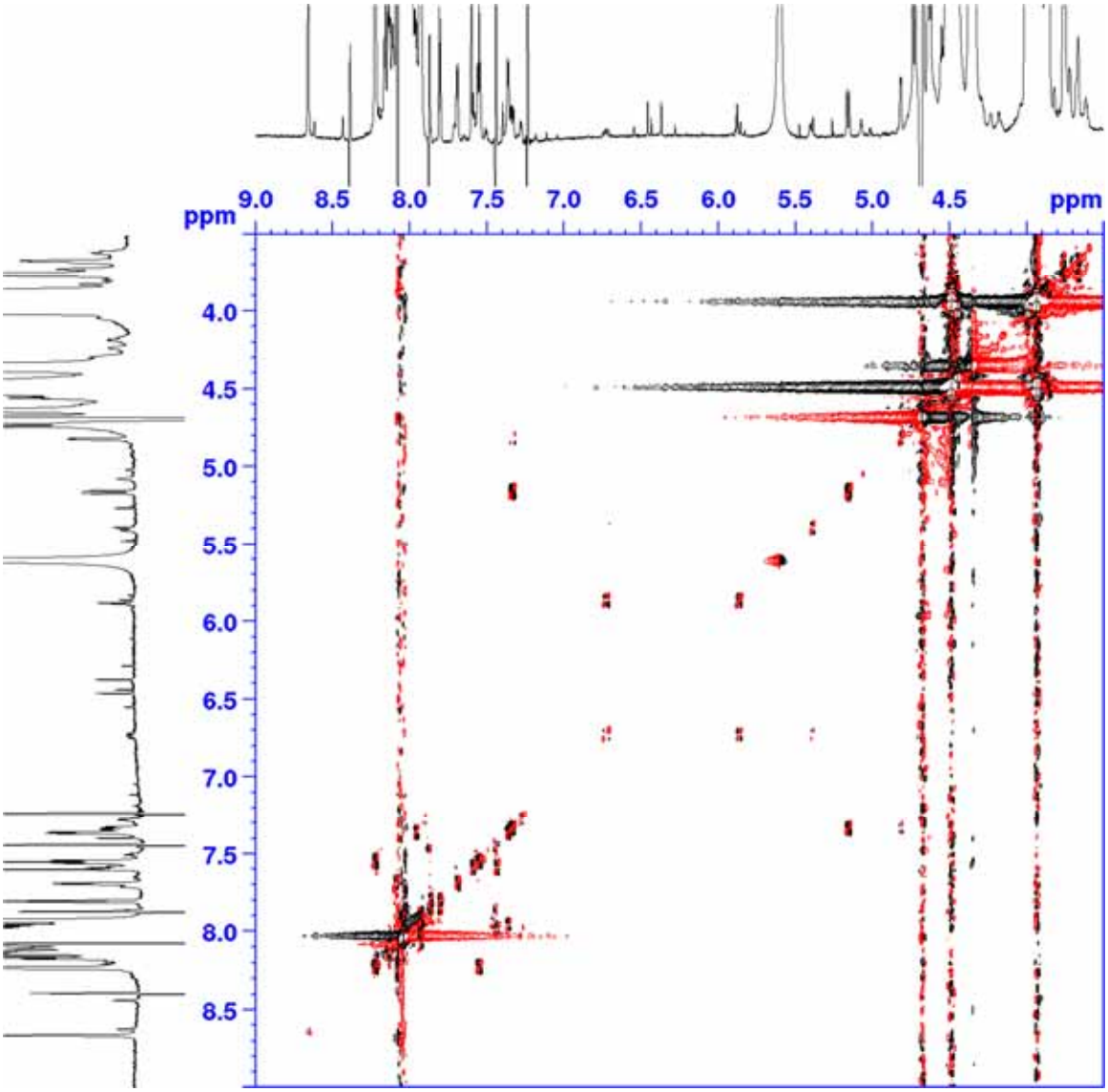

Figure S5. MWET(7) DQF-COSY spectrum of the 3% PET solution with 7 points suppression as same as Figure S4 at 900 MHz. 32 scans, repetition time: 2.4 s, total experiment time: 7 h 41 min, spectral width SW: 12,594 (^{1}H) × 12,626 (^{1}H) Hz, acquired data points: 336 (t1) × 2,048(t2) and processed data points: 1,024 (F1) × 4,096 (F2), DIGMOD: baseopt, DE: 30 μs.

