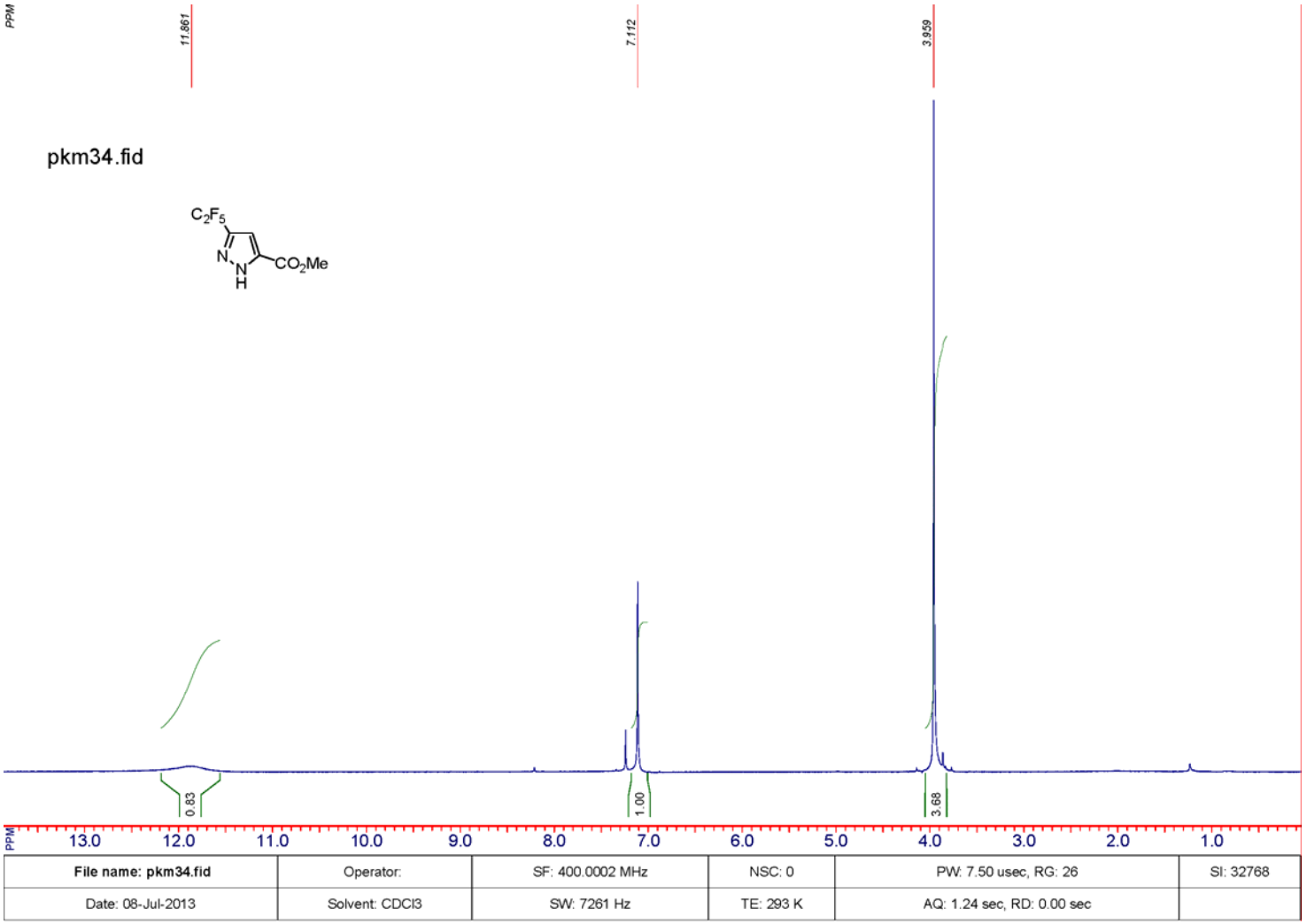
Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

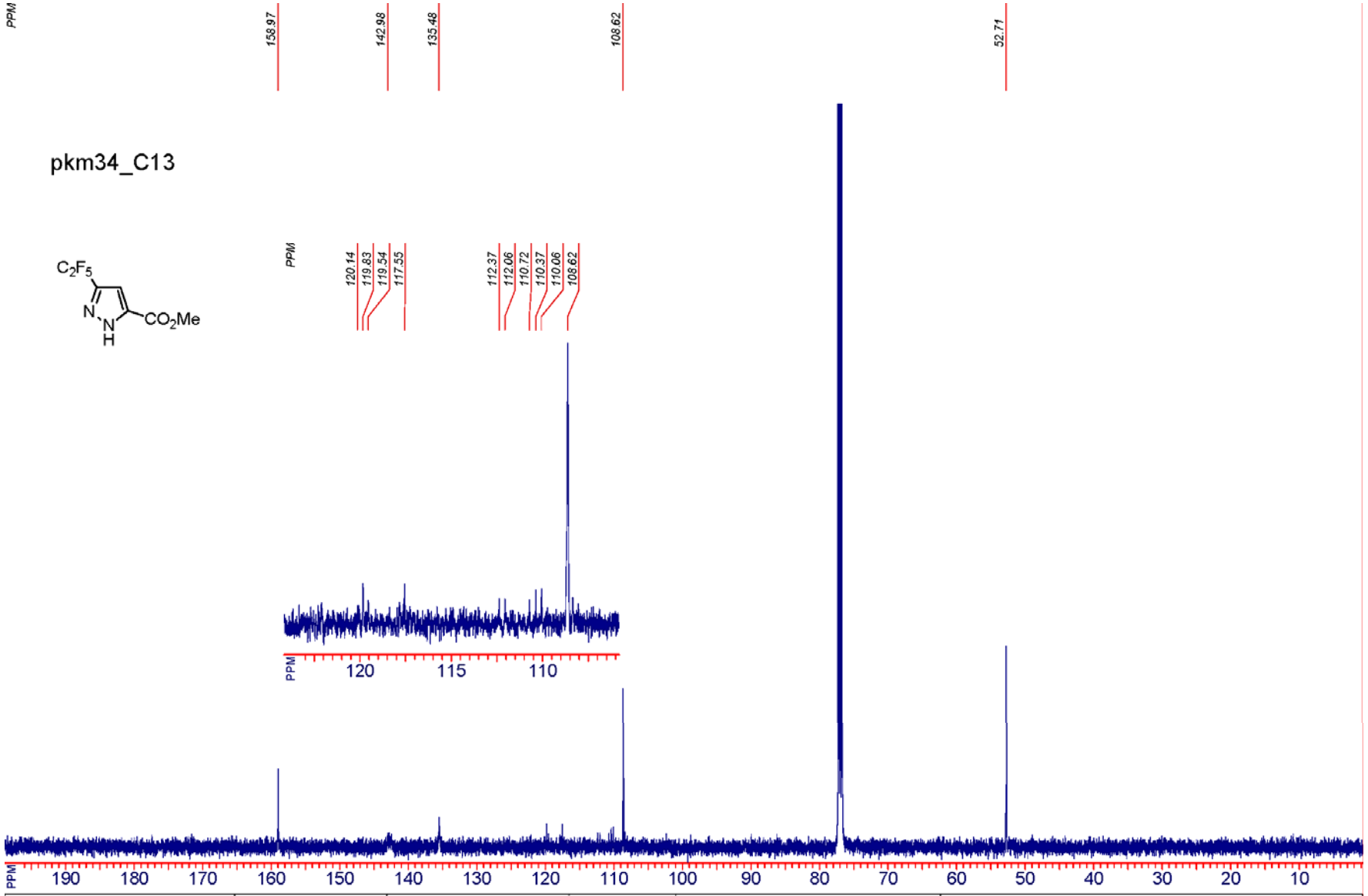
Supporting Information

Copies of NMR spectra.

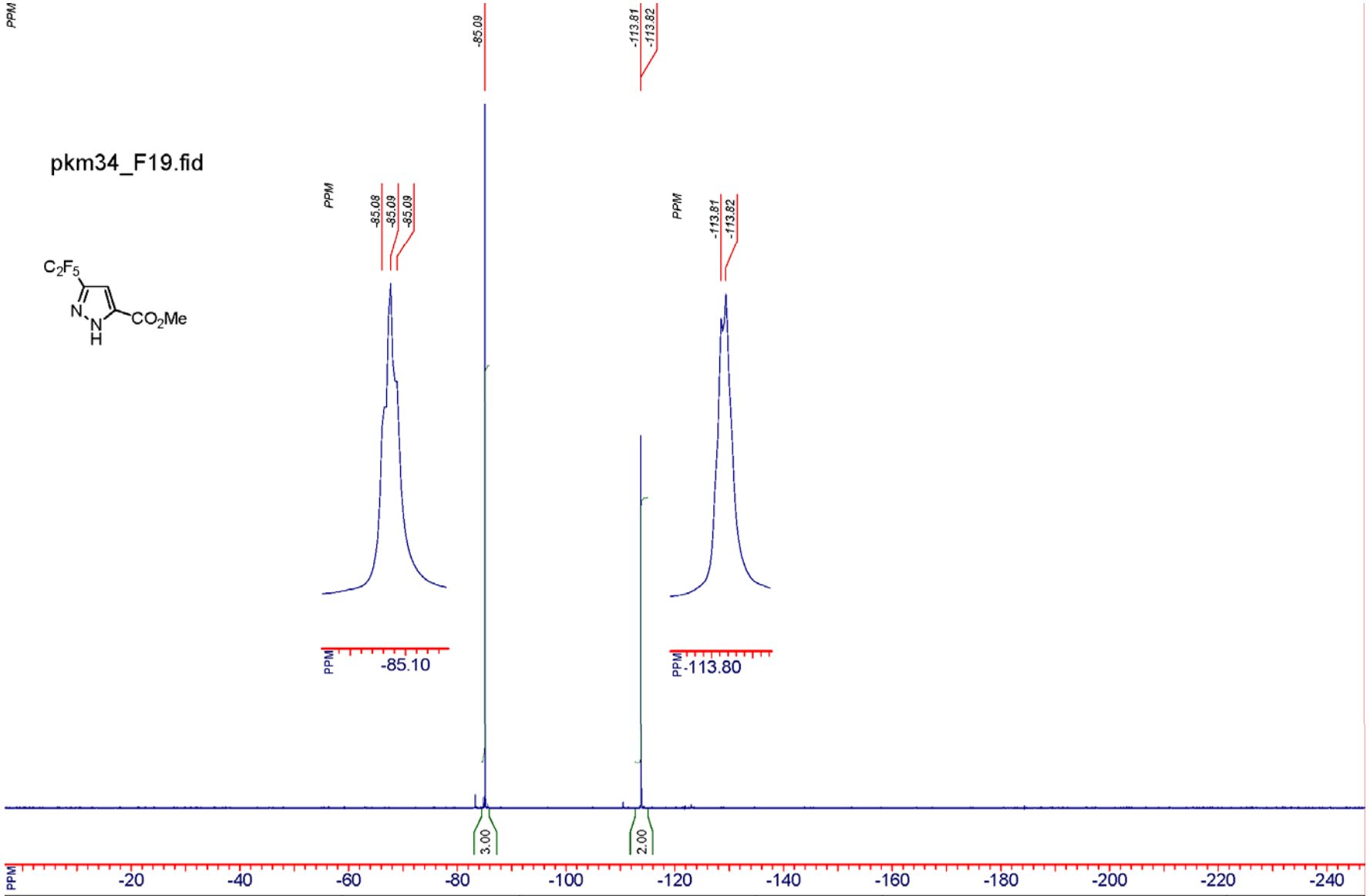
Compound 2a



File name: pkm34.fid	Operator:	SF: 400.0002 MHz	NSC: 0	PW: 7.50 usec, RG: 26	SI: 32768
Date: 08-Jul-2013	Solvent: CDCl3	SW: 7261 Hz	TE: 293 K	AQ: 1.24 sec, RD: 0.00 sec	

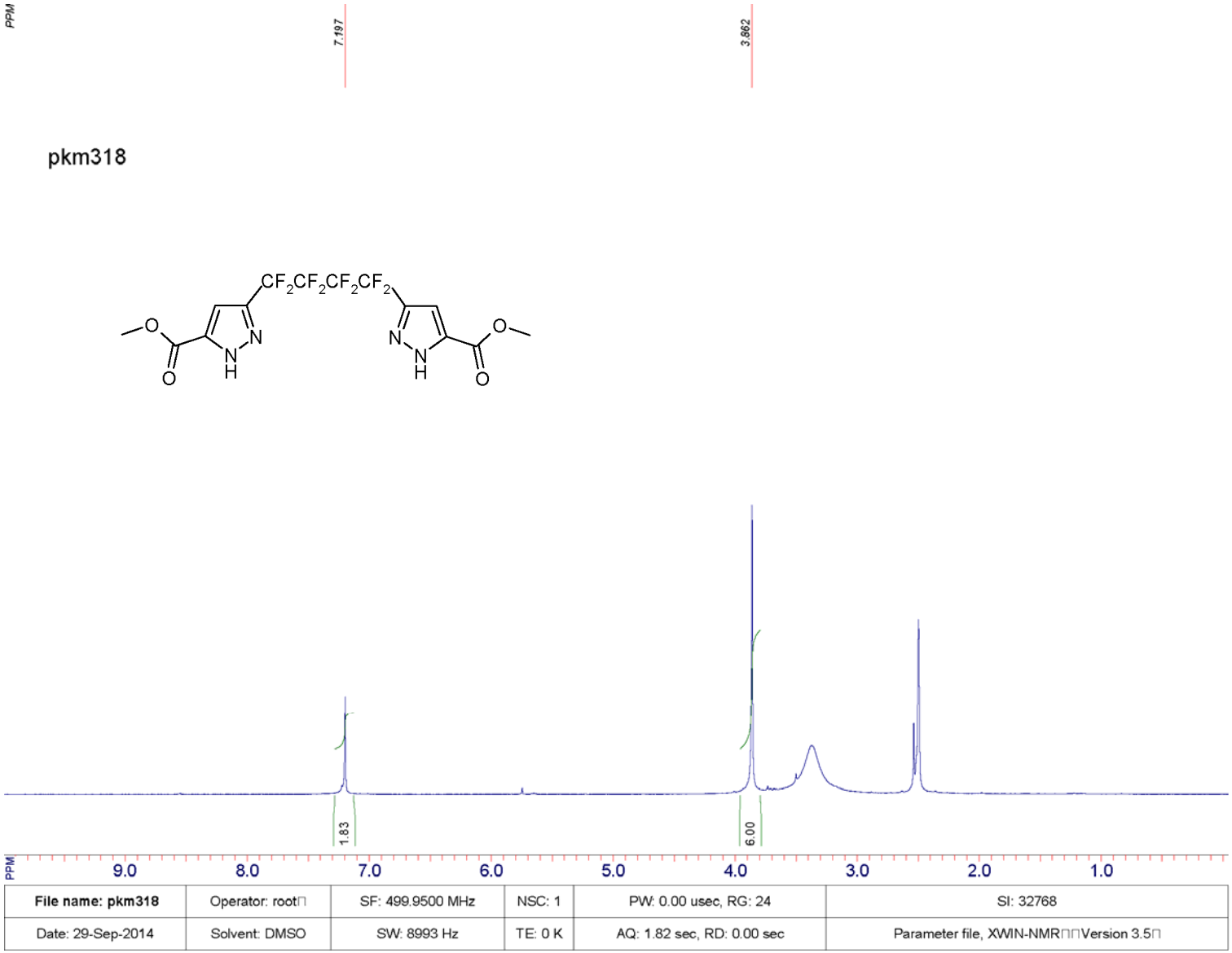


File name: pkm34_C13	Operator: root	SF: 125.7422 MHz	NSC: 1125	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 10-Jul-2013	Solvent: dmso	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMRVersion 3.5

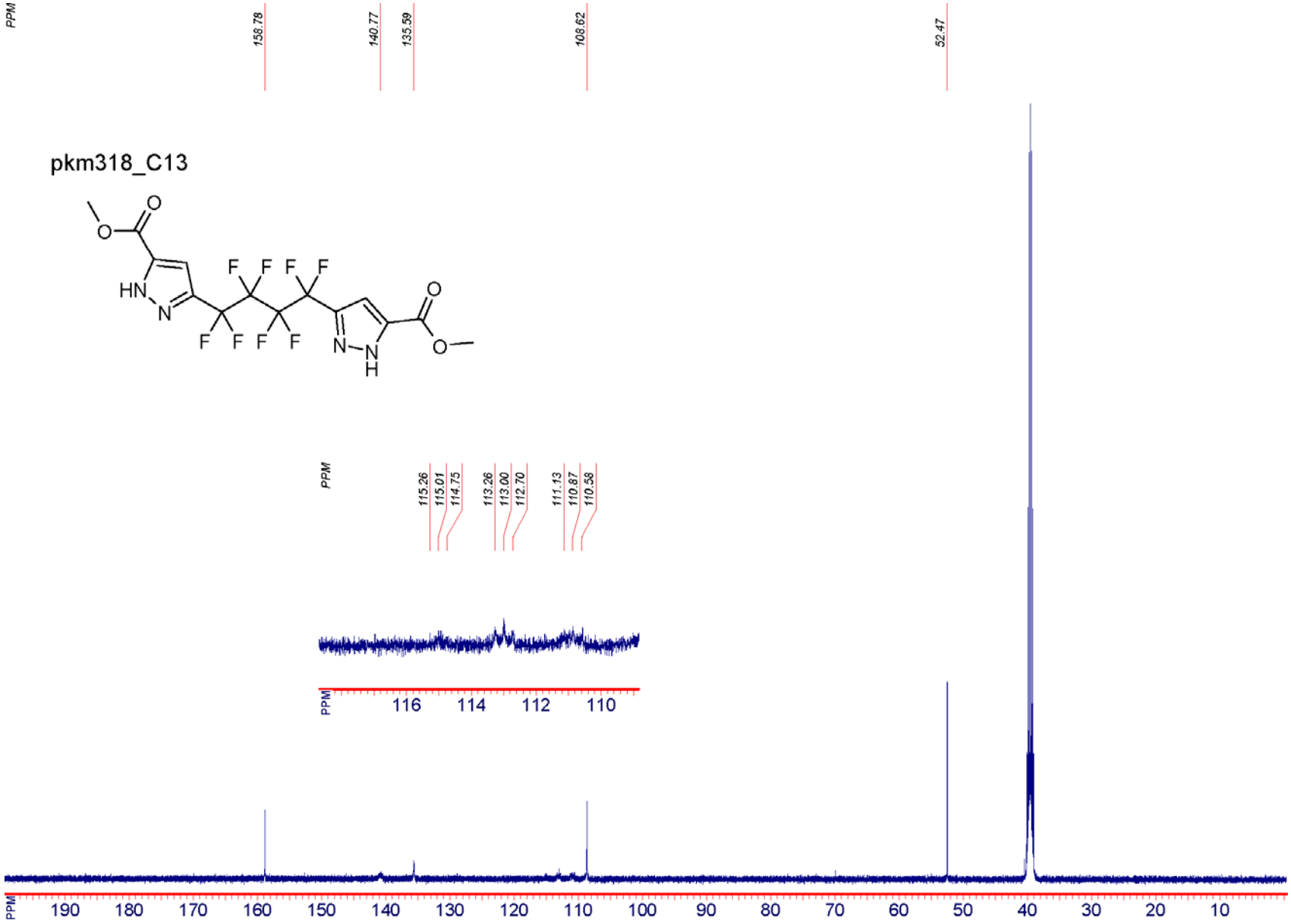


File name: pkm34_F19.fid	Operator:	SF: 376.3189 MHz	NSC: 0	PW: 3.00 usec, RG: 16	SI: 131072
Date: 11-Jul-2013	Solvent: cdcl3	SW: 94118 Hz	TE: 293 K	AQ: 0.68 sec, RD: 0.00 sec	

Compound 3a

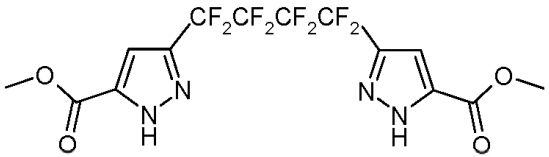


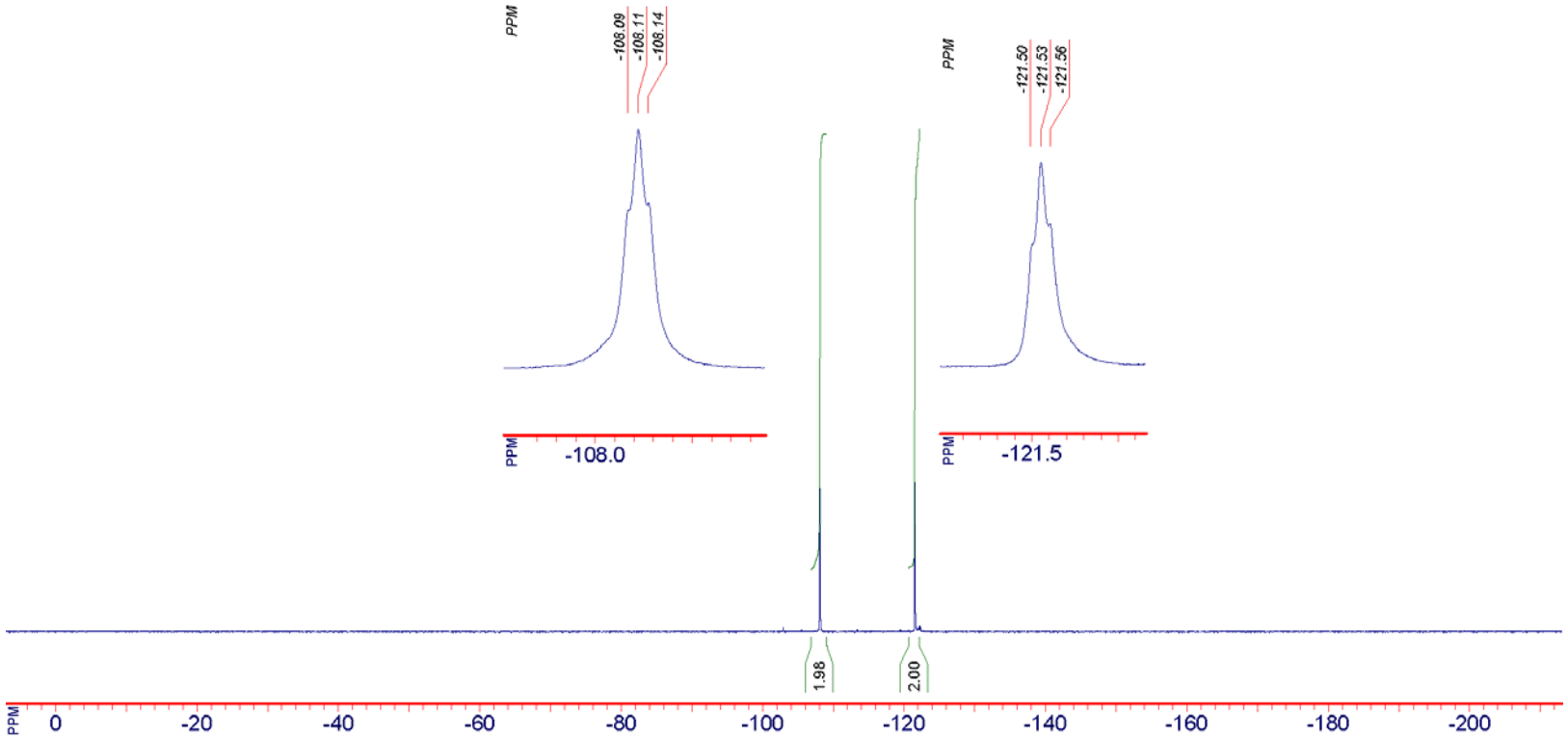
File name: pkm318	Operator: root⊓	SF: 499.9500 MHz	NSC: 1	PW: 0.00 usec, RG: 24	SI: 32768
Date: 29-Sep-2014	Solvent: DMSO	SW: 8993 Hz	TE: 0 K	AQ: 1.82 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓



