

## *Supporting Information*

### **Heterogeneous photocatalysis as a potent tool for organic synthesis: cross-dehydrogenative C–C coupling of *N*-heterocycles with ethers employing TiO<sub>2</sub>/*N*-hydroxyphthalimide system under visible light**

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\* Correspondence: krylovigor@yandex.ru (I.B.K.); alterex@yandex.ru (A.O.T.)

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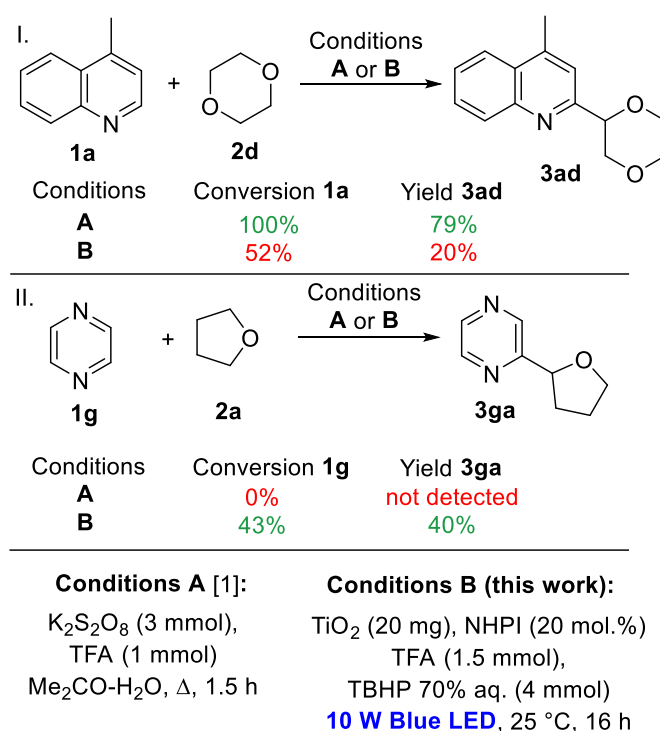
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## General

In all experiments RT stands for 23-25 °C.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a Bruker AVANCE II 300 and Bruker Fourier 300HD (300.13 and 75.47 MHz, respectively) spectrometers in  $\text{CDCl}_3$  and  $\text{DMSO-}d_6$ . Residual signals of  $\text{CDCl}_3$  (7.26 in  $^1\text{H}$  NMR, 77.16 in  $^{13}\text{C}$  NMR) were used as reference signals for precise chemical shift determination. FT-IR spectra were recorded on Bruker Alpha instrument. IR spectra were registered in KBr pellets for solid compounds, and liquid compounds were placed between two KBr windows to make a thin layer. High resolution mass spectra (HR-MS) were measured on a Bruker maXis instrument using electrospray ionization (ESI). The measurements were performed in a positive ion mode (interface capillary voltage – 4500 V); mass range from  $m/z$  50 to  $m/z$  3000 Da; external calibration with Electrospray Calibrant Solution (Fluka). A syringe injection was used for all acetonitrile solutions (flow rate 3  $\mu\text{L}/\text{min}$ ). Nitrogen was applied as a dry gas; interface temperature was set at 180 °C.

## The comparison of the developed method with the literature procedure

The coupling products of the of THF with pyrazines (**3ga**, **3ha**) have not been previously described. We decided to test other known effective procedure [1] to compare the results with the results achieved by our method. The chosen literature method [1] works reliably for the coupling of 4-methylquinoline **1a** with dioxane **2d** (I, conditions A), while our system gives lower yields of product **3ad** (I, conditions B). When switching to pyrazine **1g** as a substrate we do not observe the desired product **3ga** using the literature procedure (II, conditions A). However, it was possible to obtain **3ga** using our photochemical approach (II, conditions B).



**Experimental details for conditions A [1].** To a solution of heterocycle (1 mmol) in a mixture of acetone/ $\text{H}_2\text{O}$  (7 mL/3 mL) was added the corresponding ether (20 mmol, 20 equiv.),  $\text{K}_2\text{S}_2\text{O}_8$  (811 mg, 3 mmol, 3 equiv.) and TFA (80  $\mu\text{L}$ , 1 mmol). The mixture was then stirred for 1.5h under reflux. The reaction was quenched with saturated aqueous  $\text{NaHCO}_3$  (16 mL) and extracted with ethyl acetate ( $2 \times 25$  mL). The combined organic phases were dried over  $\text{MgSO}_4$  and concentrated under reduced pressure. The crude product was further purified by chromatography on silica gel (Petroleum ether/EtOAc = 2/1) to give the desired product.

For experimental details of conditions **B**, see the Experimental details for the Schemes 2 and 3.

## The determination of the side products of the studied process

To identify the oxidation products of **3aa**, the  $^1\text{H}$ ,  $^{13}\text{C}$  NMR and HRMS spectra were recorded for the reaction mixture in conditions for Scheme 4, A (See 3. Materials and Methods, Experimental details for Scheme 4, A). The peroxide 2-(2-hydroperoxytetrahydrofuran-2-yl)-4-methylquinoline **3aa'** was recognized as the major product. The characteristic  $^{13}\text{C}$  signal corresponding to the C-OOH of hydroperoxide is 113.3, which is in a good agreement with the literature data for  $\alpha$ -alkoxyhydroperoxides [2]. HR-MS spectrum confirmed the formation of **3aa'**.