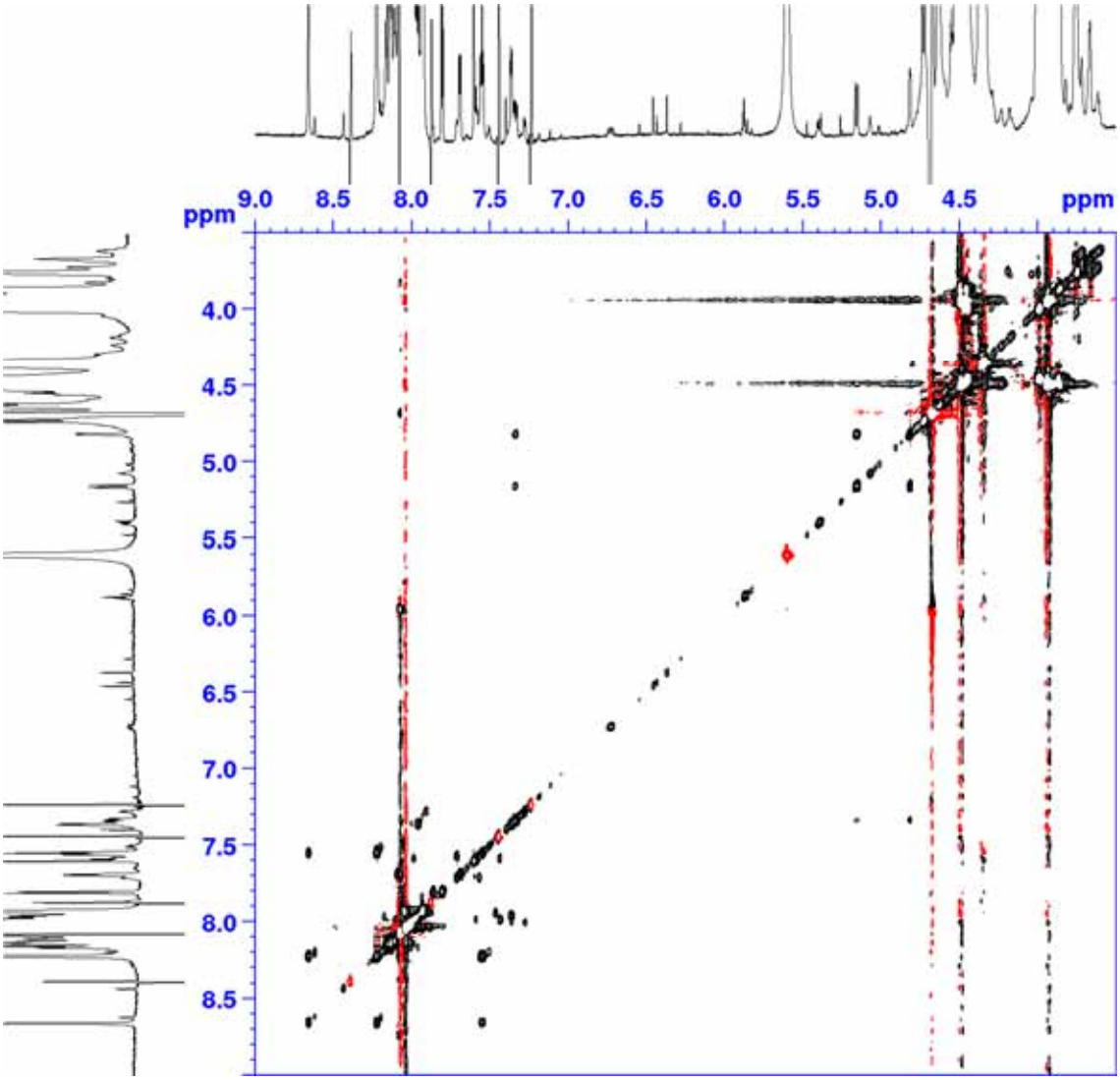

Figure S6. MWET(7) TOCSY spectrum of the 3% PET solution with 7 points suppression as same as Figure S4 at 900 MHz. 8 scans, repetition time: 2.3 s, total experiment time: 2 h 14 min, TOCSY (DIPSI2) mixing time: 140 ms, spectral width SW: 12,594 (^{1}H) × 12,626 (^{1}H) Hz, acquired data points: 384 (t1) × 2,048 (t2) and processed data points: 1,024 (F1) × 4,096 (F2) , DIGMOD: baseopt, DE: 30 μs..

a) ^{1}H-decoupled ^{13}C spectrum with NOE. 100,000 scans, 30° pulse, repetition time: 2.7 s. total experiment time: 3 d 4 h 45 min, ^{1}H-decoupling: bi_waltz65_256*[5]. Baseline was corrected by au-program “abs13c”.

