

***Supporting Information***

***t*-BuOOH / TiO<sub>2</sub> photocatalytic system as a convenient peroxy radical source at room temperature under visible light and its application for CH-peroxidation of Barbituric acids**

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## EPR experiments

EPR spectra were recorded in 100  $\mu$ L capillary tubes (inner diameter ca. 1.2 mm) using X-Band EPR spectrometer Adani Spinscan X at 31-32 °C with following settings: sweep width – 7 mT, sweep time – 60 s, modulation amplitude – 100  $\mu$ T, single scan for all experiments. For experiments with irradiation by blue LED, samples were irradiated inside the EPR cavity after instrument tuning. EPR spectra recording was started immediately after the start of irradiation. For experiments with UV irradiation, samples were irradiated by 20 W ultraviolet (365 nm) LED for 30 seconds (1 cm from the LED to the sample in a capillary tube), then samples were placed in EPR cavity, and spectra were recorded (ca. 2-3 min after irradiation). Unless otherwise noted, samples were prepared under room light.

Samples for EPR studies were prepared by mixing solutions or suspensions of interest (150  $\mu$ L, mixture A) with 150  $\mu$ L of solution of spin trap DMPO (5,5-Dimethyl-1-pyrroline N-oxide, solution B). DMPO solution (B) was prepared from DMPO (10 mg, 88.3  $\mu$ mol) and MeCN (1 mL). For EPR measurements under argon, mixture A and solution B were prepared under air, but flushed by argon flow for two minutes after mixing. Compositions of studied mixtures (A) are given below:

TiO<sub>2</sub> + *t*-BuOOH: To TiO<sub>2</sub> (10 mg) was added MeCN (1 mL), the mixture was sonicated in an ultrasonic bath for 1 minute, then *t*-BuOOH 70% aq. (257 mg, 2 mmol) was added.

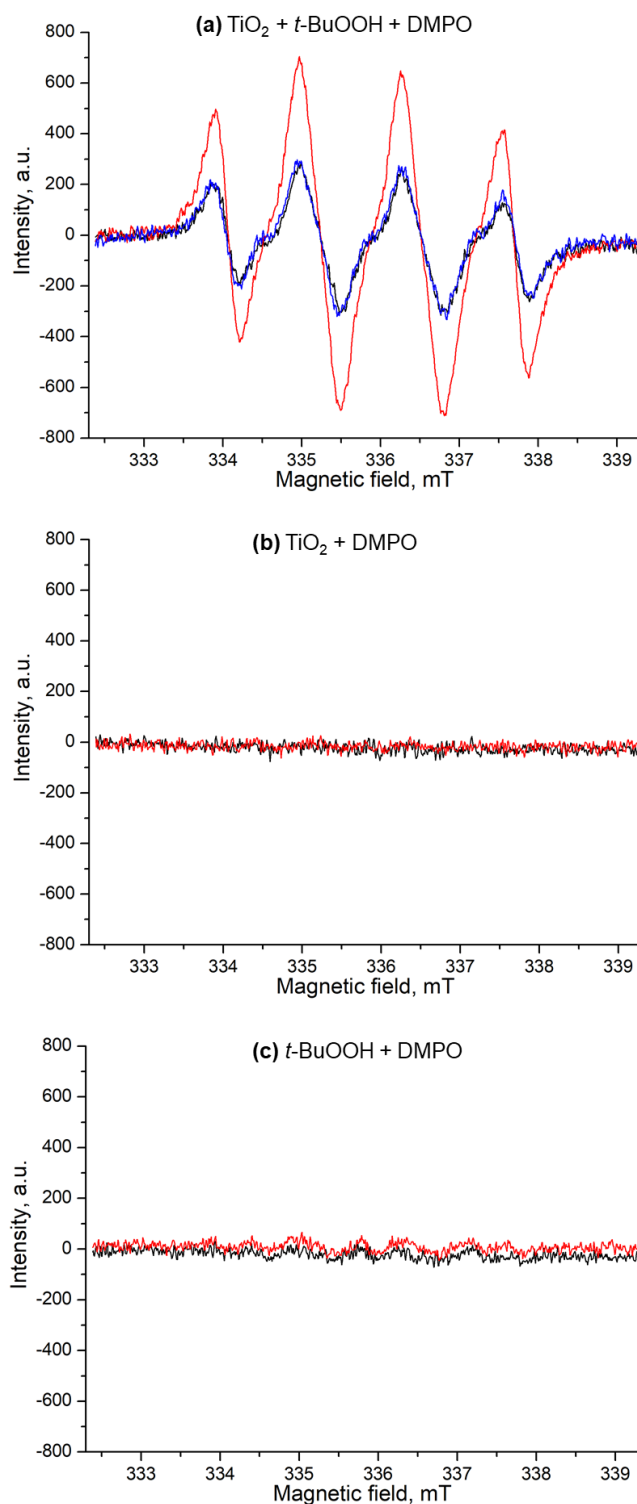
*t*-BuOOH: *t*-BuOOH 70% aq. (257 mg, 2 mmol) was dissolved in MeCN (1 mL).

TiO<sub>2</sub>: TiO<sub>2</sub> (10 mg) was dissolved in MeCN (1 mL).

*t*-BuOO*t*-Bu: di-tert-butylperoxide (292 mg, 2 mmol) was dissolved in MeCN (1 mL).

To study the formation of free radicals in the *t*-BuOOH-TiO<sub>2</sub> system EPR spin trapping experiments with DMPO were realized (Figure S1). In each experiment, spectrum was recorded before blue LED irradiation (black line) and under blue LED irradiation (red line). Intensive EPR signal was observed for the mixture of TiO<sub>2</sub>, *t*-BuOOH and DMPO (Figure S1, a) even before irradiation, which was surprising, because no peroxidation product **9a** and no Barbituric acid **8a** conversion was observed without blue light irradiation (see Table 1, entry 2). In order to check, whether radical formation is associated with the room lighting, under which samples were prepared, we performed control experiment with sample preparation in the dark room (minimal light exposure, Figure S1, a, blue line). The same signal intensity was observed as in case of sample preparation under room lighting, indicating that free radicals can be formed in *t*-BuOOH-TiO<sub>2</sub> even without light exposure. However, as we can see from Table 1, entry 2 in the main text of the paper, this is not sufficient for the preparative peroxidation to take place. Anyway, EPR signal intensity increased upon irradiation by blue LED (Figure S1, a, red line), which confirms

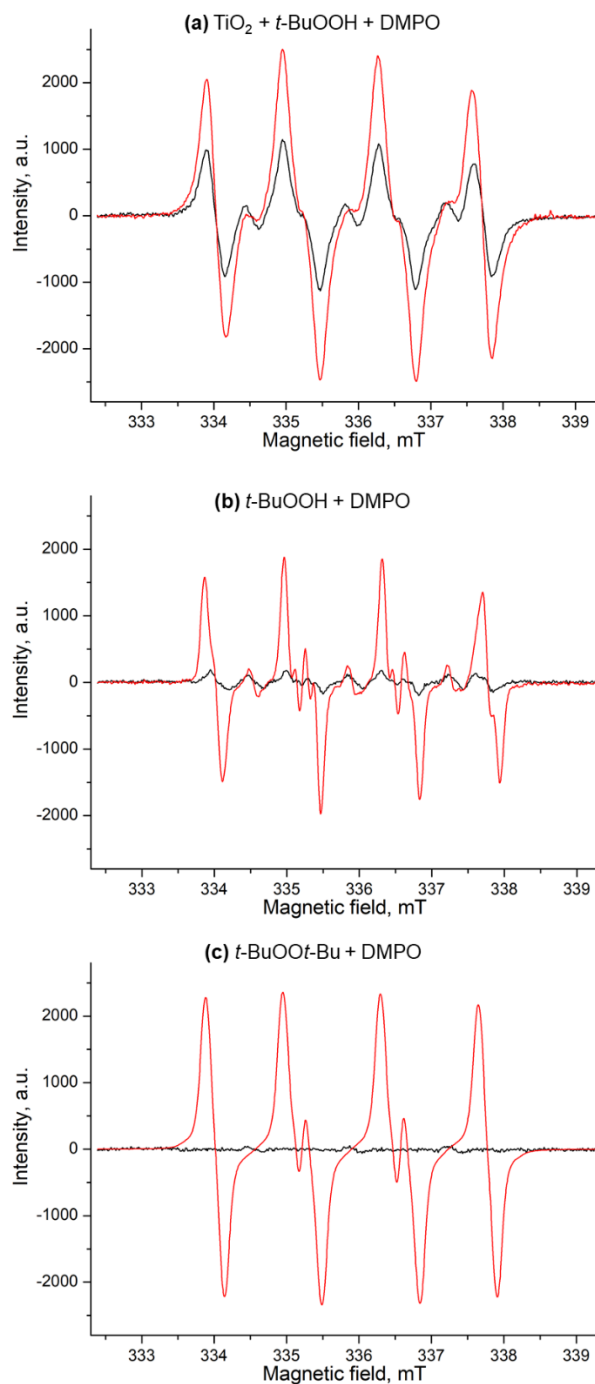
the role of blue light in the generation of free radicals from *t*-BuOOH. No signal was observed for TiO<sub>2</sub>-DMPO mixture (Figure S1, b) both without or with blue LED irradiation and only trace signals were observed for *t*-BuOOH-DMPO mixture (Figure S1, c). These results indicate that signals observed in Figure S1 (a) truly correspond to free-radical species formed upon interaction of *t*-BuOOH with TiO<sub>2</sub>.



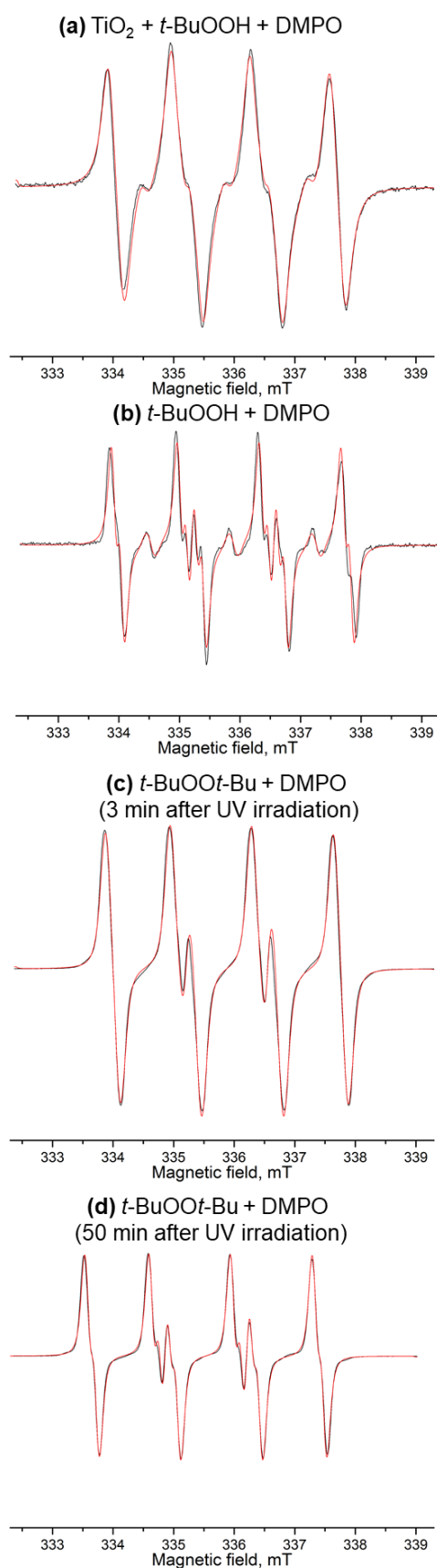
**Figure S1.** EPR spectra of spin trapping experiments under air. Black lines – EPR spectra before irradiation. Red lines – EPR spectra under Blue LED irradiation. Blue line in experiment (a) – spectrum of the sample prepared under dark conditions.

In order to propose the structure of observed DMPO spin adduct, we performed experiment analogous to the experiment of Figure 1, a, but under argon atmosphere in order to increase the resolution (Figure S2, a). Control experiments were conducted with the generation of O-centered

radical from *t*-BuOOH (Figure S2, b) and *t*-BuOO*t*-Bu (Figure S2, c) upon UV irradiation. Observed signals were simulated using EasySpin 5.2.35 program [1], results are presented in Figure S3 and Table S1.



**Figure S2.** EPR spectra of spin trapping experiments under argon atmosphere. Black lines – EPR spectra before irradiation. Red line in (a) – EPR spectrum under Blue LED irradiation. Red lines in experiments (b) and (c) – EPR spectra after UV (365 nm) irradiation. The intensity of signal upon UV irradiation in experiment (c) was significantly higher than in other experiments, it was reduced by a factor of 25 for this figure.

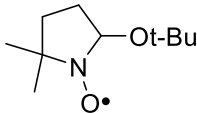


**Figure S3.** Experimental (black) and simulated (red) EPR spectra for mixtures in MeCN:  $\text{TiO}_2 + t\text{-BuOOH} + \text{DMPO}$  under blue led irradiation (a),  $t\text{-BuOOH} + \text{DMPO}$  after 365 nm UV irradiation (b), and  $t\text{-BuOO}t\text{-Bu} + \text{DMPO}$  after 365 nm UV irradiation (c) and (d)

For *t*-BuOO*t*-Bu + DMPO mixture radical concentration was significantly higher than in other cases judging one EPR intensity (ca. 25 times higher signal), which apparently resulted in line broadening (Figure S3, c). Resolution increased after partial radical decomposition over 50 minutes (Figure S3, d). In the case of TiO<sub>2</sub> + *t*-BuOOH + DMPO mixture, line broadening maybe associated with TiO<sub>2</sub> presence (heterogeneous character of the mixture). In all cases, main signals (Figure S3, a–d) are simulated quite well employing very close parameters:  $g = 2.0059$ – $2.0060$ ,  $a_N = 1.31$ – $1.36$  mT,  $a_H^\beta = 1.04$ – $1.09$  mT,  $a_H^\gamma = 0.12$ – $0.13$  mT. This signal can be assigned to the spin adduct of *t*-BuO• radical to DMPO.[2,3] However, recent studies showed that parameters of alkoxyl (for example, *t*-BuO•) and peroxy (for example, *t*-BuOO•) adducts to DMPO can be practically indistinguishable by EPR.[4] This assumption is in agreement with very similar EPR spectra observed for photolysis of *t*-BuOOH and *t*-BuOO*t*-Bu (Figure S3, b-d) in our case despite the fact that *t*-BuOO*t*-Bu can produce only *t*-BuO• radicals, whereas *t*-BuOOH can produce both *t*-BuO• and *t*-BuOO• radicals. According to simulation data (Table S1), seemingly different signals differ almost only by linewidths. Thus, unambiguous assignment of the observed signals to *t*-BuO• or *t*-BuOO• adducts to DMPO can not be made. It should also be noted that additional small signal ( $a_N = 1.37$  mT) was observed in TiO<sub>2</sub> + *t*-BuOOH + DMPO and *t*-BuOOH + DMPO systems.



**Table S1.** Simulation parameters for spectra presented in Figure S3 in comparison with literature data

Sample	g	$a_N$ , mT	$a_H^\beta$ , mT	$a_H^\gamma$ , mT	Voigtian broadenin g, mT <sup>a</sup>	Weight
TiO <sub>2</sub> + <i>t</i> -BuOOH + DMPO, blue LED	2.00595	1.3143	1.0367	0.127	0.06, 0.16	0.99
	2.00585	1.37	-	-	0.17, 0	0.01
<i>t</i> -BuOOH + DMPO, 365 nm LED	2.00593	1.356	1.088	0.122	0.017, 0.086	0.97
	2.00585	1.37	-	-	0.15, 0	0.03
<i>t</i> -BuOO <i>t</i> -Bu + DMPO, 365 nm LED, 3 min after irradiation	2.00598	1.352	1.066	0.119	0.115, 0.08	1
<i>t</i> -BuOO <i>t</i> -Bu + DMPO, 365 nm LED, 50 min after irradiation	2.00585	1.351	1.061	0.13	0.08, 0.061	1
Literature values for DMPO <i>t</i> -BuO• radical adduct in MeCN:						
						
Ref. [2]	-	1.33	0.77	0.09	-	-
Ref. [3]	-	1.35	1.075	0.127	-	-

<sup>a</sup> First value corresponds to Gaussian linewidth, the second corresponds to Lorentzian linewidth, both values are peak-to-peak in first-derivative spectrum.

## References

1. Stoll, S.; Schweiger, A. EasySpin, a Comprehensive Software Package for Spectral Simulation and Analysis in EPR. *Journal of Magnetic Resonance* **2006**, *178*, 42–55, doi:10.1016/j.jmr.2005.08.013.
2. Peng, F.; McLaughlin, M.; Liu, Y.; Mangion, I.; Tschaen, D.M.; Xu, Y. A Mild Cu(I)-Catalyzed Oxidative Aromatization of Indolines to Indoles. *J. Org. Chem.* **2016**, *81*, 10009–10015, doi:10.1021/acs.joc.6b01854.
3. Oyama, R.; Abe, M. Reactivity and Product Analysis of a Pair of Cumyloxyl and *Tert*-Butoxyl Radicals Generated in Photolysis of *Tert*-Butyl Cumyl Peroxide. *J. Org. Chem.* **2020**, *85*, 8627–8638, doi:10.1021/acs.joc.0c01016.

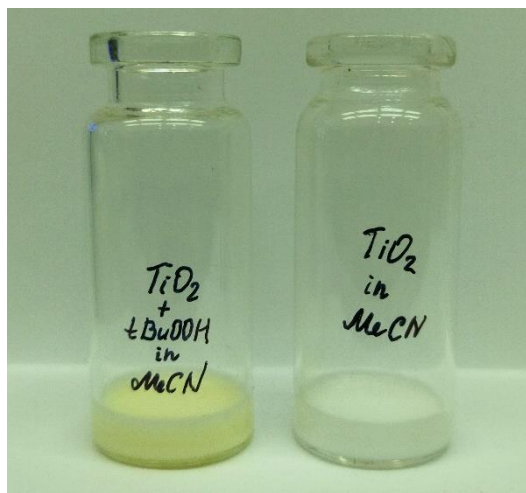
4. Dikalov, S.I.; Mason, R.P. Reassignment of Organic Peroxyl Radical Adducts. *Free Radical Biology and Medicine* **1999**, 27, 864–872, doi:10.1016/S0891-5849(99)00134-3.

### The appearance of the $\text{TiO}_2$ suspension with *t*-BuOOH

Contents of the left vial (Figure S4):  $\text{TiO}_2$  Hombikat UV100 (10 mg), MeCN (1 mL) and *t*-BuOOH (70%, 256 mg).

Contents of the right vial (Figure S4):  $\text{TiO}_2$  Hombikat UV100 (10 mg), MeCN (1 mL).

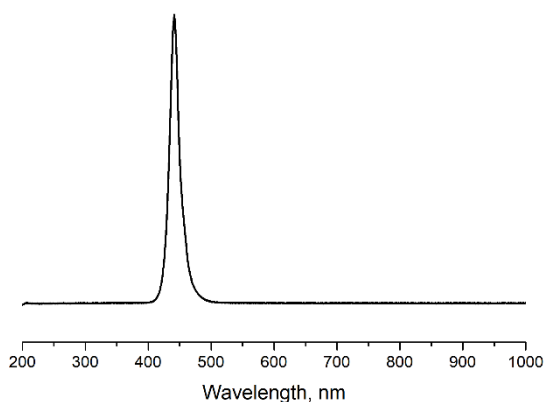
Both mixtures were sonicated for 2 minutes in an ultrasonic bath.



**Figure S4.** Appearance of  $\text{TiO}_2$  suspension in MeCN in the presence (left) and absence (right) of *t*-BuOOH

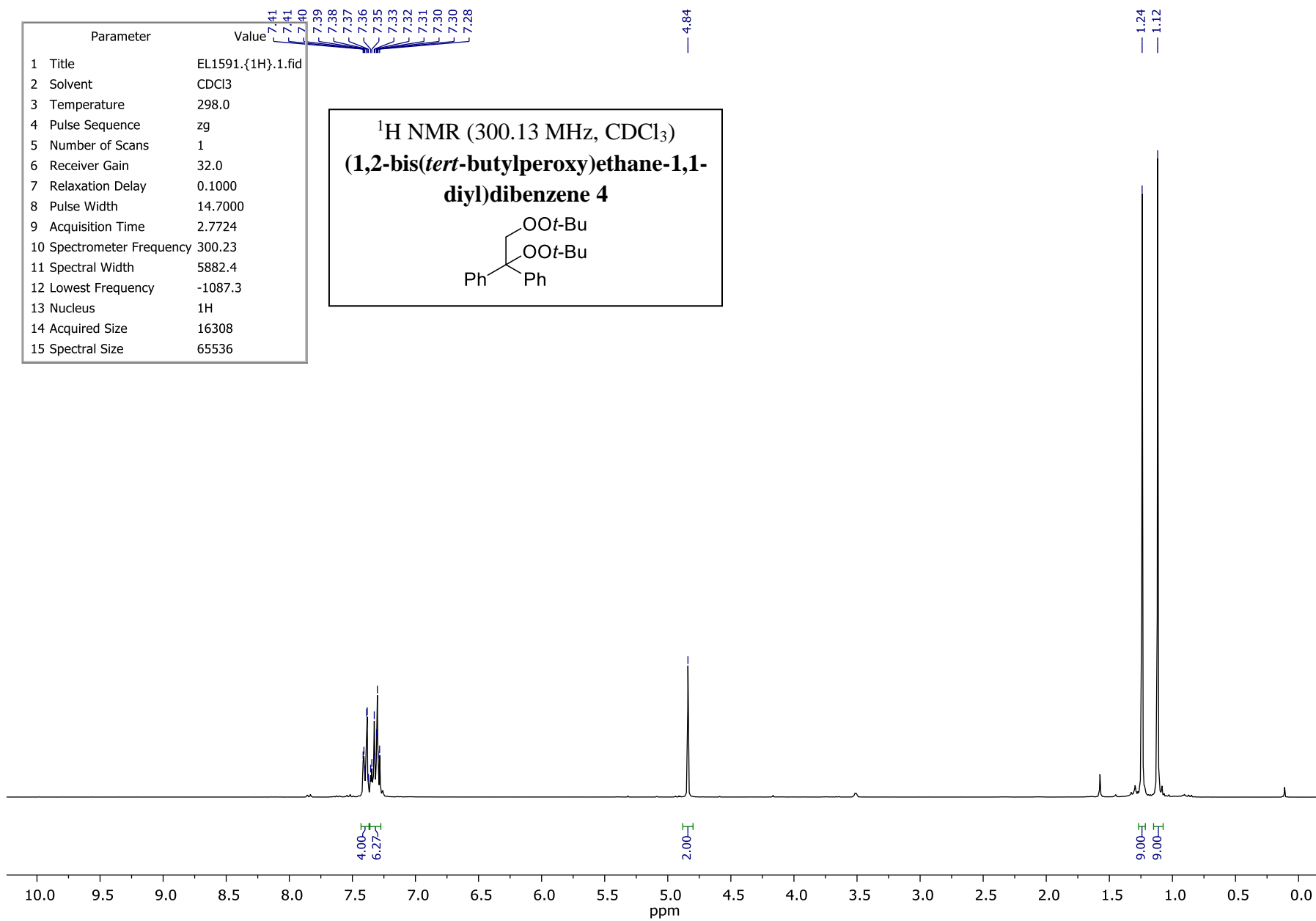
### UV-Vis spectrum of Blue LED used in the present study

The UV-Vis emission spectrum of the Blue LED (Figure S5) was recorded Ocean Optics FLAME-S-XR1 spectrometer.