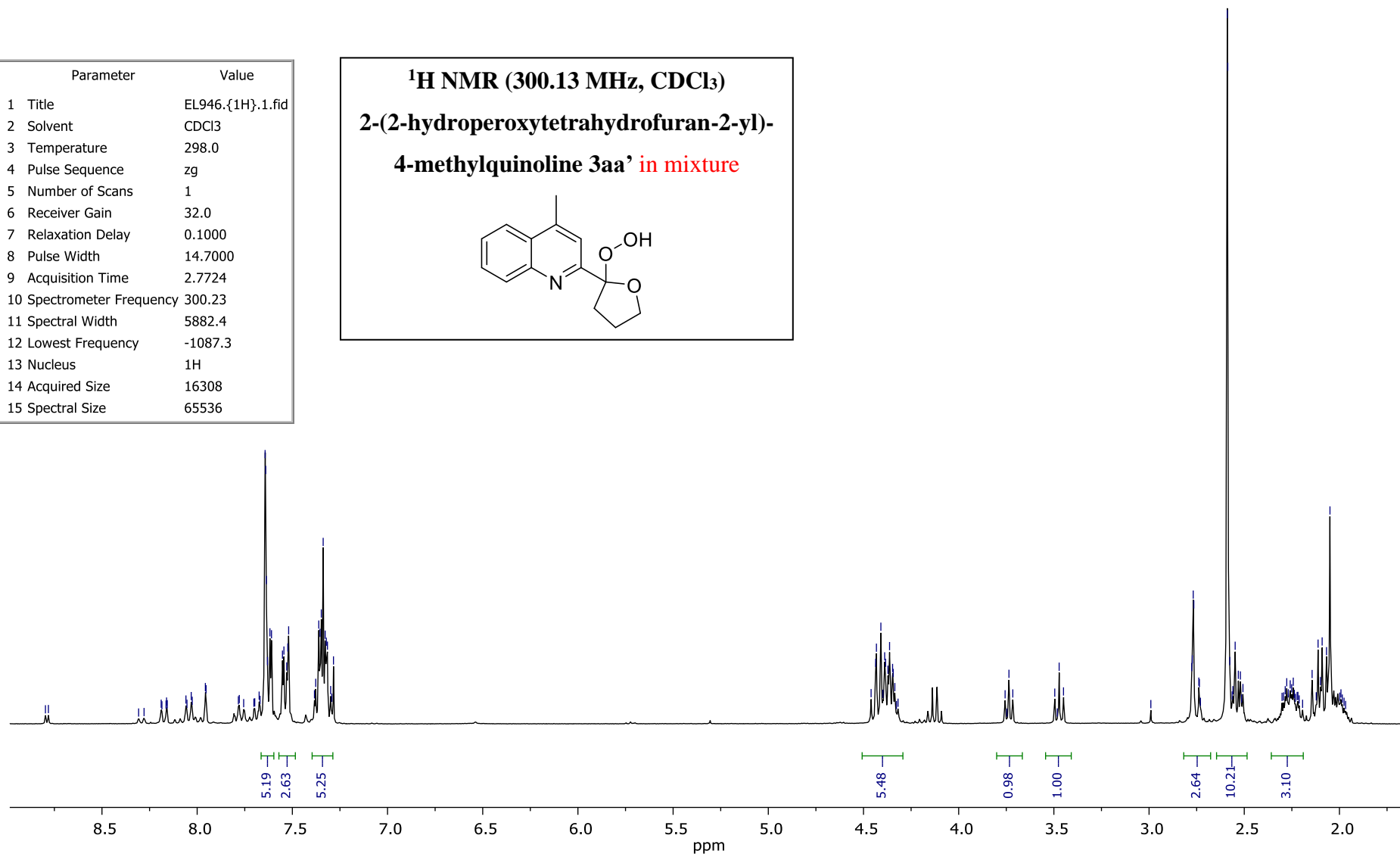
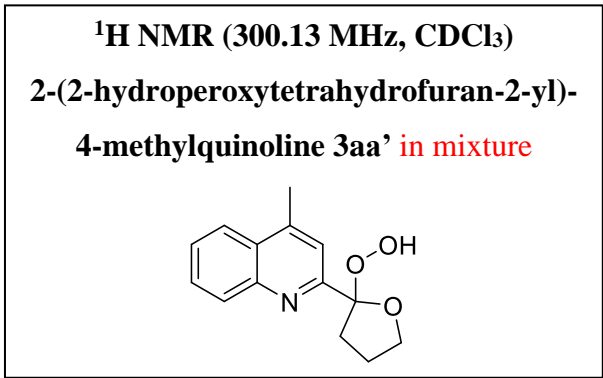