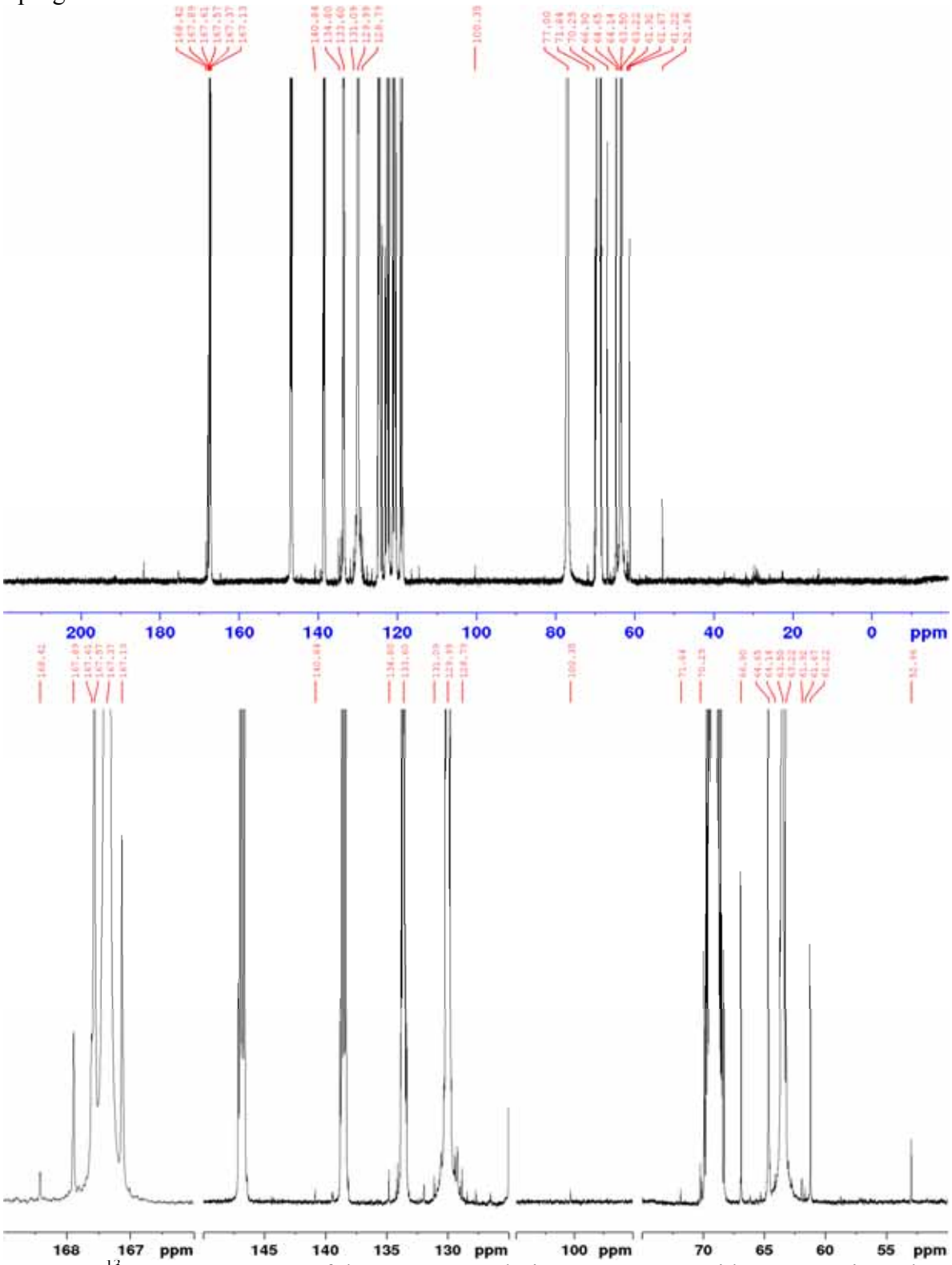

Figure S7. ^{13}C NMR spectrum of the 10% PET solution at 150 MHz with a cryogenic probe.

b) ^{1}H-decoupled ^{13}C spectrum without NOE. 42,200 scans, 30° pulse, repetition time: 5 s, AQ: 0.91 s, total experiment time: 2 d 11 h 3 min. ^{1}H-decoupling: waltz65, pre-decoupling time: 2 ms, maximum variation: 60%. Baseline was corrected by au-program "abs13c".