File name: pkm318_C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 7168	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 24-Oct-2014	Solvent: DMSO	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

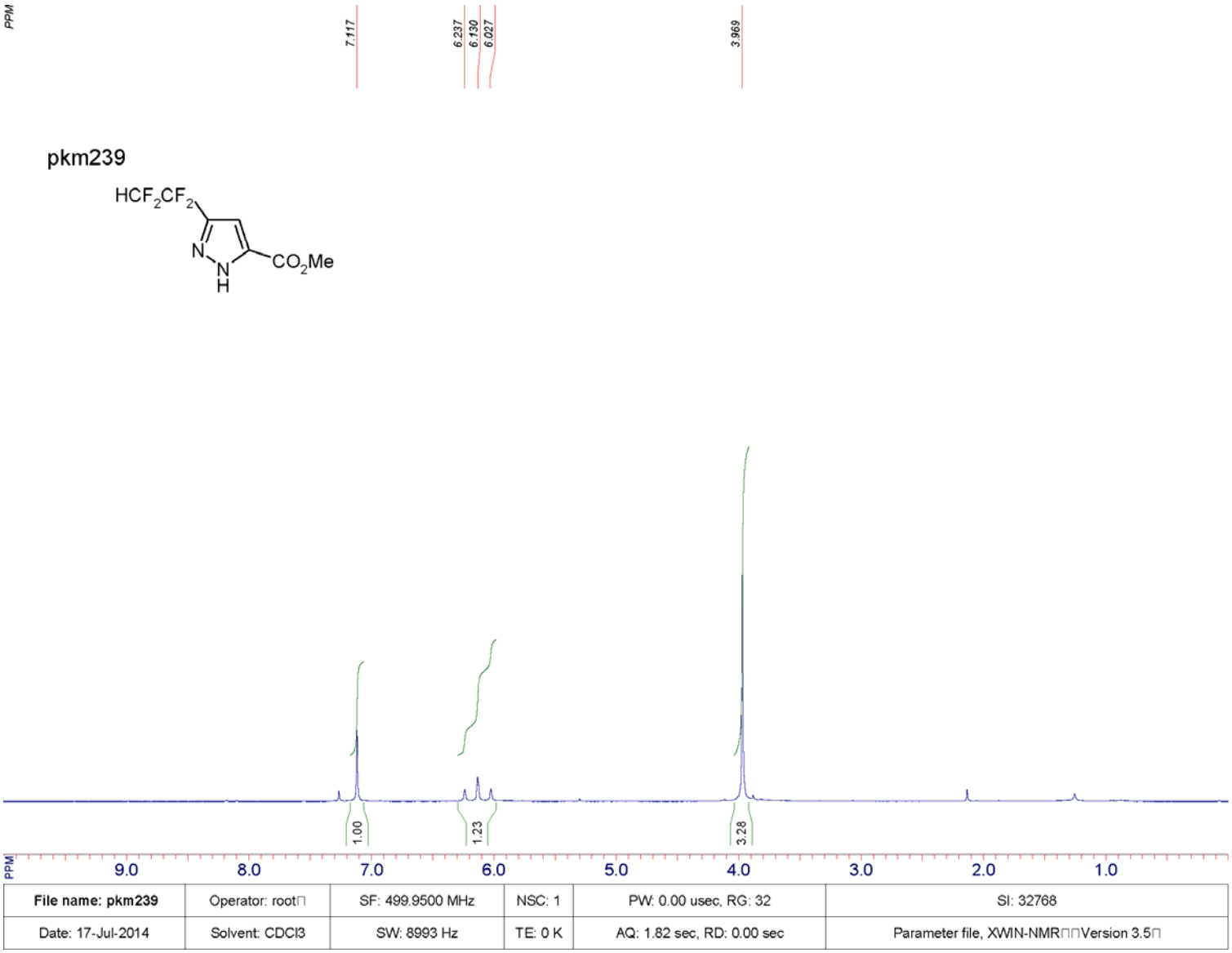
pkm318_F19.fid



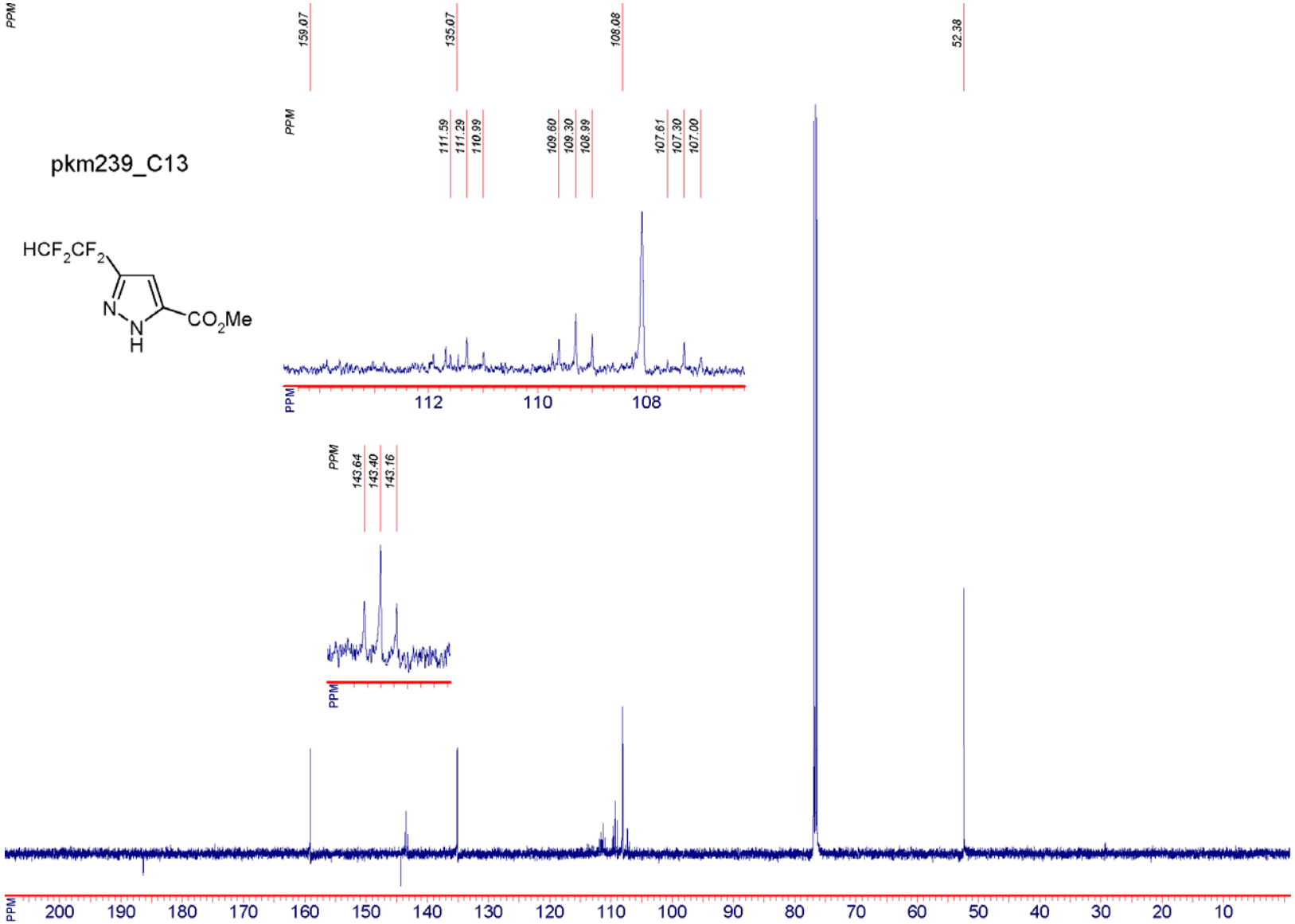


File name: pkm318_F19.fid	Operator:	SF: 376.3274 MHz	NSC: 0	PW: 3.00 usec, RG: 32	SI: 65536
Date: 07-Oct-2014	Solvent: dmso_d6	SW: 82816 Hz	TE: 293 K	AQ: 0.39 sec, RD: 0.00 sec	⊓

Compound 4a

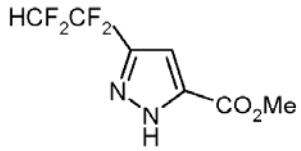


File name: pkm239	Operator: root⊓	SF: 499.9500 MHz	NSC: 1	PW: 0.00 usec, RG: 32	SI: 32768
Date: 17-Jul-2014	Solvent: CDCl3	SW: 8993 Hz	TE: 0 K	AQ: 1.82 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓



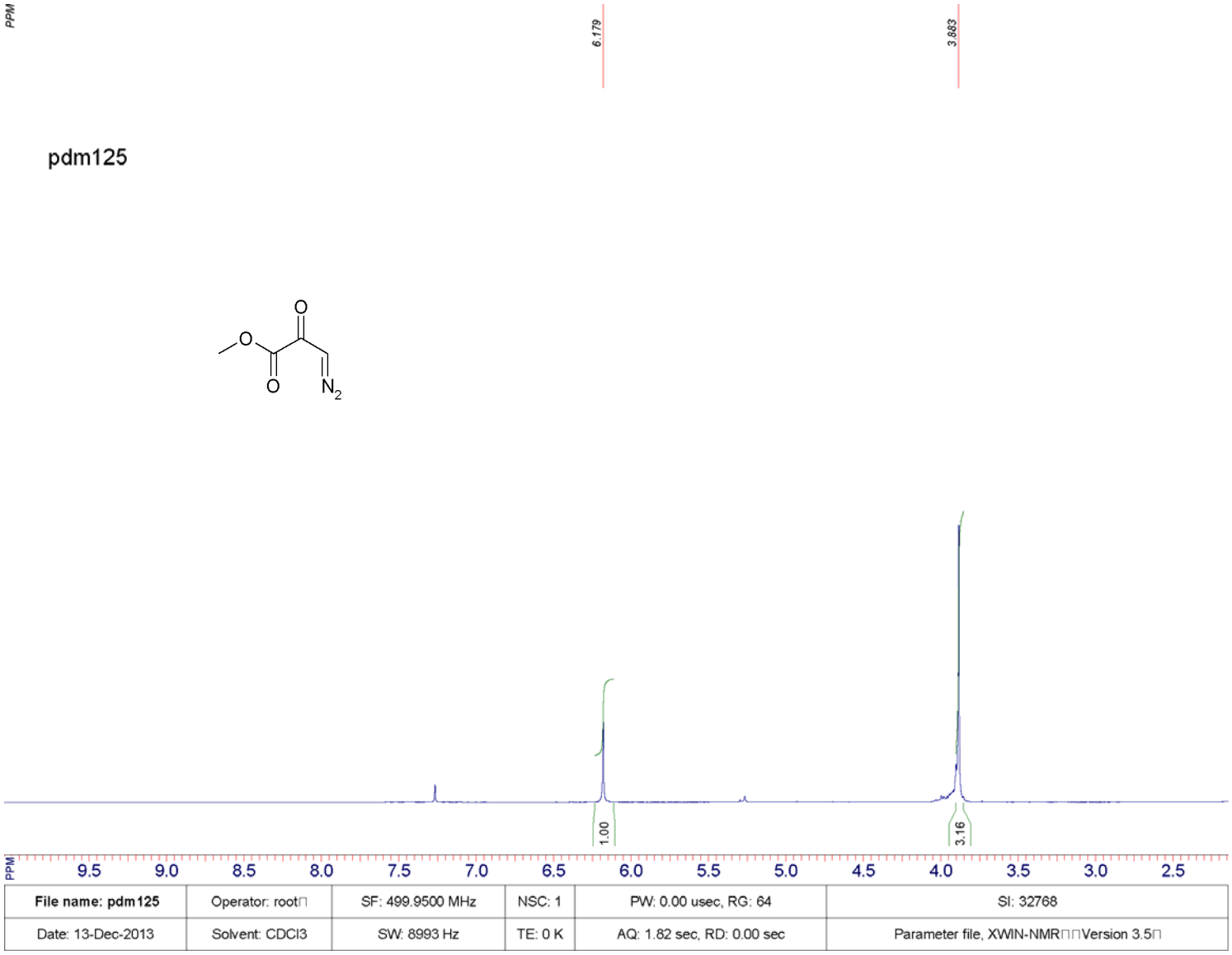
File name: pkm239_C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 475	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 31-Jul-2014	Solvent: dmso	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

pkm239_F19.fid



File name: pkm239_F19.fid	Operator:	SF: 376.3189 MHz	NSC: 0	PW: 3.00 usec, RG: 26	SI: 65536
Date: 05-Aug-2014	Solvent: cdcl3	SW: 94118 Hz	TE: 293 K	AQ: 0.34 sec, RD: 0.00 sec	⊓

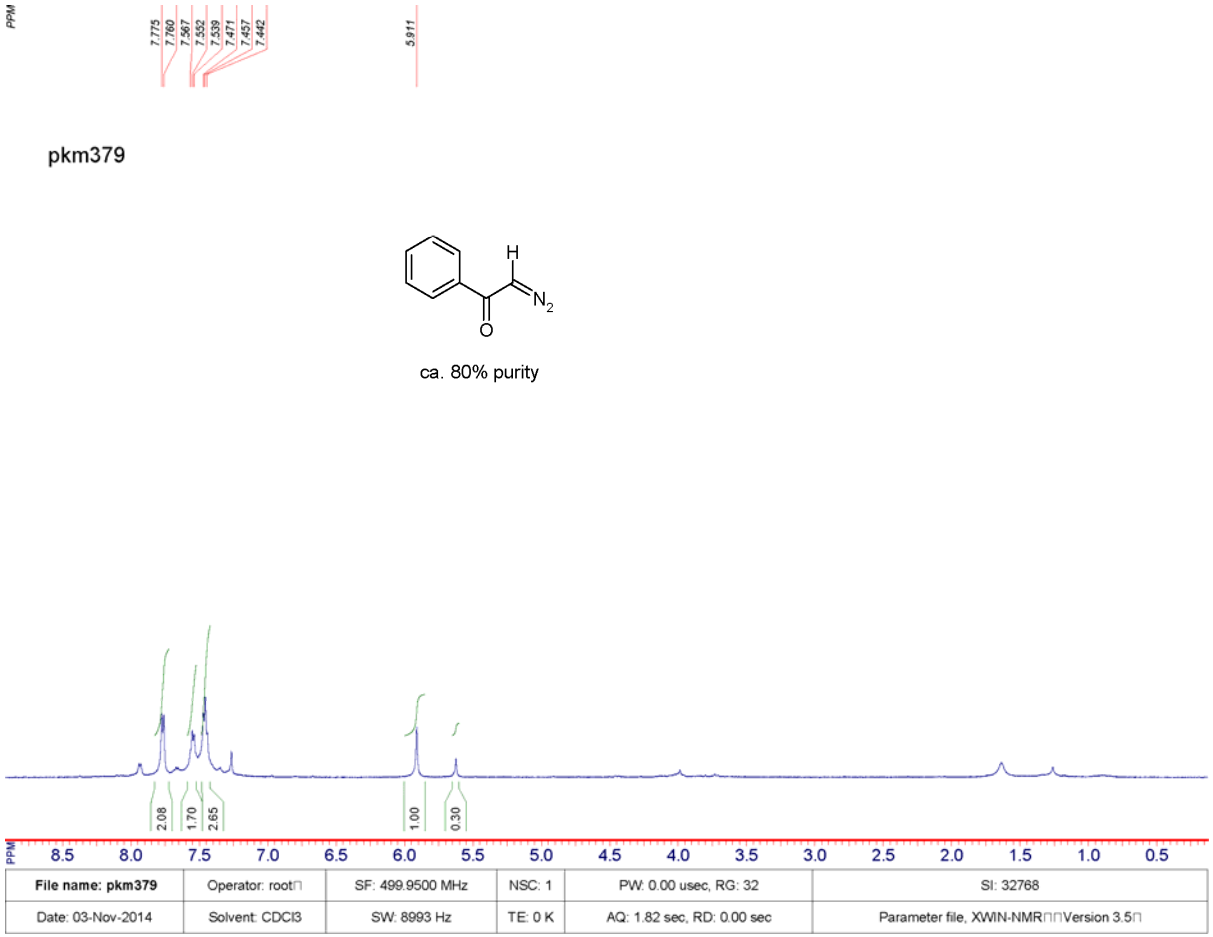
Compound 5b



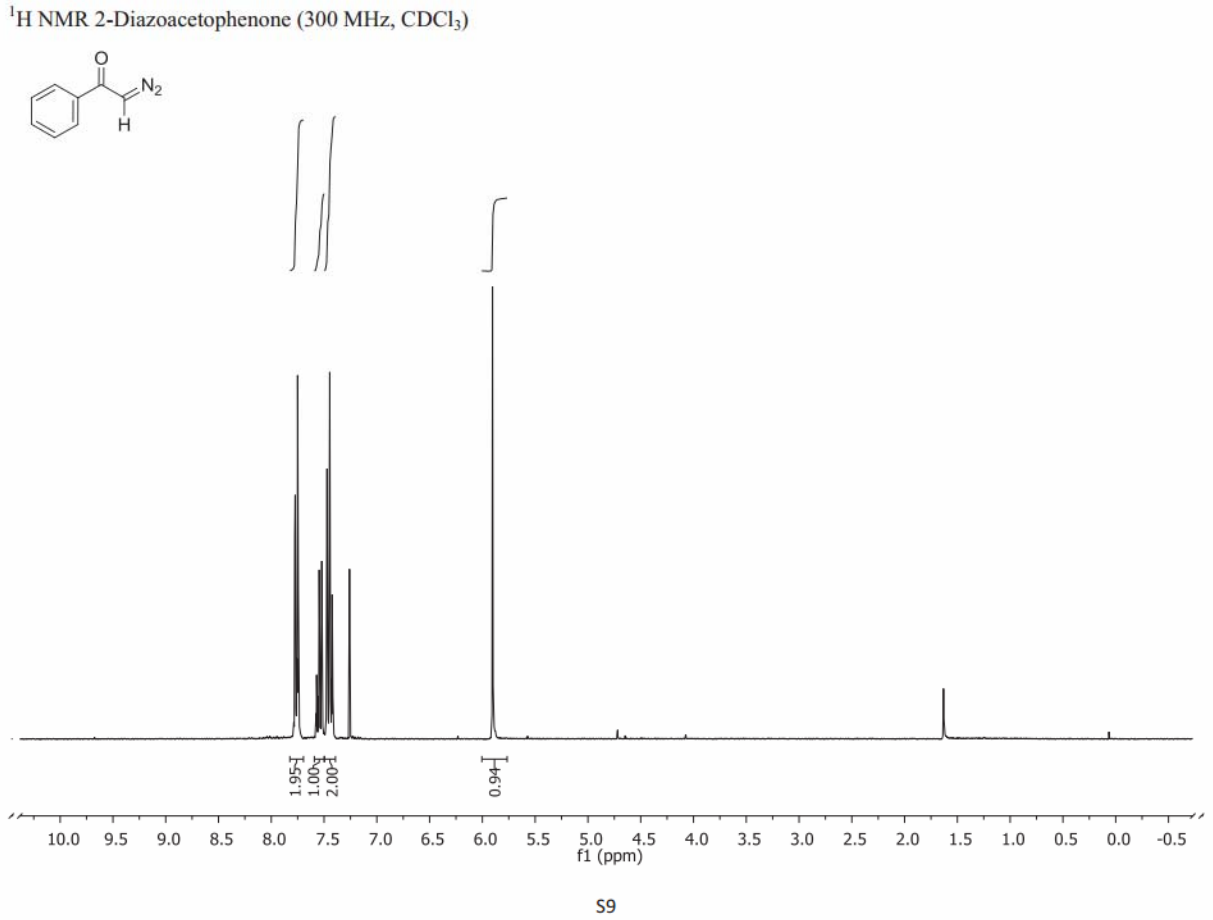
File name: pdm125	Operator: root⊓	SF: 499.9500 MHz	NSC: 1	PW: 0.00 usec, RG: 64	SI: 32768
Date: 13-Dec-2013	Solvent: CDCl3	SW: 8993 Hz	TE: 0 K	AQ: 1.82 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

File name: pkm125_C13	Operator: psm⊓	SF: 125.7126 MHz	NSC: 687	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 14-Dec-2013	Solvent: dmso	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

Compound 6b (ca. 90% purity)

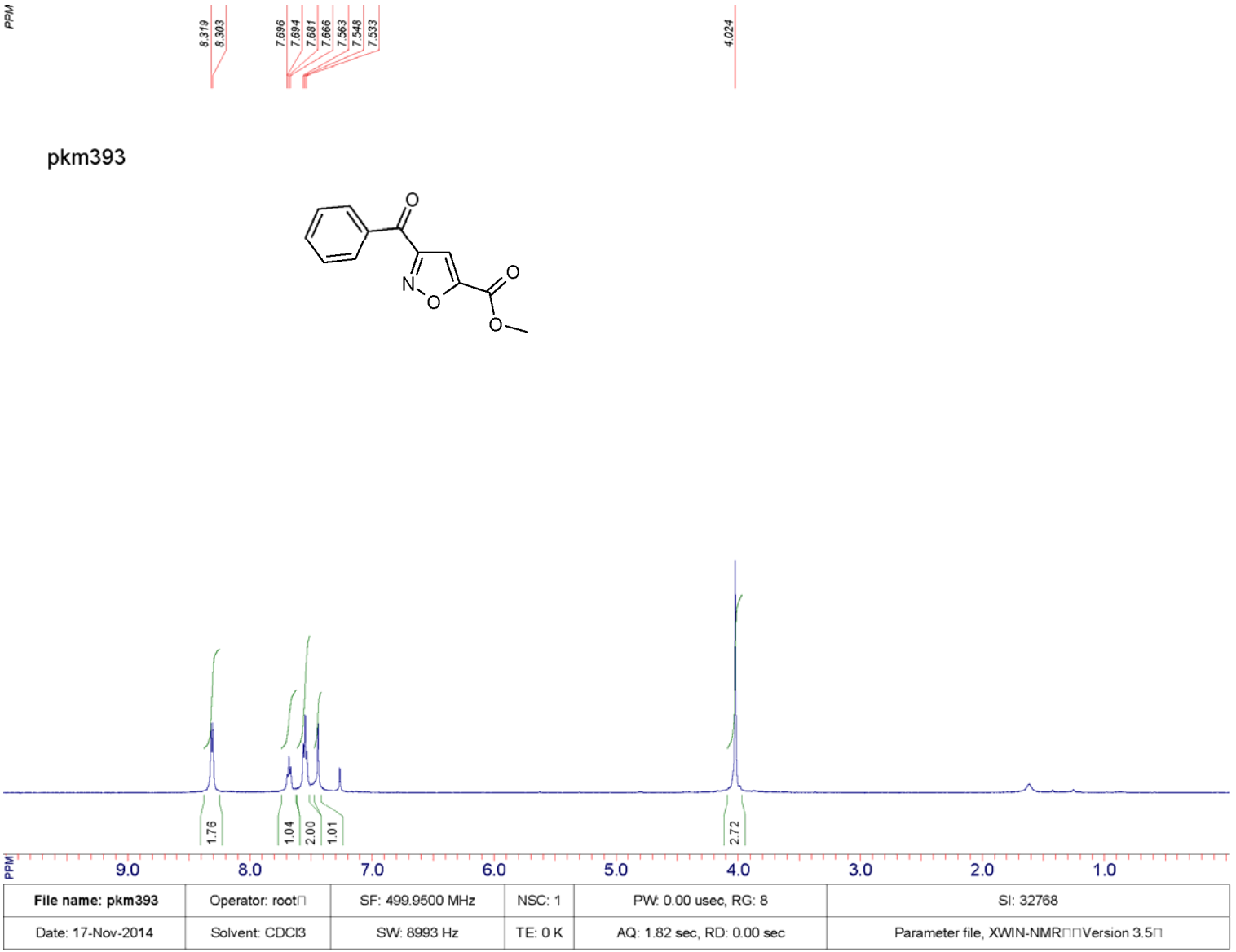


Copy of NMR of pure **6b** from Literature data.[1]