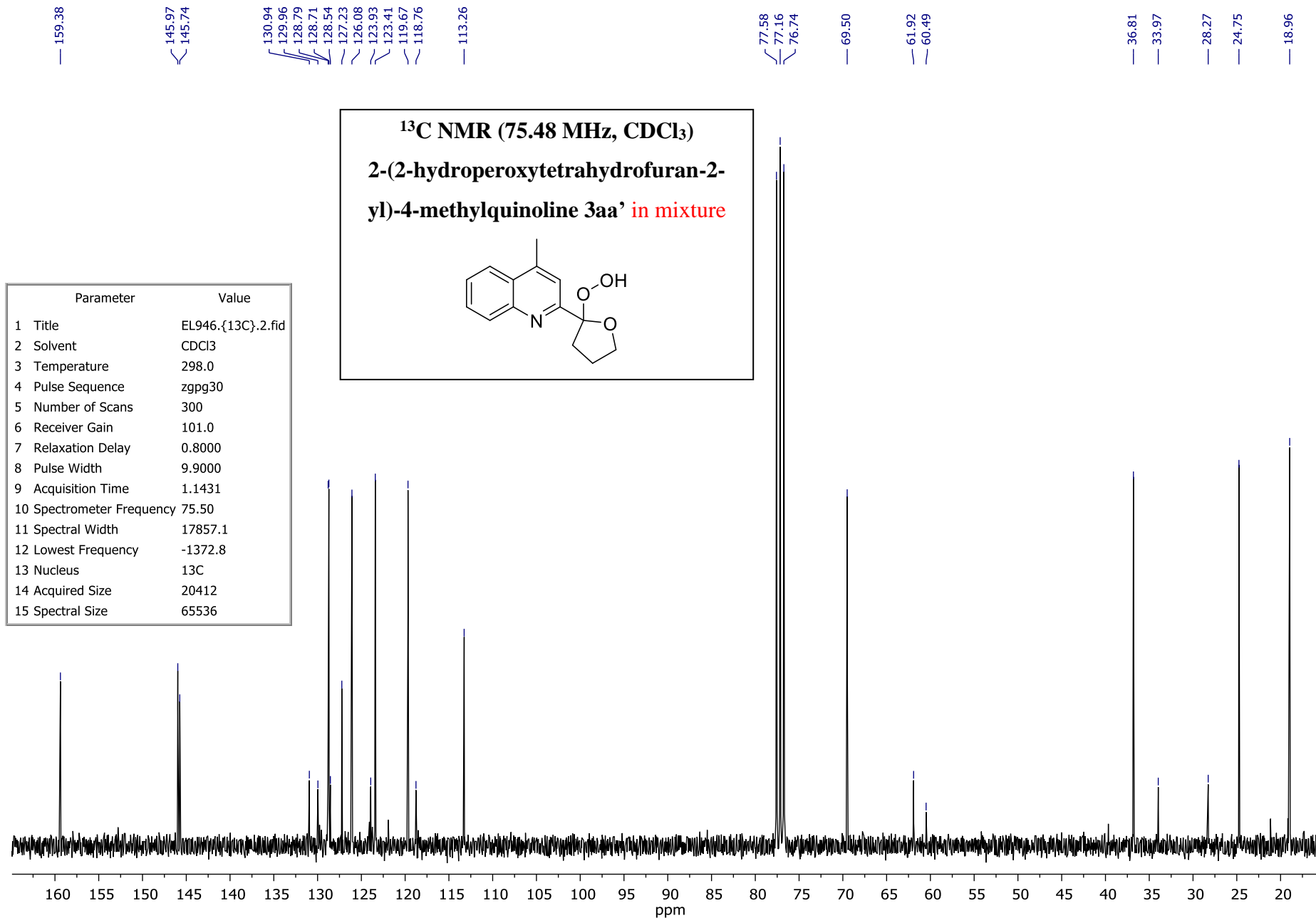
**2-(2-hydroperoxytetrahydrofuran-2-yl)-4-methylquinoline 3aa'**.  $^{13}\text{C}\{^1\text{H}\}$ NMR (75.48 MHz,  $\text{CDCl}_3$ )  $\delta$  159.4, 146.0, 145.7, 128.8, 128.7, 127.2, 126.1, 123.4, 119.7, 113.3, 69.5, 36.8, 24.8, 19.0. HR-MS (ESI):  $m/z$  = 246.1125, calcd. for  $\text{C}_{14}\text{H}_{15}\text{NO}_3+\text{H}^+$ : 246.1123.