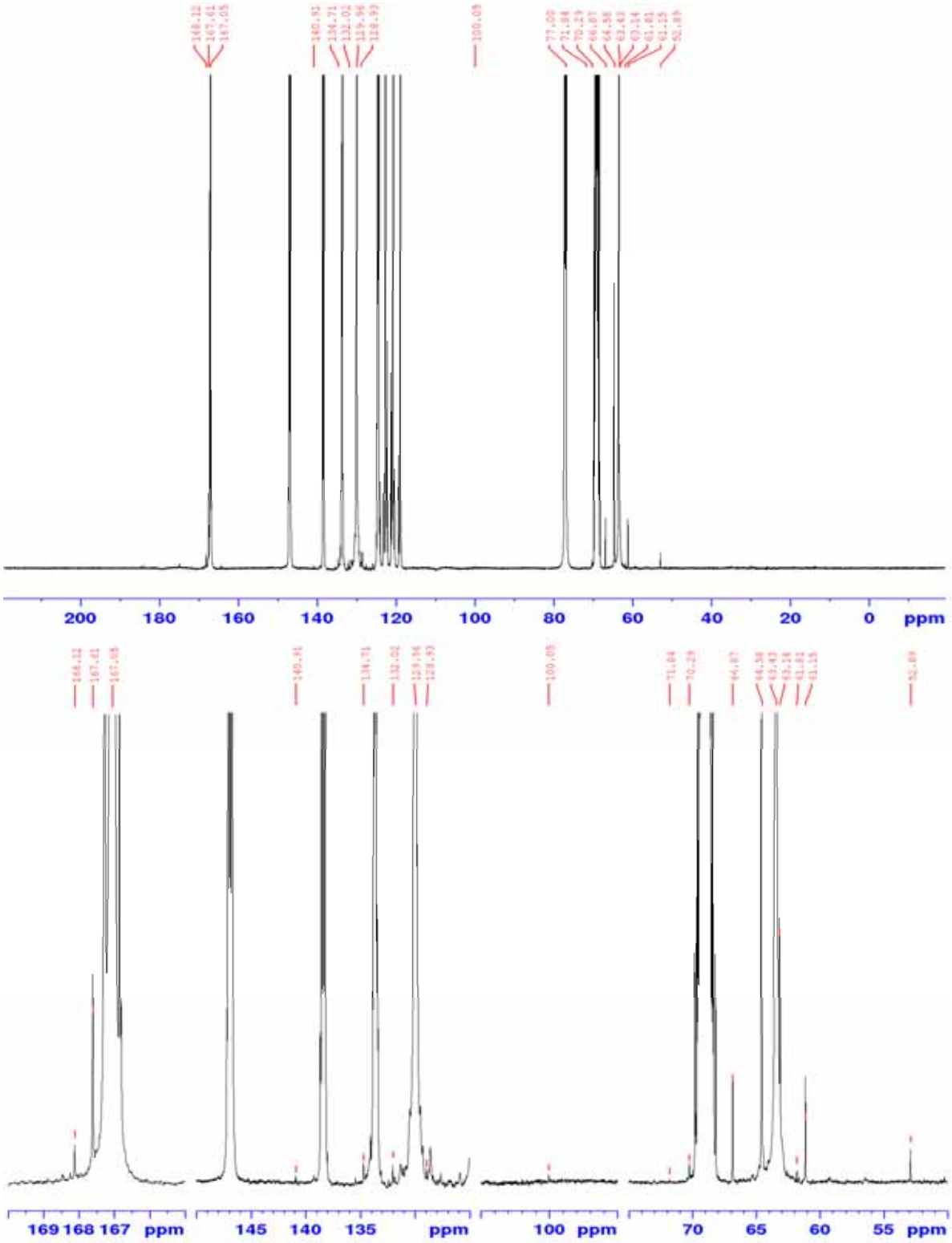

Figure S7. (Continued)

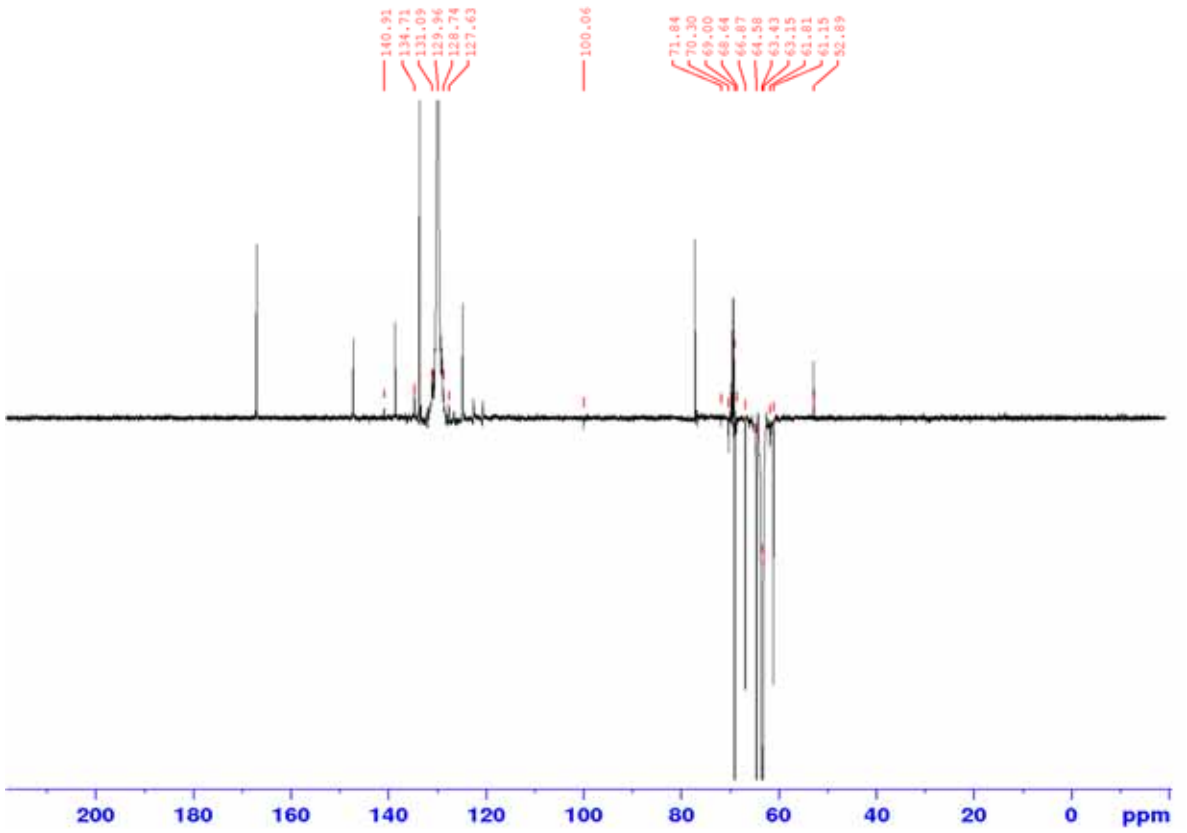

Figure S8. DEPT-135 spectrum of the 10% PET solution at 150 MHz. 4,096 scans, repetition time: 3.9 s. total experiment time: 4 h 32 min, ^{1}H-decoupling: bi_waltz65_64pl*[5](7). Baseline was corrected by au-program "abs13c".