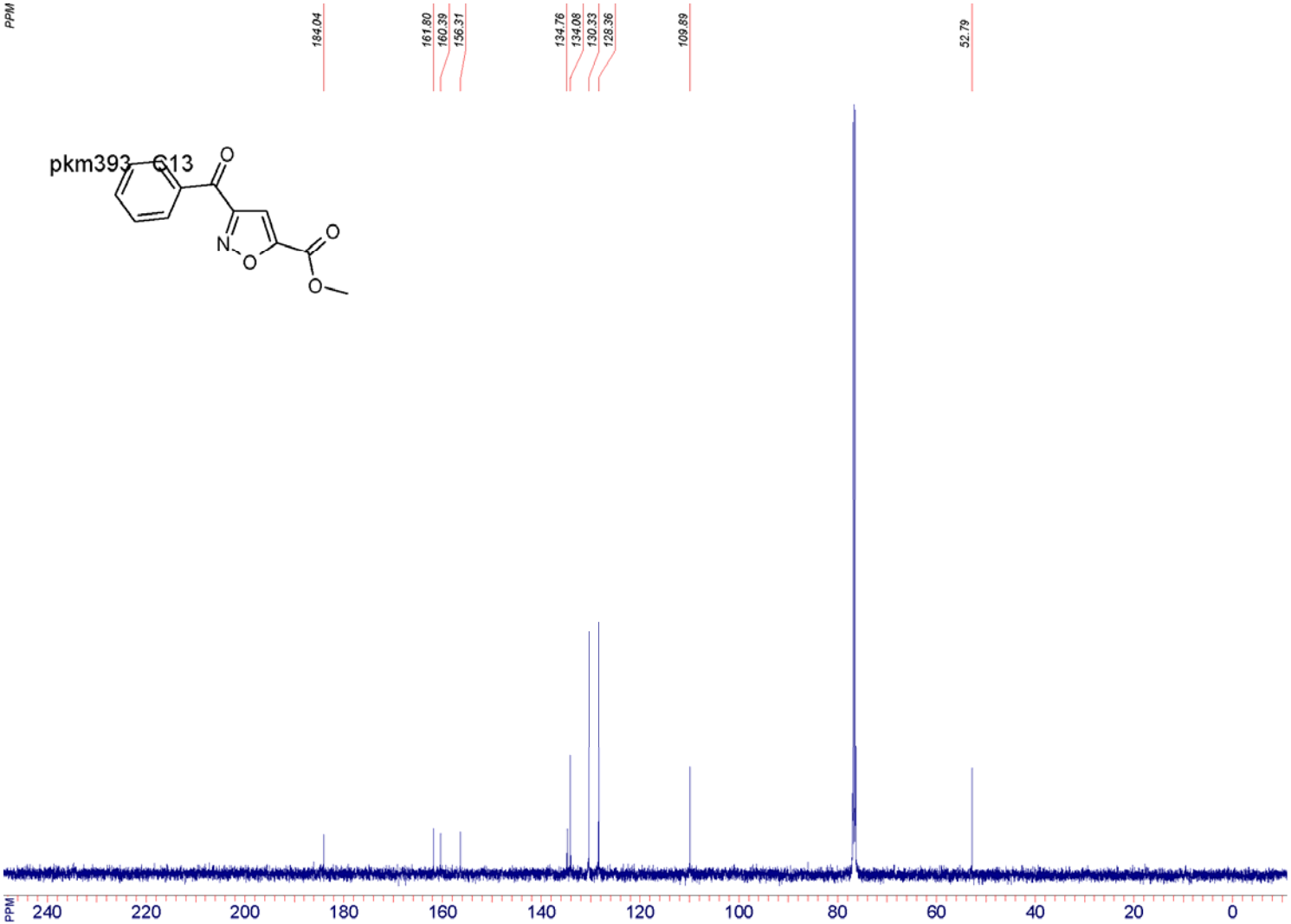


[1] S. P. Bew, P.-A. Ashford, D. U. Bachera *Synthesis* **2013**, *45*, 903.

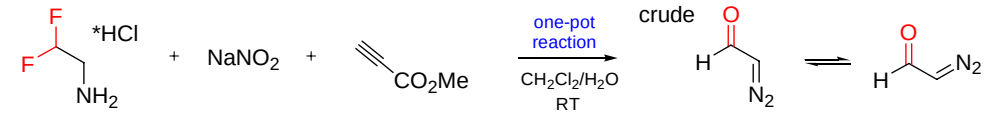
Compound 6c



File name: pkm393	Operator: root⊓	SF: 499.9500 MHz	NSC: 1	PW: 0.00 usec, RG: 8	SI: 32768
Date: 17-Nov-2014	Solvent: CDCl3	SW: 8993 Hz	TE: 0 K	AQ: 1.82 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

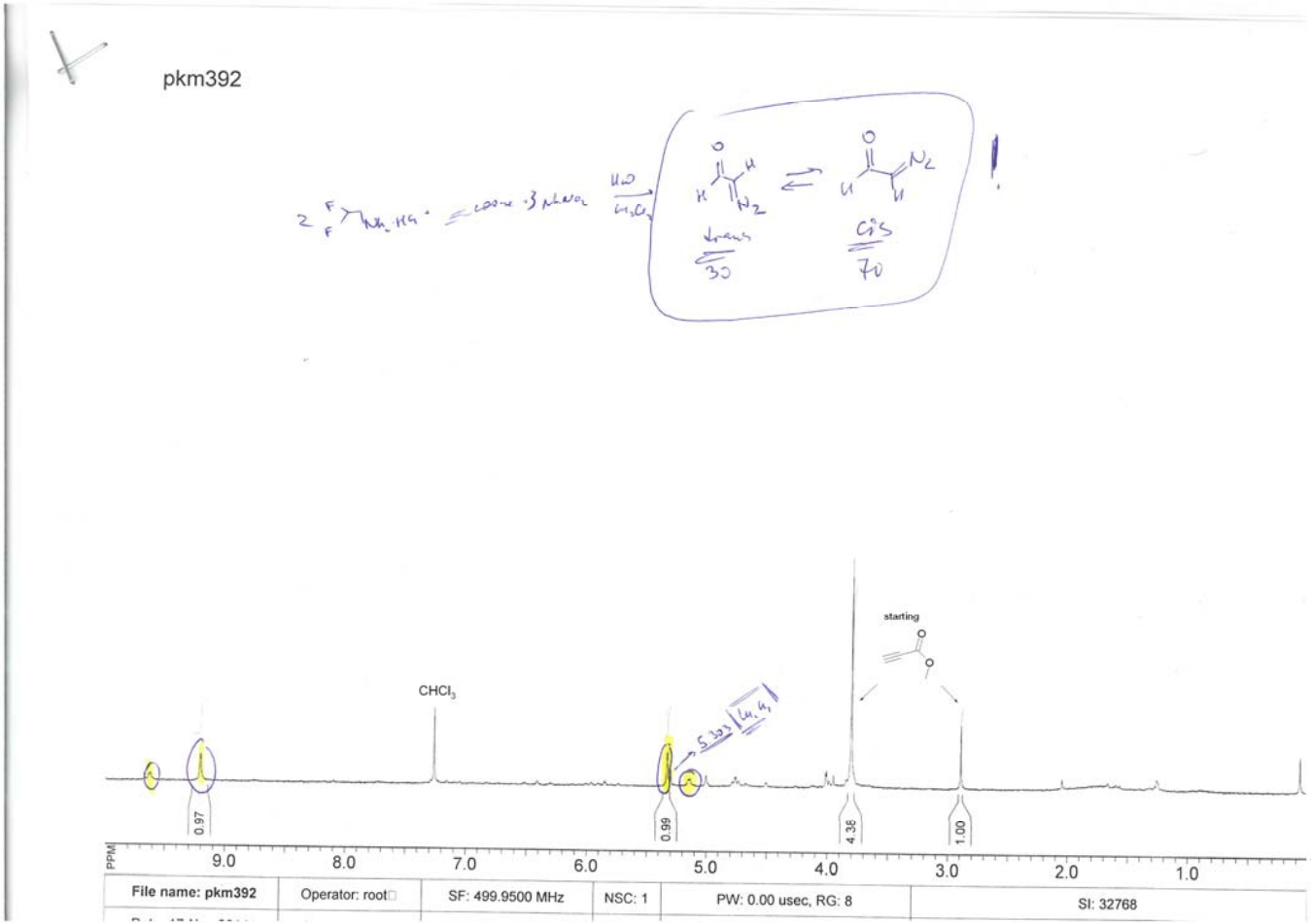


File name: pkm393 C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 578	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 20-Nov-2014	Solvent: CDCl3	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓



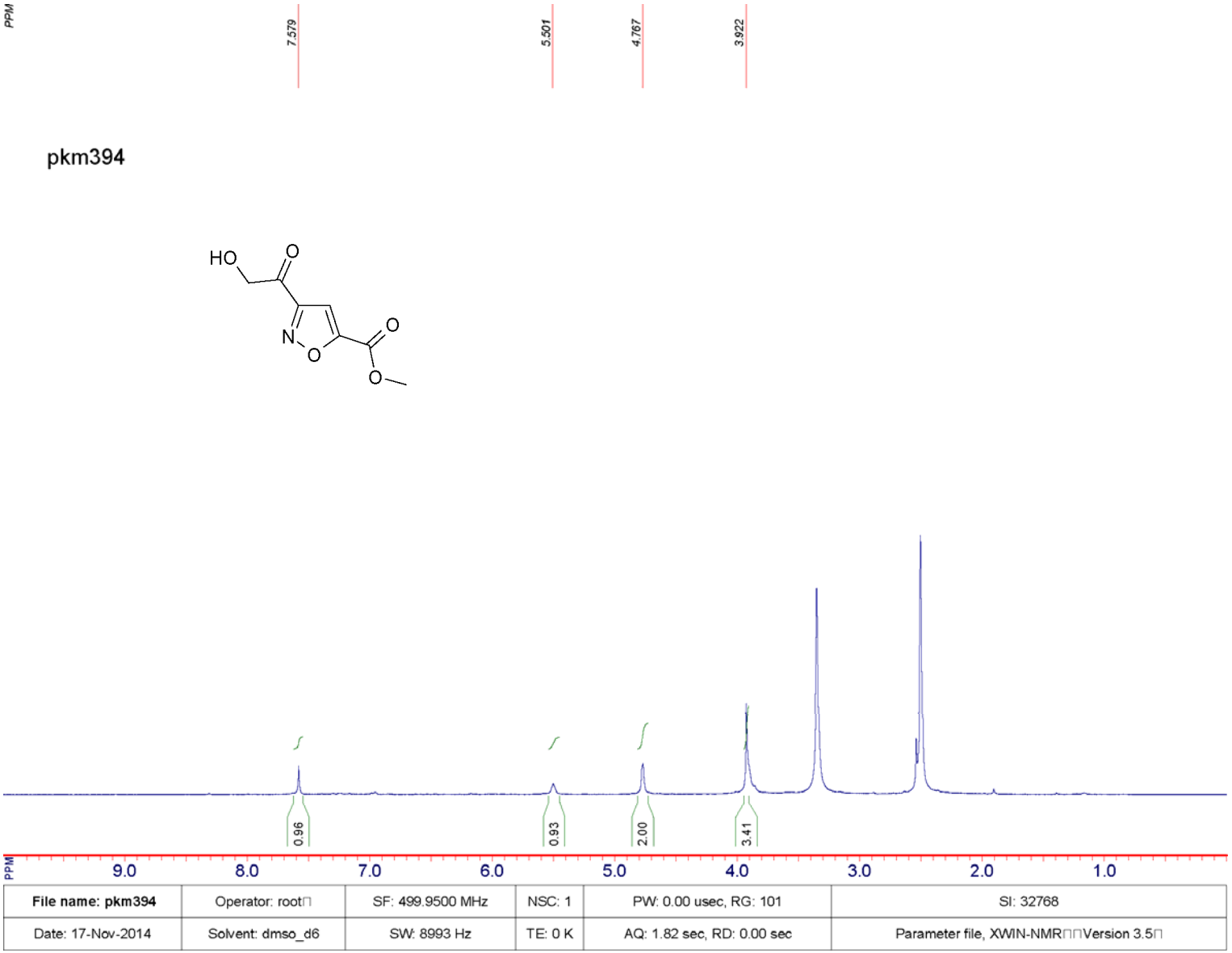
trans (30%) **7b** *cis (70%)*

Reaction mixture containing 7b (signals are marked in yellow):[2]

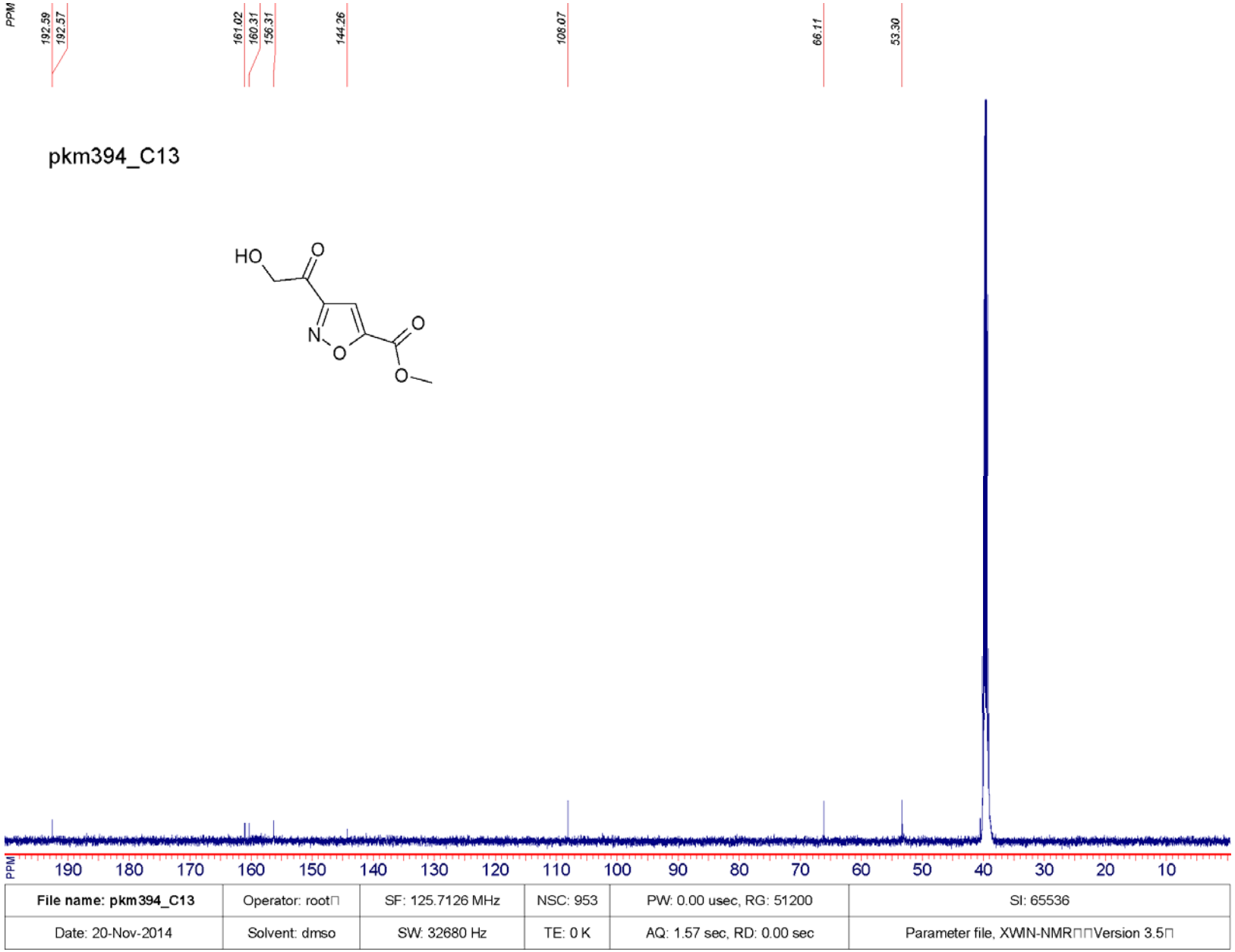


[2] Pure diazoacetaldehyde **7b** is described in: Y. Chiang, A. J. Kresge, V. V. Popik *J. Chem. Soc. Perkin Trans. 2*, **1999**, 1107. Citation from the manuscript:
"Proton NMR spectra of diazoacetaldehyde: δH (200 MHz; $CDCl_3$; Me_4Si) 9.61 (0.30 H, d, J = 6.8 Hz), 9.19 (0.67 H, s), 5.33 (0.74 H, s), 5.14 (0.29 H, d, J = 7.0 Hz); indicated restricted rotation about the C–C bond, giving rise to *s-cis* and *s-trans* isomers, present in the ratio 70 : 30 in $CDCl_3$."

Compound 8c

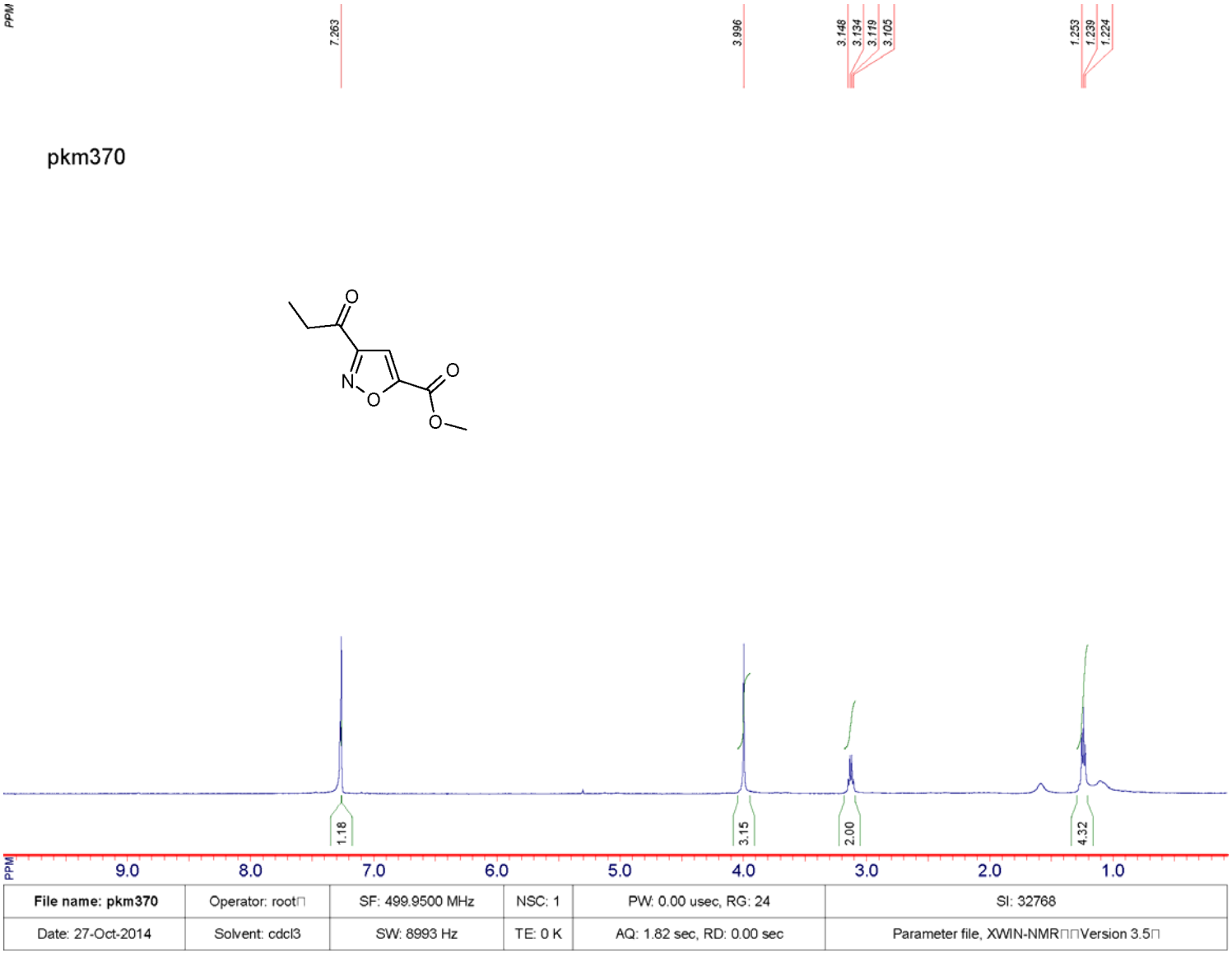


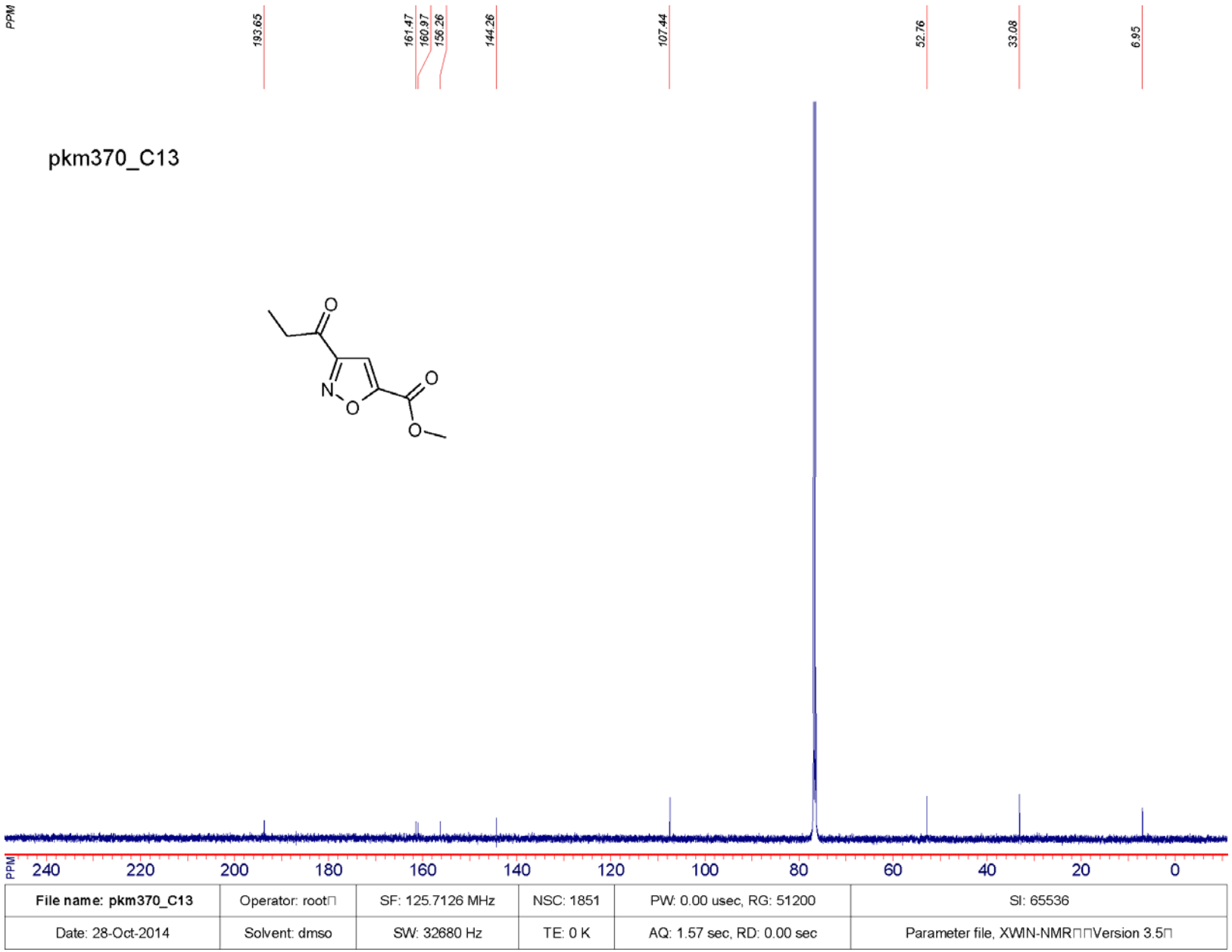
File name: pkm394	Operator: root⊓	SF: 499.9500 MHz	NSC: 1	PW: 0.00 usec, RG: 101	SI: 32768
Date: 17-Nov-2014	Solvent: dmso_d6	SW: 8993 Hz	TE: 0 K	AQ: 1.82 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓



File name: pkm394_C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 953	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 20-Nov-2014	Solvent: dmso	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

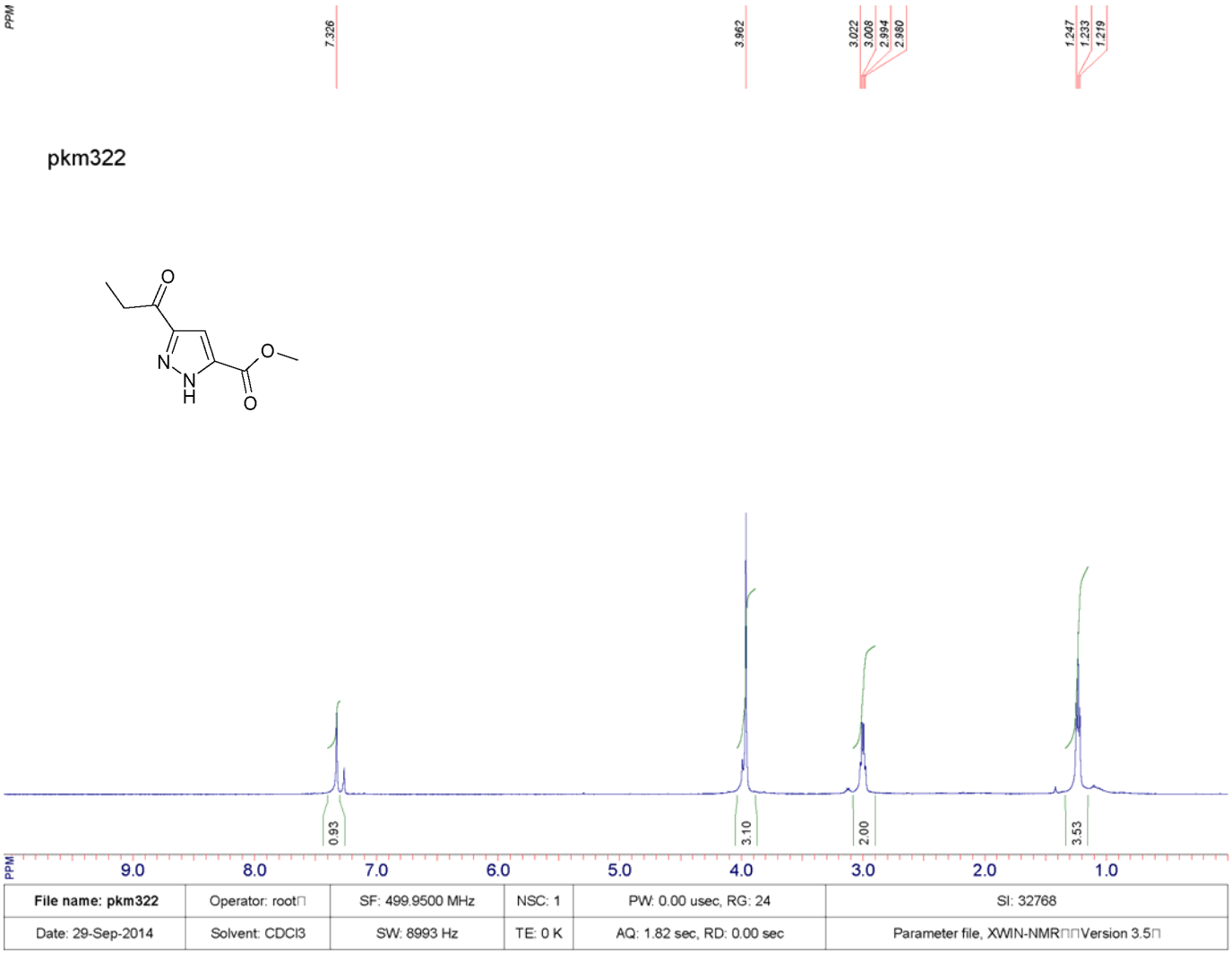
Compound 9c


PPM
7.263
3.996
3.148
3.134
3.119
3.105
1.253
1.239
1.224
pkm370
O
N
O
O
O
1.18
3.15
2.00
4.32
PPM
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
File name: pkm370
Operator: root
SF: 499.9500 MHz
NSC: 1
PW: 0.00 usec, RG: 24
SI: 32768
Date: 27-Oct-2014
Solvent: cdcl3
SW: 8993 Hz
TE: 0 K
AQ: 1.82 sec, RD: 0.00 sec
Parameter file, XWIN-NMR Version 3.5

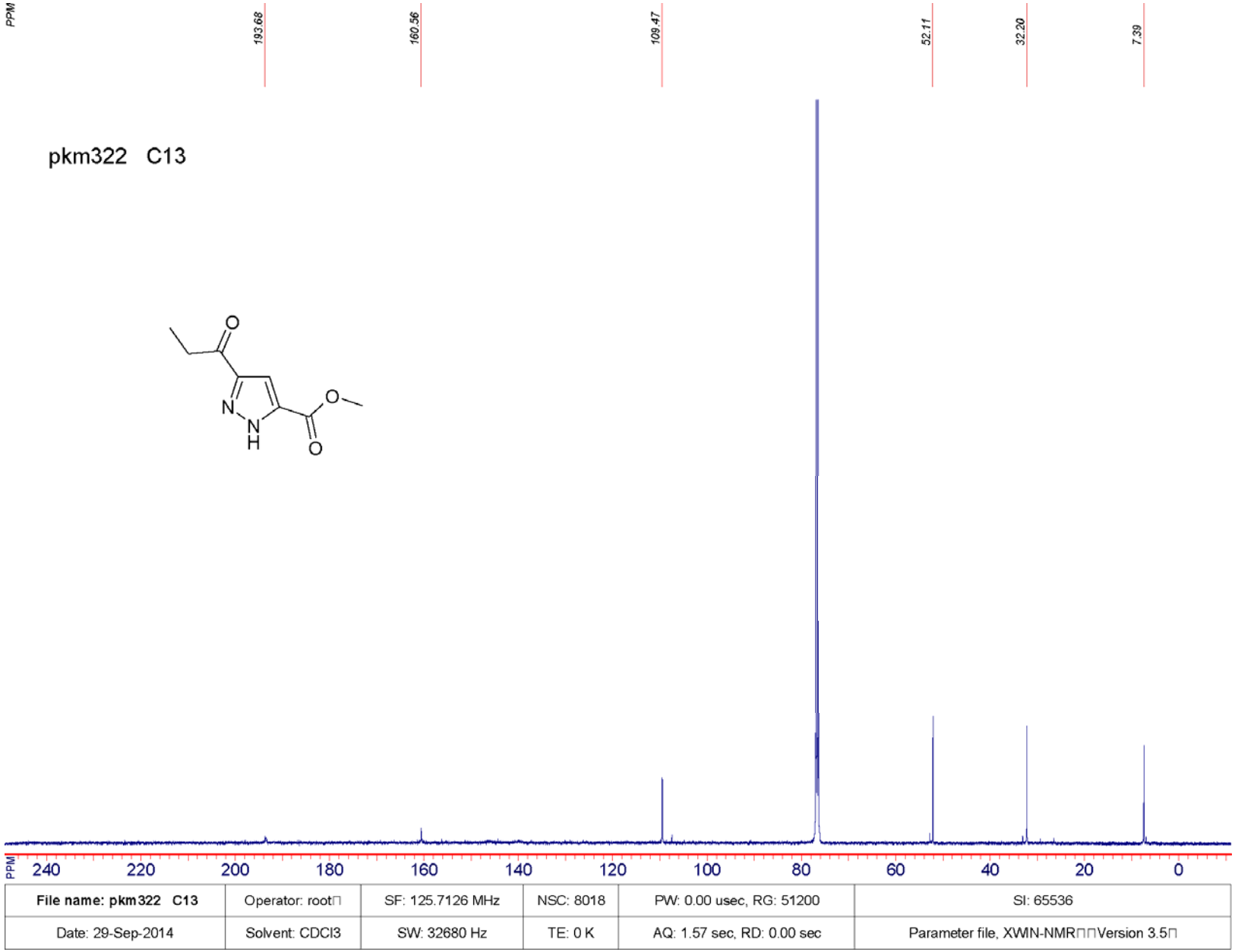


File name: pkm370_C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 1851	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 28-Oct-2014	Solvent: dmso	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

Compound 9d

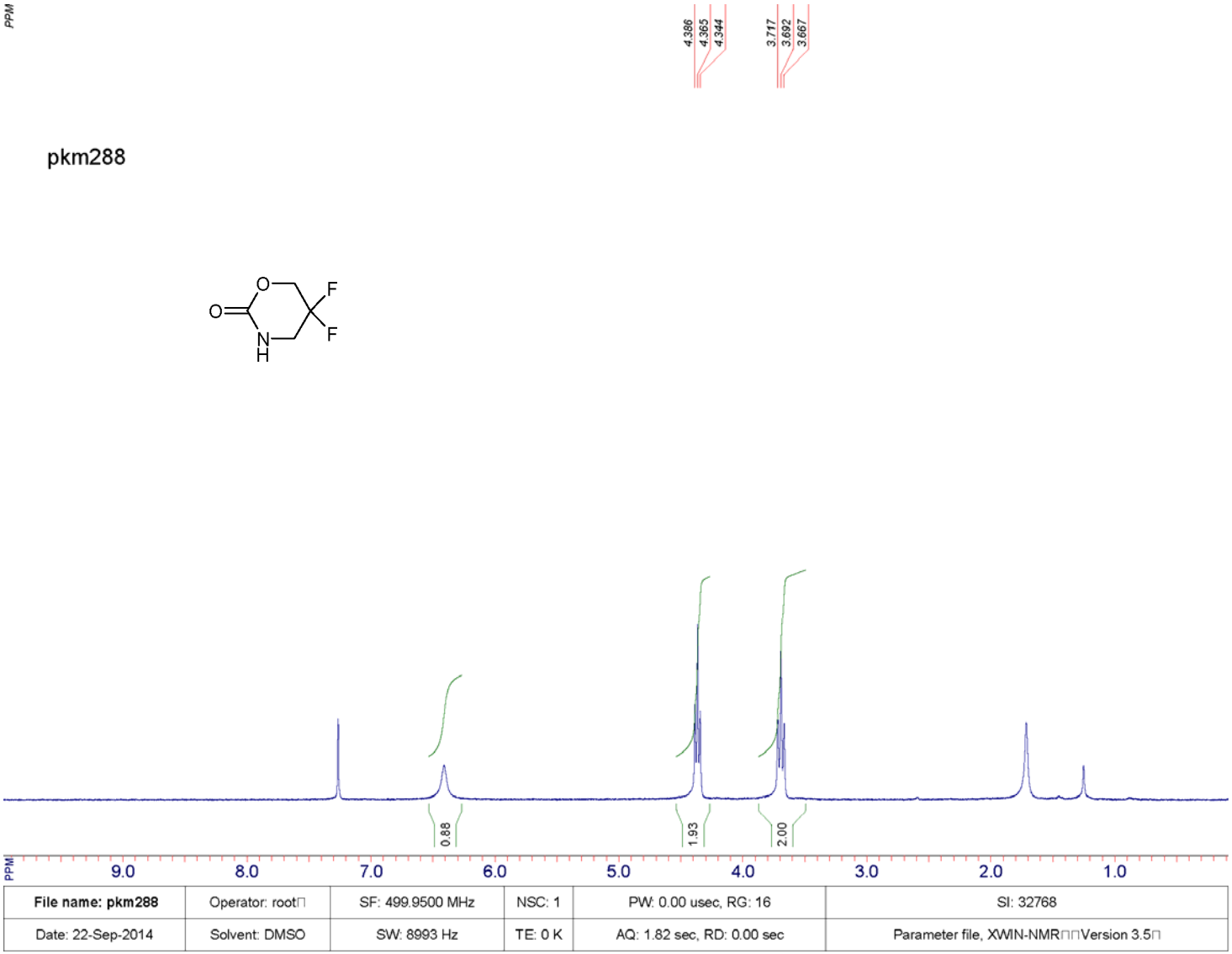


File name: pkm322	Operator: root⊓	SF: 499.9500 MHz	NSC: 1	PW: 0.00 usec, RG: 24	SI: 32768
Date: 29-Sep-2014	Solvent: CDCl3	SW: 8993 Hz	TE: 0 K	AQ: 1.82 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

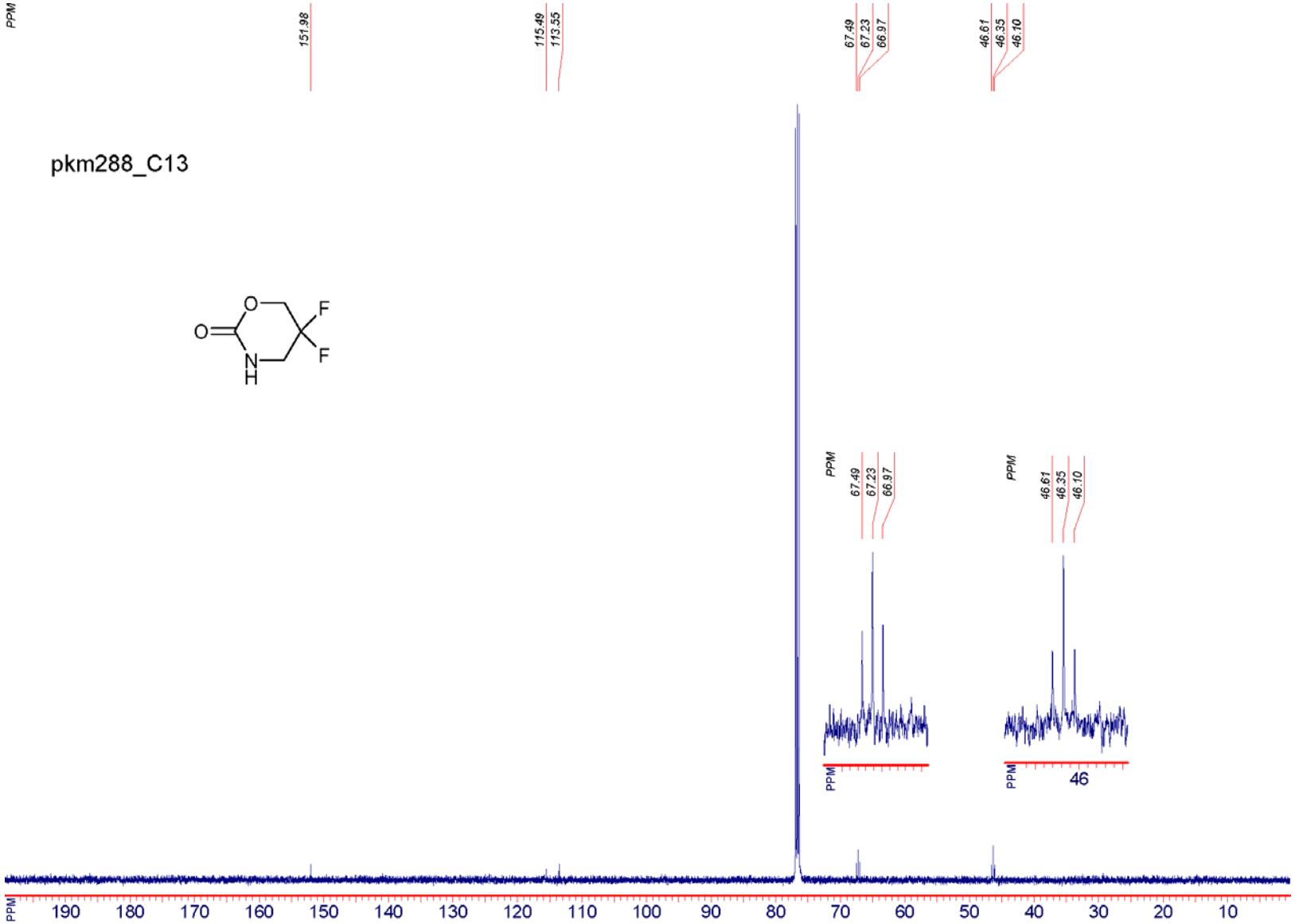


File name: pkm322 C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 8018	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 29-Sep-2014	Solvent: CDCl3	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

Compound 10e

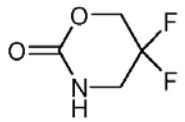


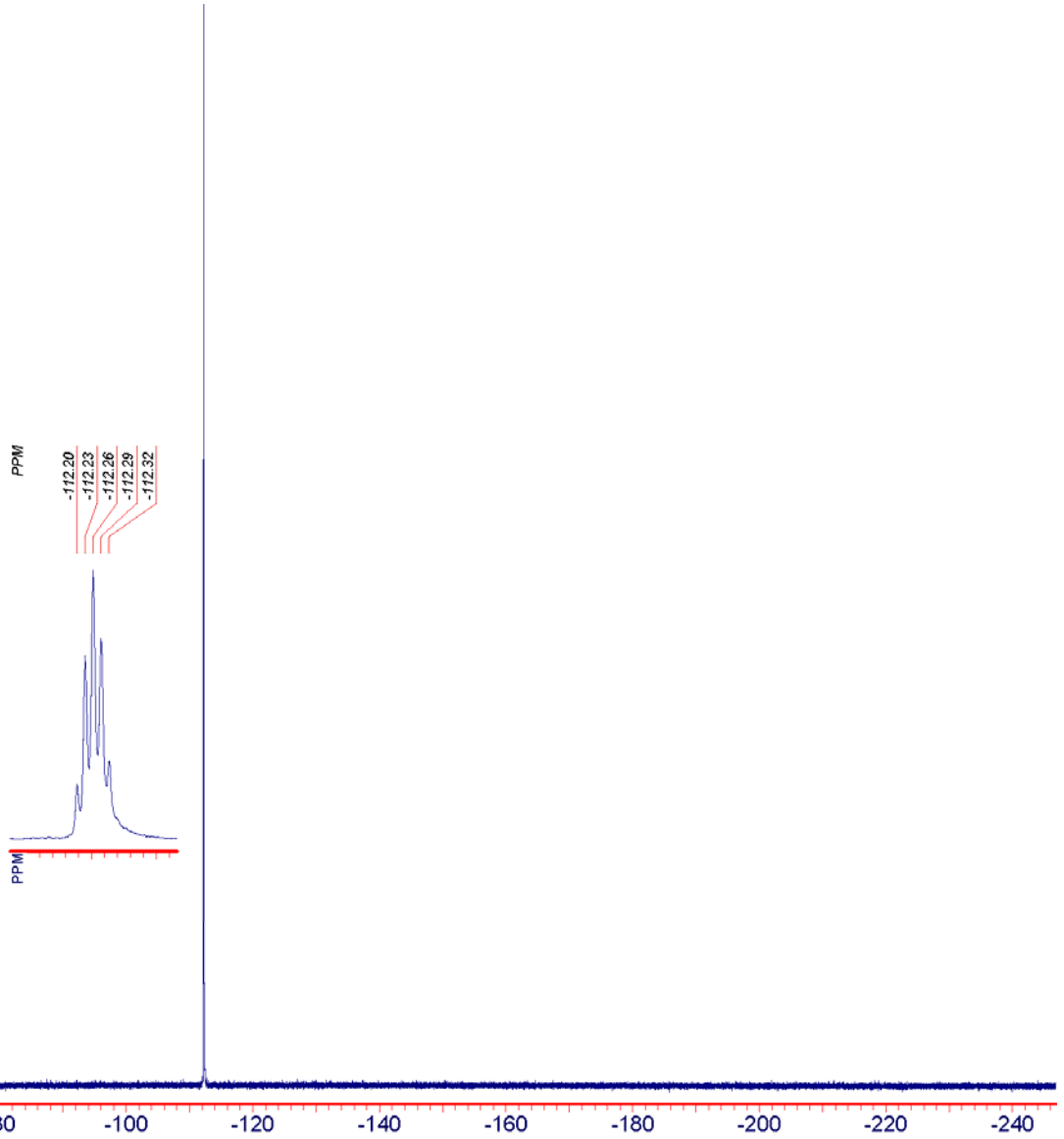
File name: pkm288	Operator: root⊓	SF: 499.9500 MHz	NSC: 1	PW: 0.00 usec, RG: 16	SI: 32768
Date: 22-Sep-2014	Solvent: DMSO	SW: 8993 Hz	TE: 0 K	AQ: 1.82 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓



File name: pkm288_C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 1305	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 22-Sep-2014	Solvent: dmso	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