**Figure S5.** UV-Vis spectrum of 10W Blue LED used for photochemical syntheses in the present study ( $\lambda_{\text{max}} = 443 \text{ nm}$ )

## **Spectral data of the synthesized compounds**



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1 Title	EL1591.{13C}.2.fid
2 Solvent	CDCl3
3 Temperature	298.0
4 Pulse Sequence	zgpg30
5 Number of Scans	512
6 Receiver Gain	101.0
7 Relaxation Delay	0.8000
8 Pulse Width	9.9000
9 Acquisition Time	1.1431
10 Spectrometer Frequency	75.50
11 Spectral Width	17857.1
12 Lowest Frequency	-1370.1
13 Nucleus	13C
14 Acquired Size	20412
15 Spectral Size	65536

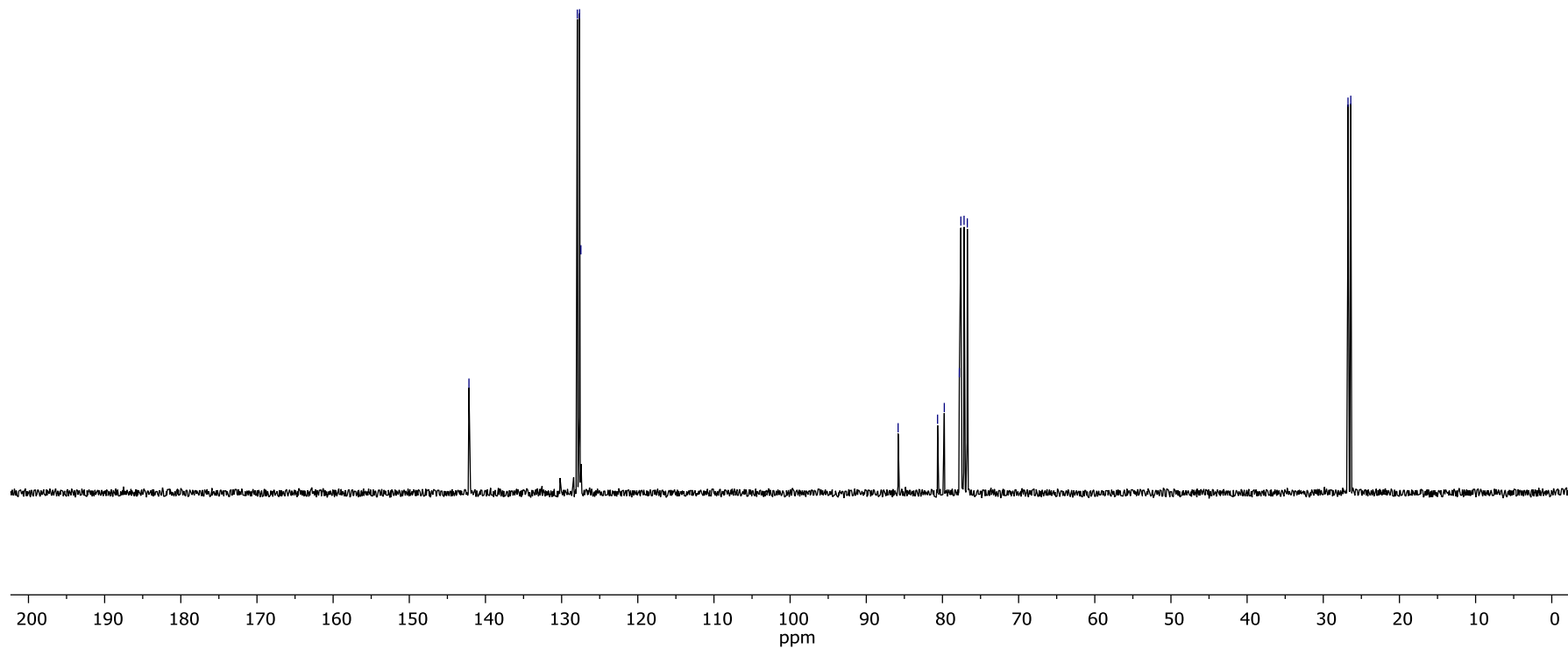
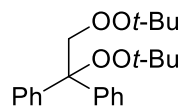
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127.65  
127.47

85.82  
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79.76  
77.78  
77.58  
77.16  
76.74

26.75  
26.39

<sup>13</sup>C NMR (75.48 MHz, CDCl<sub>3</sub>)  
(1,2-bis(tert-butylperoxy)ethane-  
1,1-diyl)dibenzene 4



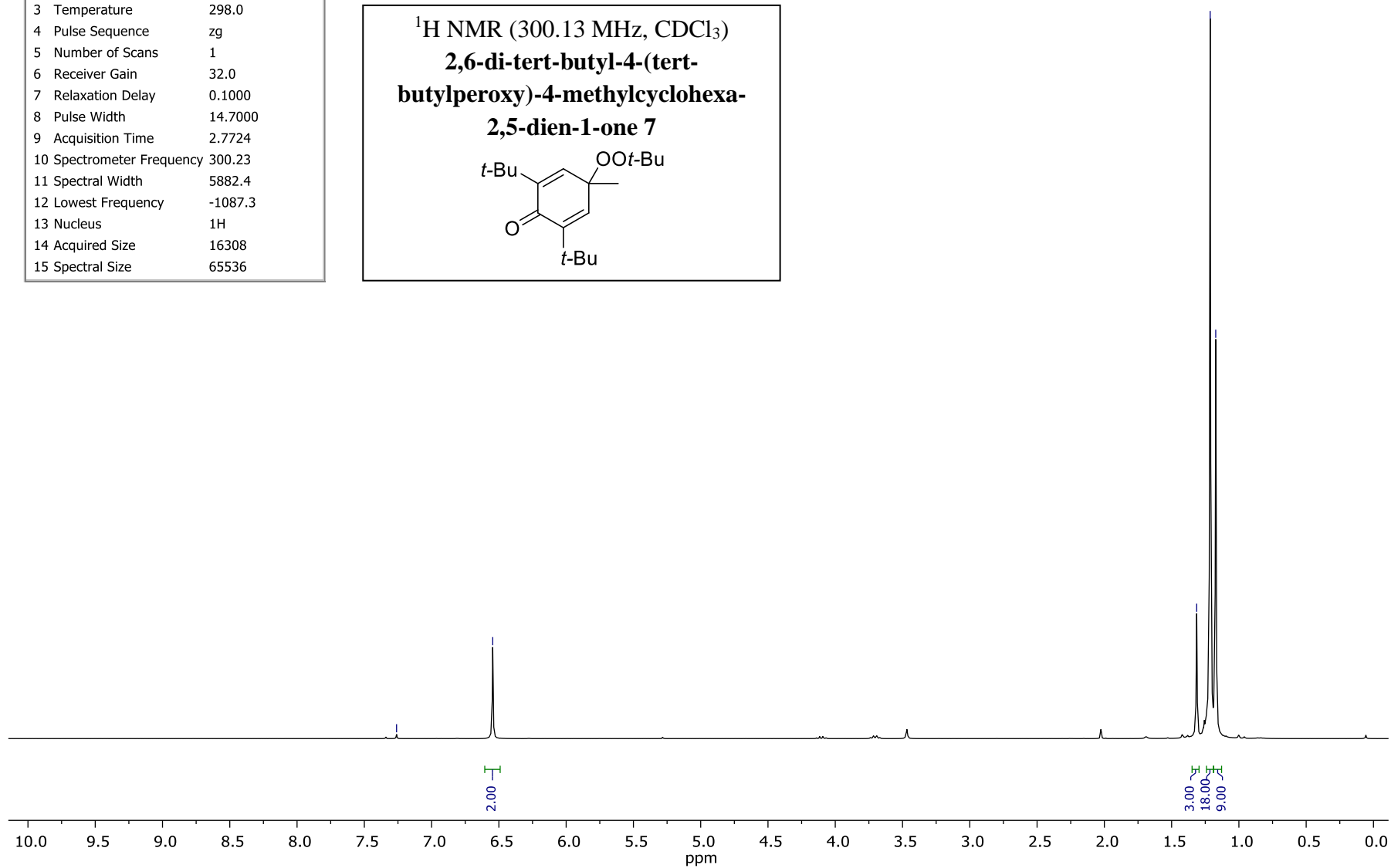
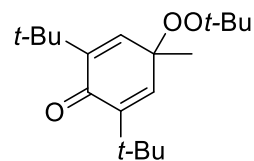
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6 Receiver Gain	32.0
7 Relaxation Delay	0.1000
8 Pulse Width	14.7000
9 Acquisition Time	2.7724
10 Spectrometer Frequency	300.23
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12 Lowest Frequency	-1087.3
13 Nucleus	<sup>1</sup> H
14 Acquired Size	16308
15 Spectral Size	65536

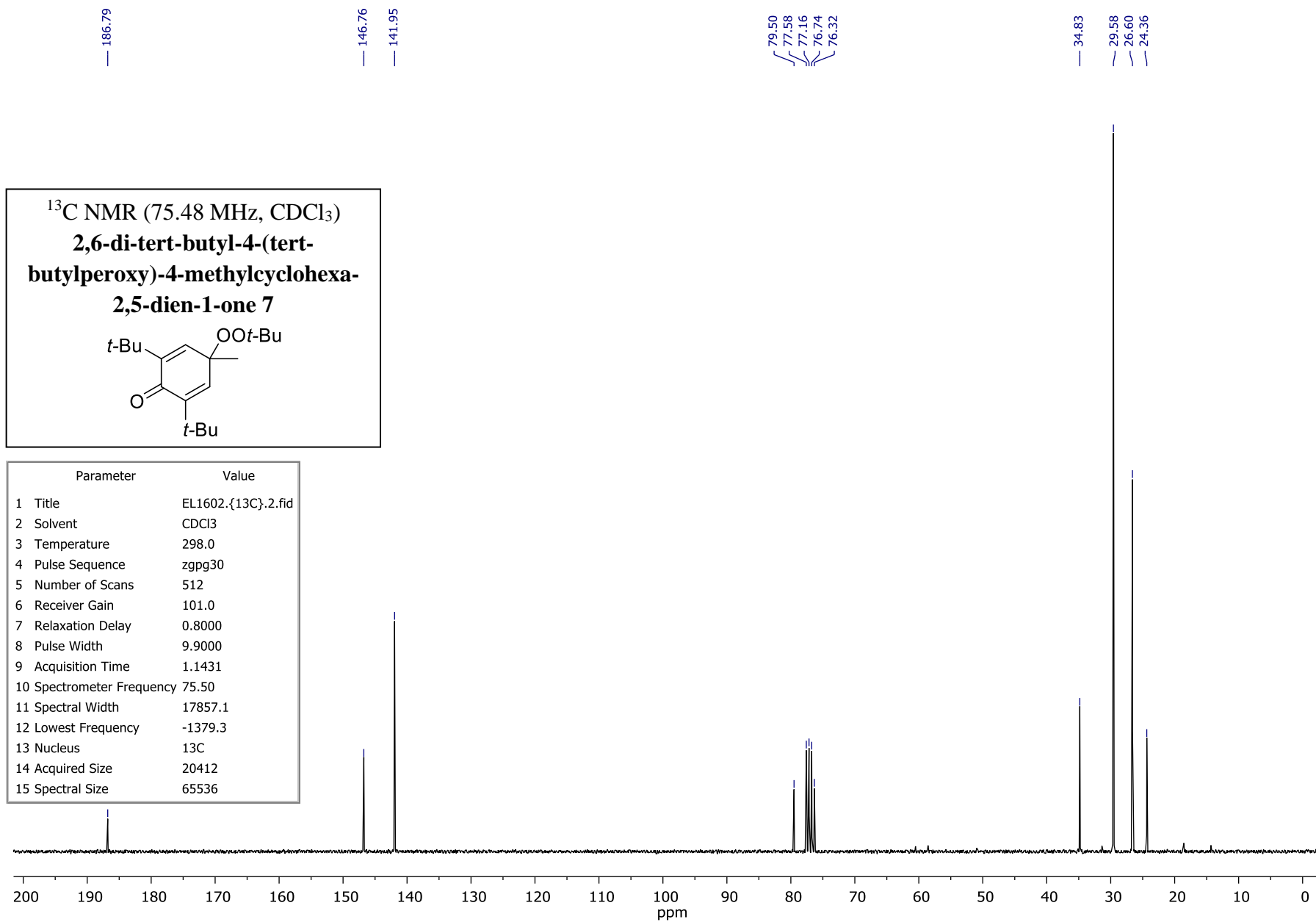
— 7.26

— 6.55

1.31  
1.21  
1.17

<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)  
**2,6-di-tert-butyl-4-(tert-butylperoxy)-4-methylcyclohexa-2,5-dien-1-one 7**





i8051.{1H}.1.fid  
 Avance-300, CDCl<sub>3</sub>

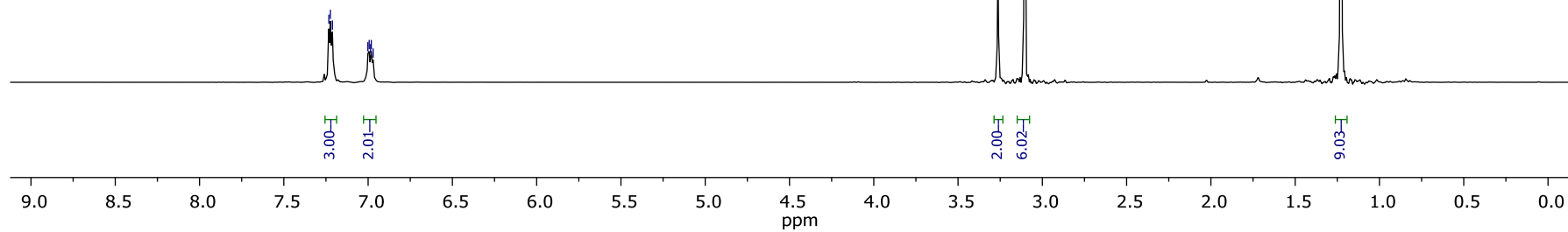
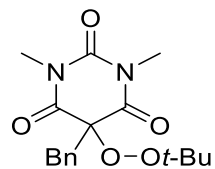
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 6.99  
 6.98  
 6.97

3.26  
 3.10

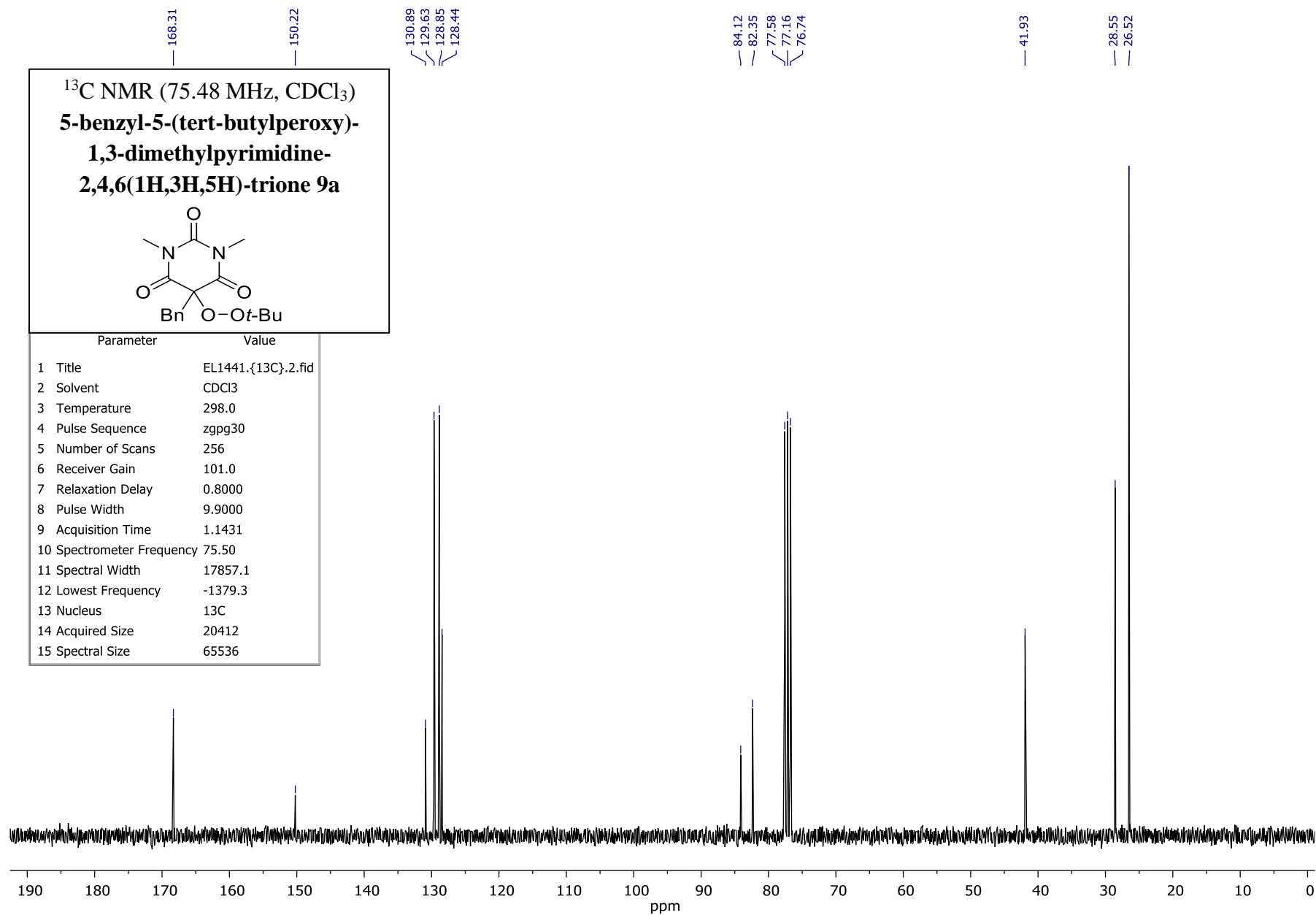
1.23

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7 Relaxation Delay	0.0050
8 Pulse Width	9.0000
9 Spectrometer Frequency	300.13
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11 Lowest Frequency	-610.2
12 Nucleus	<sup>1</sup> H
13 Acquired Size	8124
14 Spectral Size	65536

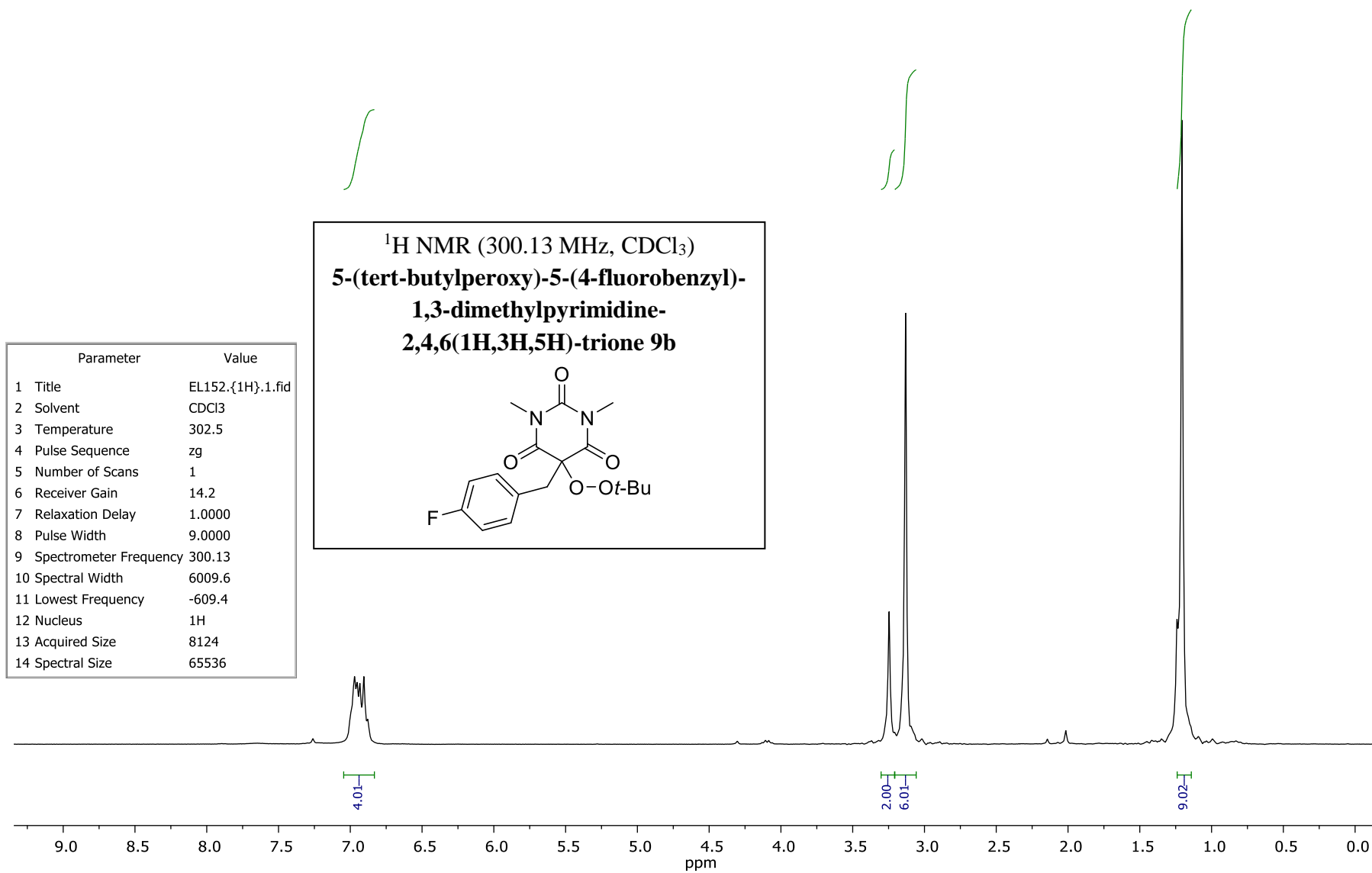
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 2,4,6(1H,3H,5H)-trione 9a**