*5: Zhou Z.; Kummerle R.; Qiu X.; Redwine D.; Cong R.; Taha A.; Baugh D.; Winniford B. A new decoupling method for accurate quantification of polyethylene copolymer composition and triad sequence distribution with ^{13}C NMR *J.Magn.Reson.* 187 (2007) 225.

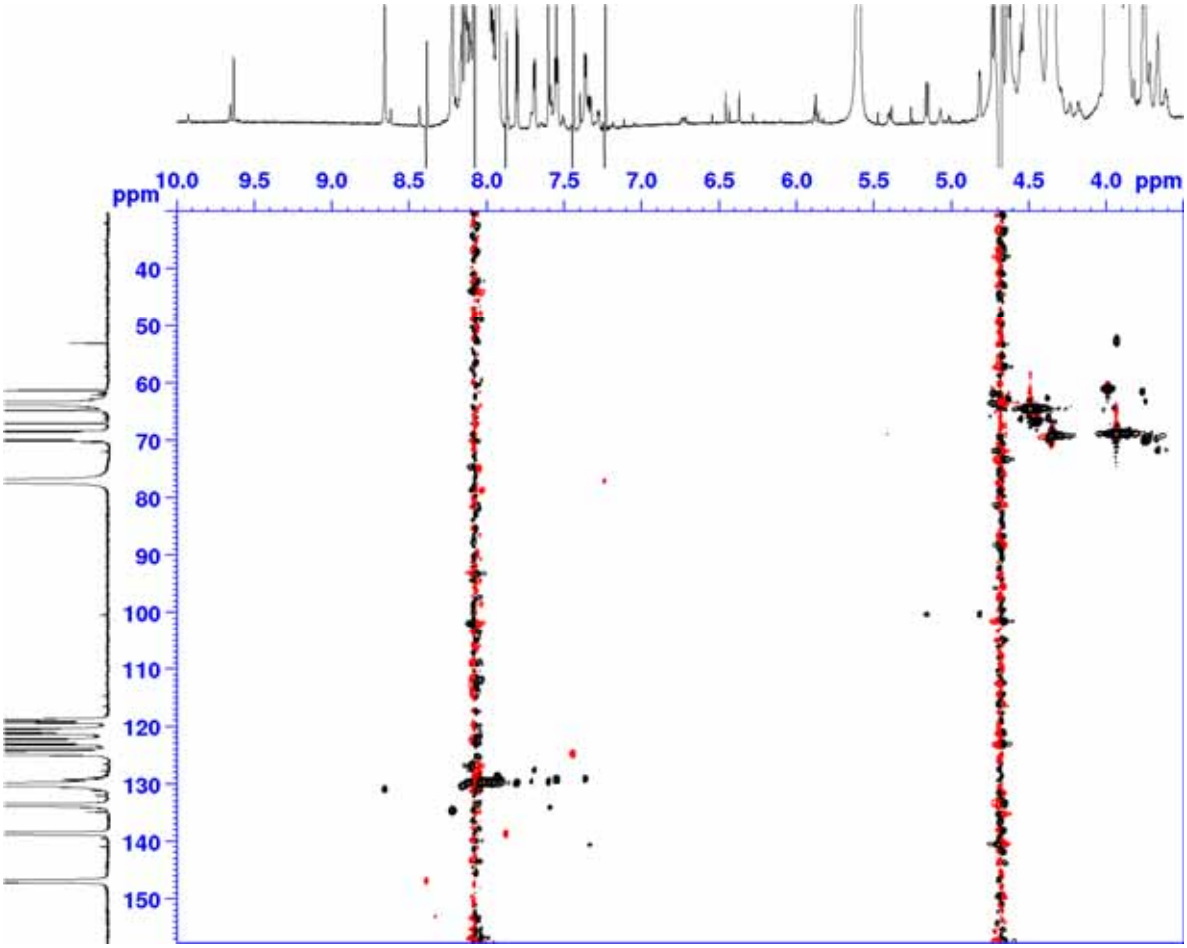

Figure S9. MWET(7) HSQC spectrum of the 3% PET solution with 7 points suppression as same as Figure S4 at 900 MHz. 88 scans, repetition time: 2.5 s, total experiment time: 12 h 23 min, spectral width SW: 37,313 (^{13}C) × 14,423 (^{1}H) Hz, acquired data points: 192 (t1) × 2,048 (t2) and processed data points: 1,024 (F1) × 4,096 (F2) , DIGMOD: baseopt, DE: 30 μs..