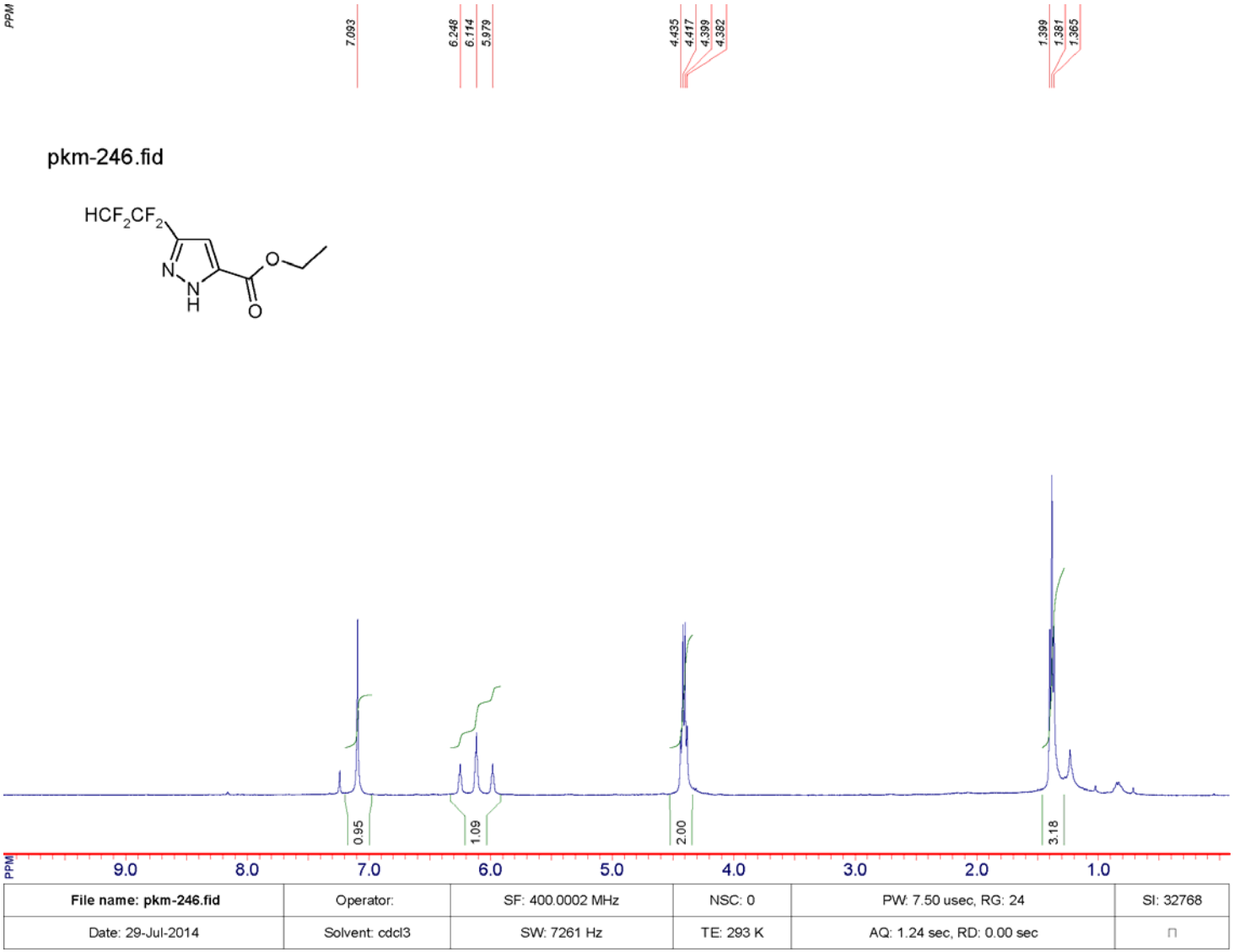
pkm288_F19.fid

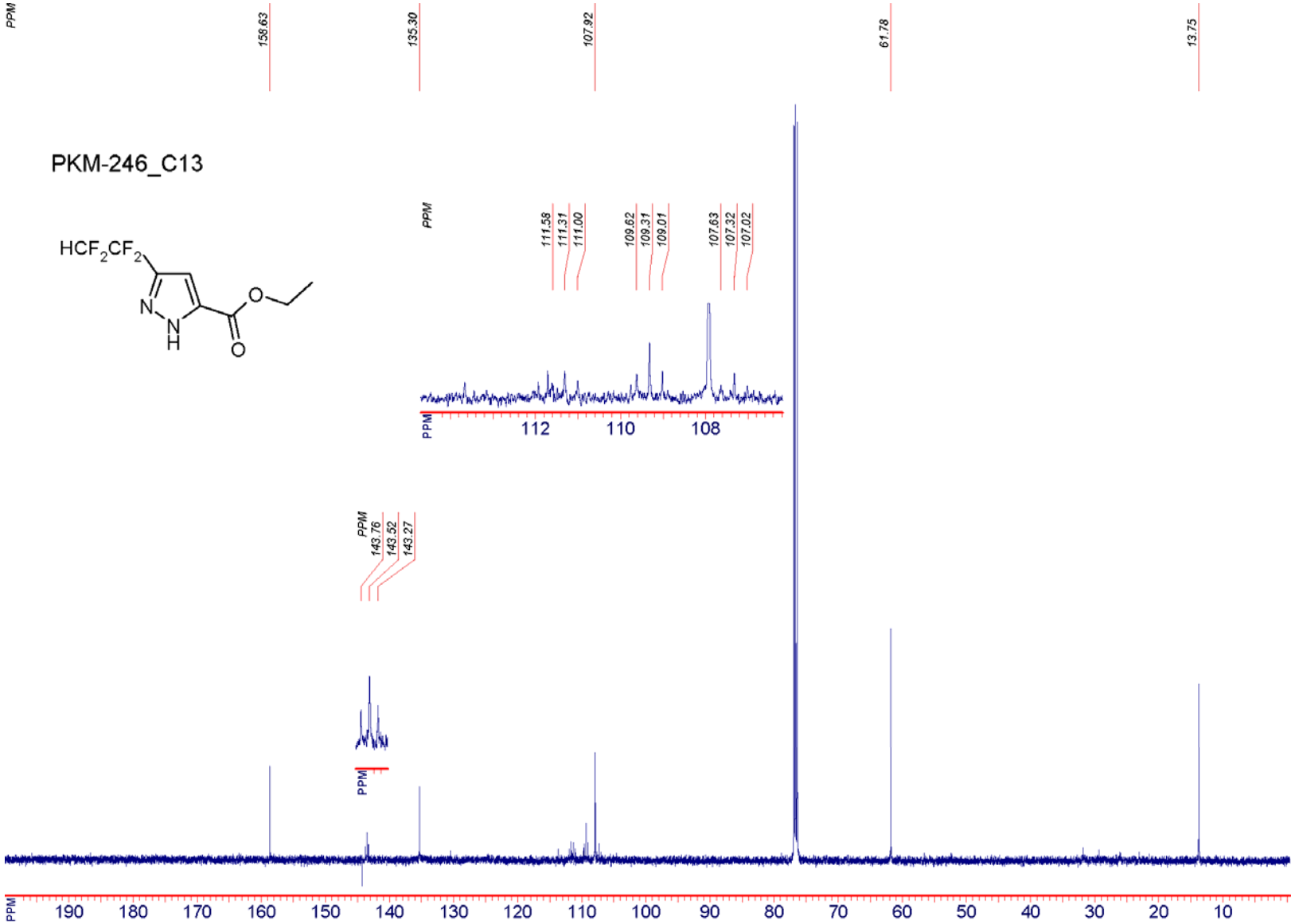




File name: pkm288_F19.fid	Operator:	SF: 376.3189 MHz	NSC: 0	PW: 3.00 usec, RG: 26	SI: 65536
Date: 25-Sep-2014	Solvent: cdcl3	SW: 94118 Hz	TE: 293 K	AQ: 0.34 sec, RD: 0.00 sec	⊓

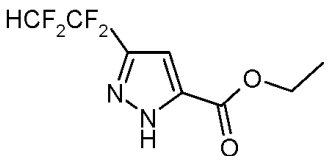
Compound 12a

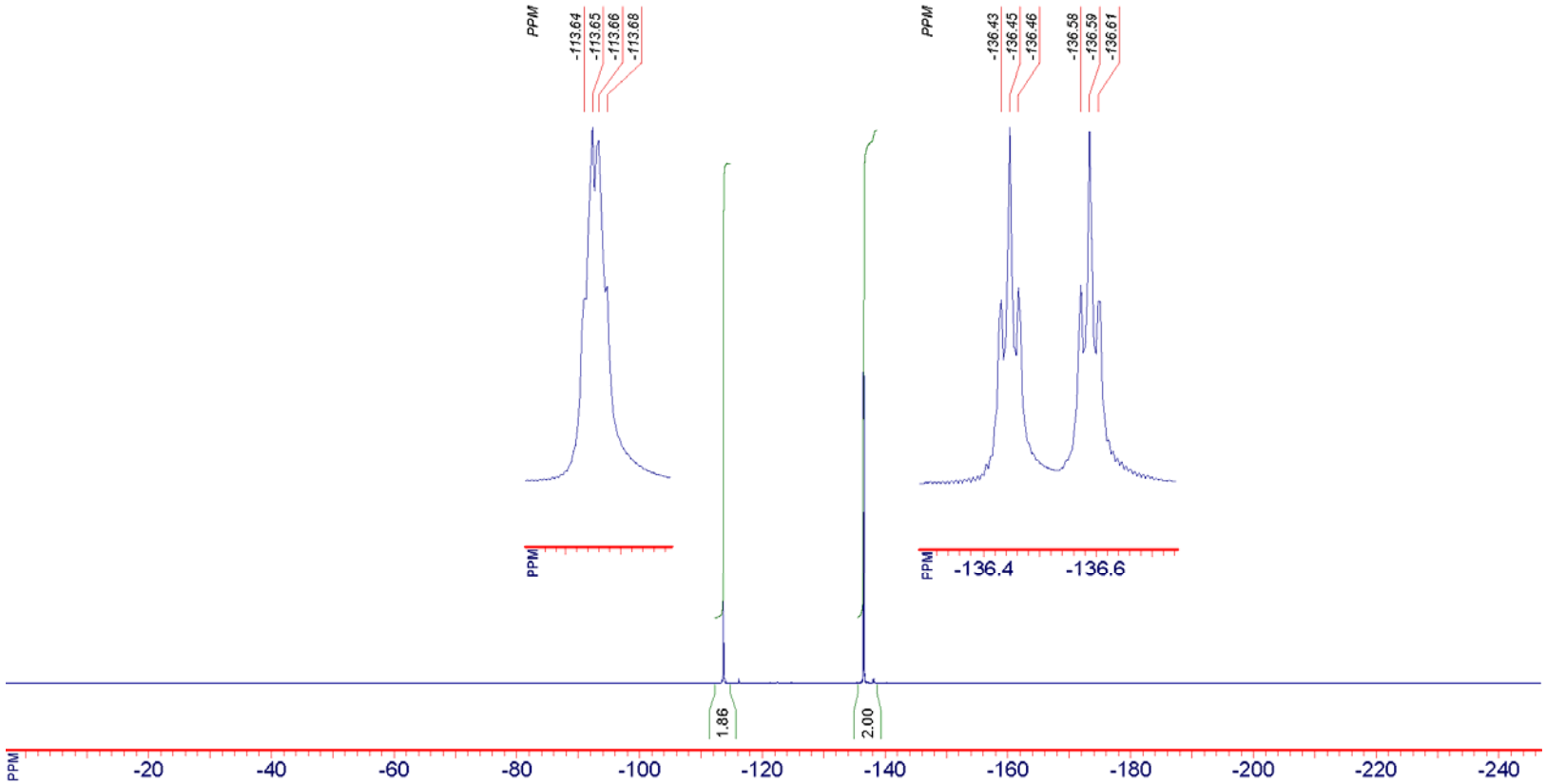




File name: PKM-246_C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 600	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 30-Jul-2014	Solvent: dmso	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

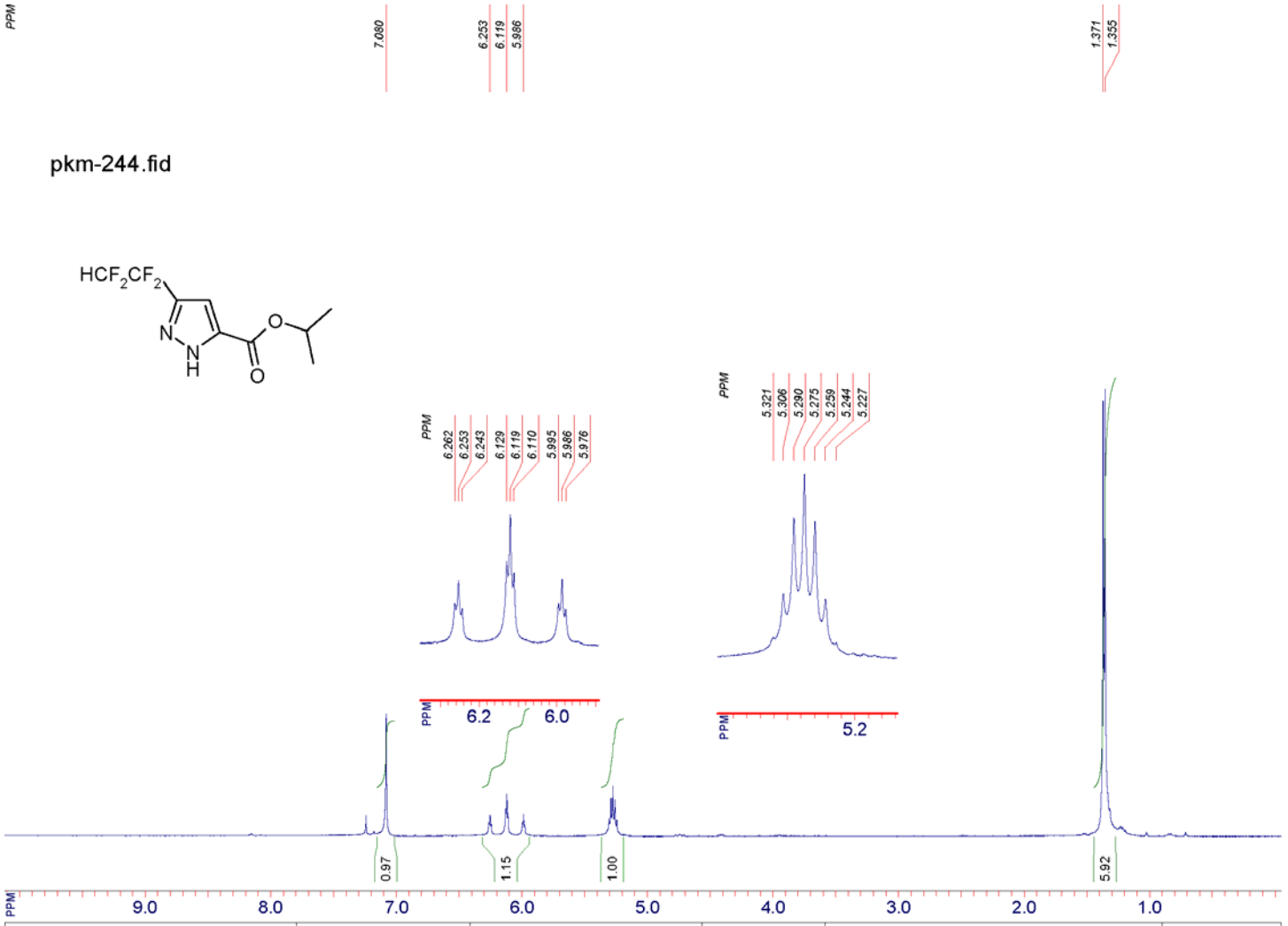
pkm246_F19.fid



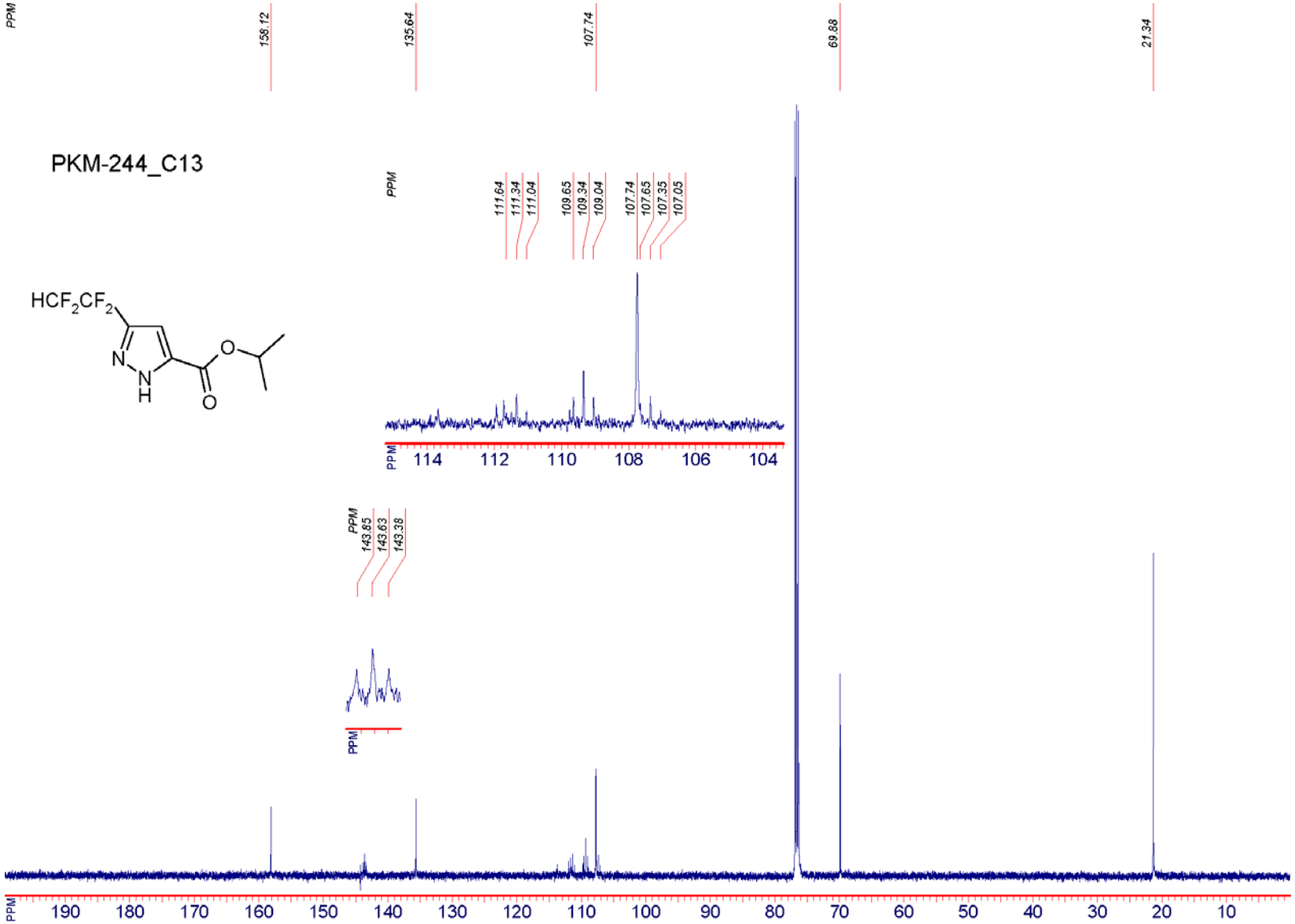


File name: pkm246_F19.fid	Operator:	SF: 376.3189 MHz	NSC: 0	PW: 3.00 usec, RG: 26	SI: 65536
Date: 31-Jul-2014	Solvent: cdcl3	SW: 94118 Hz	TE: 293 K	AQ: 0.34 sec, RD: 0.00 sec	⊓

Compound 13a

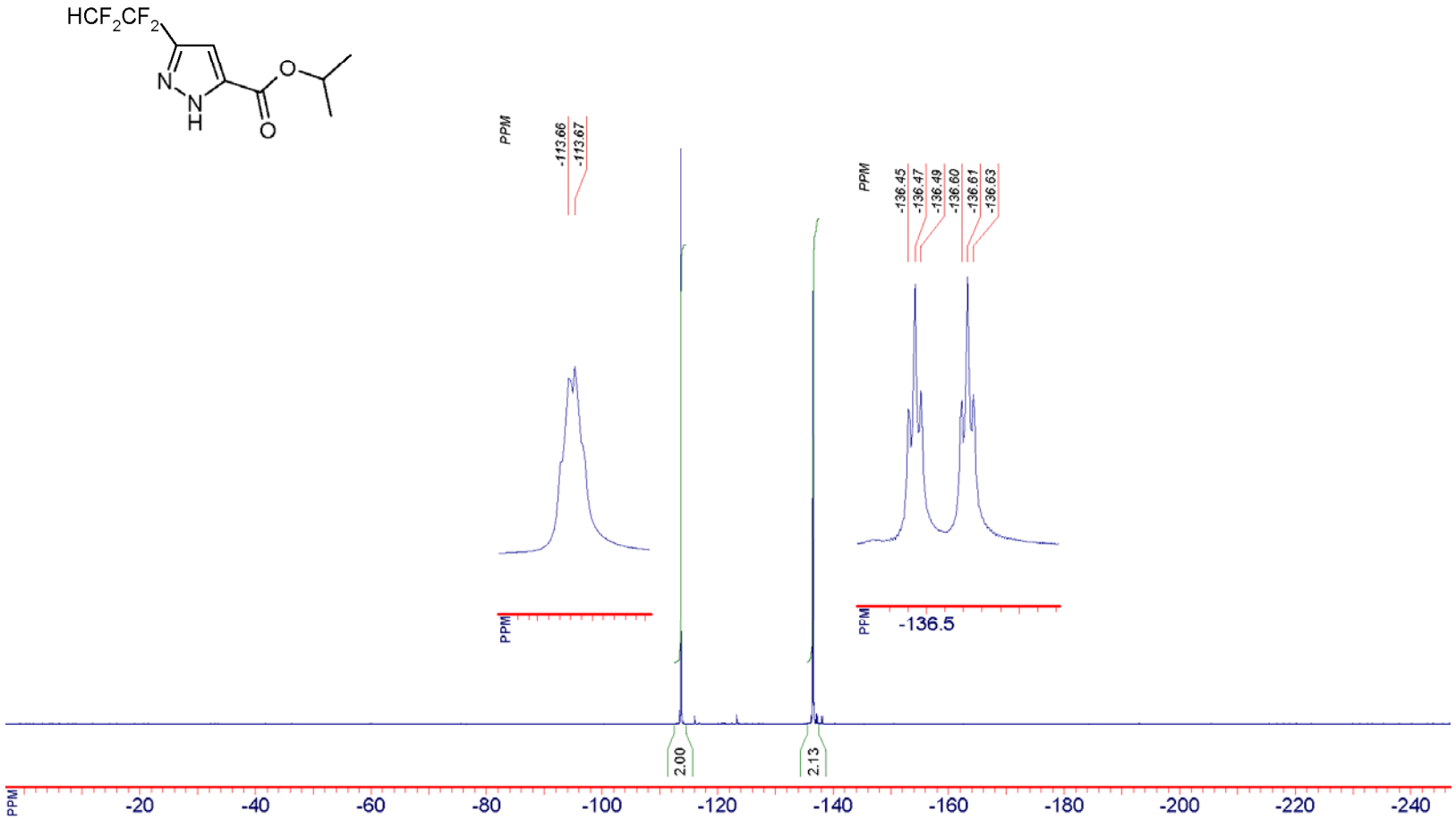


File name: pkm-244.fid	Operator:	SF: 400.0002 MHz	NSC: 0	PW: 7.50 usec, RG: 24	SI: 32768
Date: 29-Jul-2014	Solvent: cdcl3	SW: 7261 Hz	TE: 293 K	AQ: 1.24 sec, RD: 0.00 sec	⊓