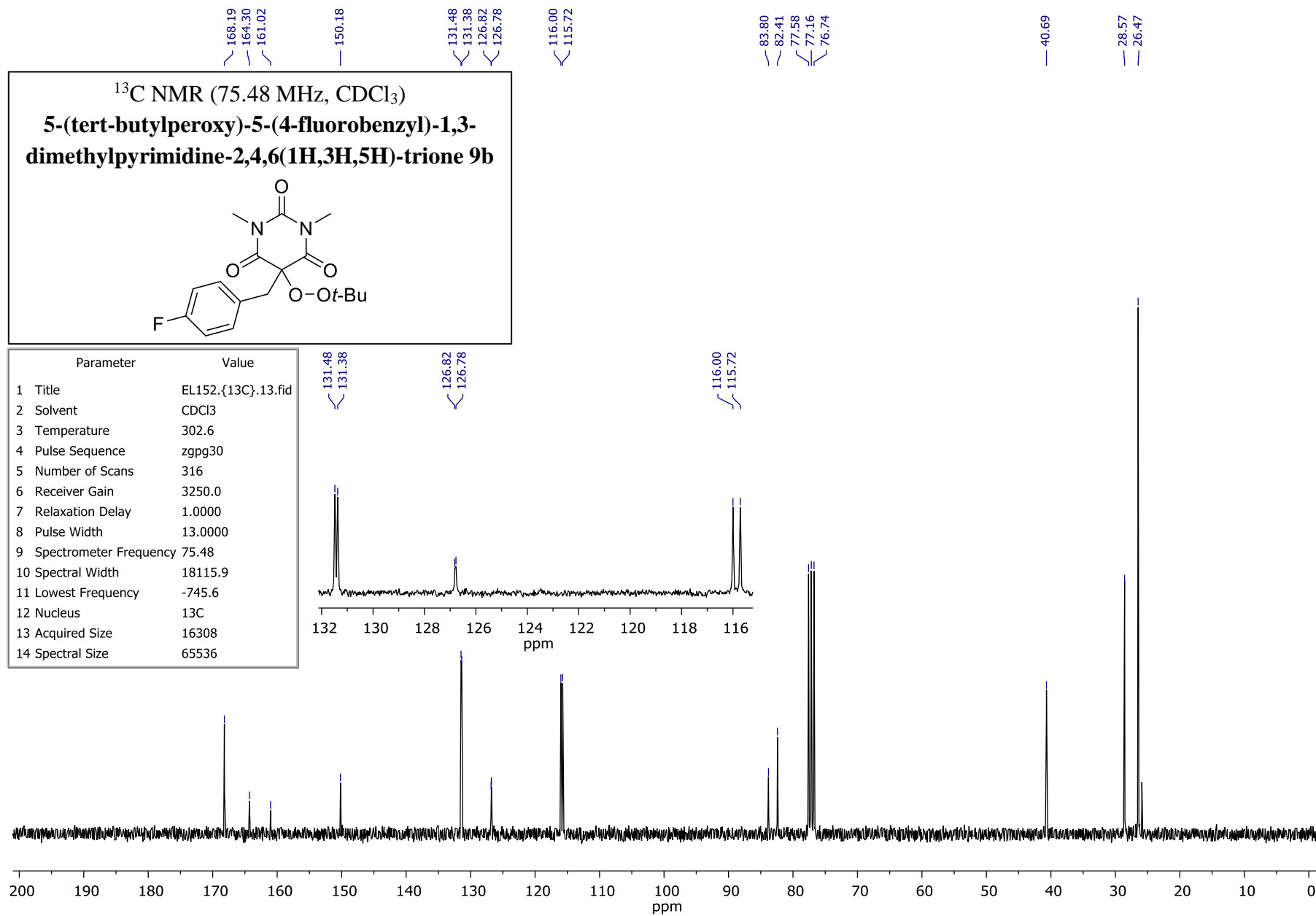


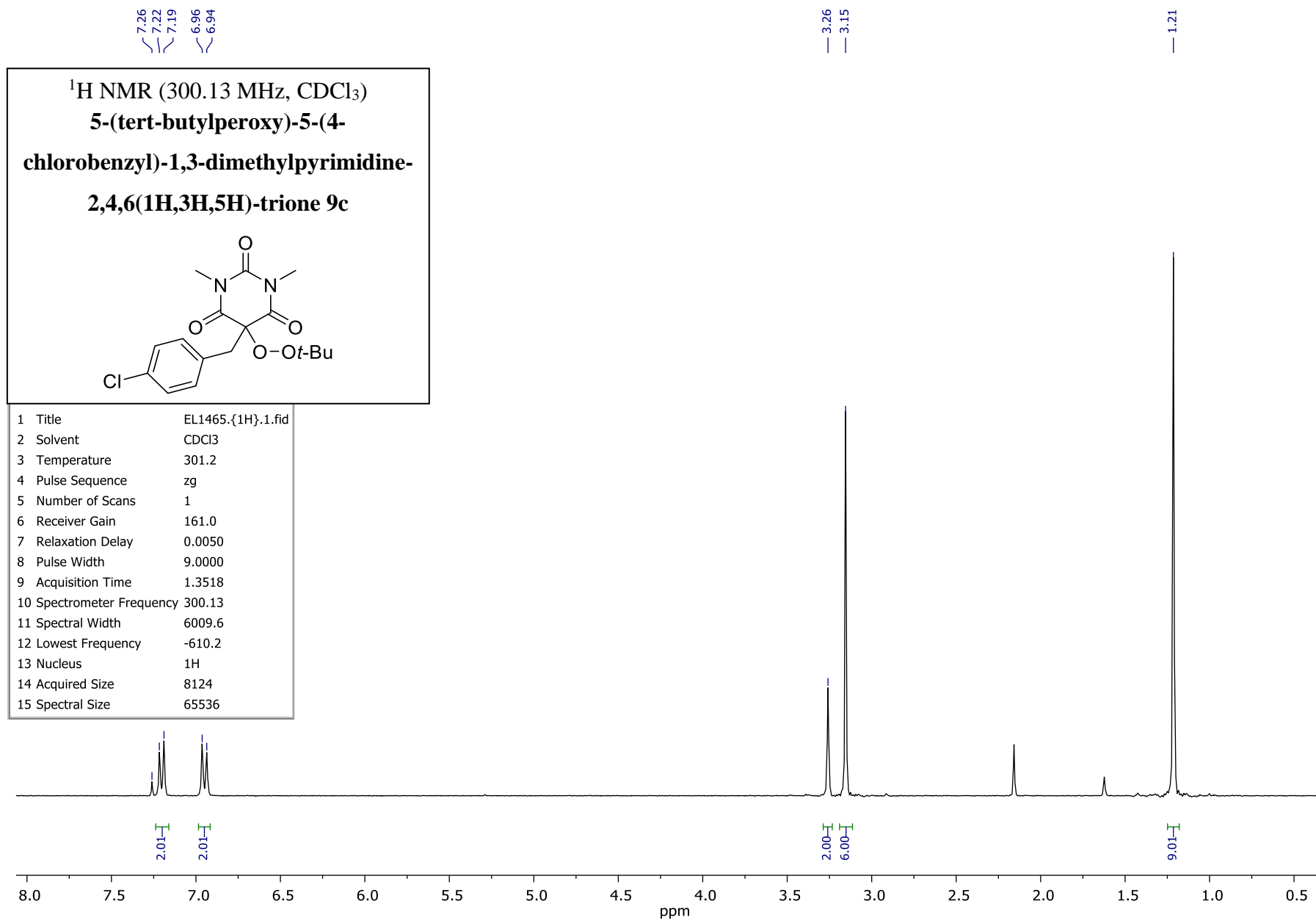


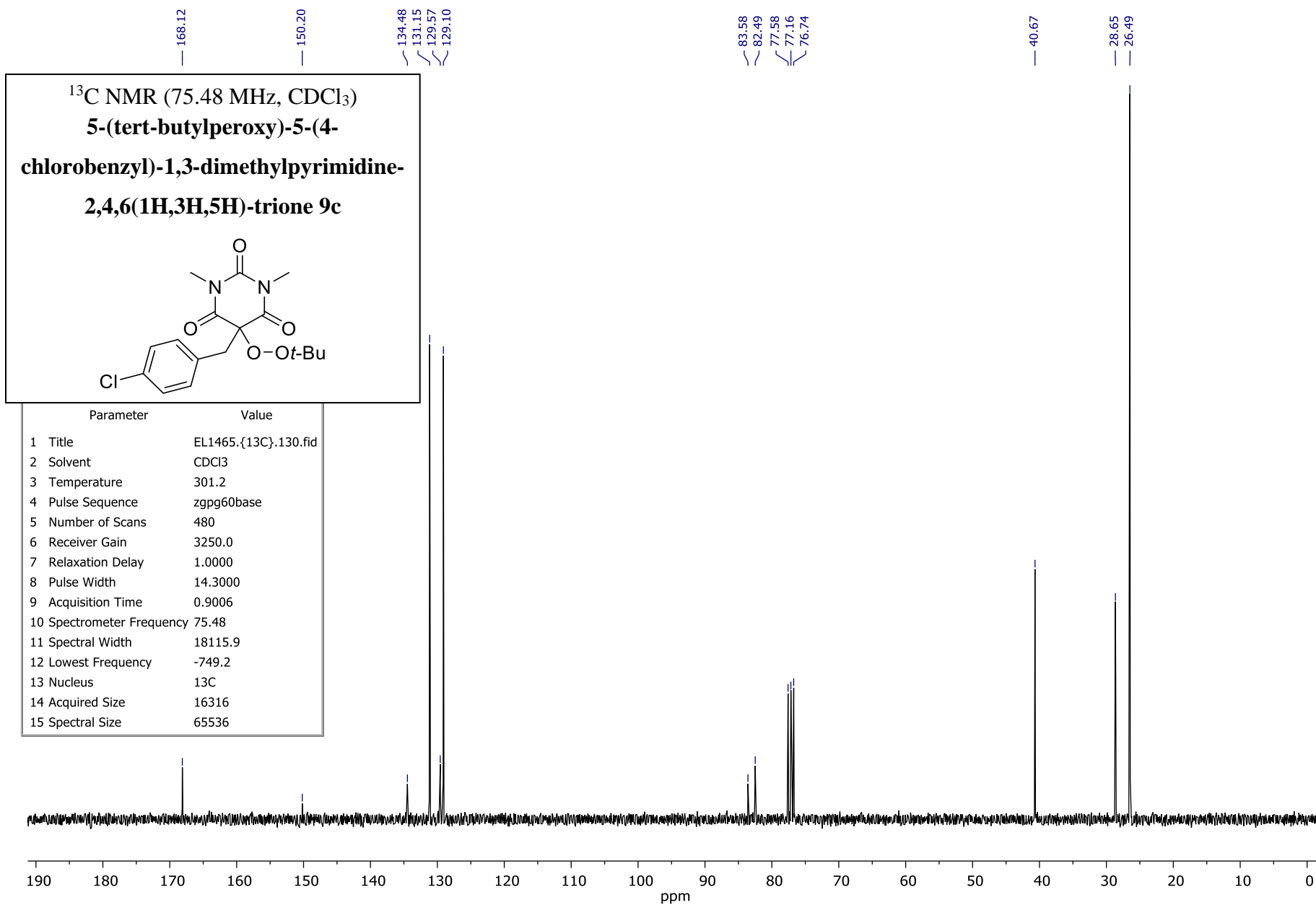


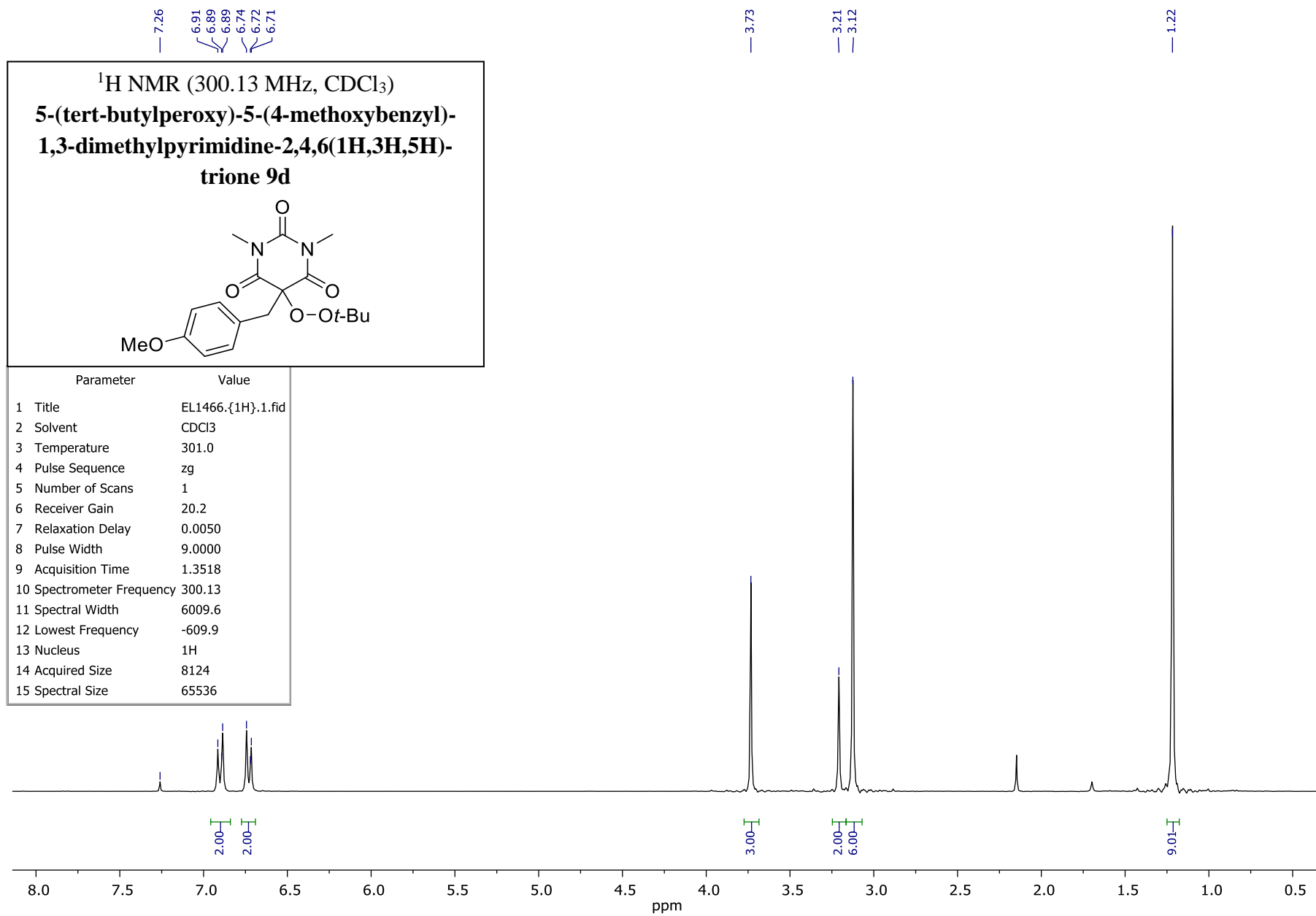
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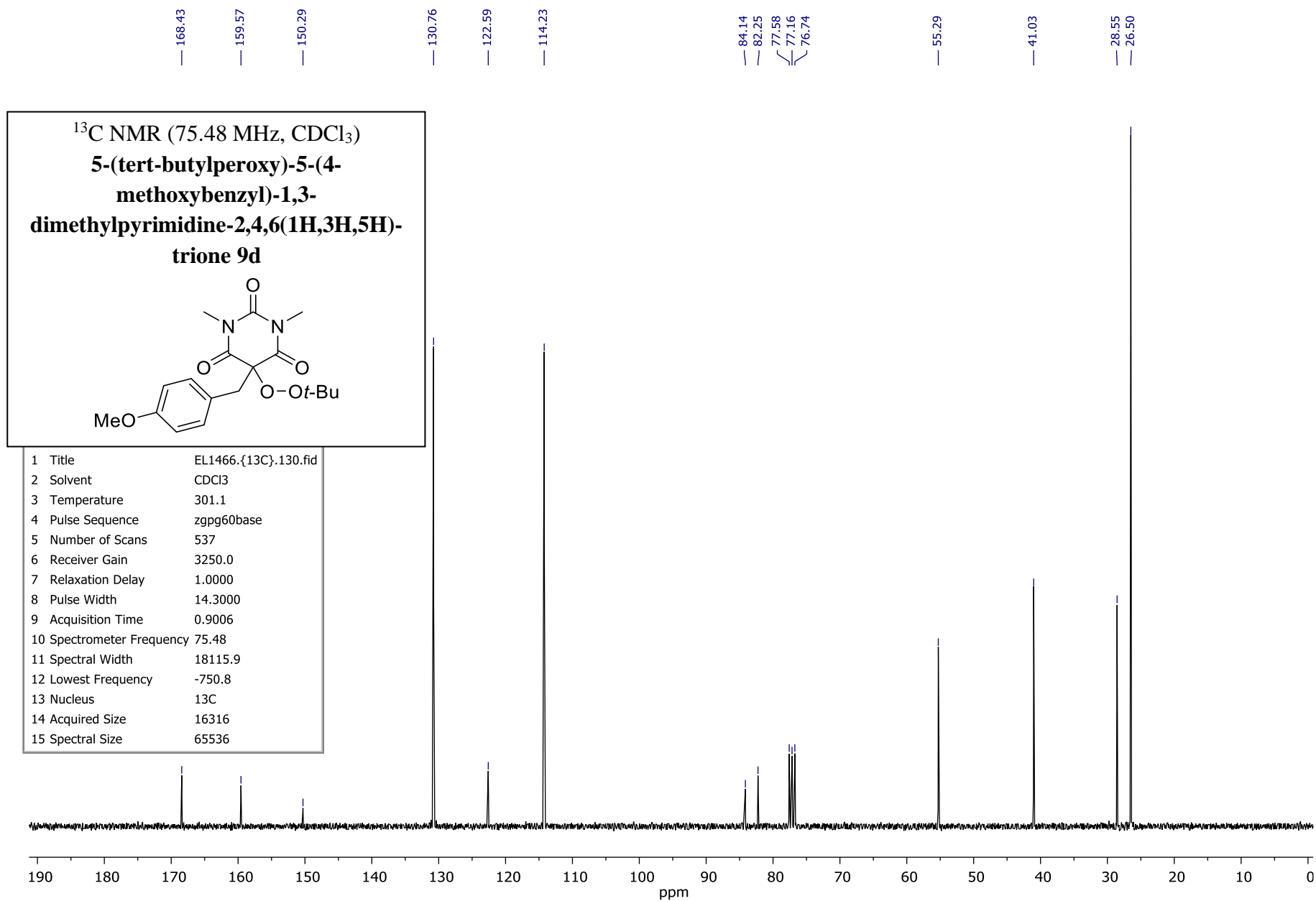