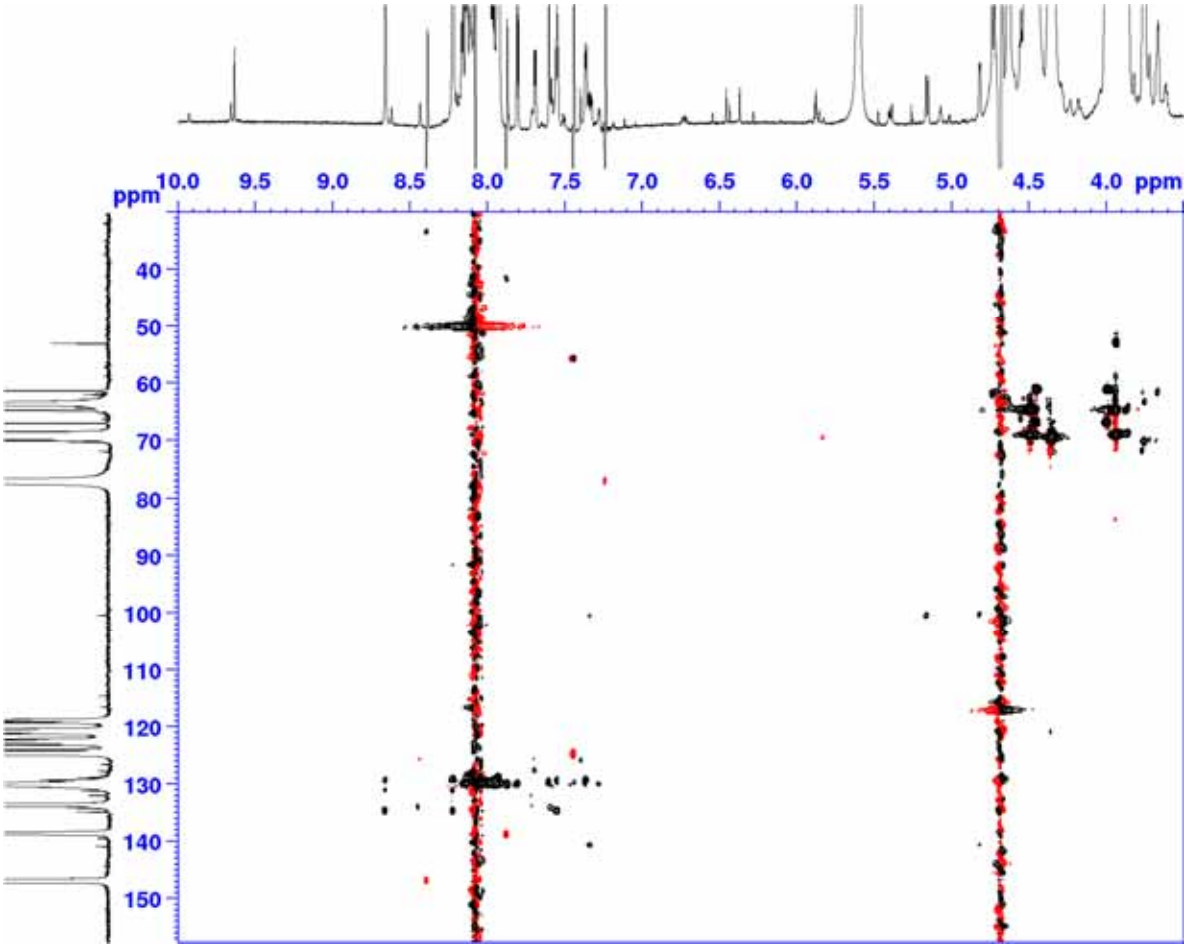

Figure S10. MWET(7) HSQC-TOCSY spectrum of the 3% PET solution with 7 points suppression as same as Figure S4 at 900 MHz. 128 scans, repetition time: 5.6 s, total experiment time: 1 d 16 h 12 min, TOCSY(DIPSI2) mixing time:140 ms, spectral width SW: 37,313 (^{13}C) × 14,423 (^{1}H) Hz, acquired data points: 192 (t1) × 2,048 (t2) and processed data points: 1,024 (F1) × 4,096 (F2) , DIGMOD: baseopt, DE: 30 μs..