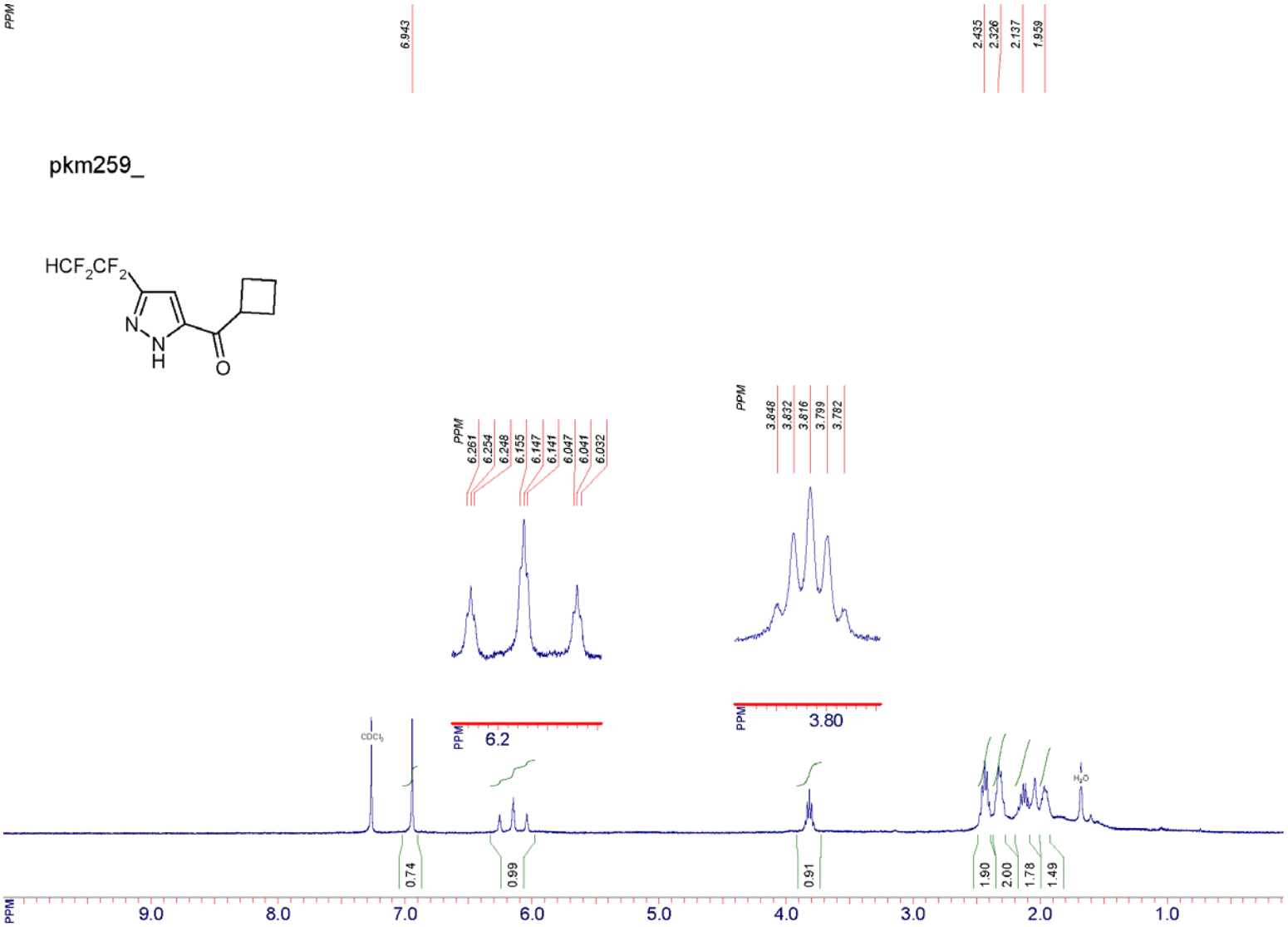
File name: PKM-244_C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 802	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 30-Jul-2014	Solvent: dmso	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

pkm244_F19.fid

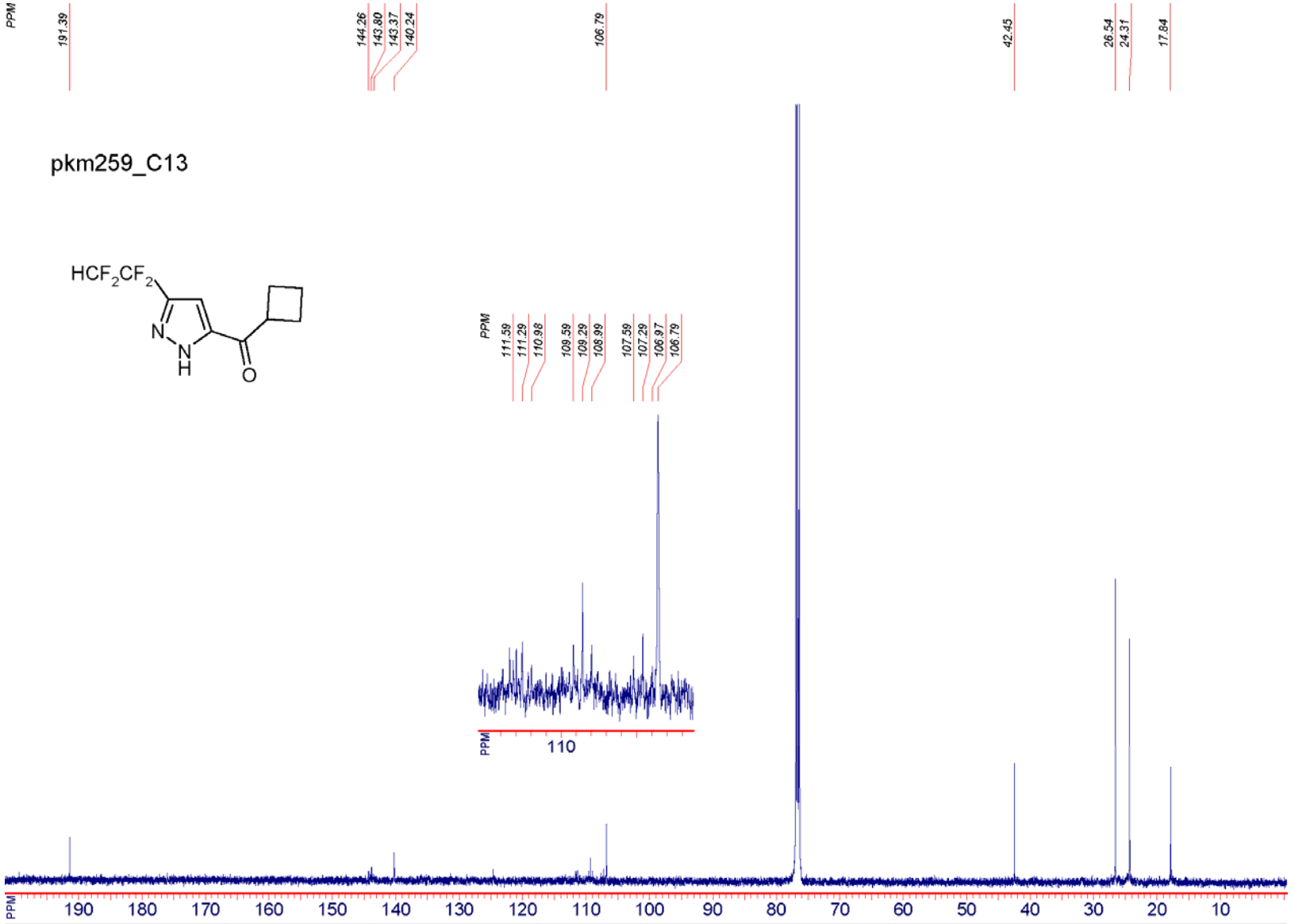


File name: pkm244_F19.fid	Operator:	SF: 376.3189 MHz	NSC: 0	PW: 3.00 usec, RG: 26	SI: 65536
Date: 31-Jul-2014	Solvent: cdcl3	SW: 94118 Hz	TE: 293 K	AQ: 0.34 sec, RD: 0.00 sec	⊓

Compound 14a

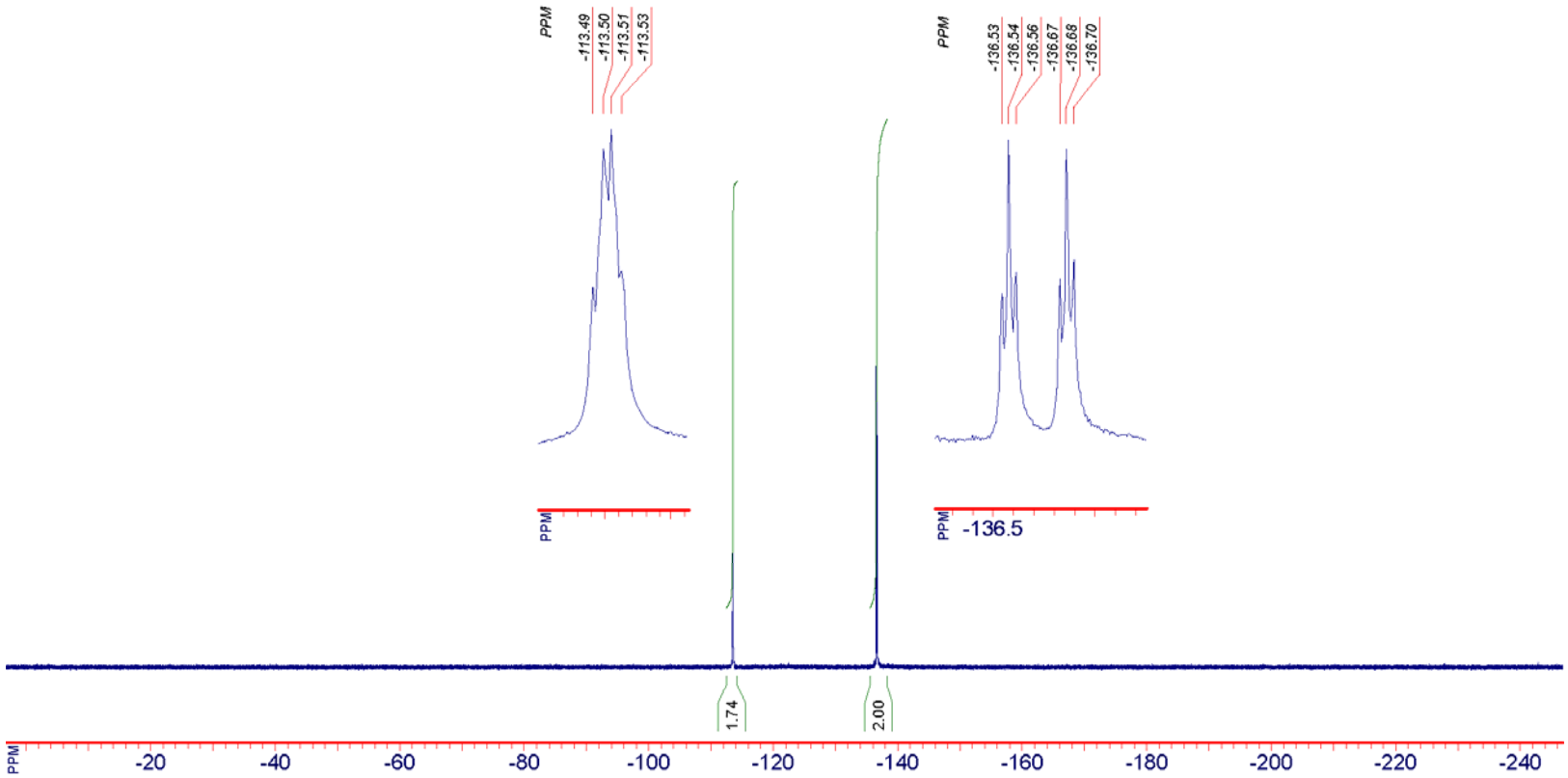


File name: pkm259_	Operator: psmП	SF: 499.9500 MHz	NSC: 1	PW: 0.00 usec, RG: 32	SI: 32768
Date: 03-Aug-2014	Solvent: CDCl3	SW: 8993 Hz	TE: 0 K	AQ: 1.82 sec, RD: 0.00 sec	Parameter file, XWIN-NMRПППVersion 3.5П



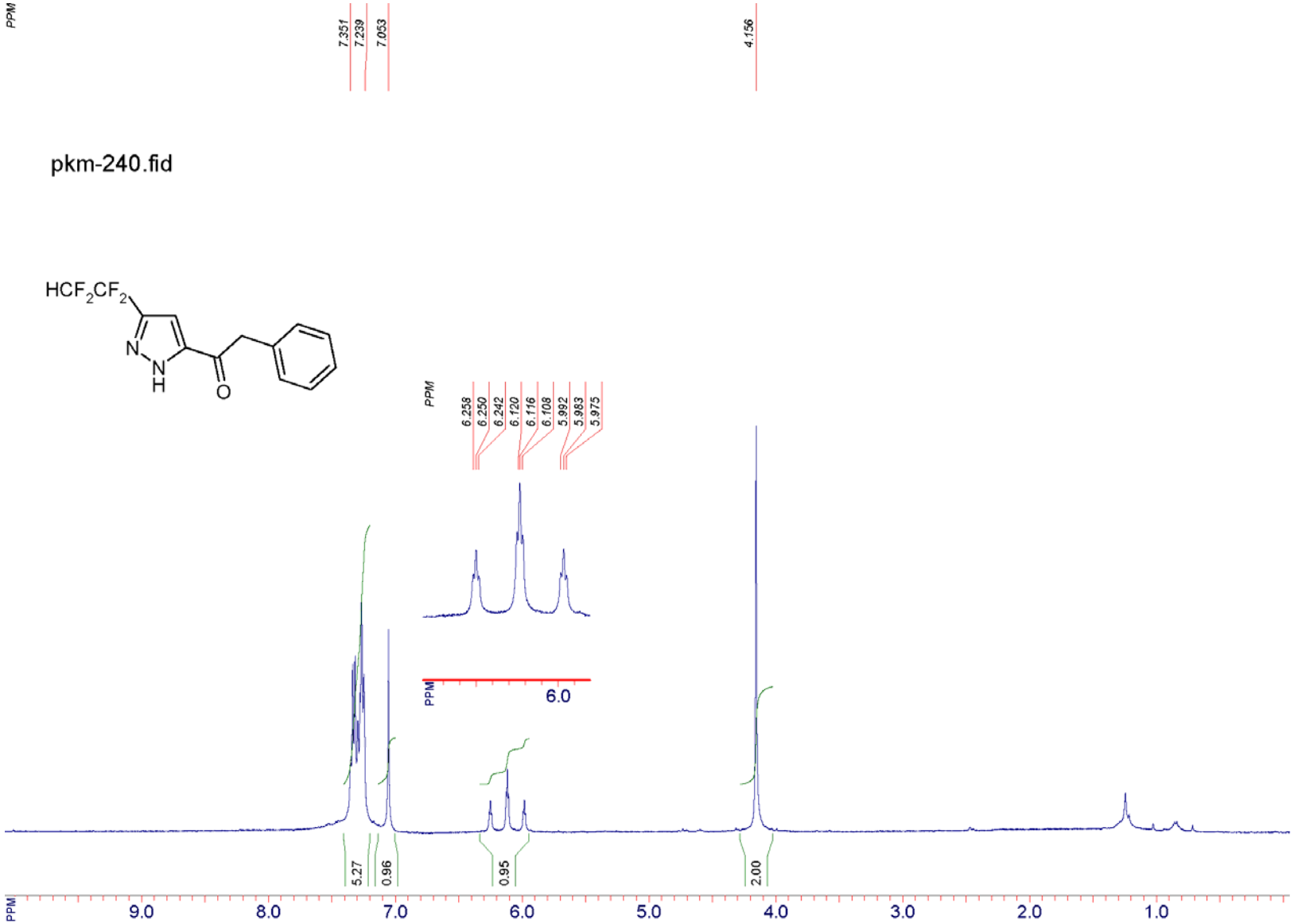
File name: pkm259_C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 4335	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 03-Aug-2014	Solvent: dmso	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

pkm259_F19.fid

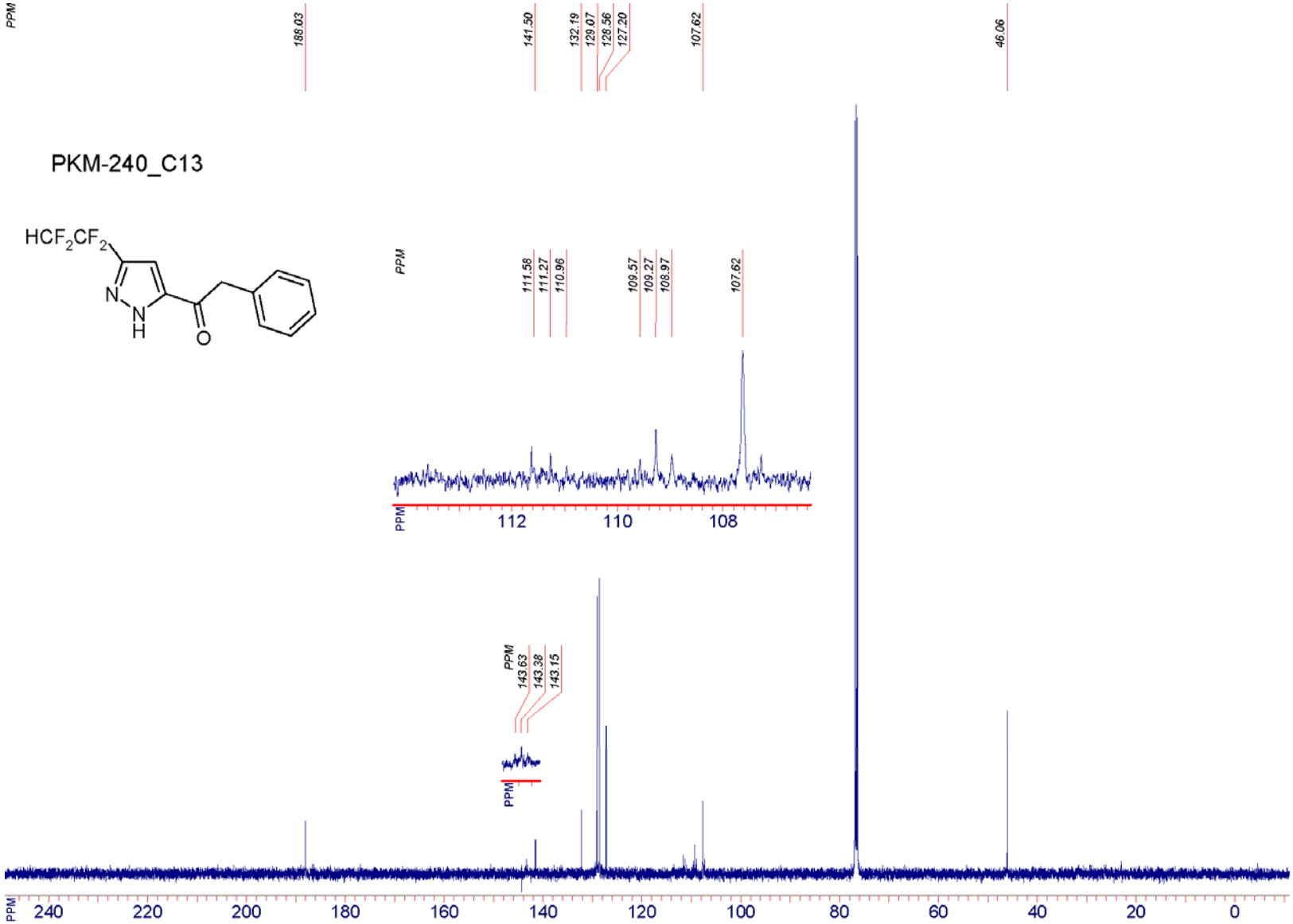


File name: pkm259_F19.fid	Operator:	SF: 376.3189 MHz	NSC: 0	PW: 3.00 usec, RG: 26	SI: 65536
Date: 07-Aug-2014	Solvent: cdcl3	SW: 94118 Hz	TE: 293 K	AQ: 0.34 sec, RD: 0.00 sec	⊓

Compound 15a

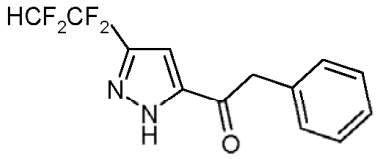


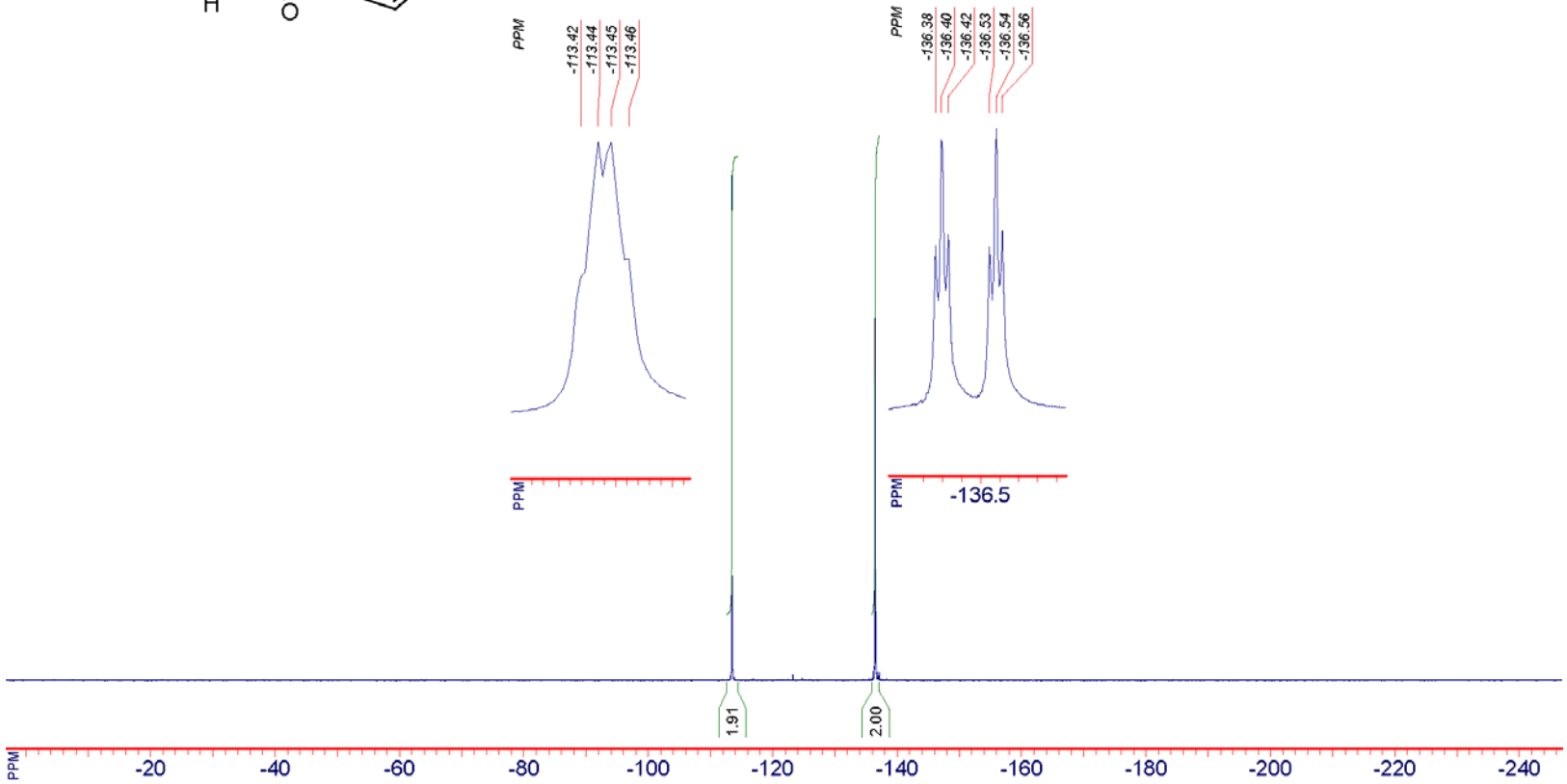
File name: pkm-240.fid	Operator:	SF: 400.0002 MHz	NSC: 0	PW: 7.50 usec, RG: 24	SI: 32768
Date: 29-Jul-2014	Solvent: cdcl3	SW: 7261 Hz	TE: 293 K	AQ: 1.24 sec, RD: 0.00 sec	□