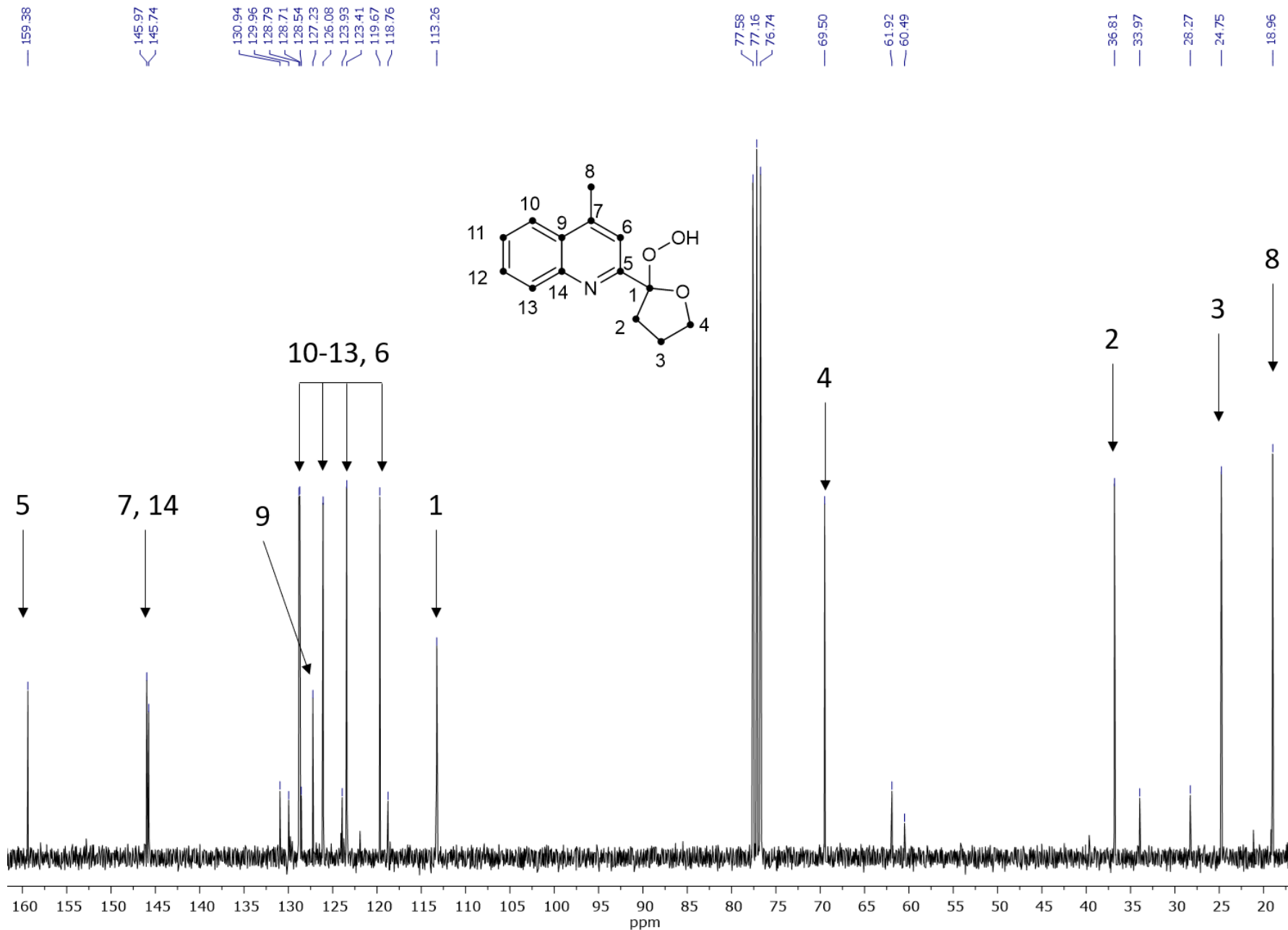
1. McCallum, T.; Jouanno, L.-A.; Cannillo, A.; Barriault, L. Persulfate-Enabled Direct C–H Alkylation of Heteroarenes with Unactivated Ethers. *Synlett* **2016**, 27, 1282–1286, doi:10.1055/s-0035-1561338.
2. Terent'ev, A.O.; Platonov, M.M.; Krylov, I.B.; Chernyshev, V.V.; Nikishin, G.I. Synthesis of 1-Hydroperoxy-1'-Alkoxyperoxides by the Iodine-Catalyzed Reactions of Geminal Bishydroperoxides with Acetals or Enol Ethers. *Org. Biomol. Chem.* **2008**, 6, 4435, doi:10.1039/b809661a.



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| 5 Number of Scans         | 1                |
| 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           |
| 11 Spectral Width         | 5882.4           |
| 12 Lowest Frequency       | -1087.3          |
| 13 Nucleus                | 1H               |
| 14 Acquired Size          | 16308            |
| 15 Spectral Size          | 65536            |









## Display Report

### Analysis Info

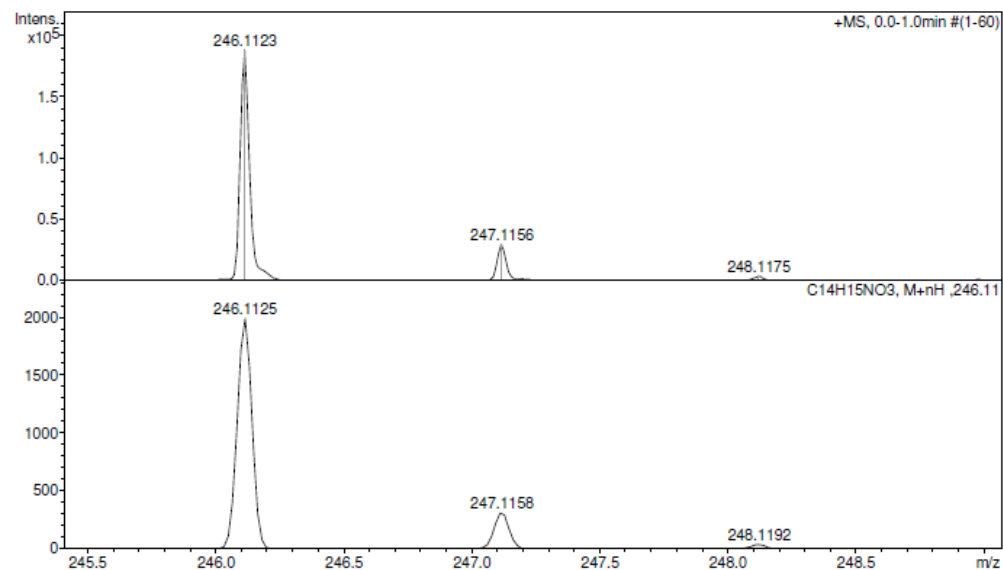
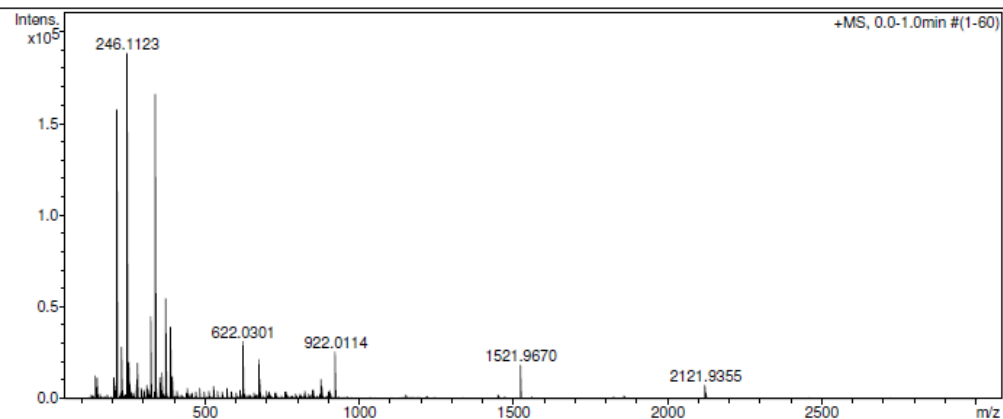
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Comment CH3CN 100 %, dil. >2000, calibrant added