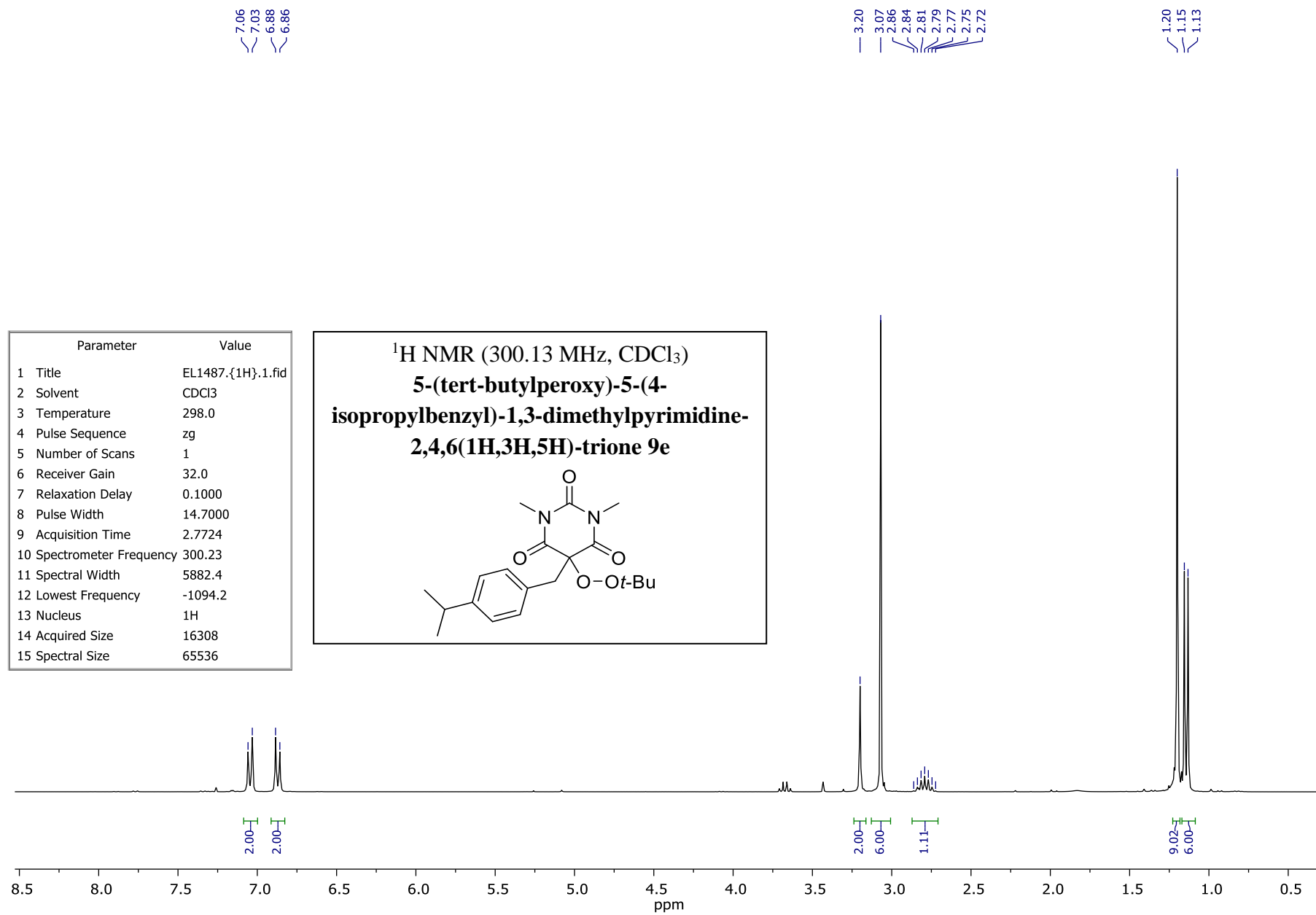




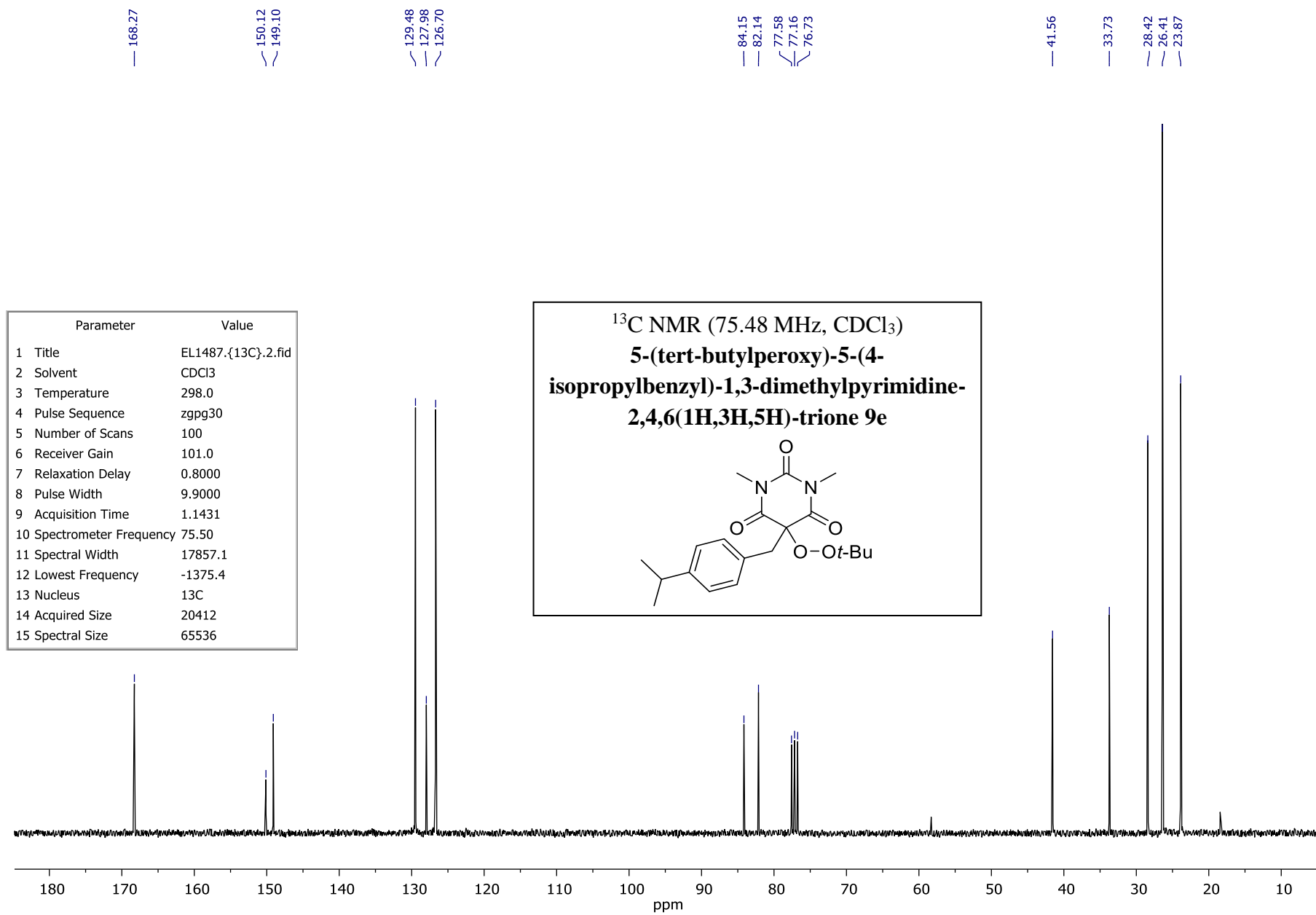


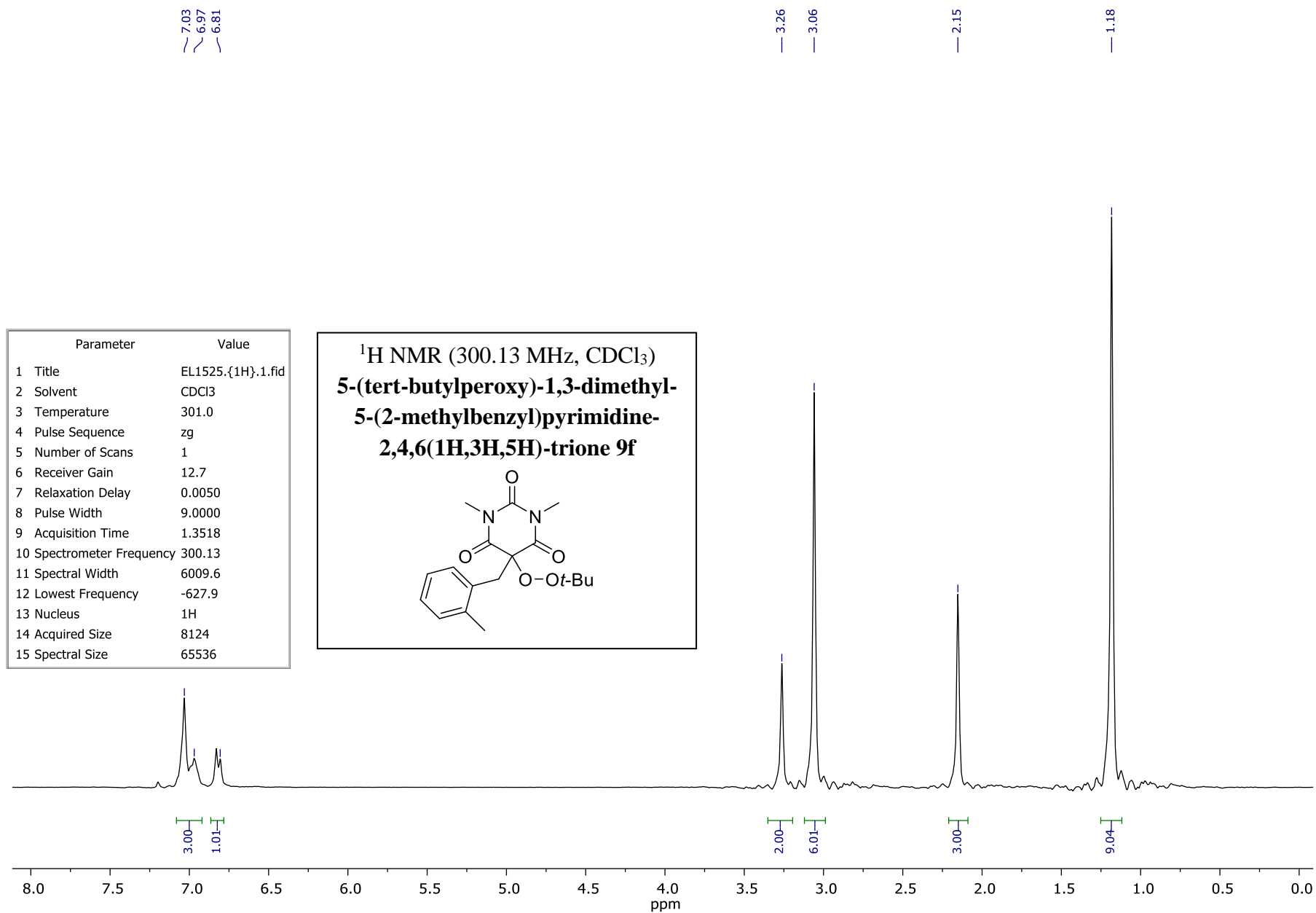


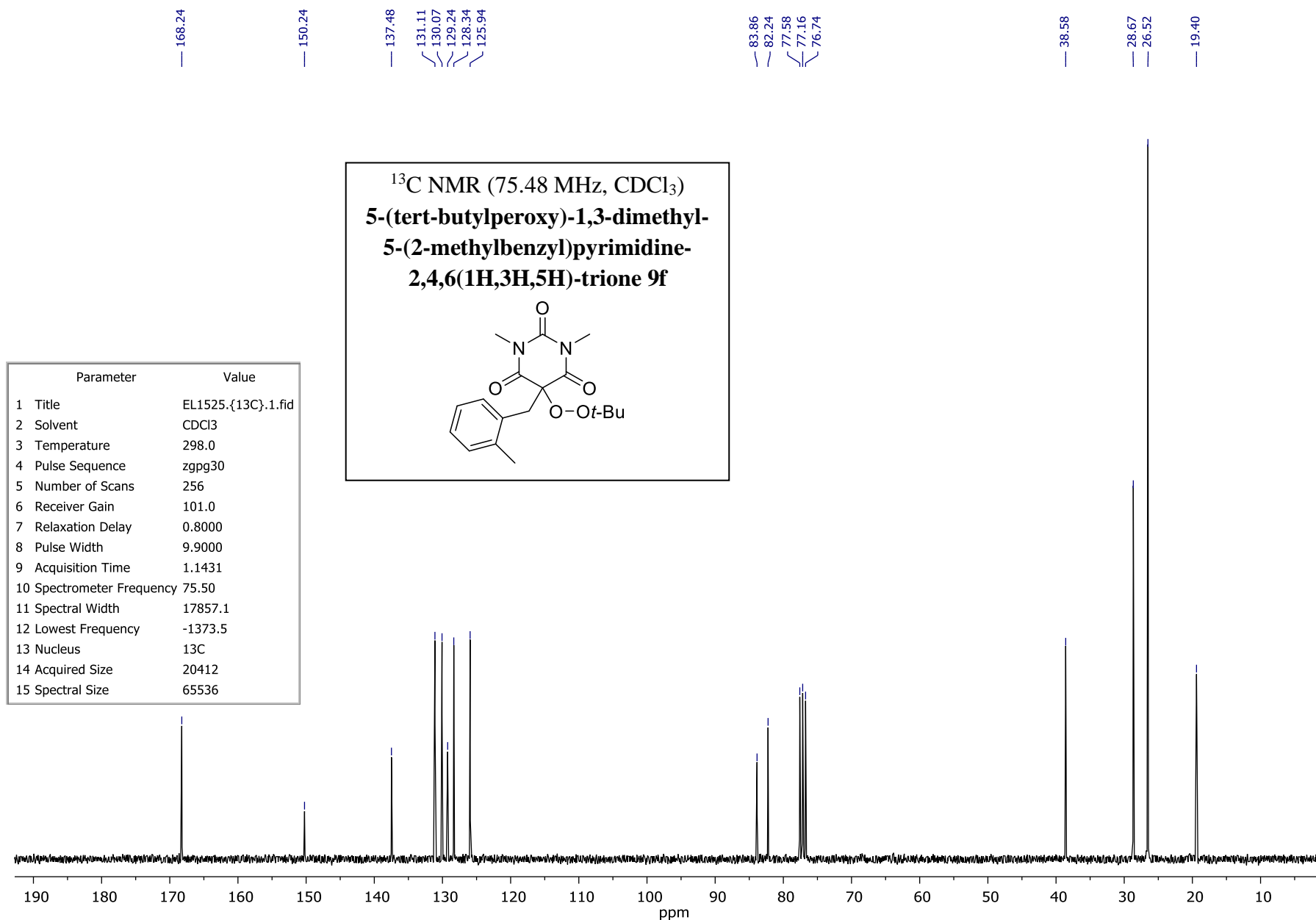












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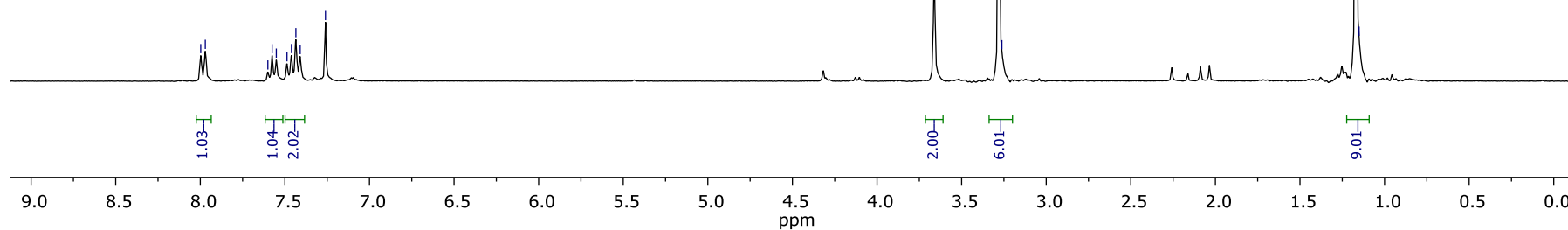
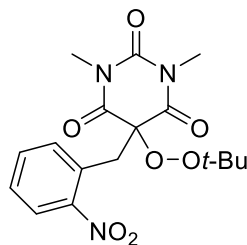
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3.66  
 3.28  
 3.26

1.17  
 1.15

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10 Spectral Width	6009.6
11 Lowest Frequency	-619.0
12 Nucleus	<sup>1</sup> H
13 Acquired Size	8124
14 Spectral Size	65536

<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)  
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EL043.{13C}.13.fid  
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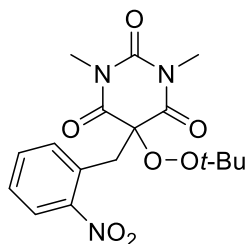
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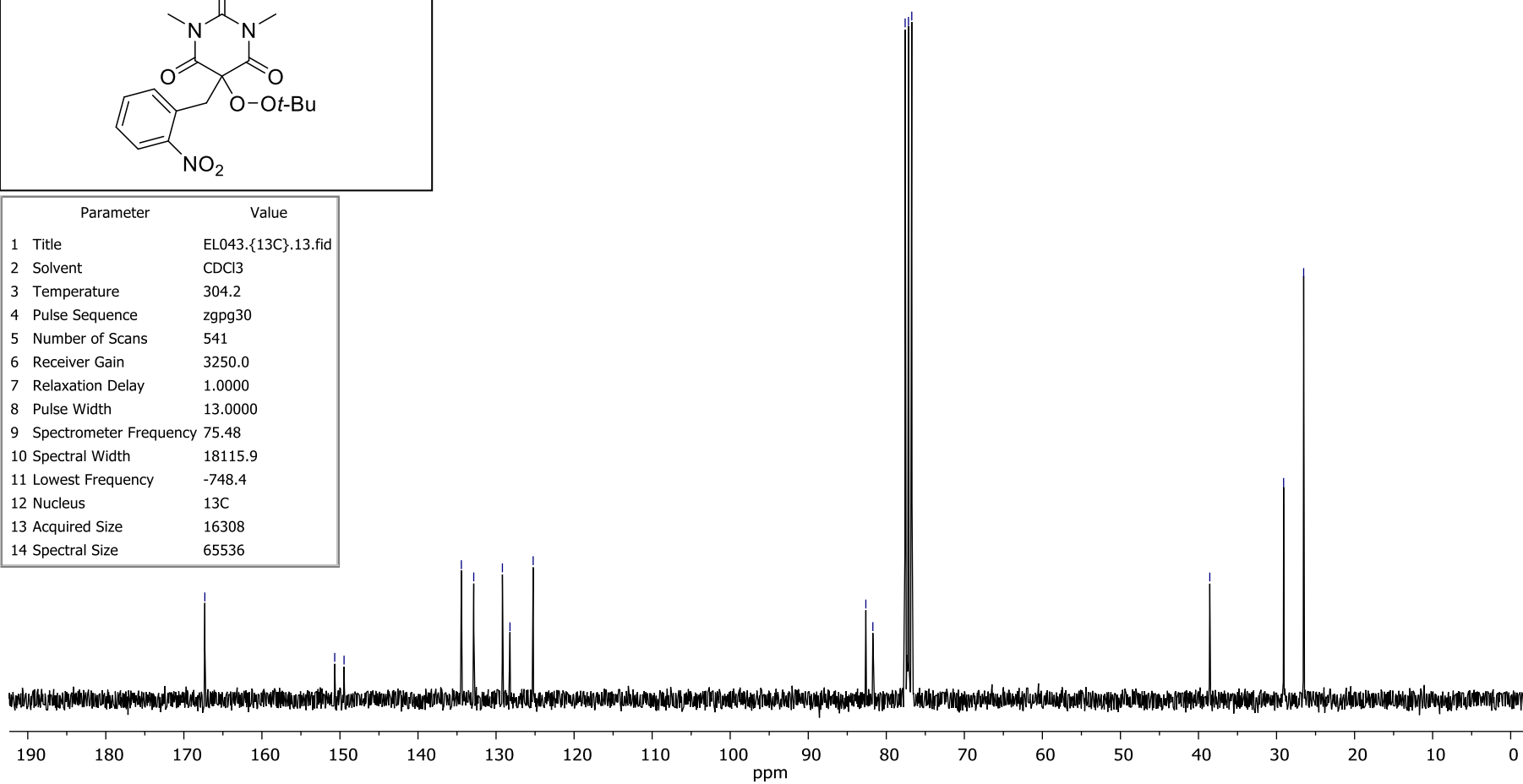
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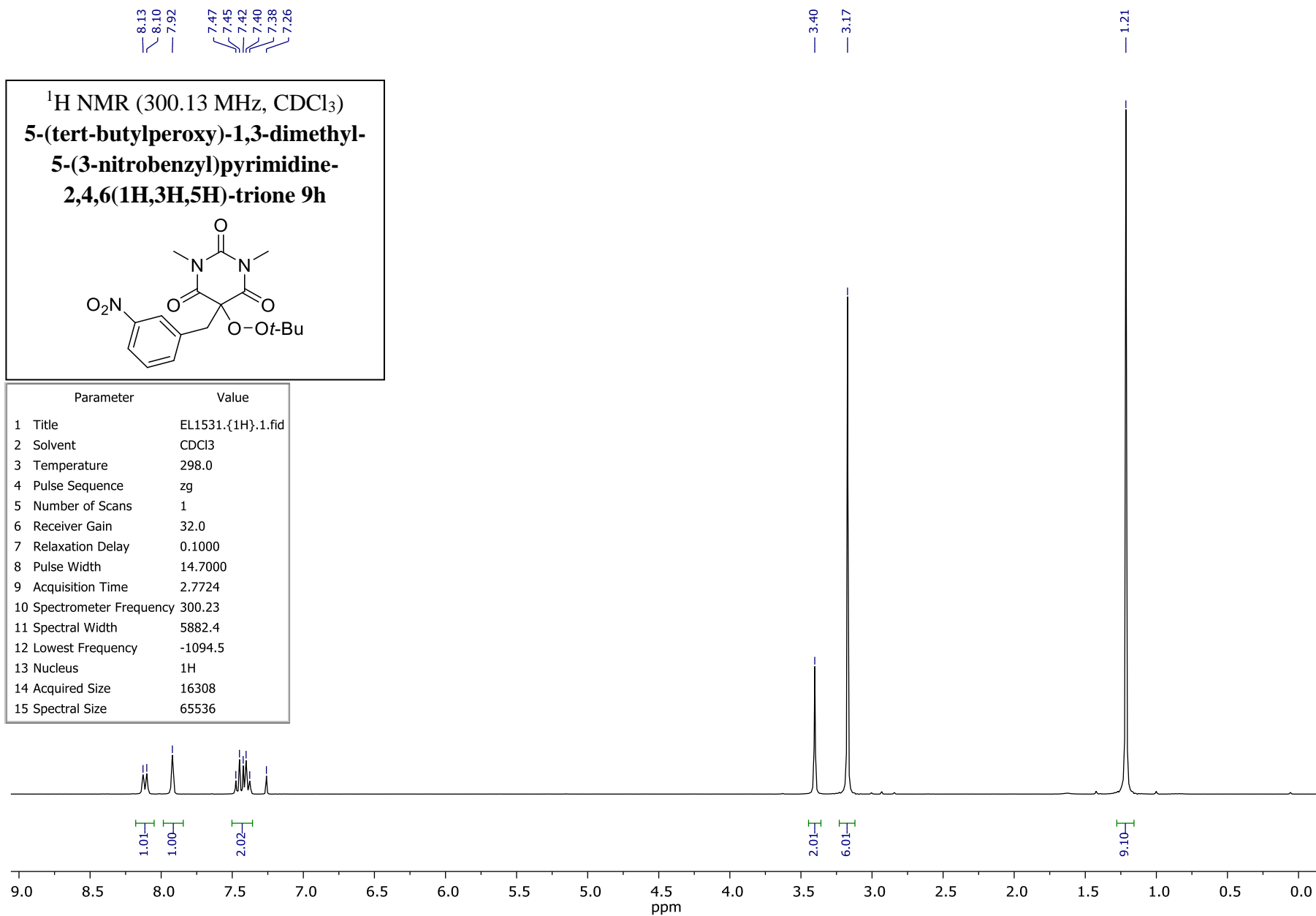
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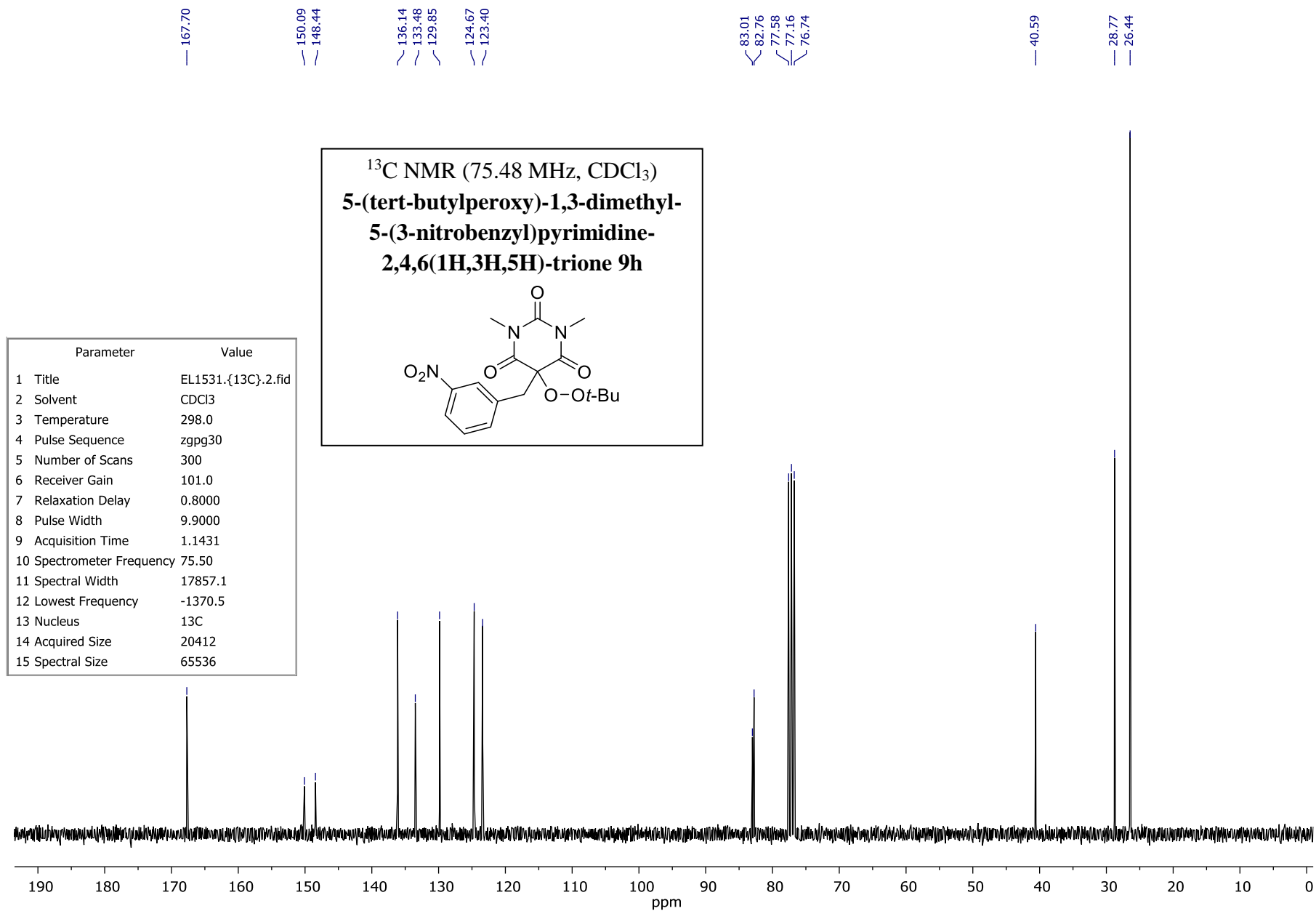
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5-(2-nitrobenzyl)pyrimidine-  
2,4,6(1H,3H,5H)-trione 9g**