File name: PKM-240_C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 388	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 30-Jul-2014	Solvent: dmso	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

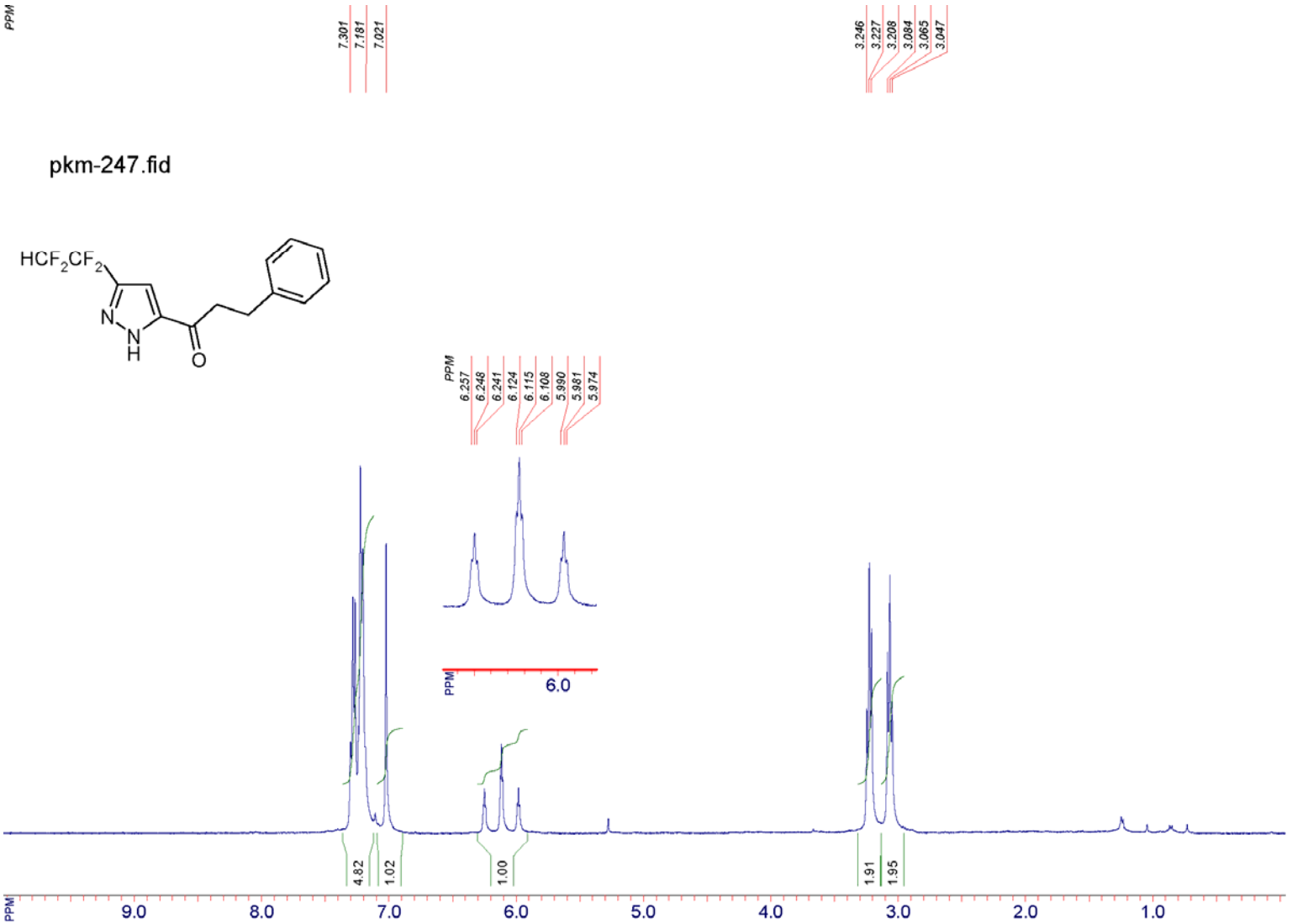
pkm240_F19.fid



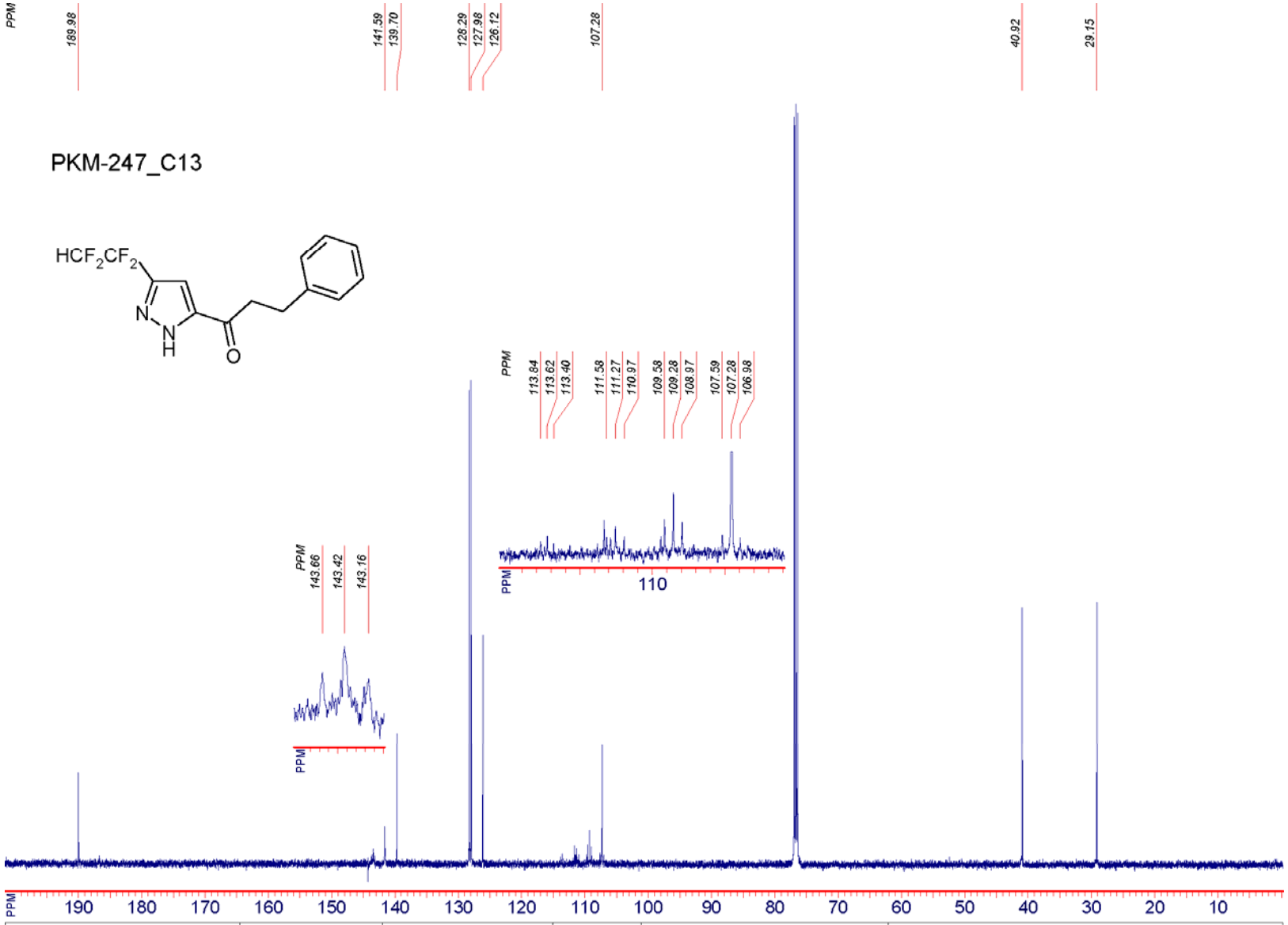


File name: pkm240_F19.fid	Operator:	SF: 376.3189 MHz	NSC: 0	PW: 3.00 usec, RG: 26	SI: 65536
Date: 31-Jul-2014	Solvent: cdcl3	SW: 94118 Hz	TE: 293 K	AQ: 0.34 sec, RD: 0.00 sec	⊓

Compound 16a

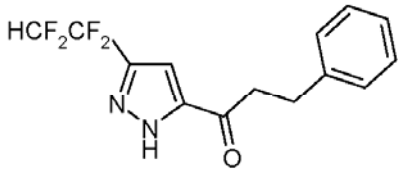


File name: pkm-247.fid	Operator:	SF: 400.0002 MHz	NSC: 0	PW: 7.50 usec, RG: 24	SI: 32768
Date: 29-Jul-2014	Solvent: cdcl3	SW: 7261 Hz	TE: 293 K	AQ: 1.24 sec, RD: 0.00 sec	



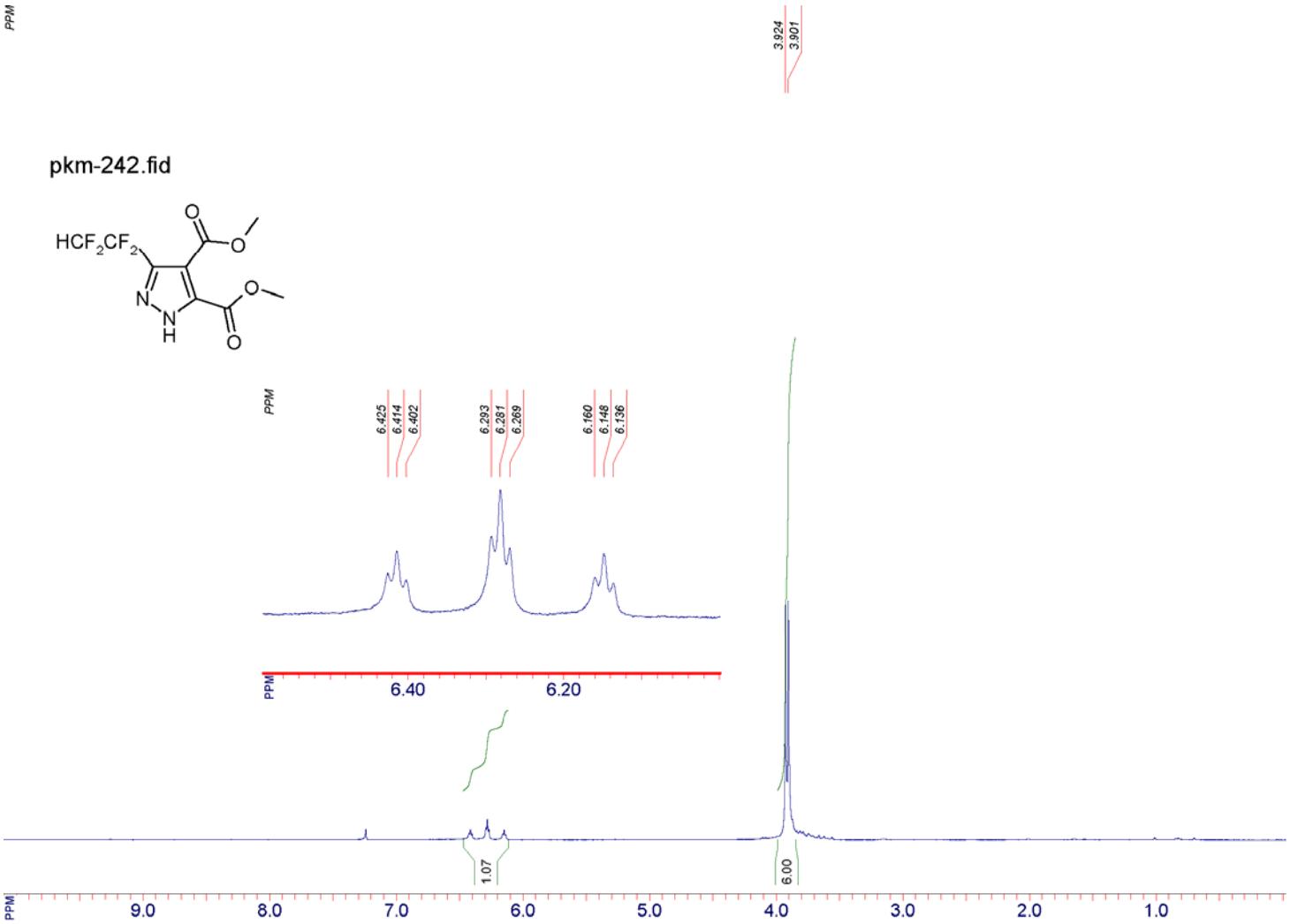
File name: PKM-247_C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 859	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 30-Jul-2014	Solvent: dmso	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

pkm247_F19.fid

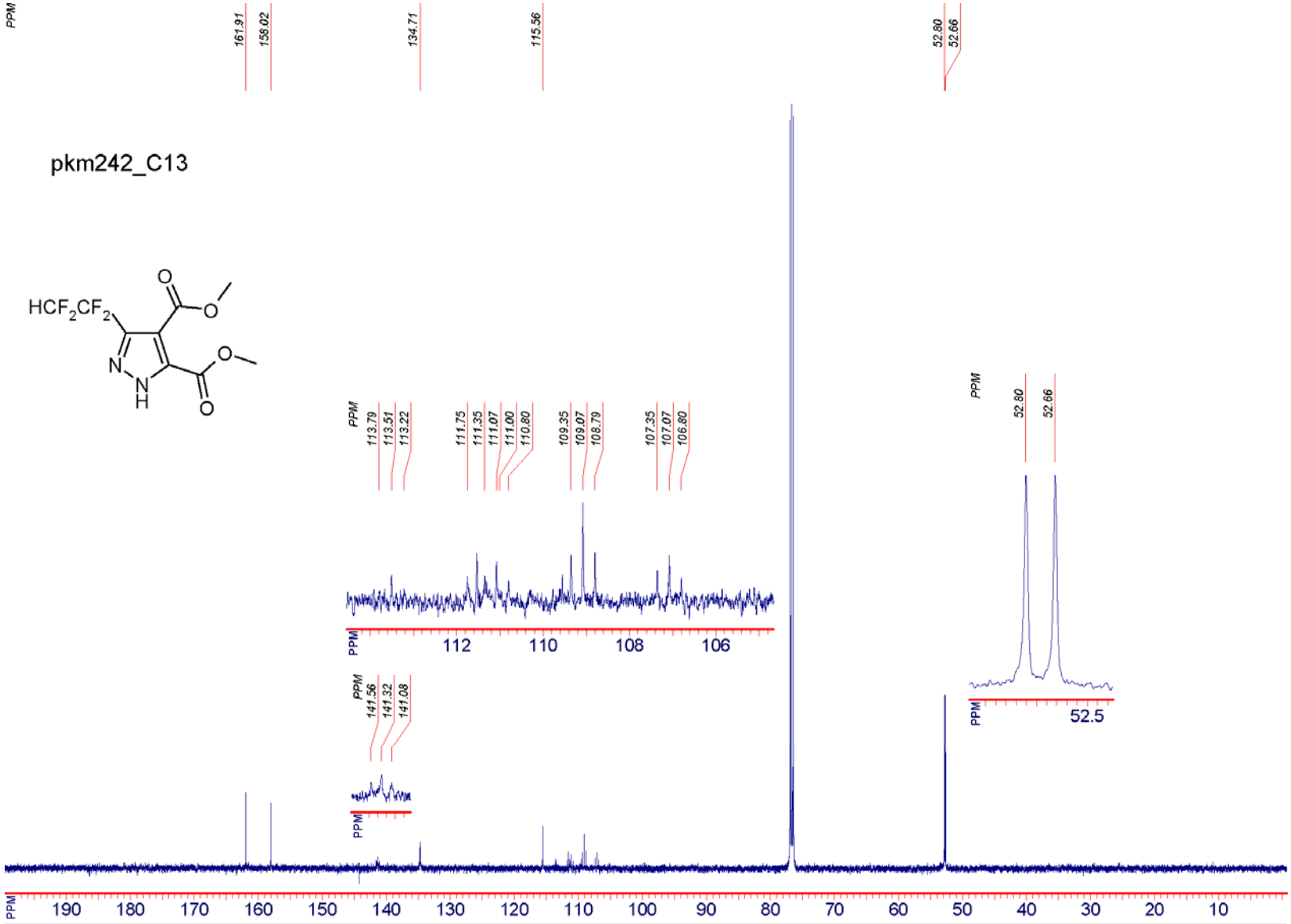


File name: pkm247_F19.fid	Operator:	SF: 376.3189 MHz	NSC: 0	PW: 3.00 usec, RG: 26	SI: 65536
Date: 31-Jul-2014	Solvent: cdcl3	SW: 94118 Hz	TE: 293 K	AQ: 0.34 sec, RD: 0.00 sec	⊓

Compound 17a

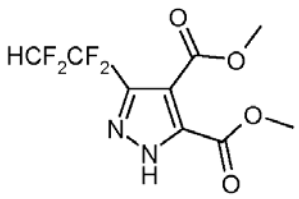


File name: pkm-242.fid	Operator:	SF: 400.0002 MHz	NSC: 0	PW: 7.50 usec, RG: 24	SI: 32768
Date: 29-Jul-2014	Solvent: cdcl3	SW: 7261 Hz	TE: 293 K	AQ: 1.24 sec, RD: 0.00 sec	⊓



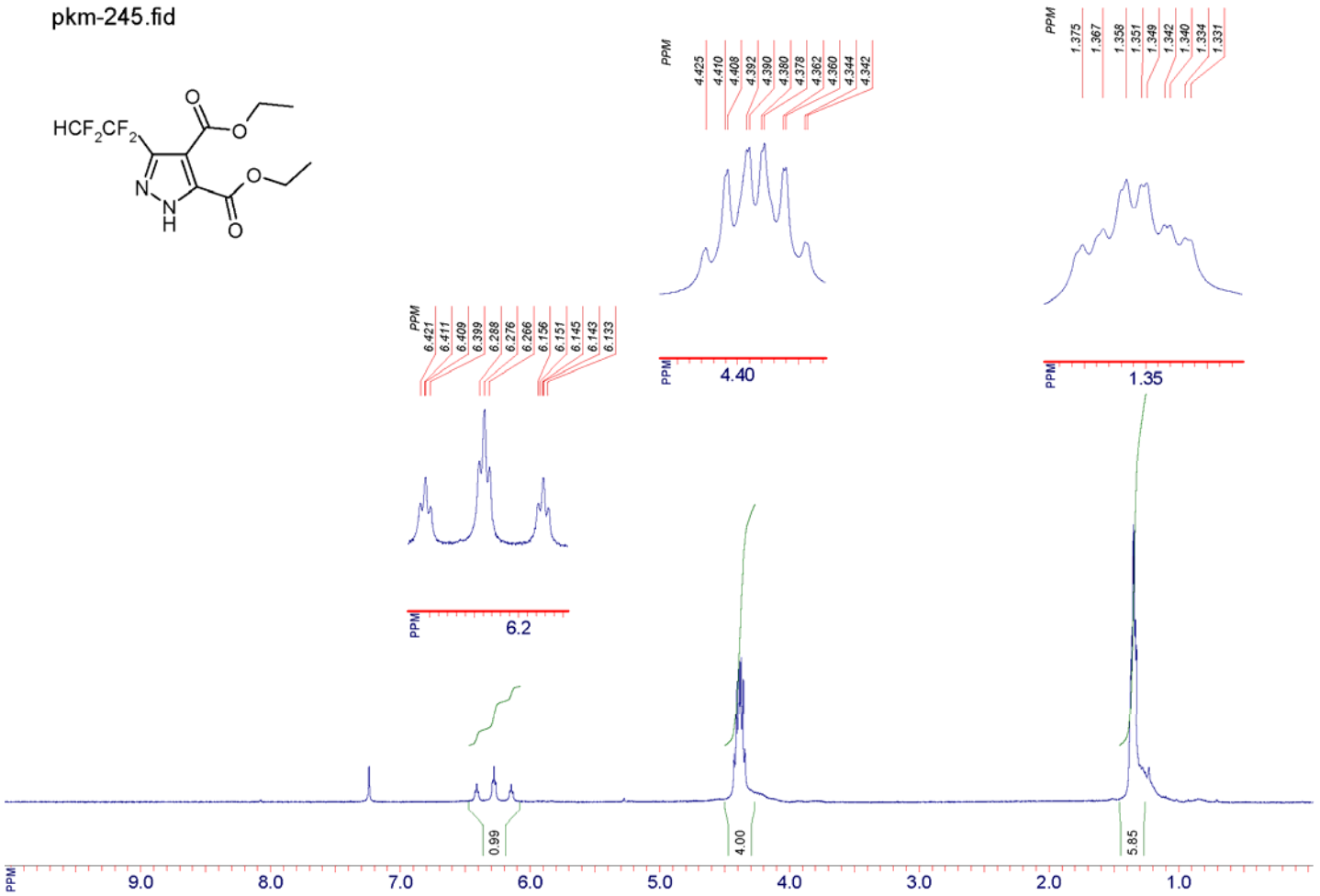
File name: pkm242_C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 739	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 31-Jul-2014	Solvent: CDCl3	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

pkm242_F19.fid

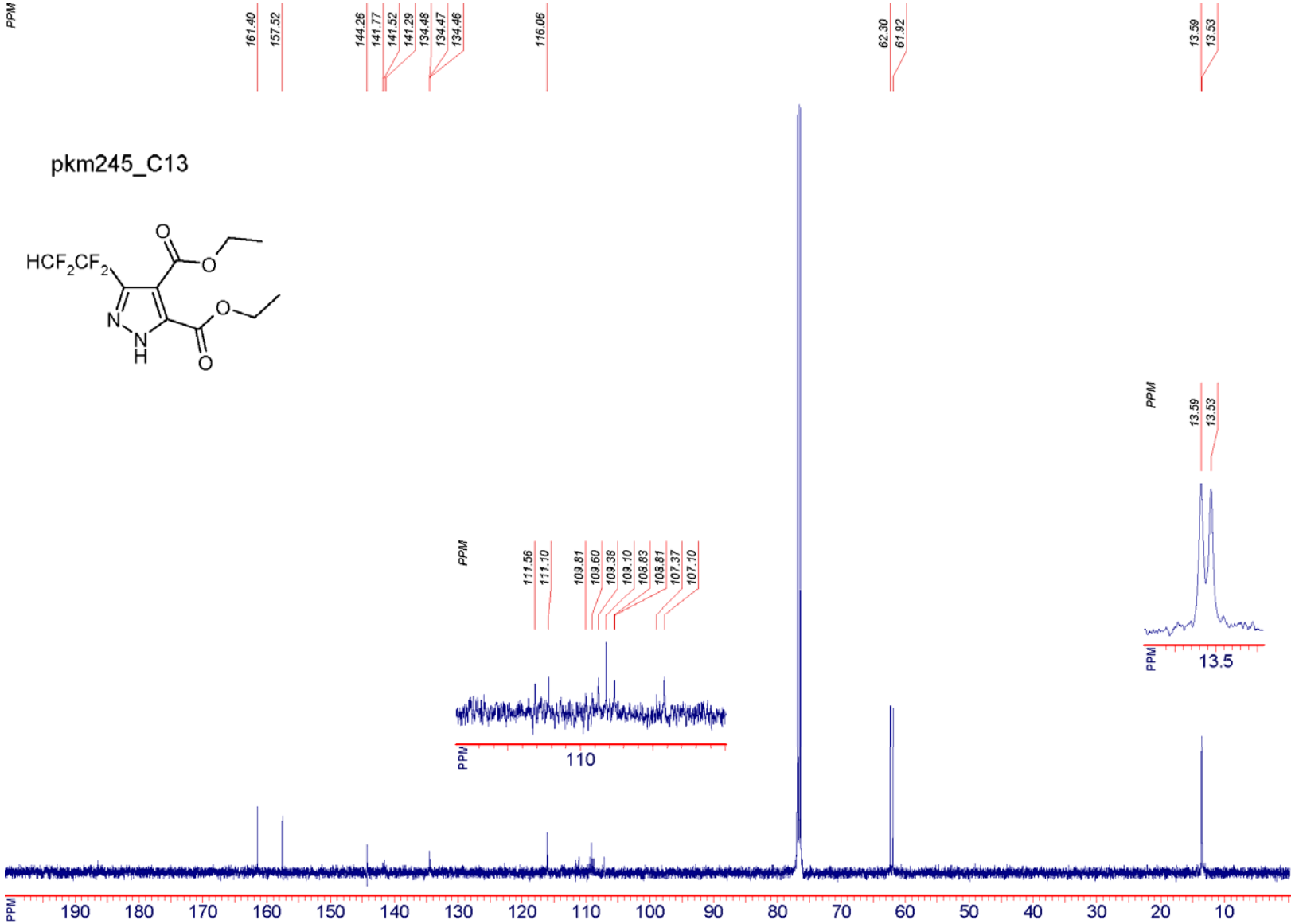


File name: pkm242_F19.fid	Operator:	SF: 376.3189 MHz	NSC: 0	PW: 3.00 usec, RG: 26	SI: 65536
Date: 05-Aug-2014	Solvent: cdcl3	SW: 94118 Hz	TE: 293 K	AQ: 0.34 sec, RD: 0.00 sec	⊓