Acquisition Date 14.10.2022 16:08:12

Operator BDAL@DE  
Instrument / Ser# micrOTOF 10248

### Acquisition Parameter

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|-------------|------------|----------------------|----------|------------------|-----------|
| Source Type | ESI        | Ion Polarity         | Positive | Set Nebulizer    | 0.4 Bar   |
| Focus       | Not active |                      |          | Set Dry Heater   | 180 °C    |
| Scan Begin  | 50 m/z     | Set Capillary        | 4500 V   | Set Dry Gas      | 4.0 l/min |
| Scan End    | 3000 m/z   | Set End Plate Offset | -500 V   | Set Divert Valve | Waste     |



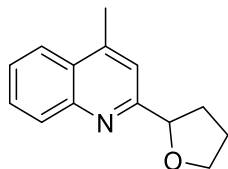
# Spectral data of the Minisci reaction products

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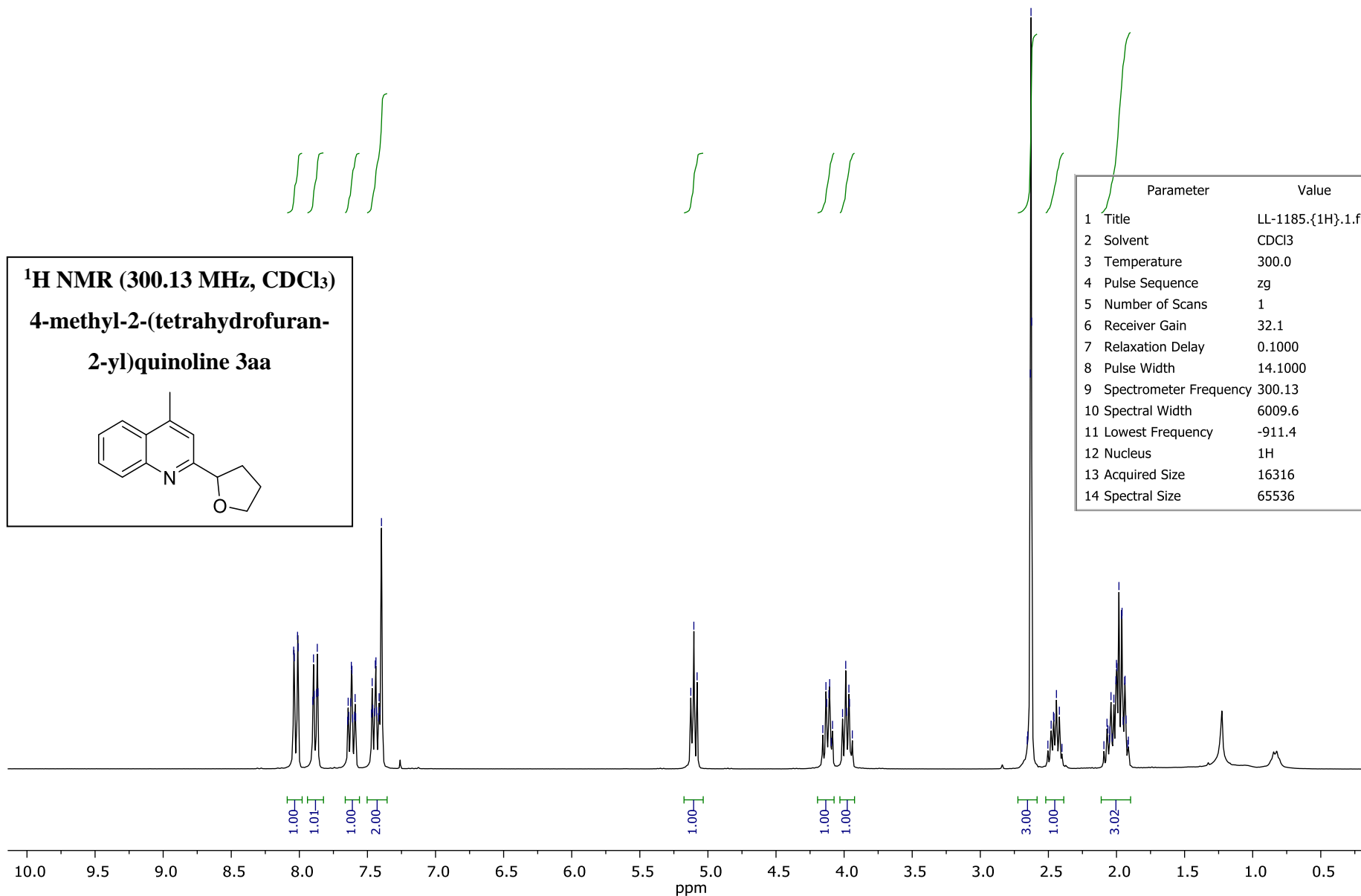
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3.96  
3.94  
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2.03  
2.02  
2.00  
2.00  
1.98  
1.96  
1.95  
1.94  
1.93  
1.91