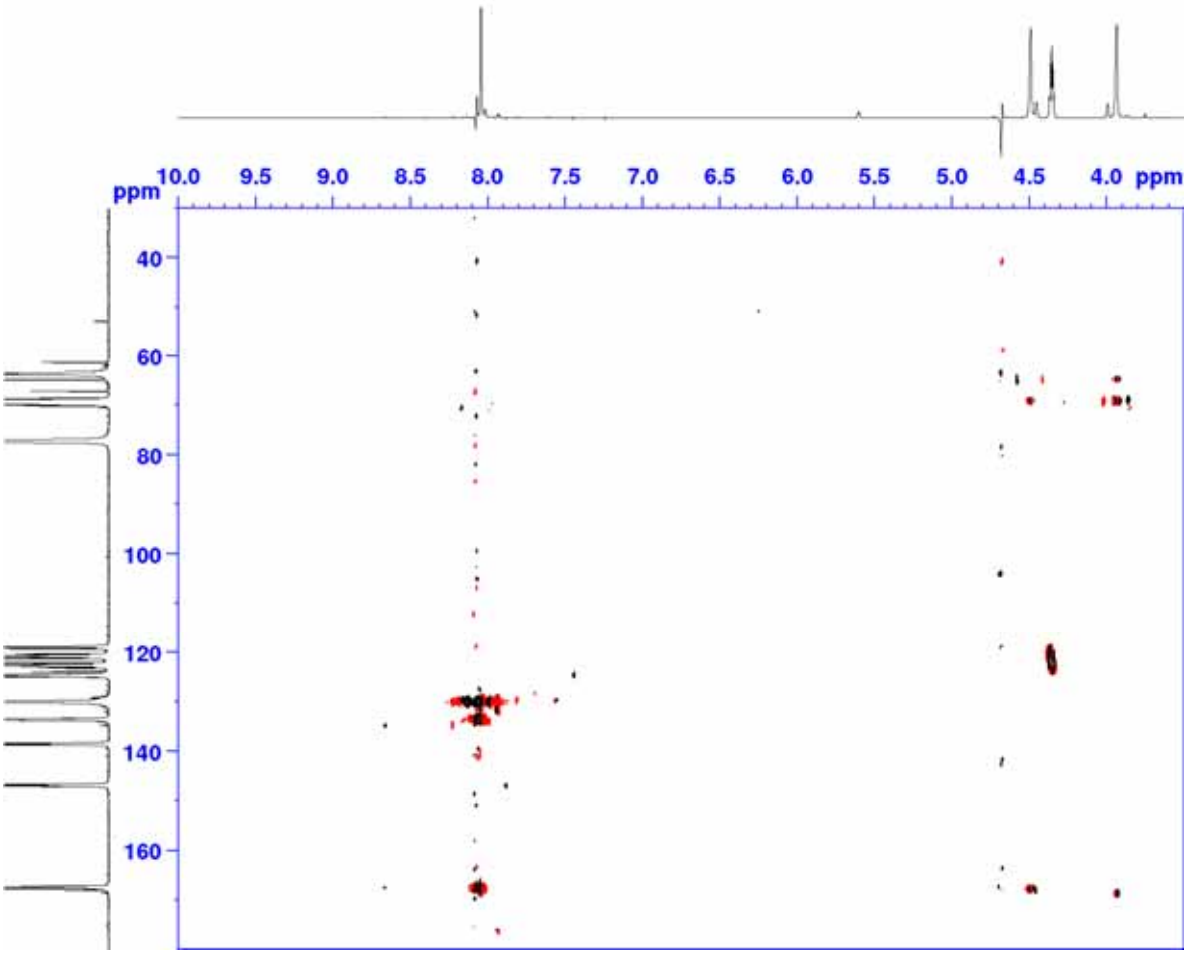

Figure S11. MWET(7) HMBC spectrum of the 3% PET solution with 7 points suppression as same as Figure S4 at 900 MHz. 80 scans, repetition time: 2.9 s, total experiment time: 13 h 11 min, long range J_{CH}: 10Hz, spectral width SW: 50,000 (^{13}C) × 12,626 (^{1}H) Hz, acquired data points: 192 (t1) × 2,048 (t2) and processed data points: 1,024 (F1) × 2,048 (F2), DIGMOD: baseopt, DE: 30 μs..