Compound 18a

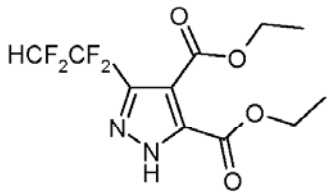


File name: pkm-245.fid	Operator:	SF: 400.0002 MHz	NSC: 0	PW: 7.50 usec, RG: 24	SI: 32768
Date: 29-Jul-2014	Solvent: cdcl3	SW: 7261 Hz	TE: 293 K	AQ: 1.24 sec, RD: 0.00 sec	n



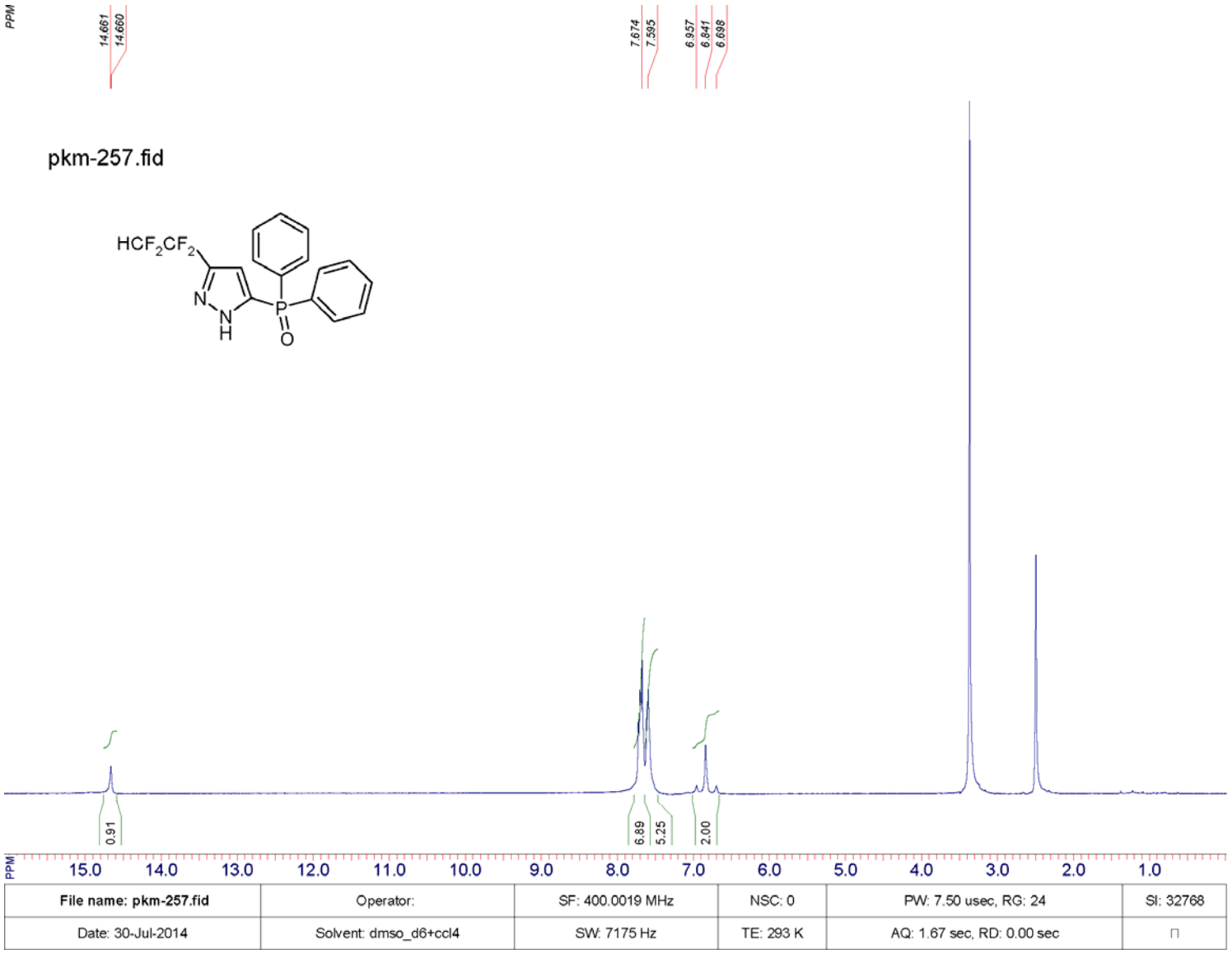
File name: pkm245_C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 798	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 31-Jul-2014	Solvent: dmso	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

pkm245_F19.fid

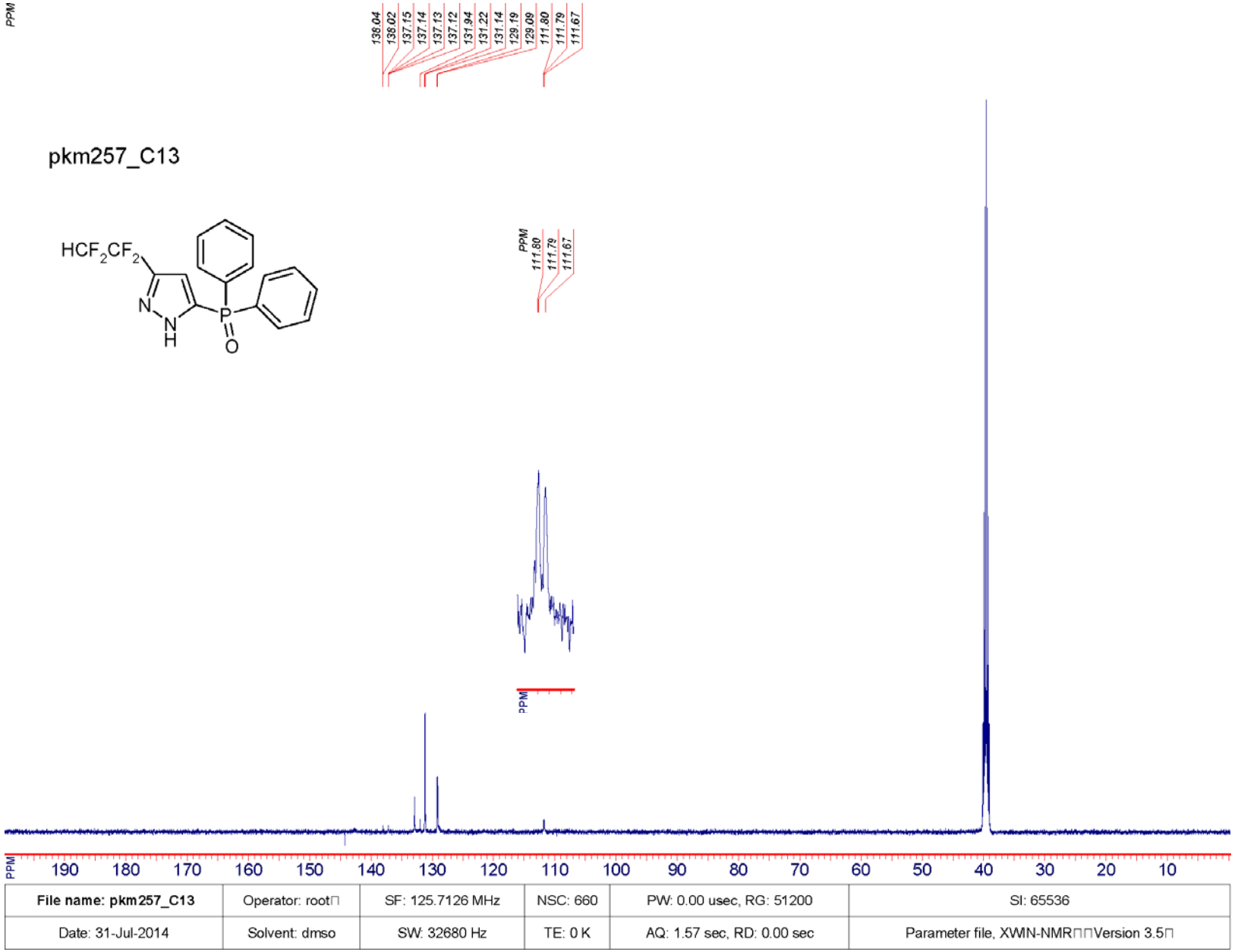


File name: pkm245_F19.fid	Operator:	SF: 376.3189 MHz	NSC: 0	PW: 3.00 usec, RG: 26	SI: 65536
Date: 05-Aug-2014	Solvent: cdcl3	SW: 94118 Hz	TE: 293 K	AQ: 0.34 sec, RD: 0.00 sec	⊓

Compound 19a

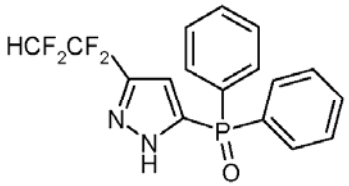


File name: pkm-257.fid	Operator:	SF: 400.0019 MHz	NSC: 0	PW: 7.50 usec, RG: 24	SI: 32768
Date: 30-Jul-2014	Solvent: dmso_d6+ccl4	SW: 7175 Hz	TE: 293 K	AQ: 1.67 sec, RD: 0.00 sec	



File name: pkm257_C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 660	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 31-Jul-2014	Solvent: dmso	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

pkm257_F19.fid

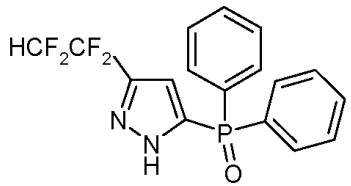


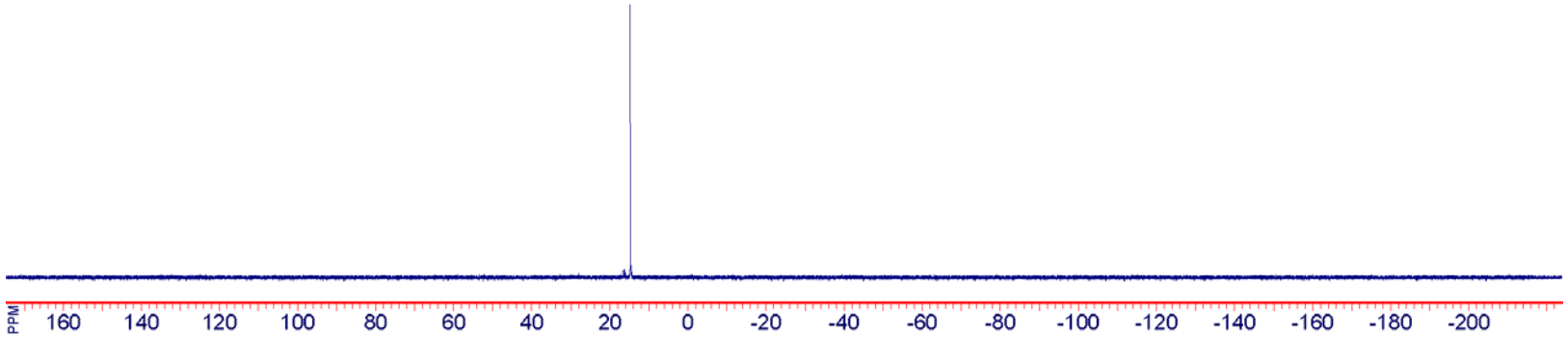
File name: pkm257_F19.fid	Operator:	SF: 376.3274 MHz	NSC: 0	PW: 3.00 usec, RG: 32	SI: 65536
Date: 05-Aug-2014	Solvent: dmso_d6	SW: 82816 Hz	TE: 293 K	AQ: 0.39 sec, RD: 0.00 sec	⊓

PPM

14.68

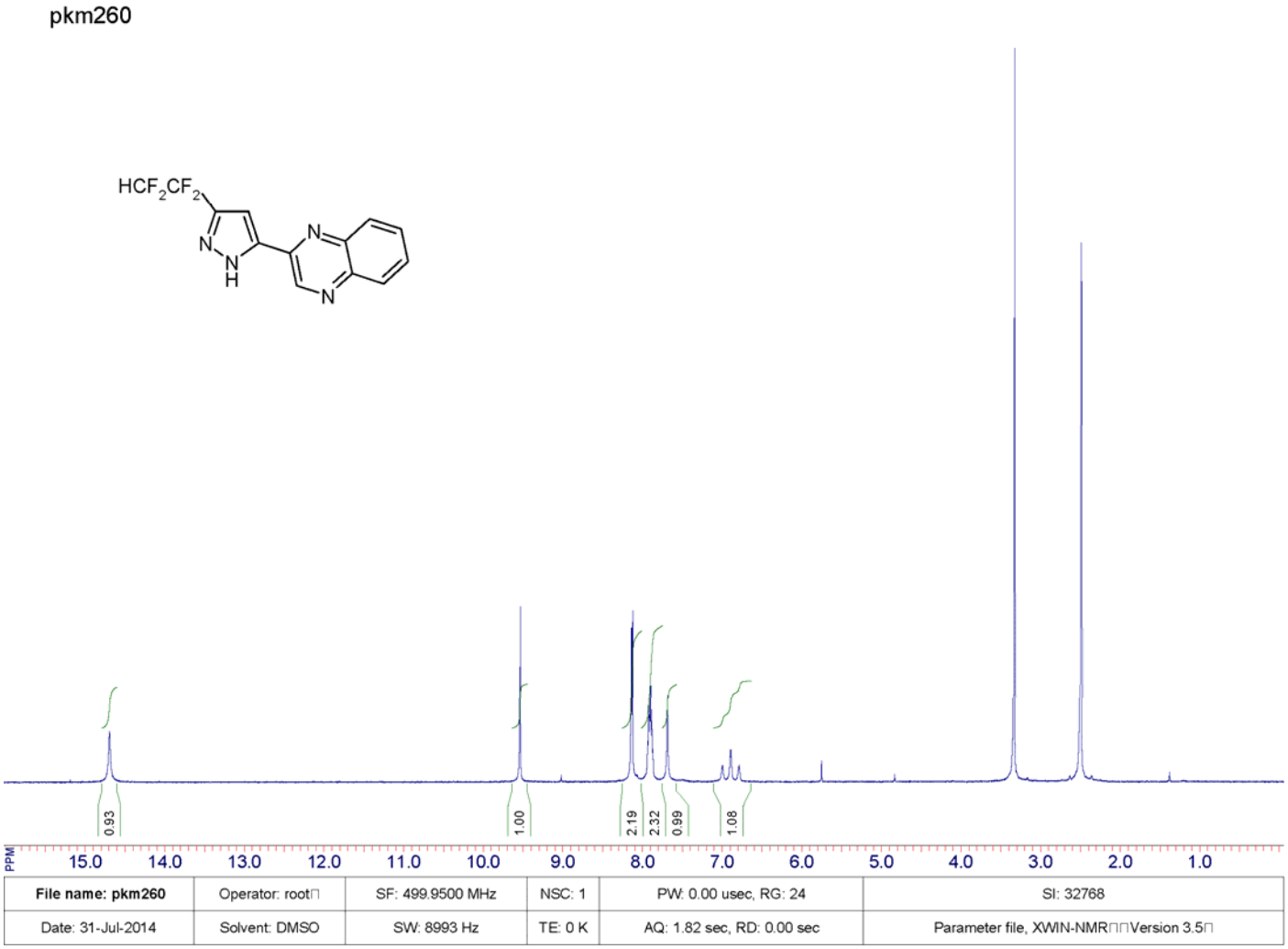
pkm257_P31



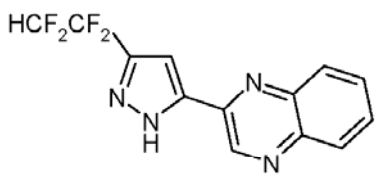


File name: pkm257_P31	Operator: root⊓	SF: 202.3834 MHz	NSC: 22	PW: 0.00 usec, RG: 2048	SI: 65536
Date: 04-Aug-2014	Solvent: d2o	SW: 80645 Hz	TE: 0 K	AQ: 0.81 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

Compound 20a

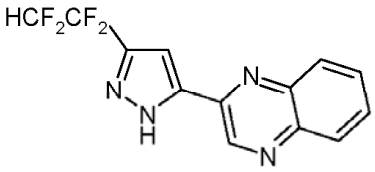


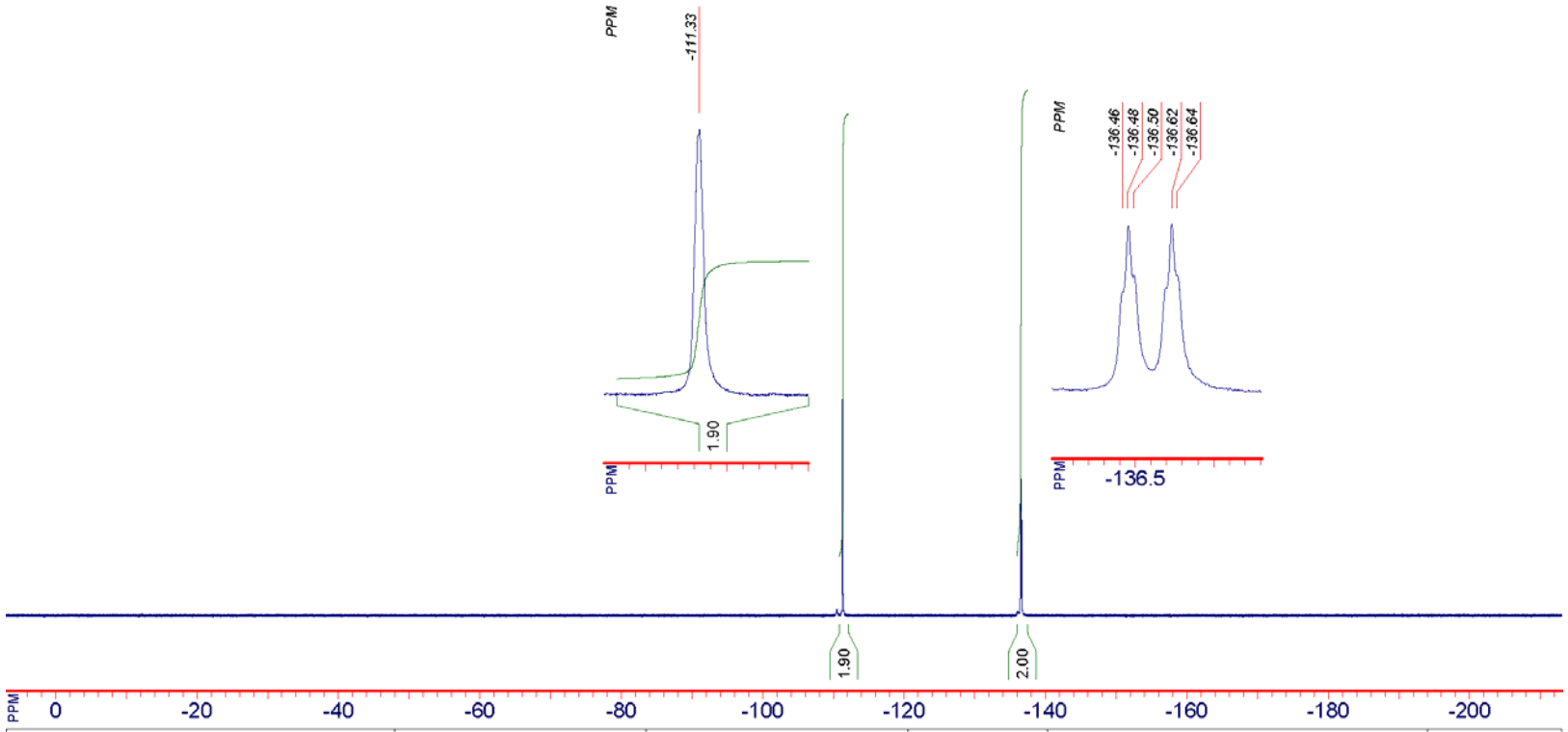
pkm260_C13



File name: pkm260_C13	Operator: root⊓	SF: 125.7126 MHz	NSC: 8415	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 03-Aug-2014	Solvent: dmso	SW: 32680 Hz	TE: 0 K	AQ: 1.57 sec, RD: 0.00 sec	Parameter file, XWIN-NMR⊓⊓Version 3.5⊓

pkm260_F19.fid





File name: pkm260_F19.fid	Operator:	SF: 376.3274 MHz	NSC: 0	PW: 3.00 usec, RG: 32	SI: 65536
Date: 07-Aug-2014	Solvent: dmso_d6	SW: 82816 Hz	TE: 293 K	AQ: 0.39 sec, RD: 0.00 sec	⊓