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

**4-methyl-2-(tetrahydrofuran-2-yl)quinoline 3aa**



| Parameter                | Value              |
|--------------------------|--------------------|
| 1 Title                  | LL-1185.{1H}.1.fid |
| 2 Solvent                | CDCl <sub>3</sub>  |
| 3 Temperature            | 300.0              |
| 4 Pulse Sequence         | zg                 |
| 5 Number of Scans        | 1                  |
| 6 Receiver Gain          | 32.1               |
| 7 Relaxation Delay       | 0.1000             |
| 8 Pulse Width            | 14.1000            |
| 9 Spectrometer Frequency | 300.13             |
| 10 Spectral Width        | 6009.6             |
| 11 Lowest Frequency      | -911.4             |
| 12 Nucleus               | <sup>1</sup> H     |
| 13 Acquired Size         | 16316              |
| 14 Spectral Size         | 65536              |



LL-1185.{13C}.2.fid  
/TERN LL-1185 Terentyev-04417

— 162.98

— 147.27

— 144.79

— 129.49

— 129.01

— 127.36

— 125.72

— 123.59

— 118.55

— 81.99

— 77.59

— 77.16

— 76.74

— 69.13

— 33.19

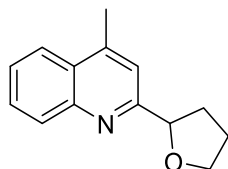
— 25.90

— 18.78

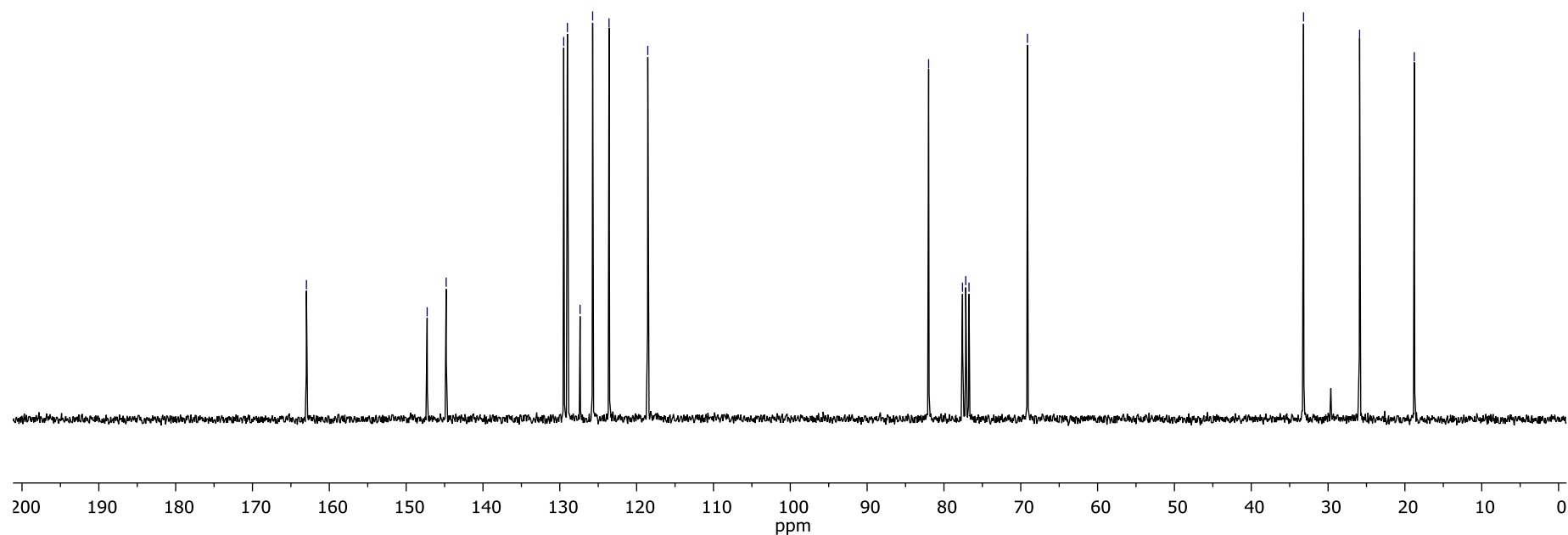
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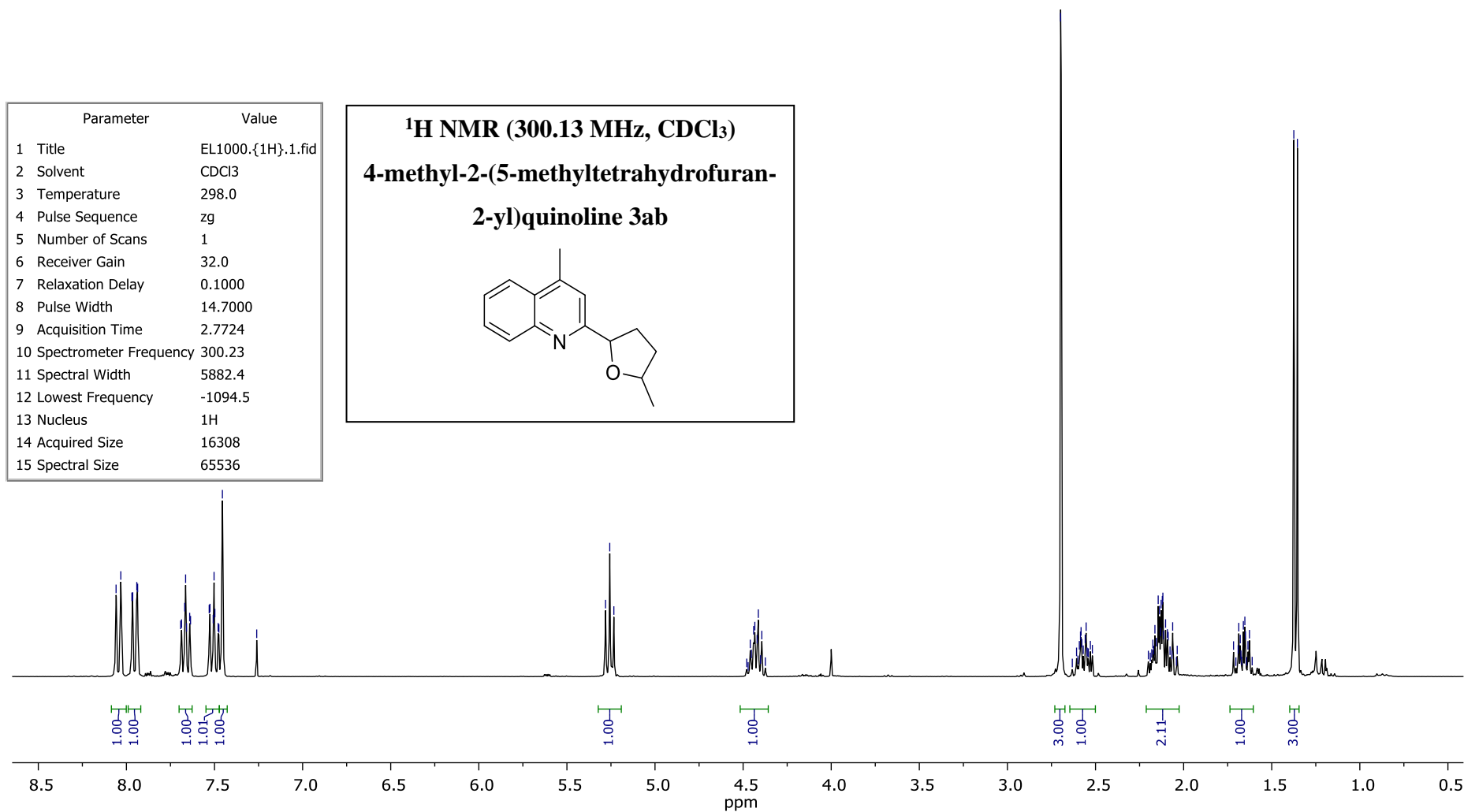
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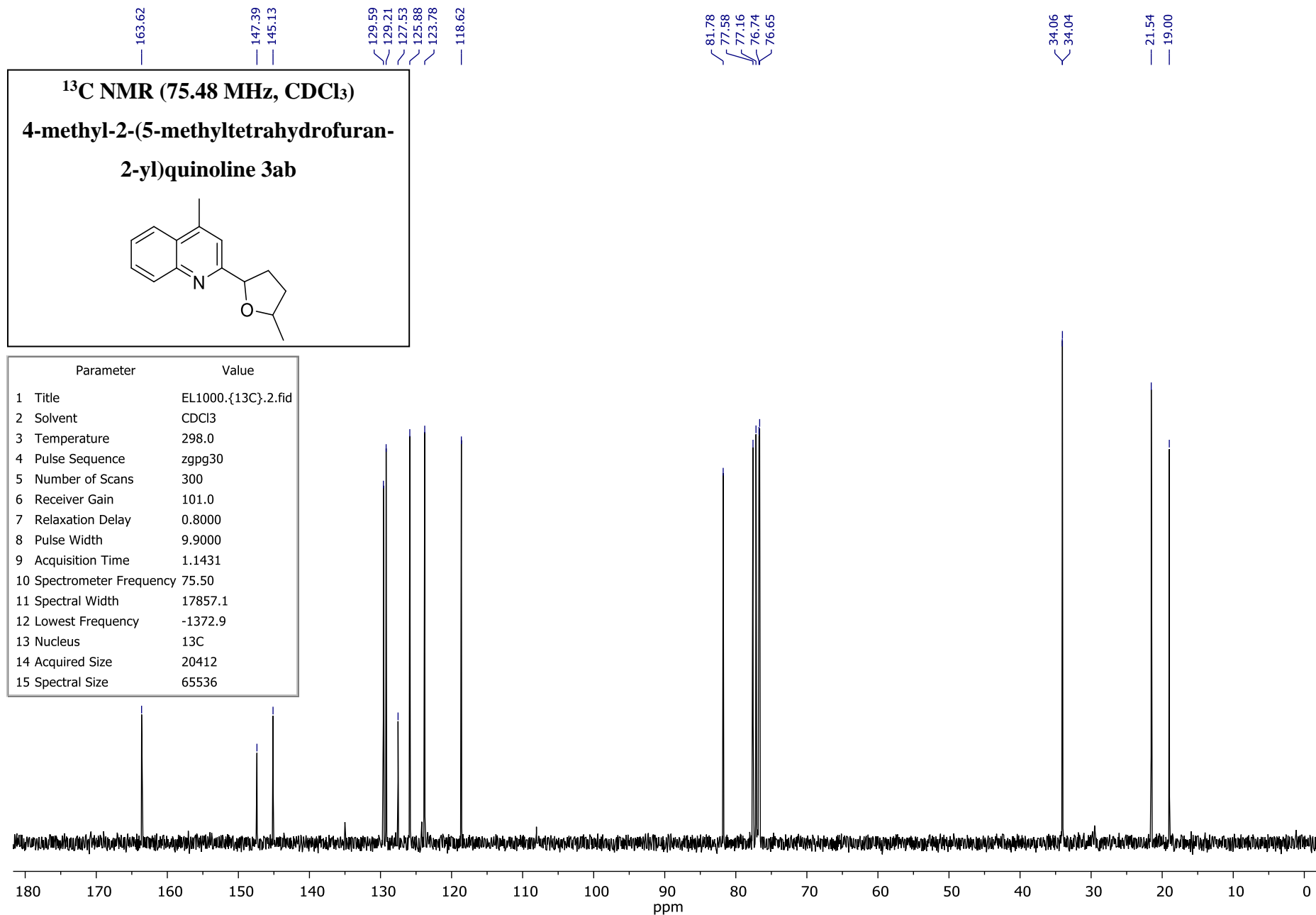
**2-yl)quinoline 3aa**