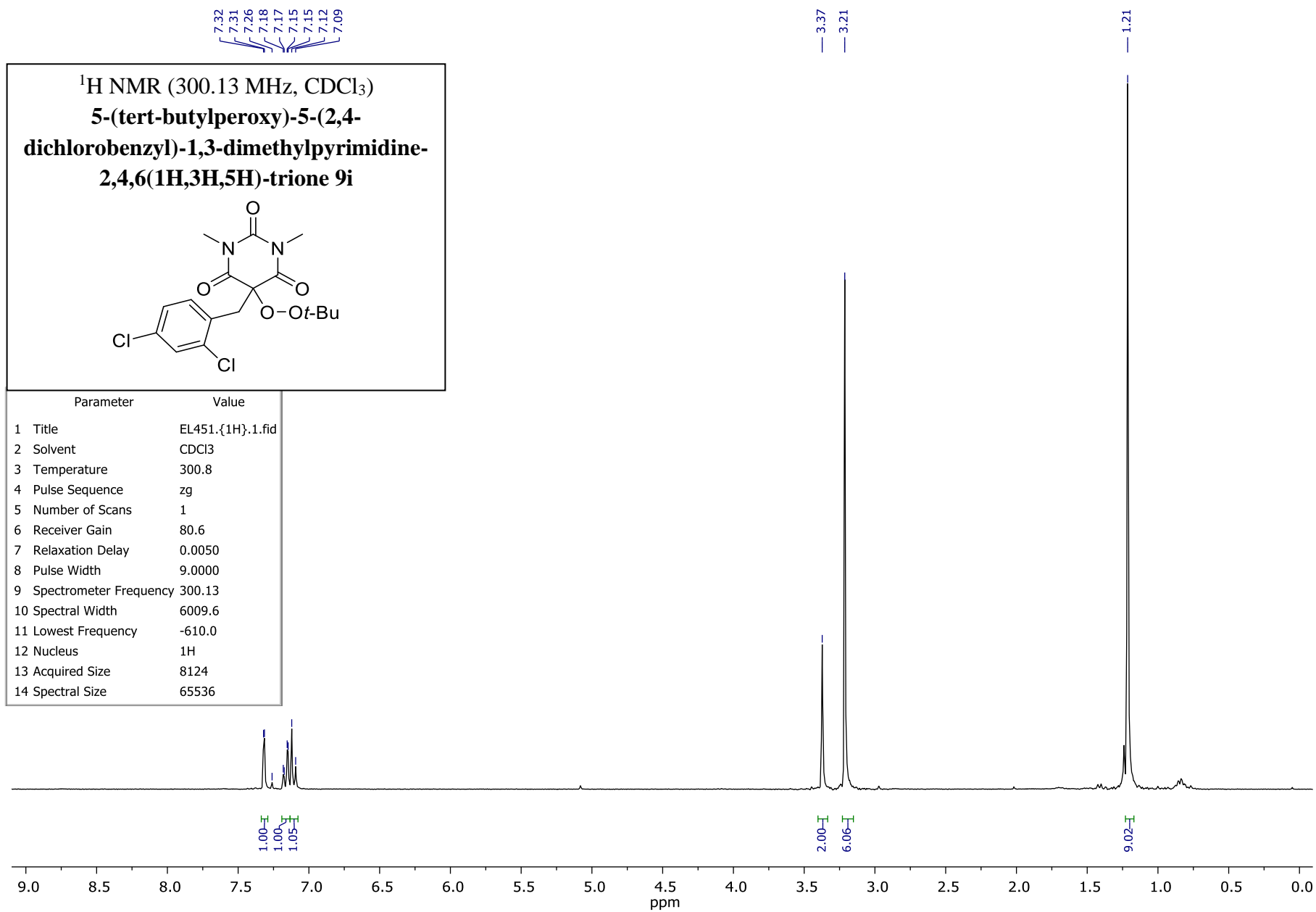


Parameter	Value
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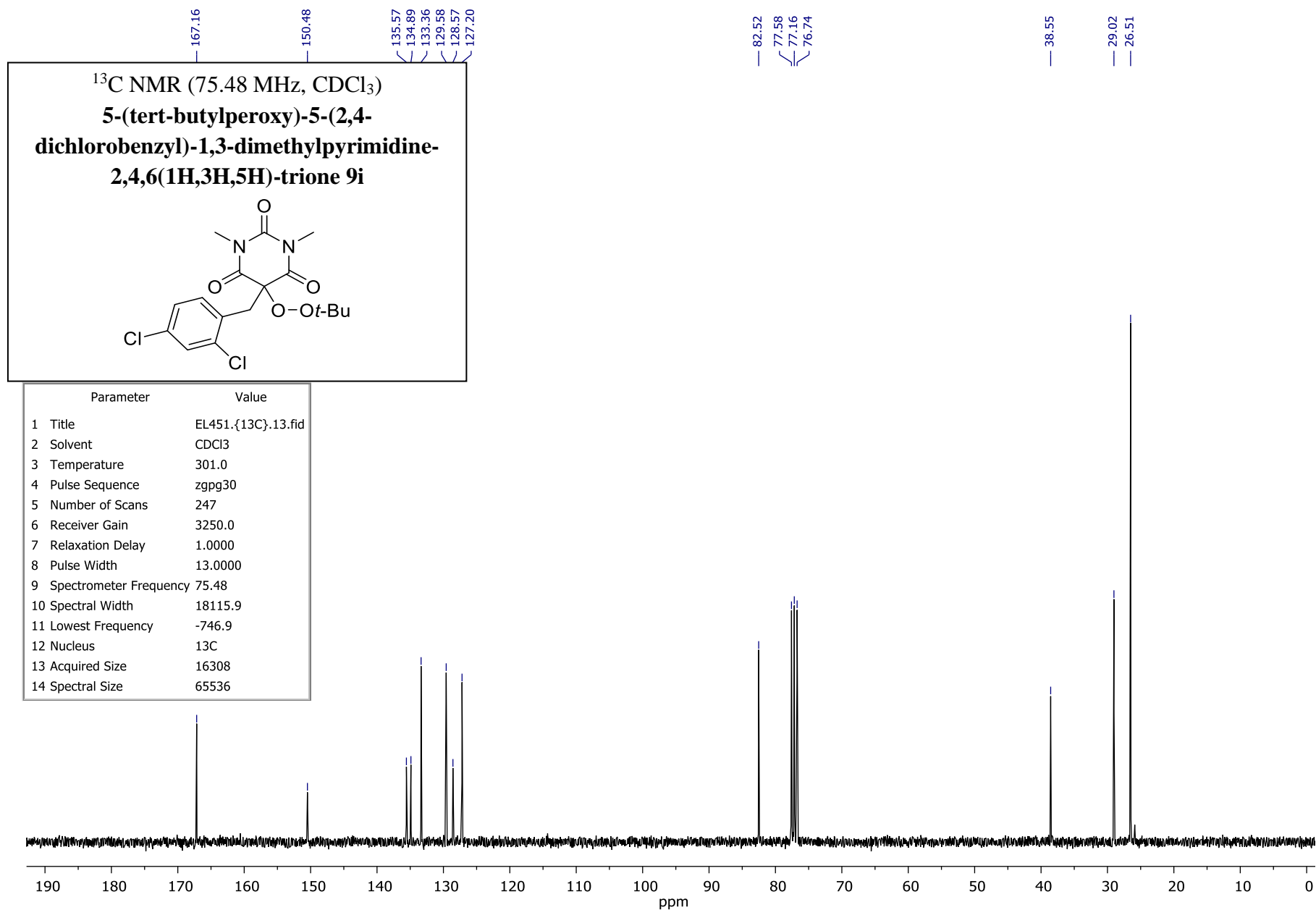






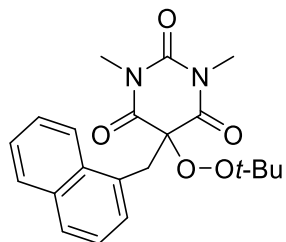




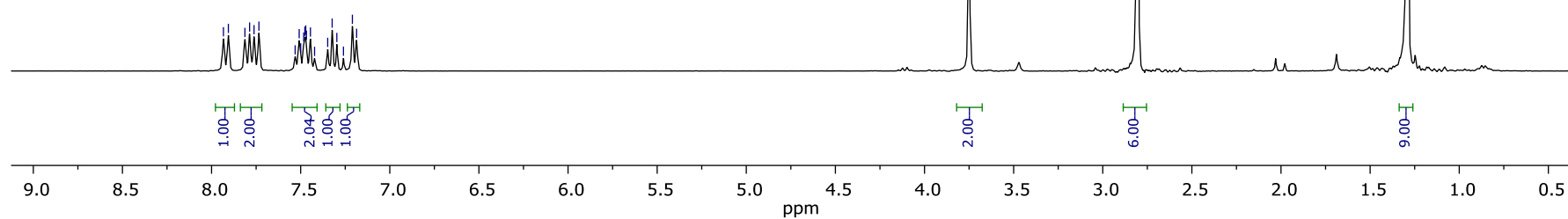


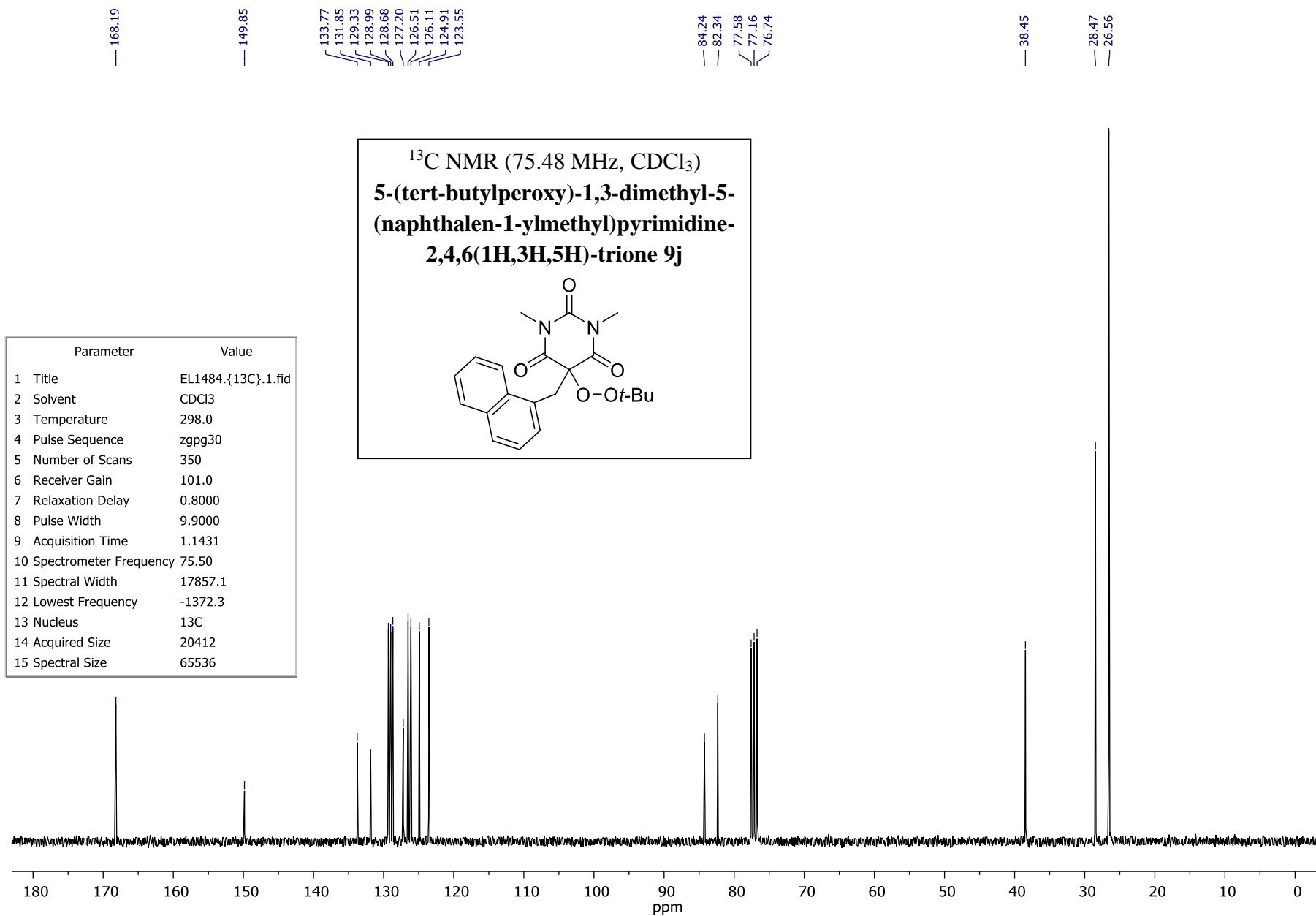
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7.35  
7.32  
7.30  
7.26  
7.21  
7.19

<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)  
**5-(tert-butylperoxy)-1,3-dimethyl-5-(naphthalen-1-ylmethyl)pyrimidine-2,4,6(1H,3H,5H)-trione 9j**

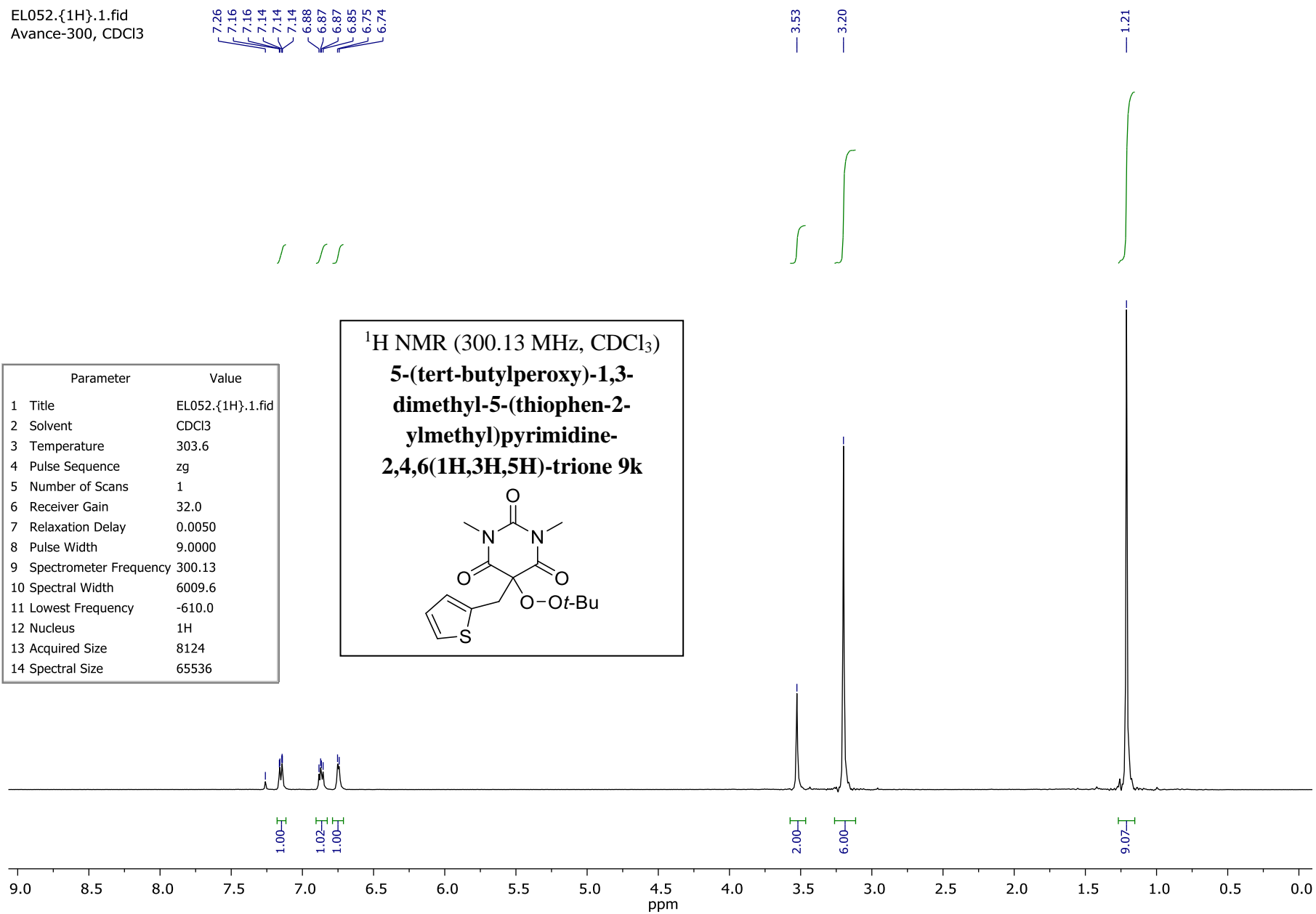


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9 Acquisition Time	1.3518
10 Spectrometer Frequency	300.13
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14 Acquired Size	8124
15 Spectral Size	65536





EL052.{1H}.1.fid  
Avance-300, CDCl<sub>3</sub>



EL052.{13C}.13.fid  
/LB58 IYT1588

— 168.19

— 150.48

— 132.04  
— 128.57  
— 127.37  
— 126.40

— 83.27  
— 82.51  
— 77.58  
— 77.16  
— 76.74

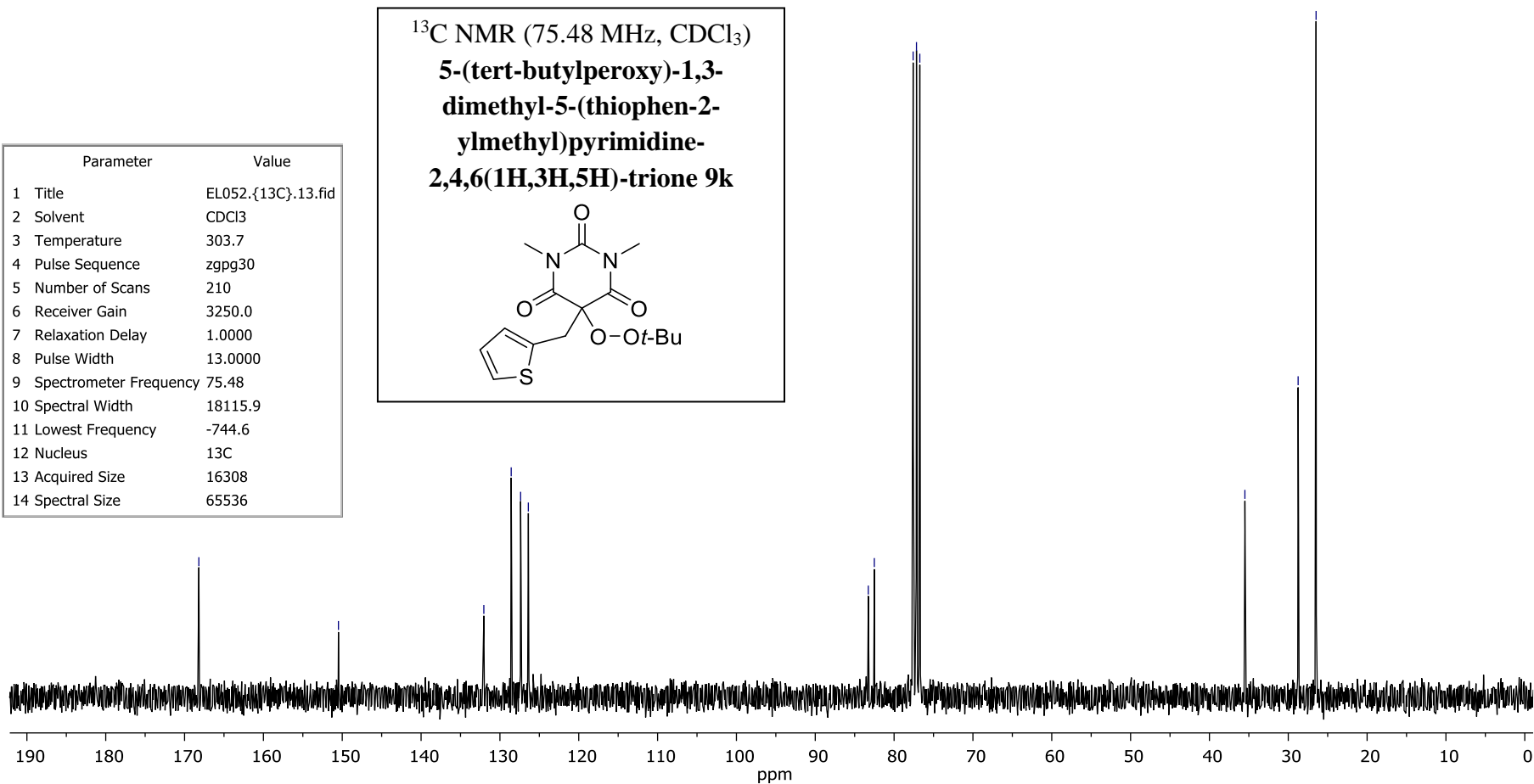
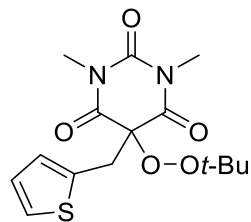
— 35.50

— 28.75  
— 26.47

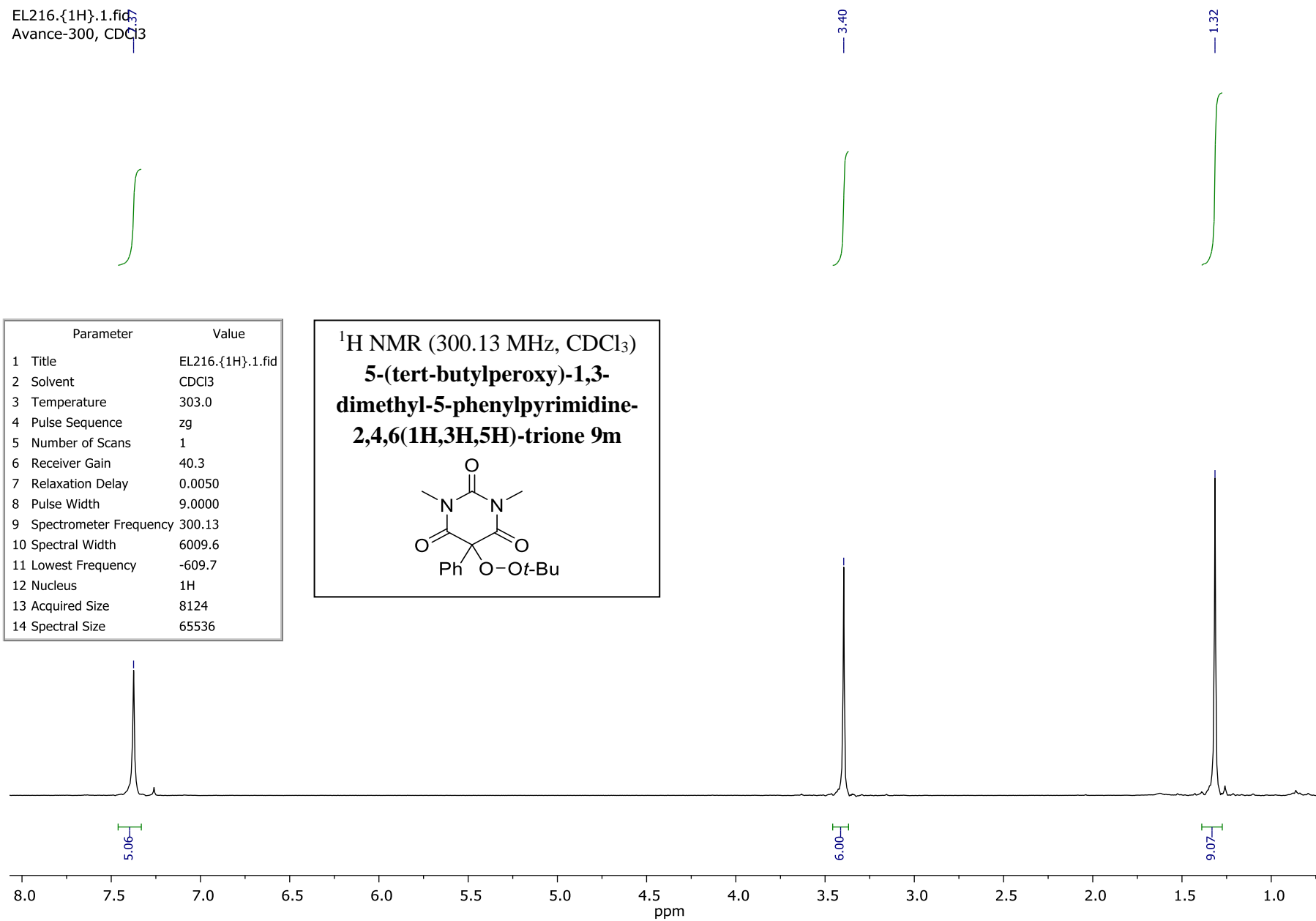
Parameter	Value
1 Title	EL052.{13C}.13.fid
2 Solvent	CDCl <sub>3</sub>
3 Temperature	303.7
4 Pulse Sequence	zgpg30
5 Number of Scans	210
6 Receiver Gain	3250.0
7 Relaxation Delay	1.0000
8 Pulse Width	13.0000
9 Spectrometer Frequency	75.48
10 Spectral Width	18115.9
11 Lowest Frequency	-744.6
12 Nucleus	<sup>13</sup> C
13 Acquired Size	16308
14 Spectral Size	65536