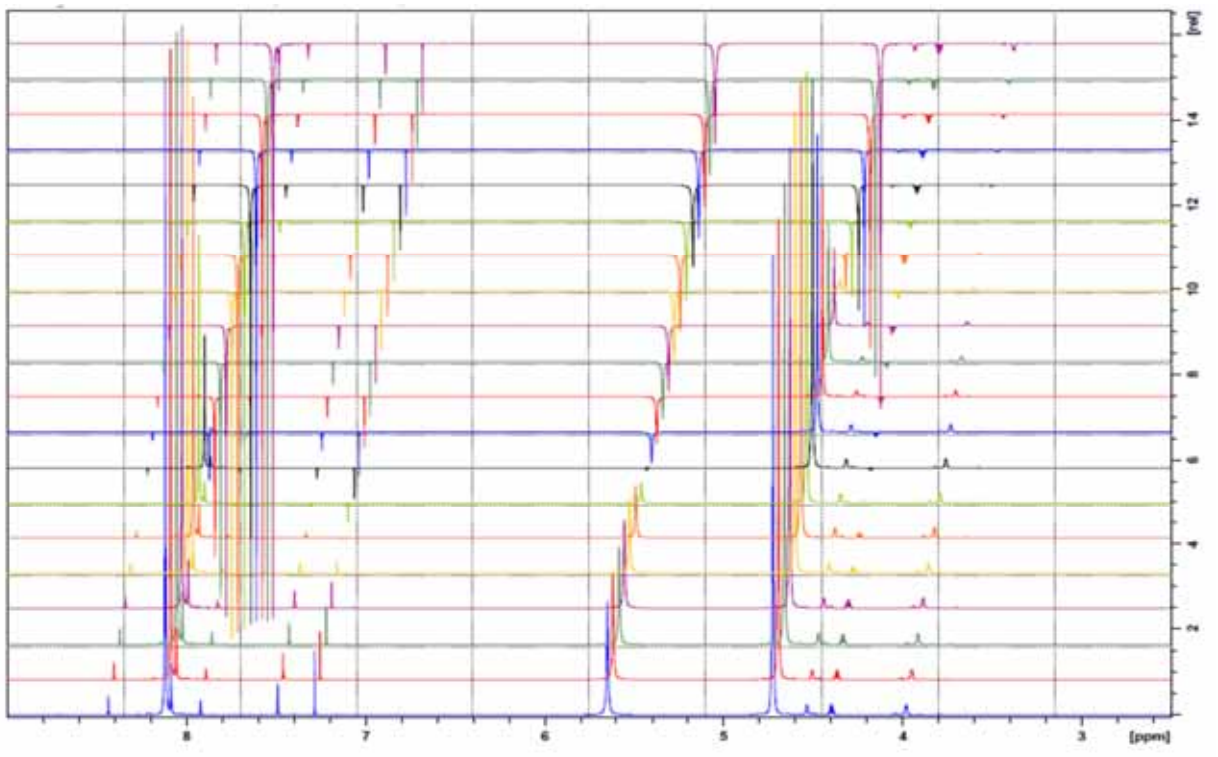

Figure S12. Stacked plot of ^{1}H inversion recovery data of the 3% PET solution at 900 MHz. Interval times τ (top to bottom): 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 1.0, 1.2, 1.5, 2.0, 3.0, 4.0, 6.0, 8.0, 10, 12, 15 and 25 s, DIGMOD: baseopt, DE: 55 μs.

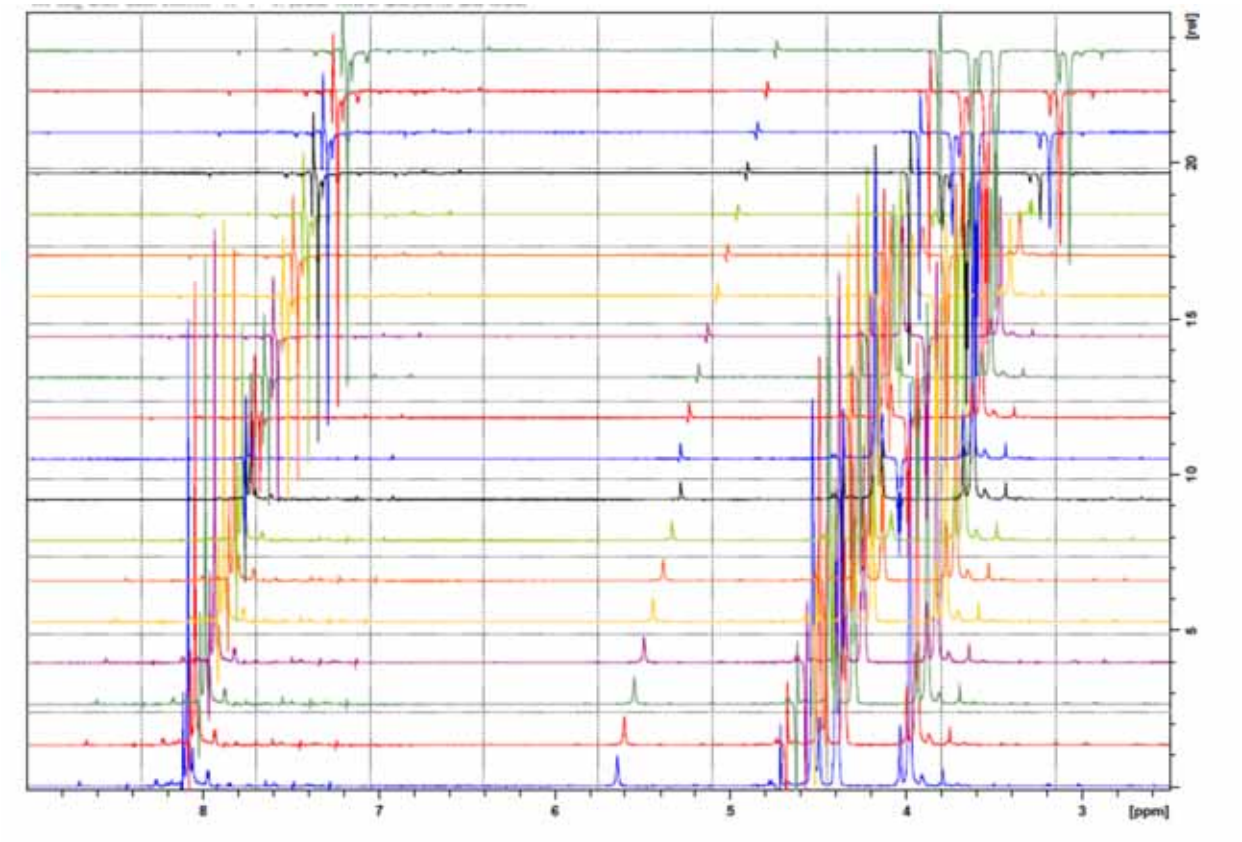

Figure S13. Stacked plot of MWET(7)-IR data of the 3% PET solution with 7 points suppression as same as Figure S4 at 900 MHz. Interval times τ (top to bottom): 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 1.0, 1.2, 1.5, 2.0, 3.0, 4.0, 6.0, 8.0, 10, 12, 15 and 25 s, DIGMOD: baseopt, DE: 55 μs.