<sup>13</sup>C NMR (75.48 MHz, CDCl<sub>3</sub>)

**5-(tert-butylperoxy)-1,3-dimethyl-5-(thiophen-2-ylmethyl)pyrimidine-2,4,6(1H,3H,5H)-trione 9k**



EL216.{1H}.1.fid  
Avance-300, CDCl<sub>3</sub>



EL216.{13C}.13.fid  
/LB58 IYT1588

167.66

150.94

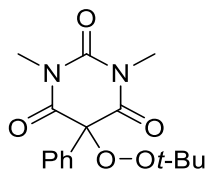
130.37  
129.14  
126.66

82.69  
77.58  
77.16  
76.74

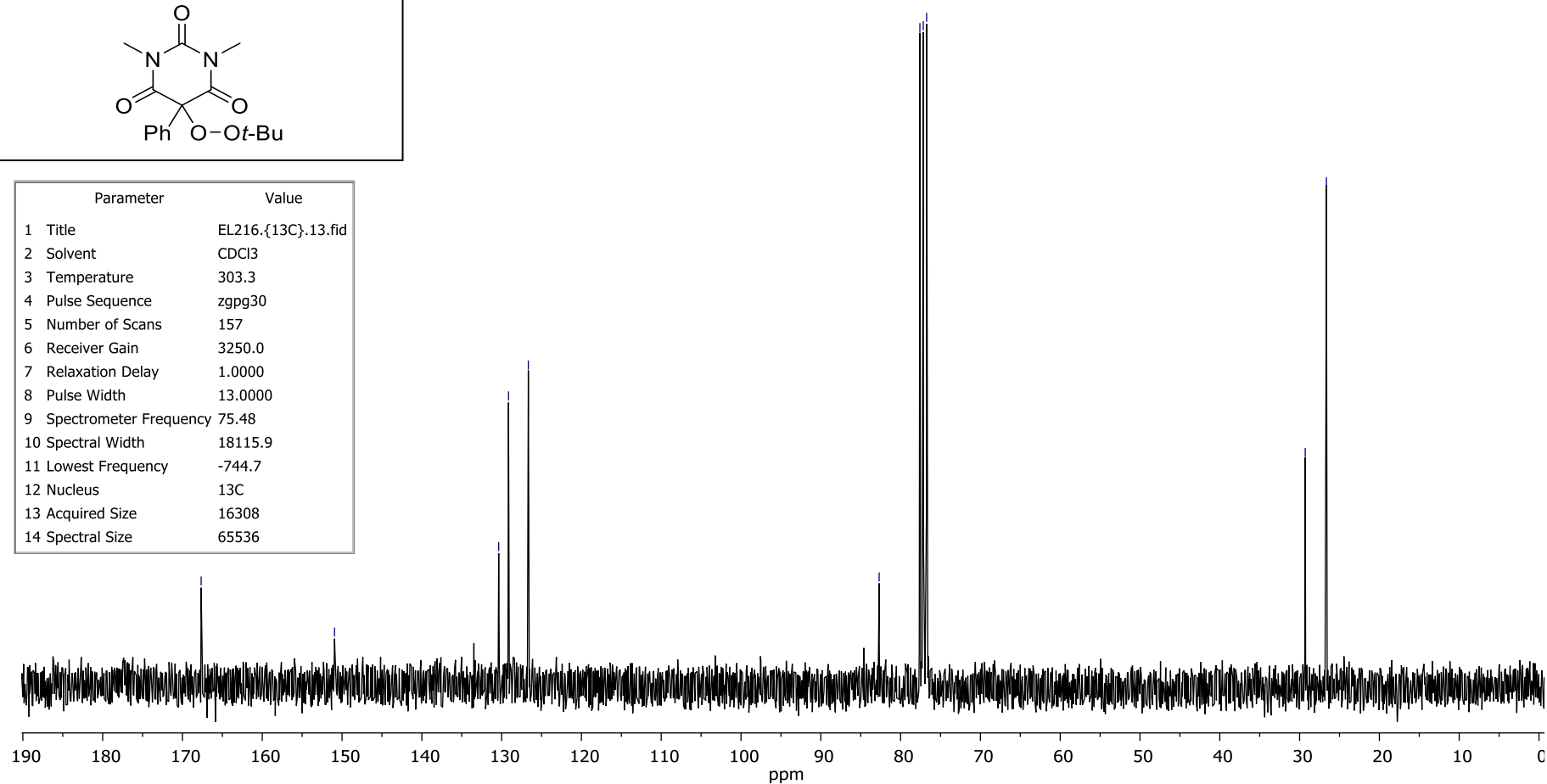
29.29  
26.64

<sup>13</sup>C NMR (75.48 MHz, CDCl<sub>3</sub>)

**5-(tert-butylperoxy)-1,3-dimethyl-5-phenylpyrimidine-2,4,6(1H,3H,5H)-trione 9m**



Parameter	Value
1 Title	EL216.{13C}.13.fid
2 Solvent	CDCl <sub>3</sub>
3 Temperature	303.3
4 Pulse Sequence	zgpg30
5 Number of Scans	157
6 Receiver Gain	3250.0
7 Relaxation Delay	1.0000
8 Pulse Width	13.0000
9 Spectrometer Frequency	75.48
10 Spectral Width	18115.9
11 Lowest Frequency	-744.7
12 Nucleus	<sup>13</sup> C
13 Acquired Size	16308
14 Spectral Size	65536



i8084.{1H}.1.fid  
/TERN i8199

— 7.26

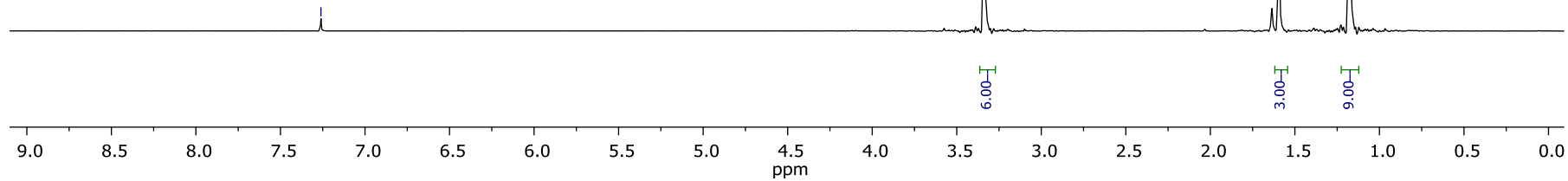
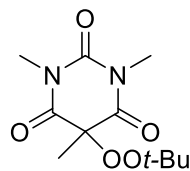
— 3.34

— 1.60

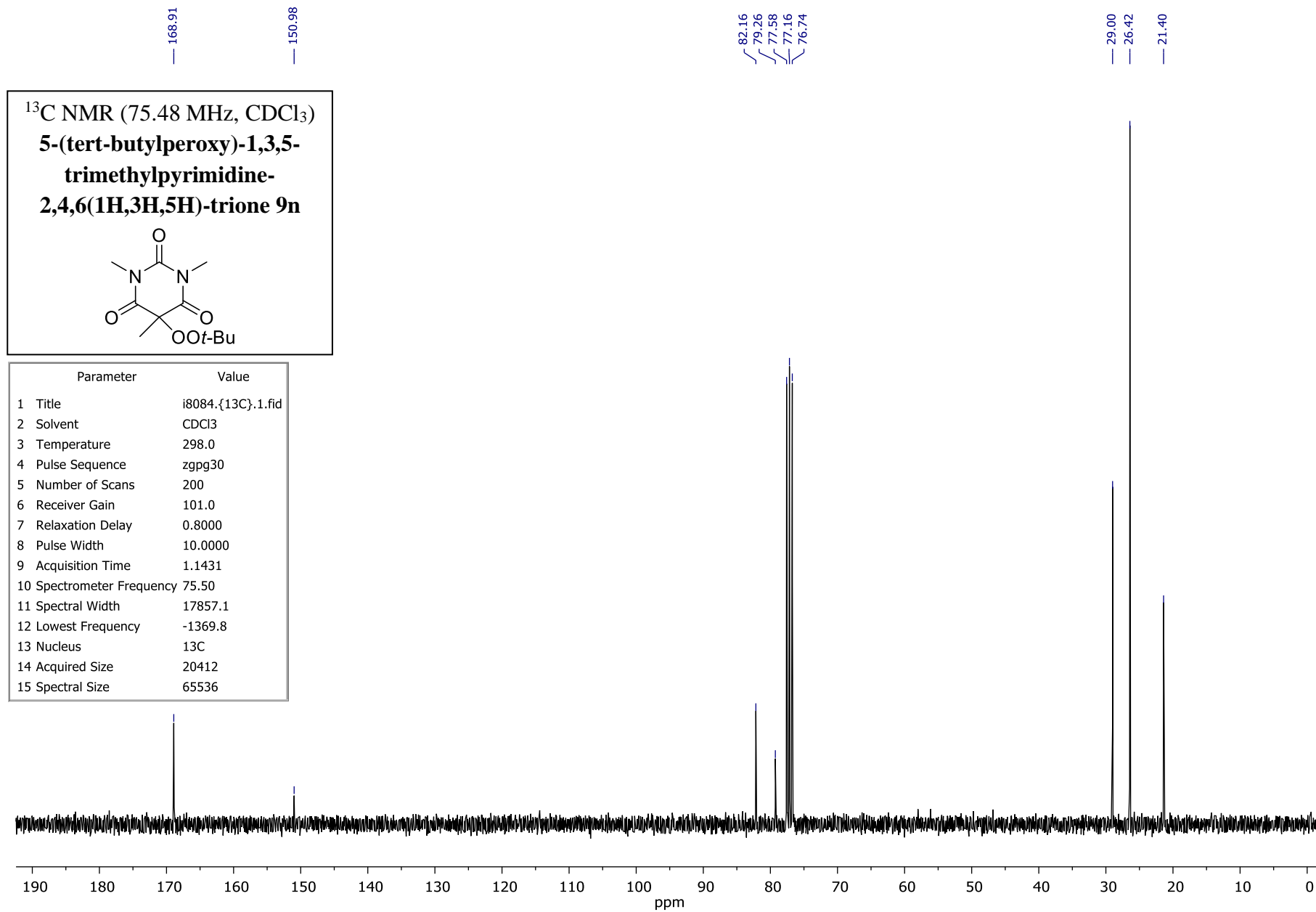
— 1.18

Parameter	Value
1 Title	i8084.{1H}.1.fid
2 Solvent	CDCl <sub>3</sub>
3 Temperature	297.1
4 Pulse Sequence	zg
5 Number of Scans	1
6 Receiver Gain	32.0
7 Relaxation Delay	0.0050
8 Pulse Width	9.0000
9 Spectrometer Frequency	300.13
10 Spectral Width	6009.6
11 Lowest Frequency	-602.4
12 Nucleus	<sup>1</sup> H
13 Acquired Size	8124
14 Spectral Size	65536

<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)  
**5-(tert-butylperoxy)-1,3,5-trimethylpyrimidine-2,4,6(1H,3H,5H)-trione 9n**



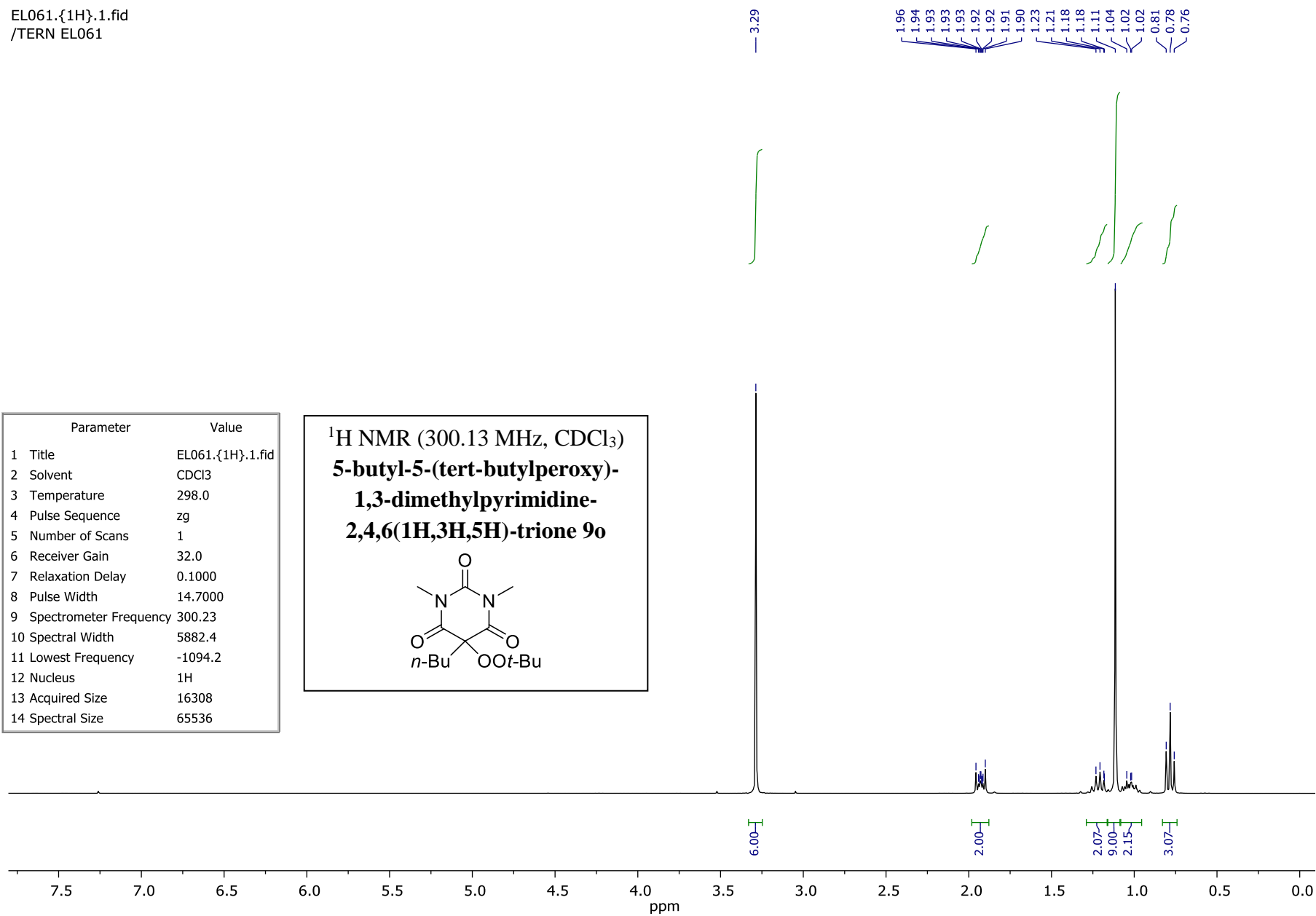
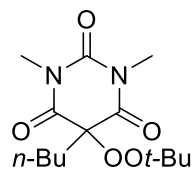




EL061.{1H}.1.fid  
/TERN EL061

Parameter	Value
1 Title	EL061.{1H}.1.fid
2 Solvent	CDCl <sub>3</sub>
3 Temperature	298.0
4 Pulse Sequence	zg
5 Number of Scans	1
6 Receiver Gain	32.0
7 Relaxation Delay	0.1000
8 Pulse Width	14.7000
9 Spectrometer Frequency	300.23
10 Spectral Width	5882.4
11 Lowest Frequency	-1094.2
12 Nucleus	<sup>1</sup> H
13 Acquired Size	16308
14 Spectral Size	65536

<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)  
**5-butyl-5-(tert-butylperoxy)-  
1,3-dimethylpyrimidine-  
2,4,6(1H,3H,5H)-trione 9o**



EL061.{13C}.2.fid  
/TERN EL061

— 168.60

— 150.89

82.82  
81.89  
77.59  
77.16  
76.74

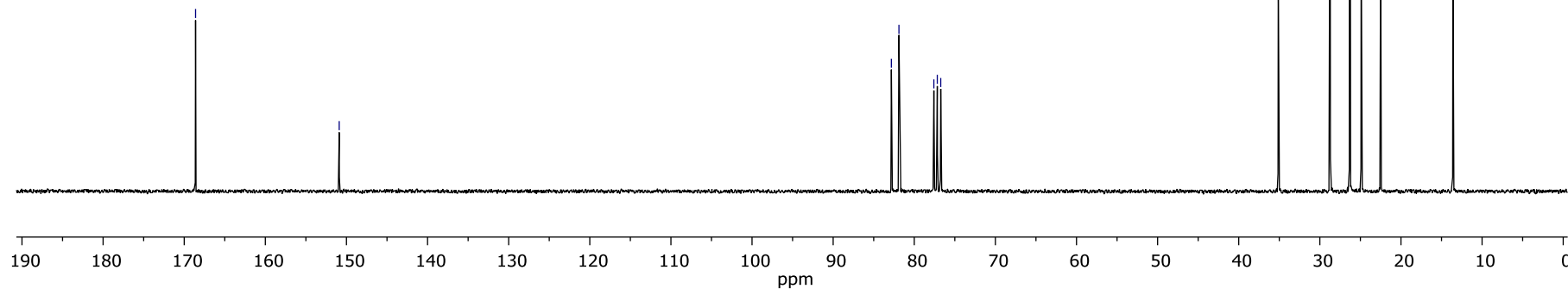
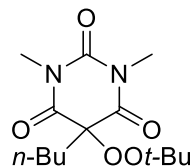
— 35.13

28.73  
26.32  
24.91  
22.51

— 13.60

Parameter	Value
1 Title	EL061.{13C}.2.fid
2 Solvent	CDCl <sub>3</sub>
3 Temperature	298.0
4 Pulse Sequence	zgpg30
5 Number of Scans	256
6 Receiver Gain	101.0
7 Relaxation Delay	0.8000
8 Pulse Width	9.9000
9 Spectrometer Frequency	75.50
10 Spectral Width	17857.1
11 Lowest Frequency	-1374.6
12 Nucleus	13C
13 Acquired Size	20412
14 Spectral Size	65536

<sup>13</sup>C NMR (75.48 MHz, CDCl<sub>3</sub>)  
**5-butyl-5-(tert-butylperoxy)-  
1,3-dimethylpyrimidine-  
2,4,6(1H,3H,5H)-trione 9o**



EL069.{1H}.1.fid  
Avance-300, CDCl<sub>3</sub>

7.26

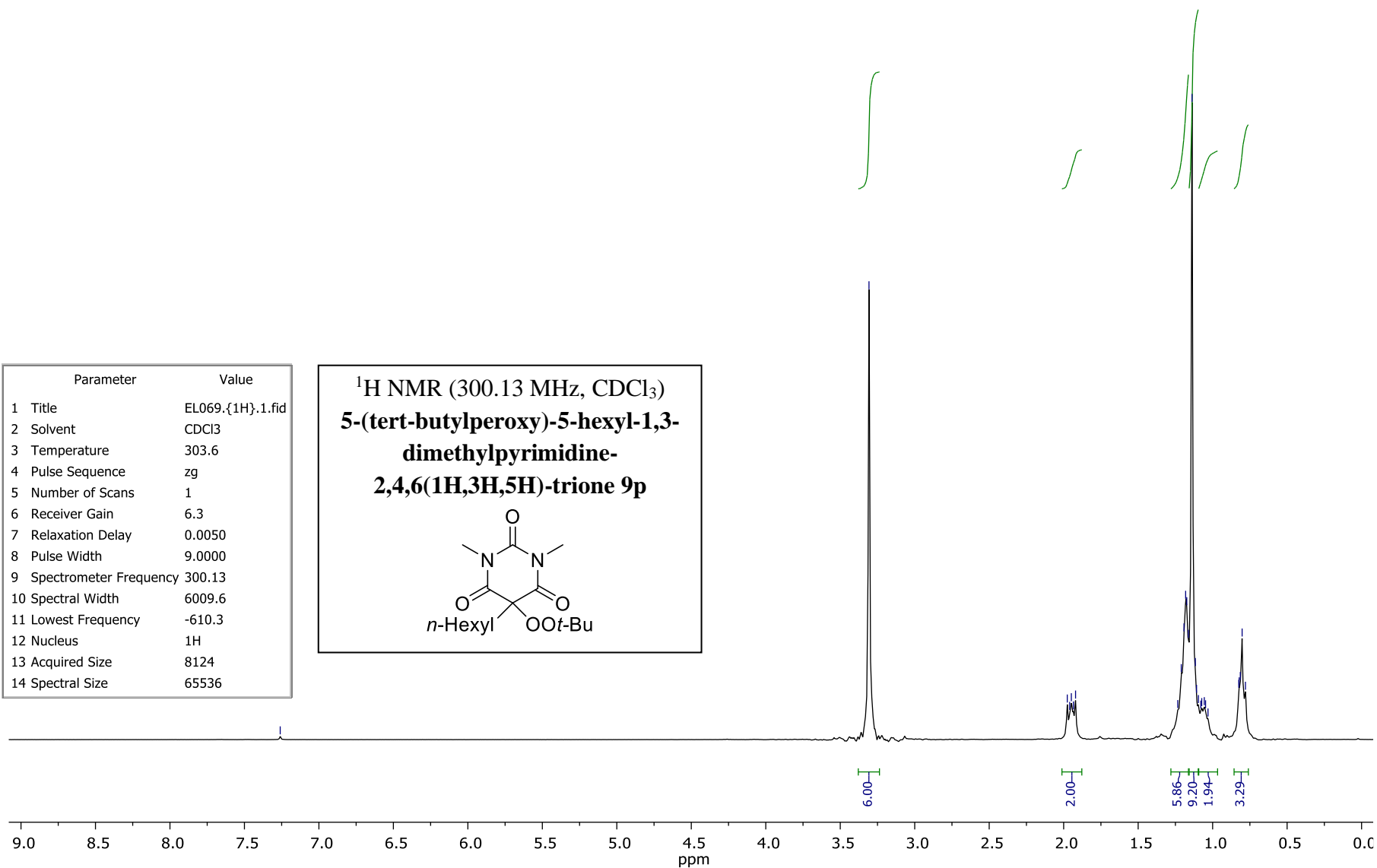
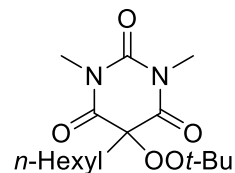
3.31

1.98  
1.96  
1.95  
1.93  
1.92

1.21  
1.19  
1.18  
1.17  
1.16  
1.14  
1.12  
1.11  
1.10  
1.07  
1.06  
0.82  
0.81  
0.80  
0.78

Parameter	Value
1 Title	EL069.{1H}.1.fid
2 Solvent	CDCl <sub>3</sub>
3 Temperature	303.6
4 Pulse Sequence	zg
5 Number of Scans	1
6 Receiver Gain	6.3
7 Relaxation Delay	0.0050
8 Pulse Width	9.0000
9 Spectrometer Frequency	300.13
10 Spectral Width	6009.6
11 Lowest Frequency	-610.3
12 Nucleus	<sup>1</sup> H
13 Acquired Size	8124
14 Spectral Size	65536

<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)  
**5-(tert-butylperoxy)-5-hexyl-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione 9p**



EL069.{13C}.13.fid  
/LB58 IYT1588

— 168.66

— 150.99

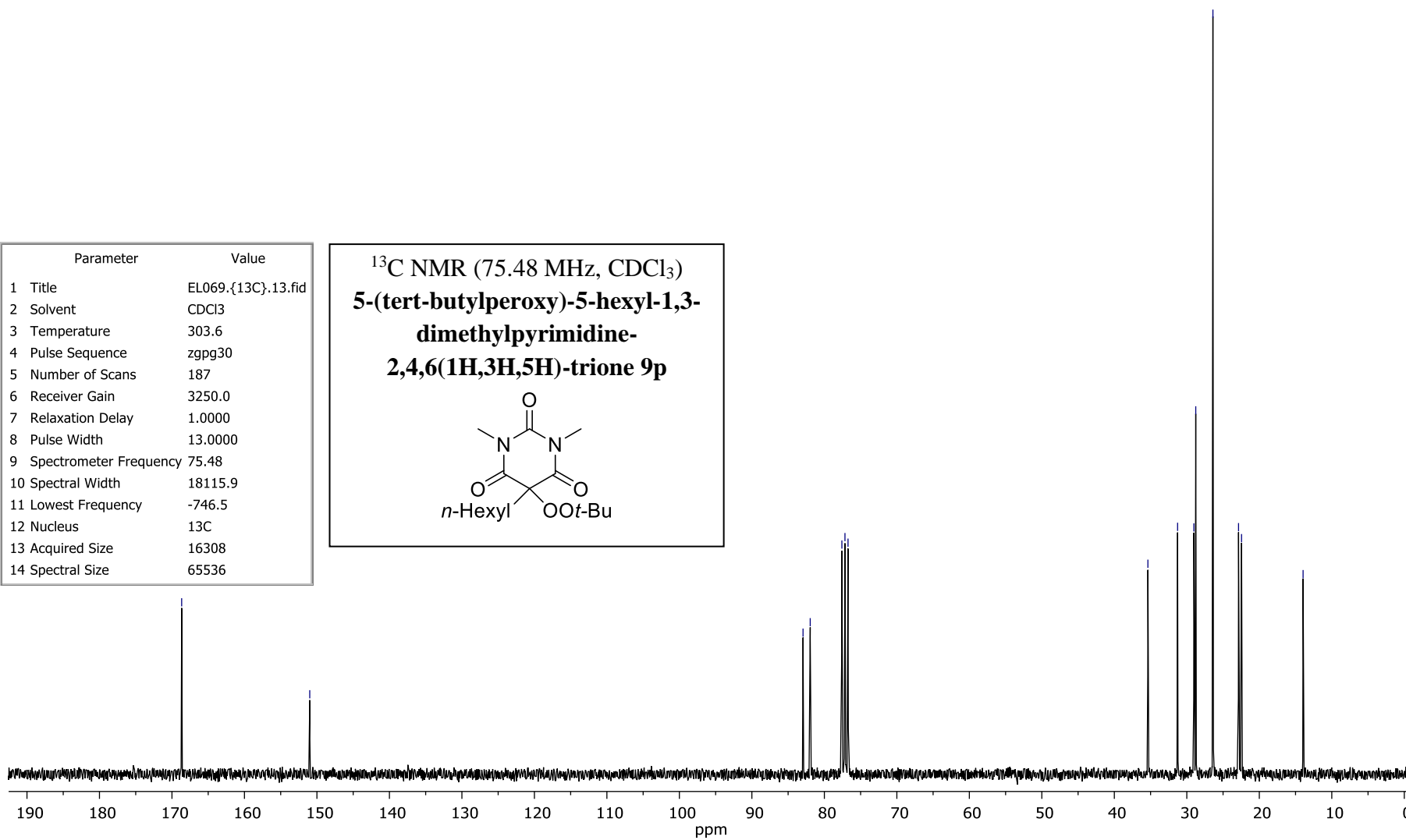
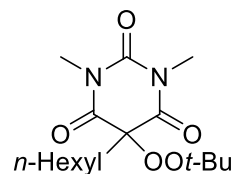
82.94  
81.95  
77.58  
77.16  
76.74

35.37  
31.27  
29.03  
28.77  
26.40  
22.87  
22.45

— 13.96

Parameter	Value
1 Title	EL069.{13C}.13.fid
2 Solvent	CDCl <sub>3</sub>
3 Temperature	303.6
4 Pulse Sequence	zgpg30
5 Number of Scans	187
6 Receiver Gain	3250.0
7 Relaxation Delay	1.0000
8 Pulse Width	13.0000
9 Spectrometer Frequency	75.48
10 Spectral Width	18115.9
11 Lowest Frequency	-746.5
12 Nucleus	<sup>13</sup> C
13 Acquired Size	16308
14 Spectral Size	65536

<sup>13</sup>C NMR (75.48 MHz, CDCl<sub>3</sub>)  
**5-(tert-butylperoxy)-5-hexyl-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione 9p**



EL196.{1H}.1.fid  
Avance-300, CDCl<sub>3</sub>

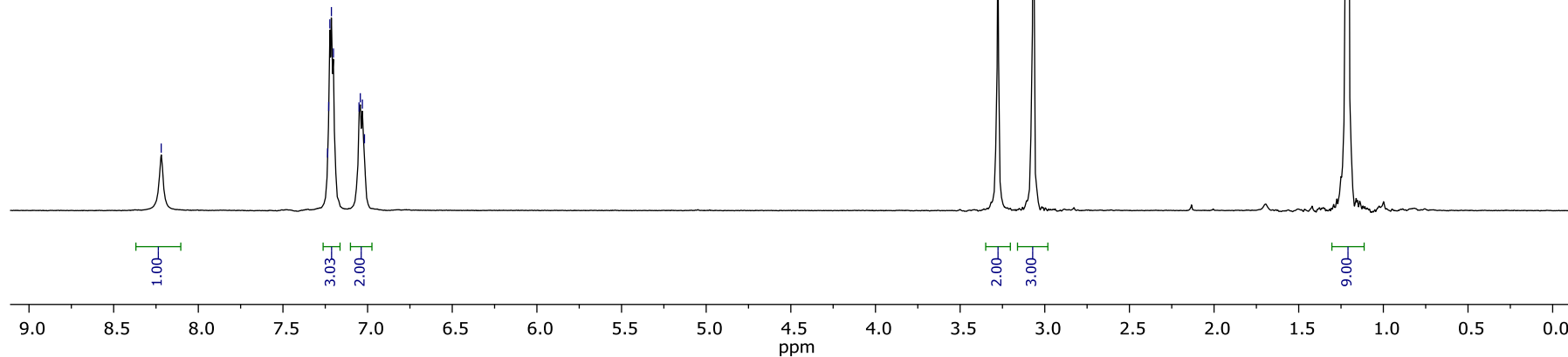
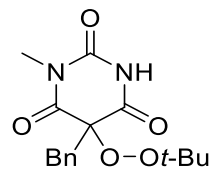
7.24  
7.23  
7.22  
7.21  
7.20  
7.05  
7.04  
7.03  
7.02

3.28  
3.07

1.21

Parameter	Value
1 Title	EL196.{1H}.1.fid
2 Solvent	CDCl <sub>3</sub>
3 Temperature	302.3
4 Pulse Sequence	zg
5 Number of Scans	1
6 Receiver Gain	25.4
7 Relaxation Delay	0.0050
8 Pulse Width	9.0000
9 Spectrometer Frequency	300.13
10 Spectral Width	6009.6
11 Lowest Frequency	-619.0
12 Nucleus	<sup>1</sup> H
13 Acquired Size	8124
14 Spectral Size	65536

<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)  
**5-benzyl-5-(tert-butylperoxy)-  
1-methylpyrimidine-  
2,4,6(1H,3H,5H)-trione 9r**



EL196.{13C}.13.fid  
/LB58 IYT1588

168.97  
167.60

148.99

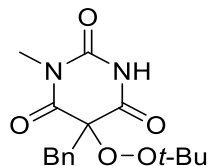
130.60  
129.95  
129.01  
128.45

84.07  
82.60  
77.58  
77.16  
76.74

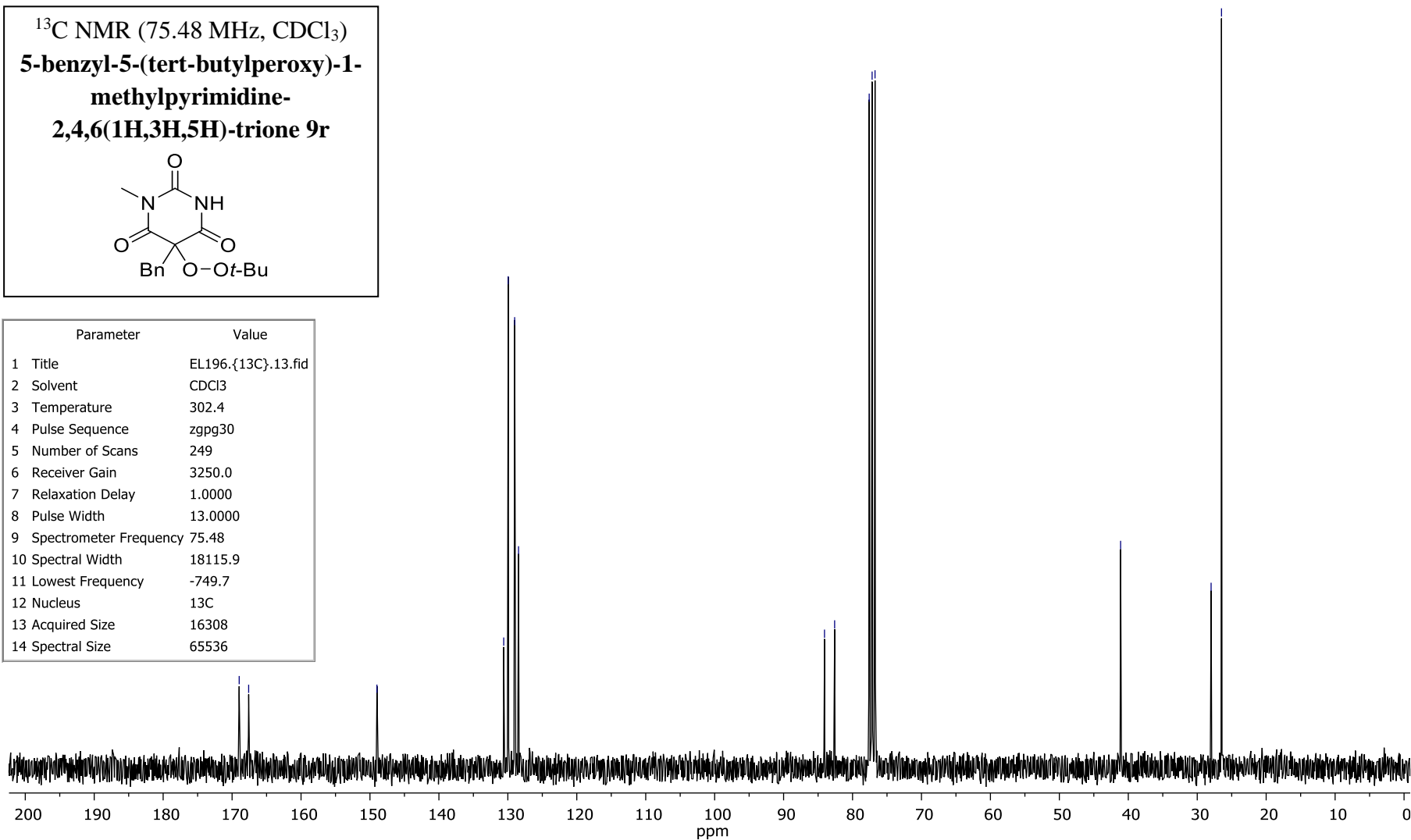
41.11

27.99  
26.48

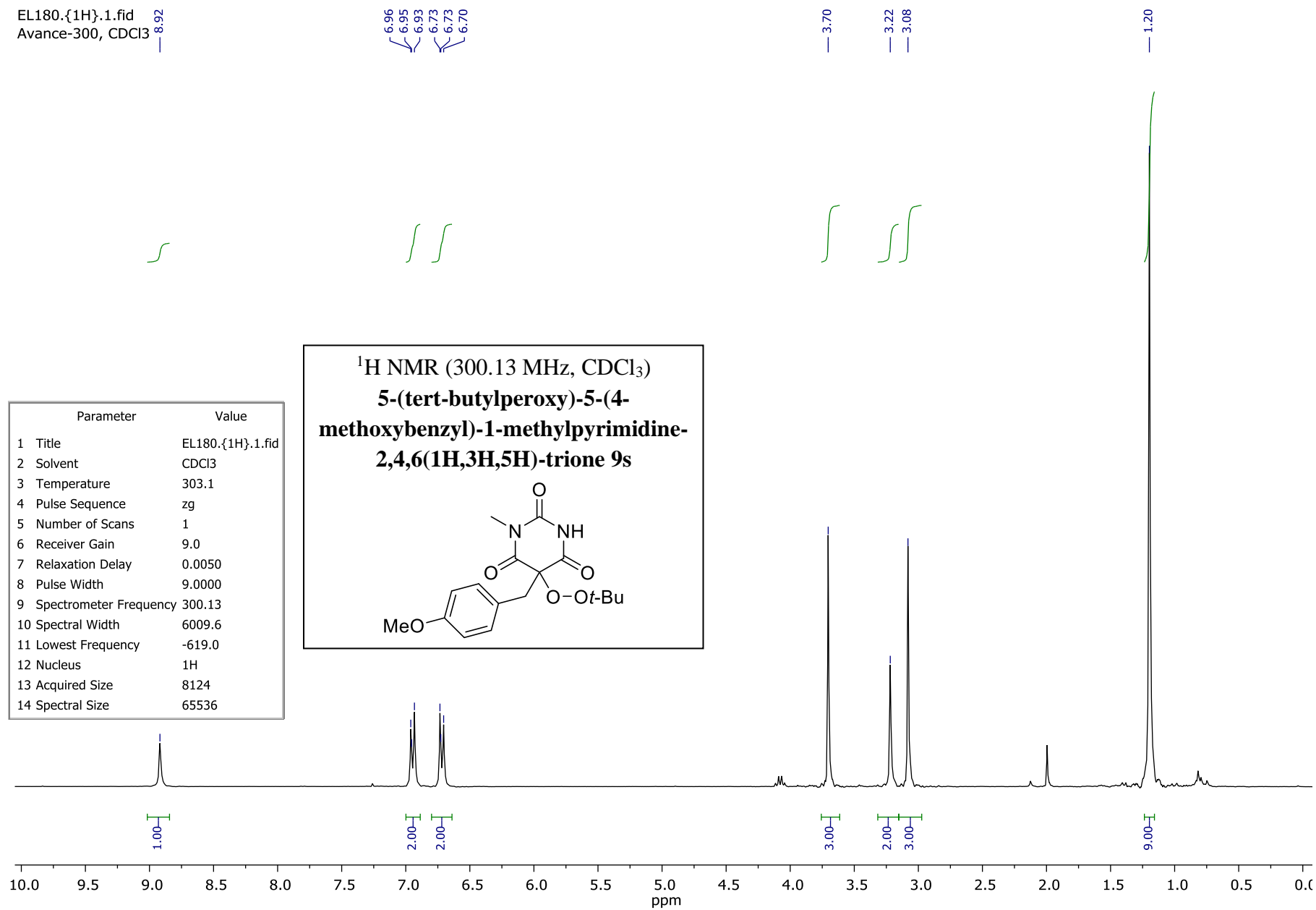
<sup>13</sup>C NMR (75.48 MHz, CDCl<sub>3</sub>)  
**5-benzyl-5-(tert-butylperoxy)-1-methylpyrimidine-2,4,6(1H,3H,5H)-trione 9r**



Parameter	Value
1 Title	EL196.{13C}.13.fid
2 Solvent	CDCl <sub>3</sub>
3 Temperature	302.4
4 Pulse Sequence	zgpg30
5 Number of Scans	249
6 Receiver Gain	3250.0
7 Relaxation Delay	1.0000
8 Pulse Width	13.0000
9 Spectrometer Frequency	75.48
10 Spectral Width	18115.9
11 Lowest Frequency	-749.7
12 Nucleus	<sup>13</sup> C
13 Acquired Size	16308
14 Spectral Size	65536



EL180.{1H}.1.fid  
Avance-300, CDCl<sub>3</sub>





EL180.{13C}.13.fid  
/LB58 IYT1588

169.06  
167.99

159.45

149.27

130.97

122.23

114.30

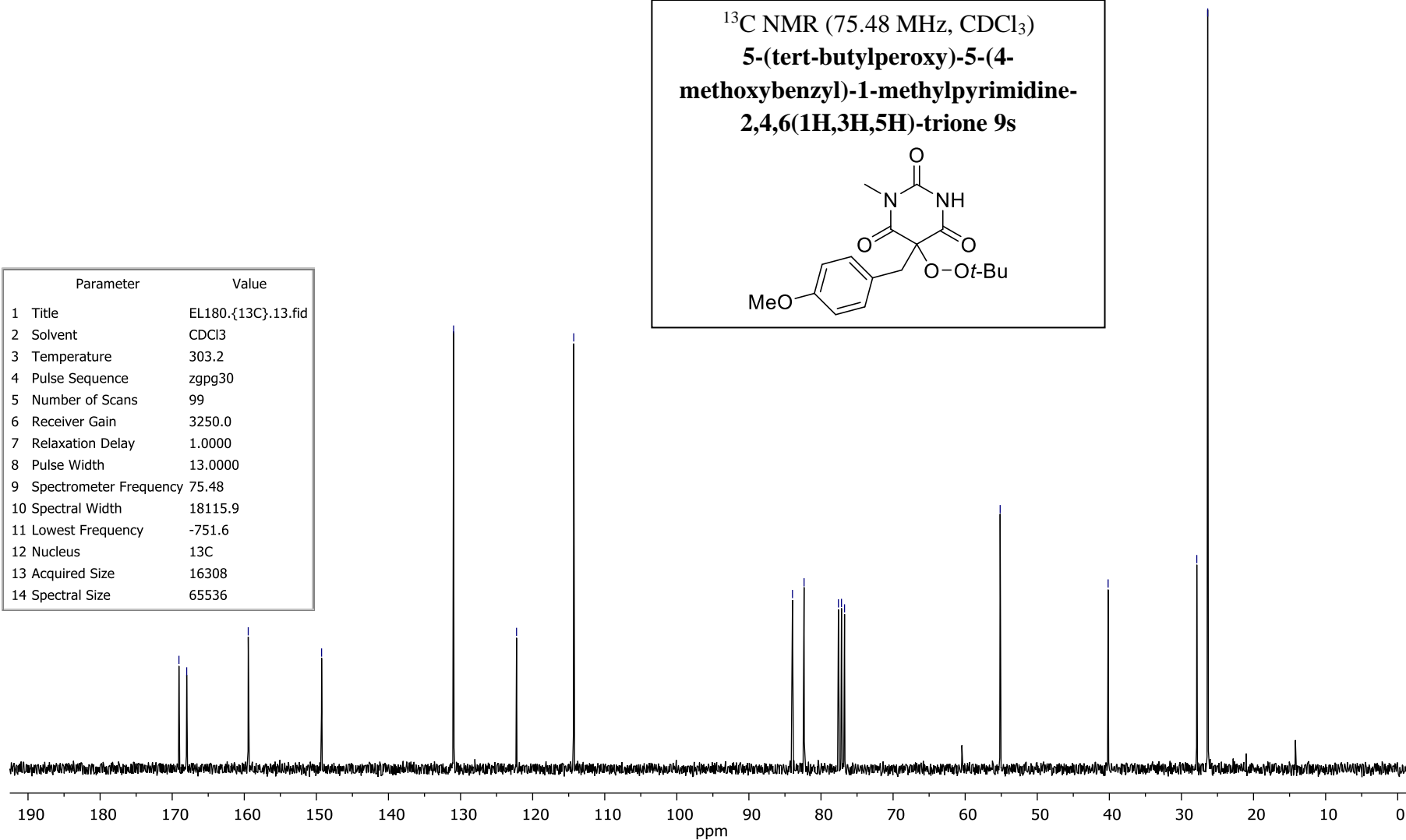
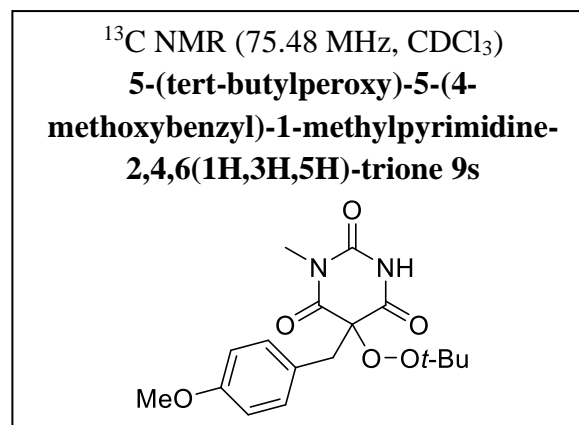
83.96  
82.35  
77.58  
77.16  
76.74

55.15

40.17

27.87  
26.36

Parameter	Value
1 Title	EL180.{13C}.13.fid
2 Solvent	CDCl3
3 Temperature	303.2
4 Pulse Sequence	zgpg30
5 Number of Scans	99
6 Receiver Gain	3250.0
7 Relaxation Delay	1.0000
8 Pulse Width	13.0000
9 Spectrometer Frequency	75.48
10 Spectral Width	18115.9
11 Lowest Frequency	-751.6
12 Nucleus	13C
13 Acquired Size	16308
14 Spectral Size	65536



7.23  
7.22  
7.21  
7.21  
7.20  
6.92  
6.92  
6.90  
6.89

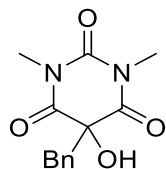
3.75

3.21

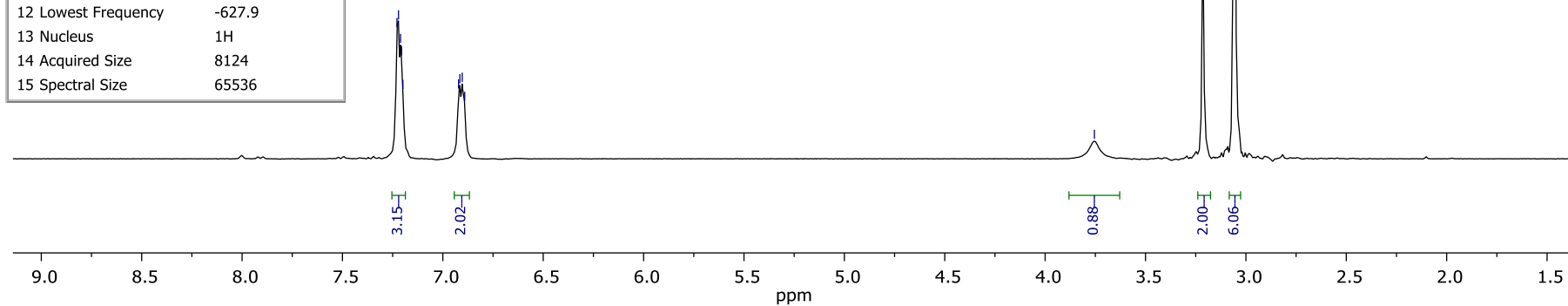
3.06

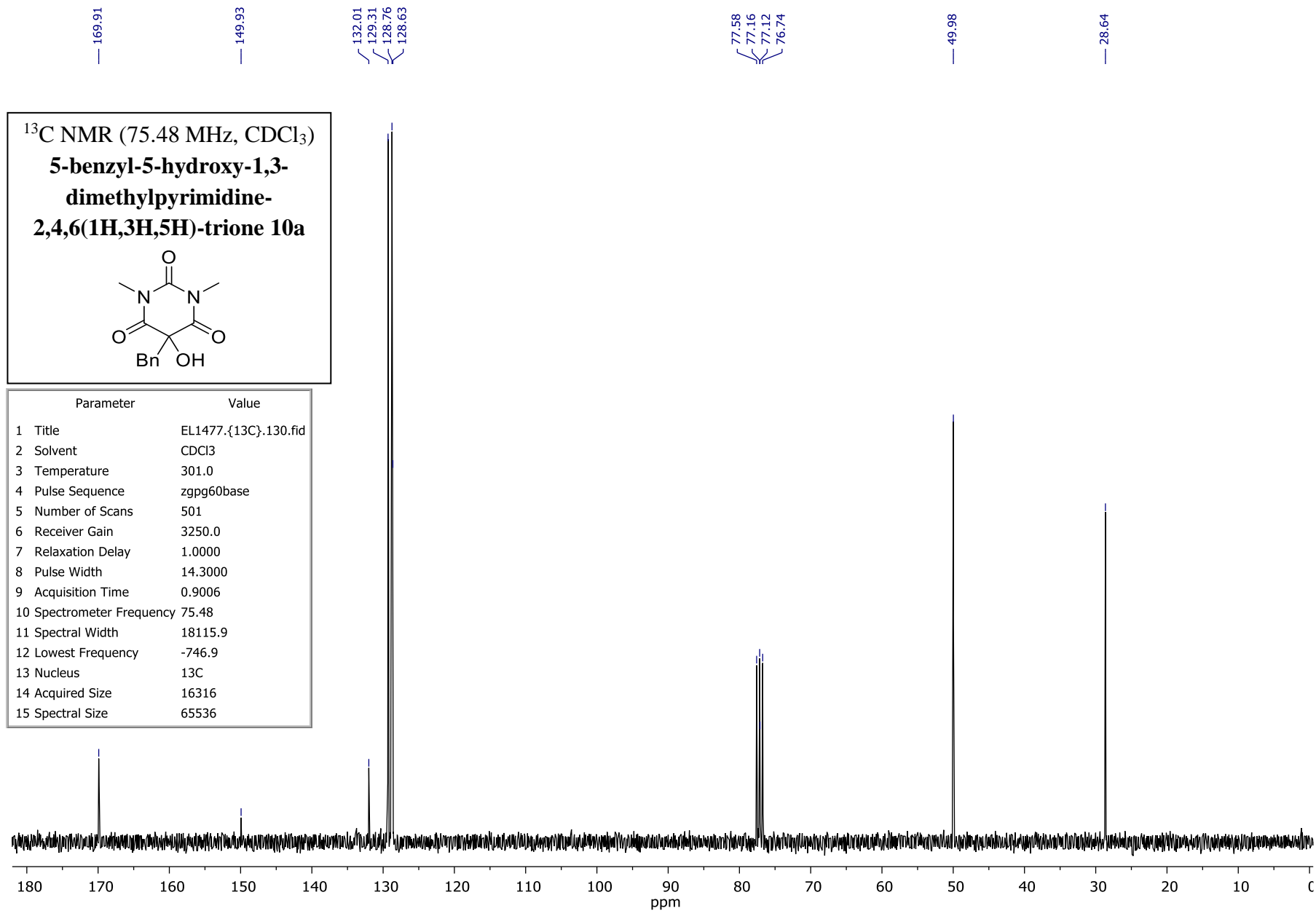
<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)

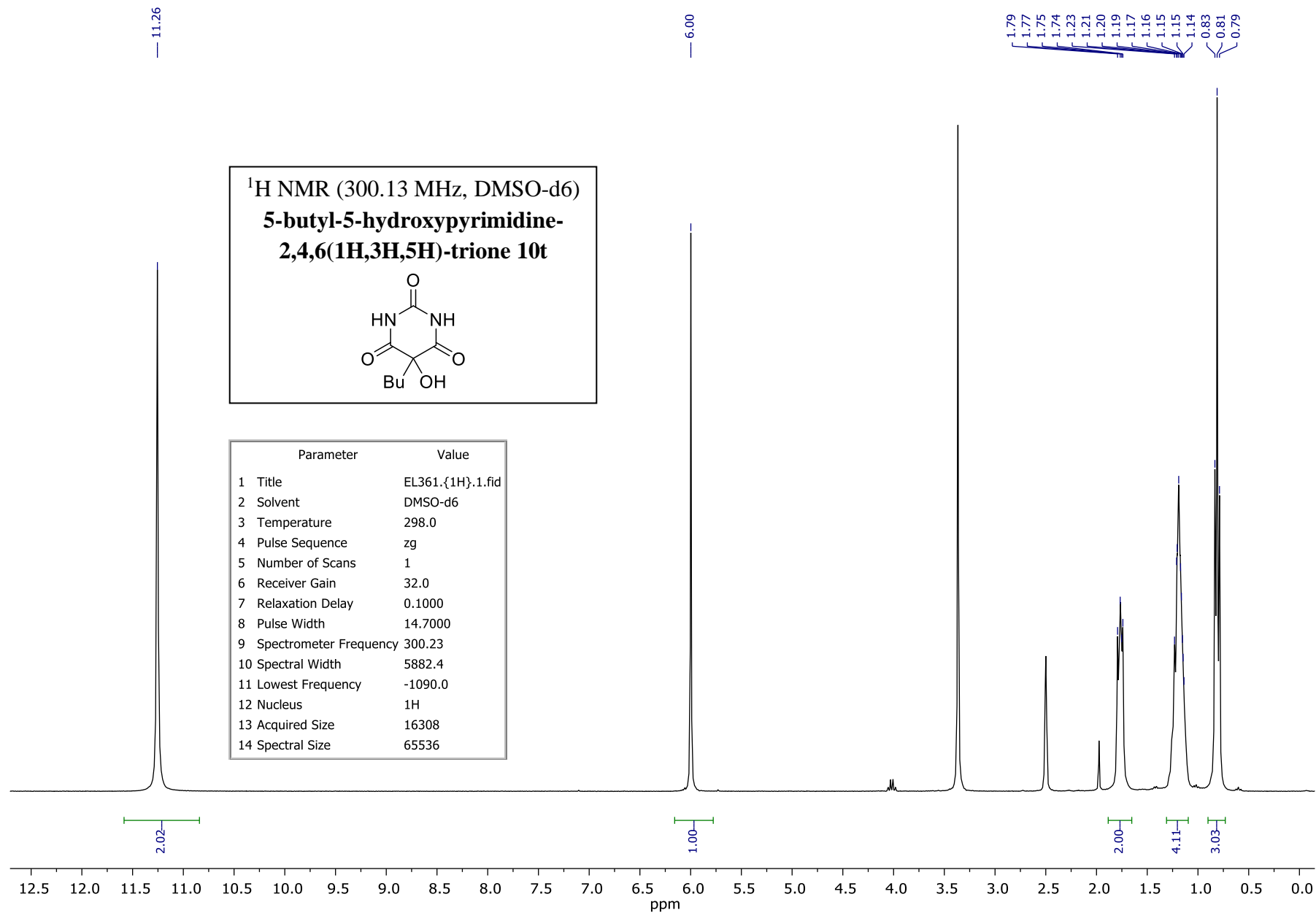
**5-benzyl-5-hydroxy-1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione 10a**

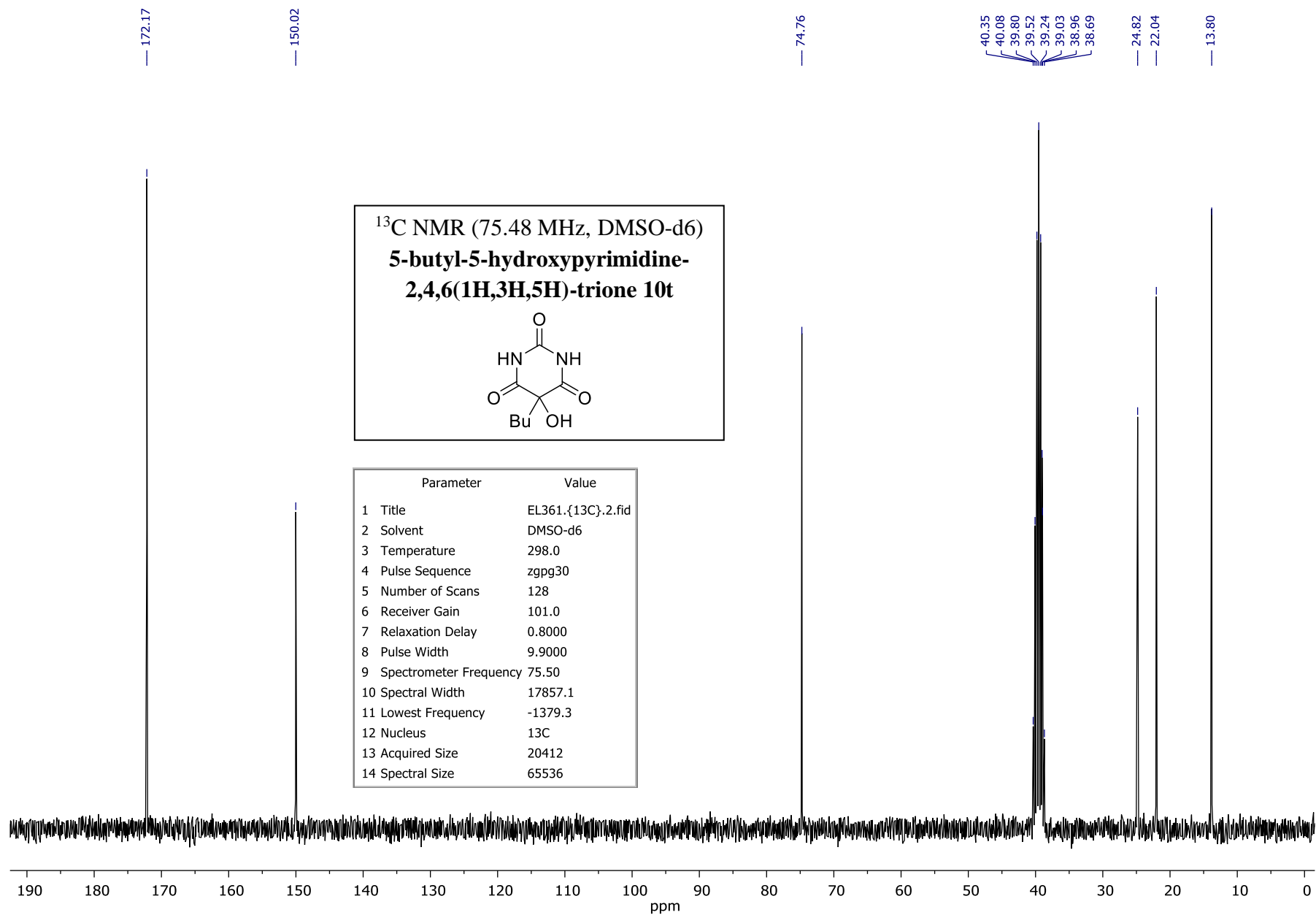


Parameter	Value
1 Title	EL1477.{1H}.1.fid
2 Solvent	CDCl <sub>3</sub>
3 Temperature	300.9
4 Pulse Sequence	zg
5 Number of Scans	1
6 Receiver Gain	40.3
7 Relaxation Delay	0.0050
8 Pulse Width	9.0000
9 Acquisition Time	1.3518
10 Spectrometer Frequency	300.13
11 Spectral Width	6009.6
12 Lowest Frequency	-627.9
13 Nucleus	<sup>1</sup> H
14 Acquired Size	8124
15 Spectral Size	65536

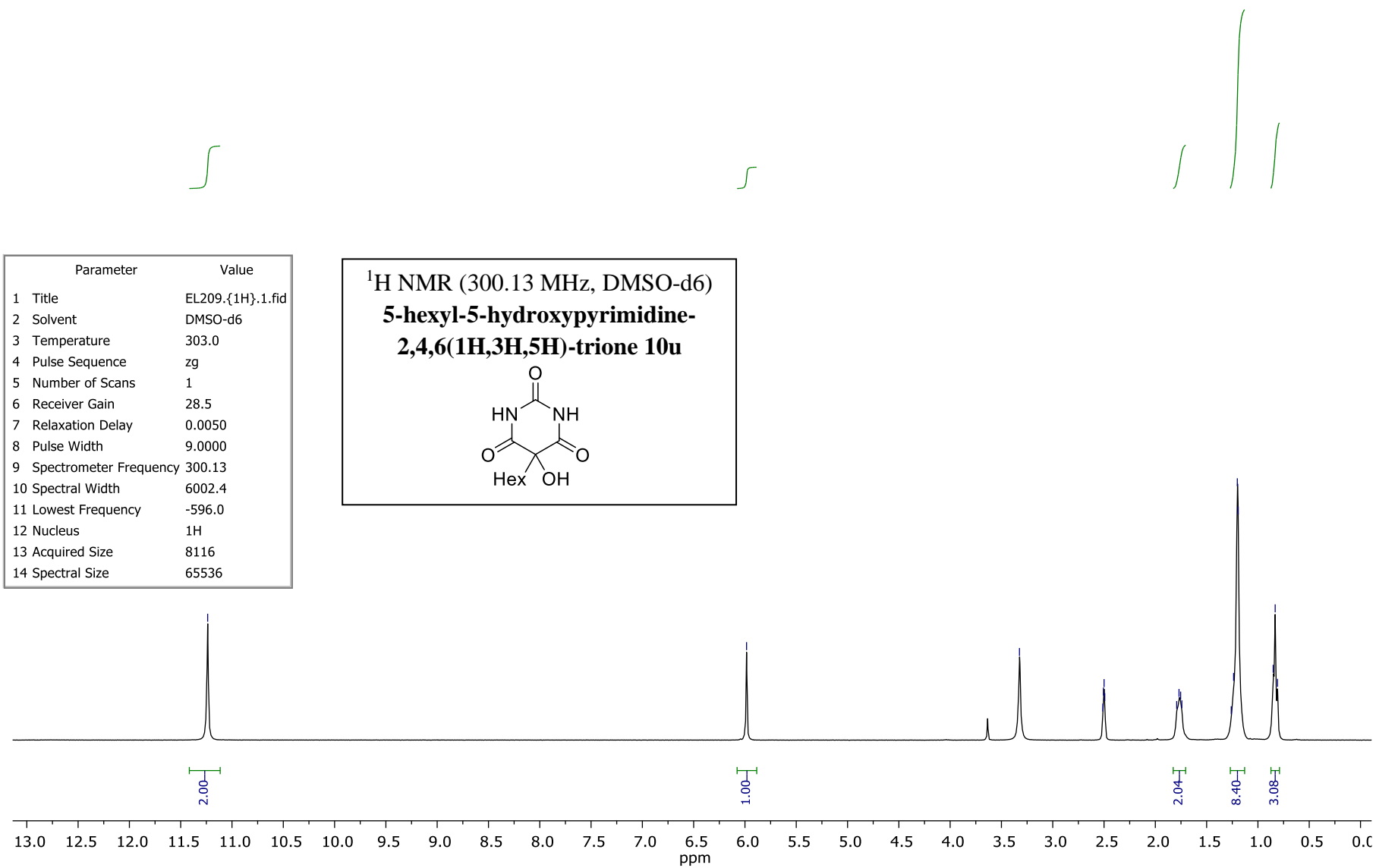








EL209.{1H}.1.fid  
/MBCI 4MEOGL4MEBNDME



EL209.{13C}.13.fid  
/KANI VEE06.1

— 172.6

— 149.91

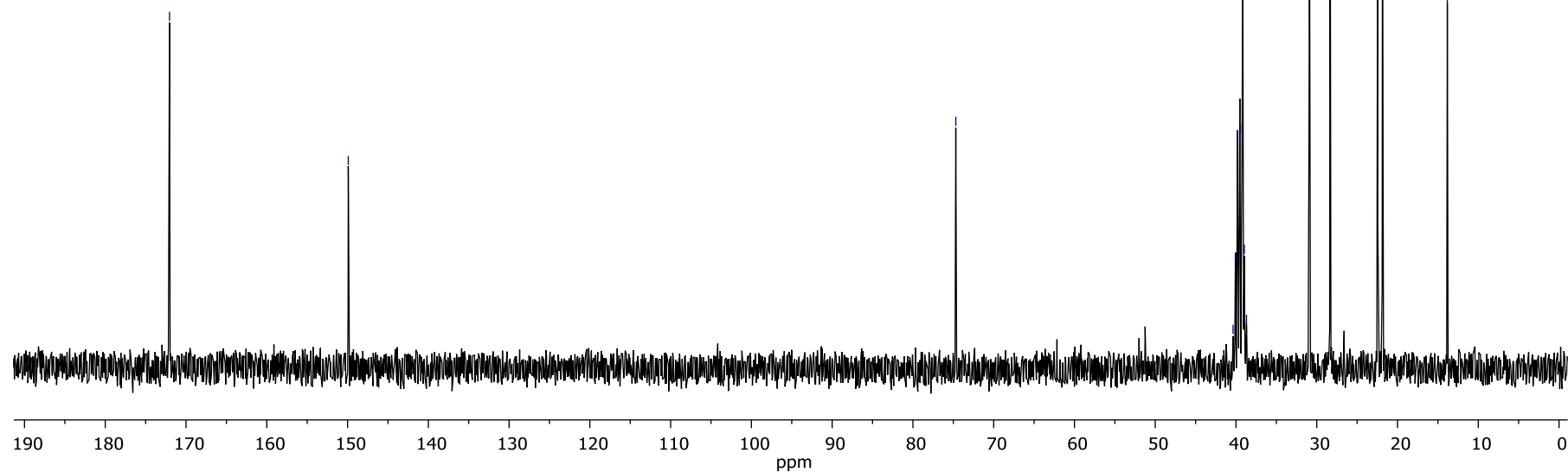
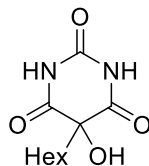
— 74.69

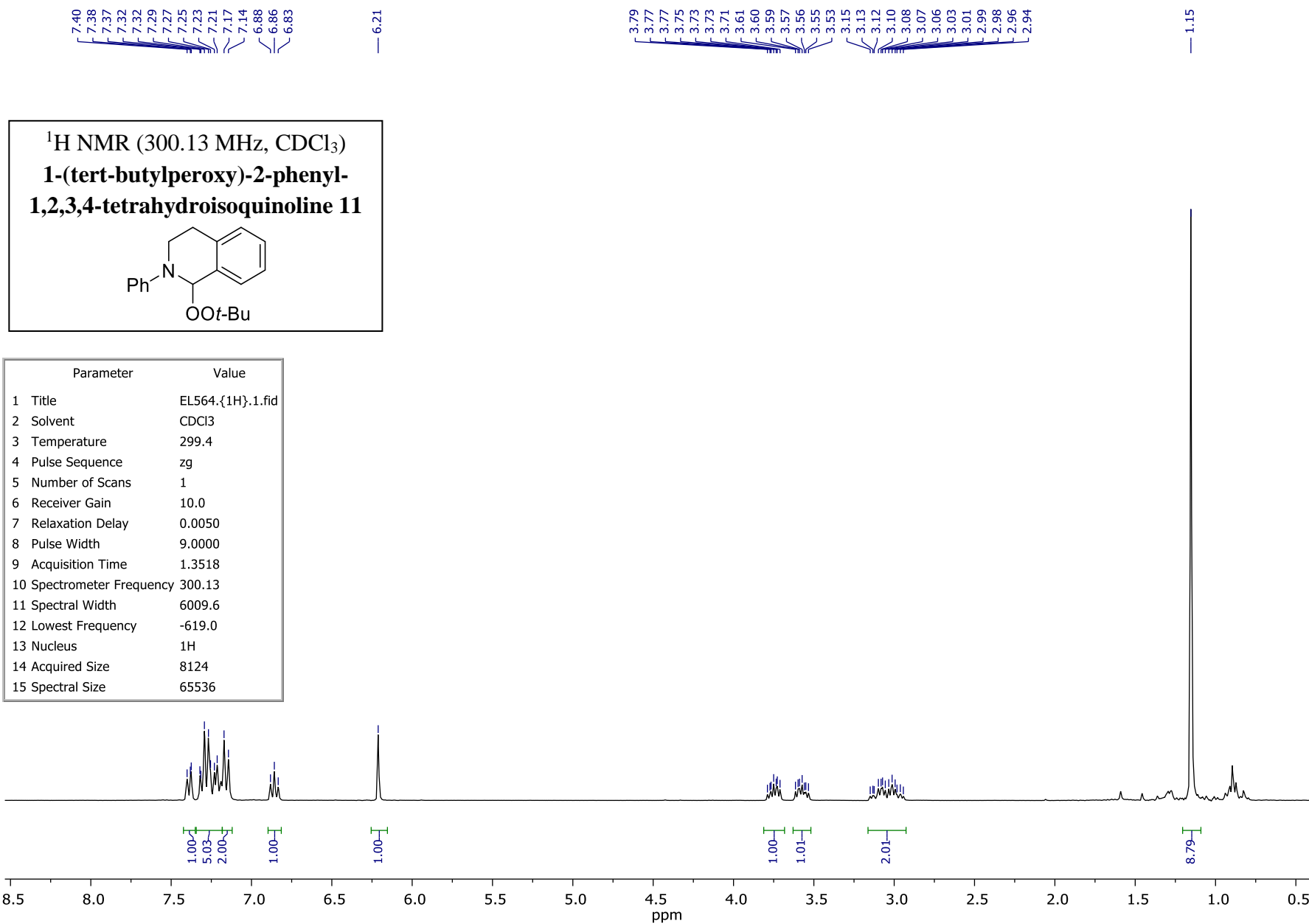
40.36  
40.08  
39.81  
39.53  
39.25  
39.16  
38.97  
38.70  
30.92  
28.35  
22.47  
21.87  
— 13.83

Parameter	Value
1 Title	EL209.{13C}.13.fid
2 Solvent	DMSO-d6
3 Temperature	303.0
4 Pulse Sequence	zgpg60base
5 Number of Scans	146
6 Receiver Gain	3250.0
7 Relaxation Delay	1.0000
8 Pulse Width	14.3000
9 Spectrometer Frequency	75.48
10 Spectral Width	18115.9
11 Lowest Frequency	-792.2
12 Nucleus	13C
13 Acquired Size	16316
14 Spectral Size	65536

<sup>13</sup>C NMR (75.48 MHz, DMSO-d6)

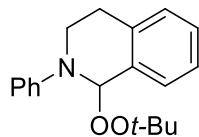
**5-hexyl-5-hydroxypyrimidine-  
2,4,6(1H,3H,5H)-trione 10u**







<sup>13</sup>C NMR (75.48 MHz, CDCl<sub>3</sub>)  
**1-(tert-butylperoxy)-2-phenyl-  
 1,2,3,4-tetrahydroisoquinoline 11**



Parameter	Value
1 Title	EL564.{ <sup>13</sup> C}.13.fid
2 Solvent	CDCl <sub>3</sub>
3 Temperature	299.2
4 Pulse Sequence	zgpg30
5 Number of Scans	145
6 Receiver Gain	3250.0
7 Relaxation Delay	1.0000
8 Pulse Width	13.0000
9 Acquisition Time	0.9002
10 Spectrometer Frequency	75.48
11 Spectral Width	18115.9
12 Lowest Frequency	-756.1
13 Nucleus	<sup>13</sup> C
14 Acquired Size	16308
15 Spectral Size	65536