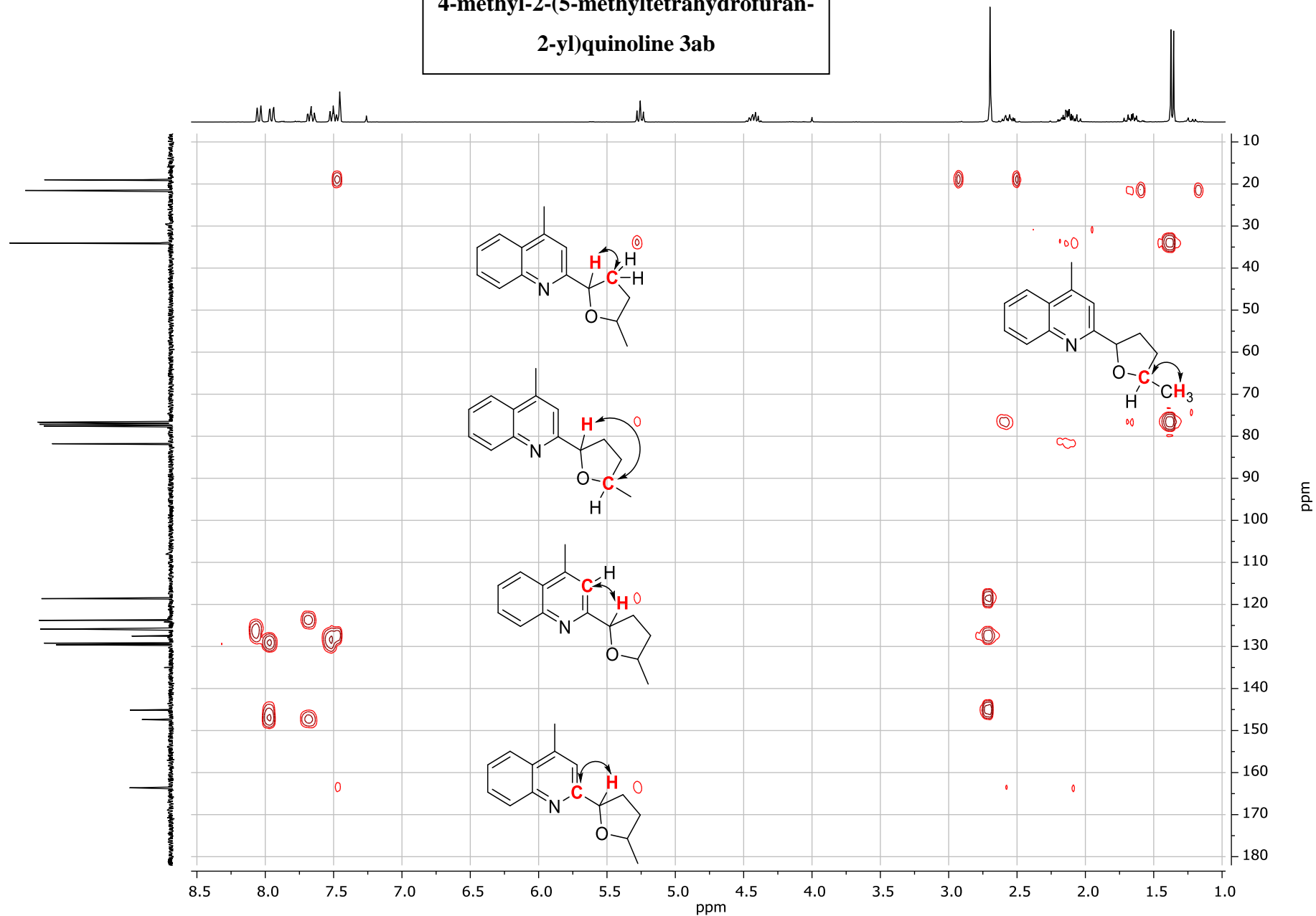
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| 3  | Temperature            | 300.0               |
| 4  | Pulse Sequence         | zgpg30              |
| 5  | Number of Scans        | 80                  |
| 6  | Receiver Gain          | 214.2               |
| 7  | Relaxation Delay       | 0.8000              |
| 8  | Pulse Width            | 9.3000              |
| 9  | Spectrometer Frequency | 75.48               |
| 10 | Spectral Width         | 18028.8             |
| 11 | Lowest Frequency       | -1472.6             |
| 12 | Nucleus                | $^{13}\text{C}$     |
| 13 | Acquired Size          | 16316               |
| 14 | Spectral Size          | 65536               |



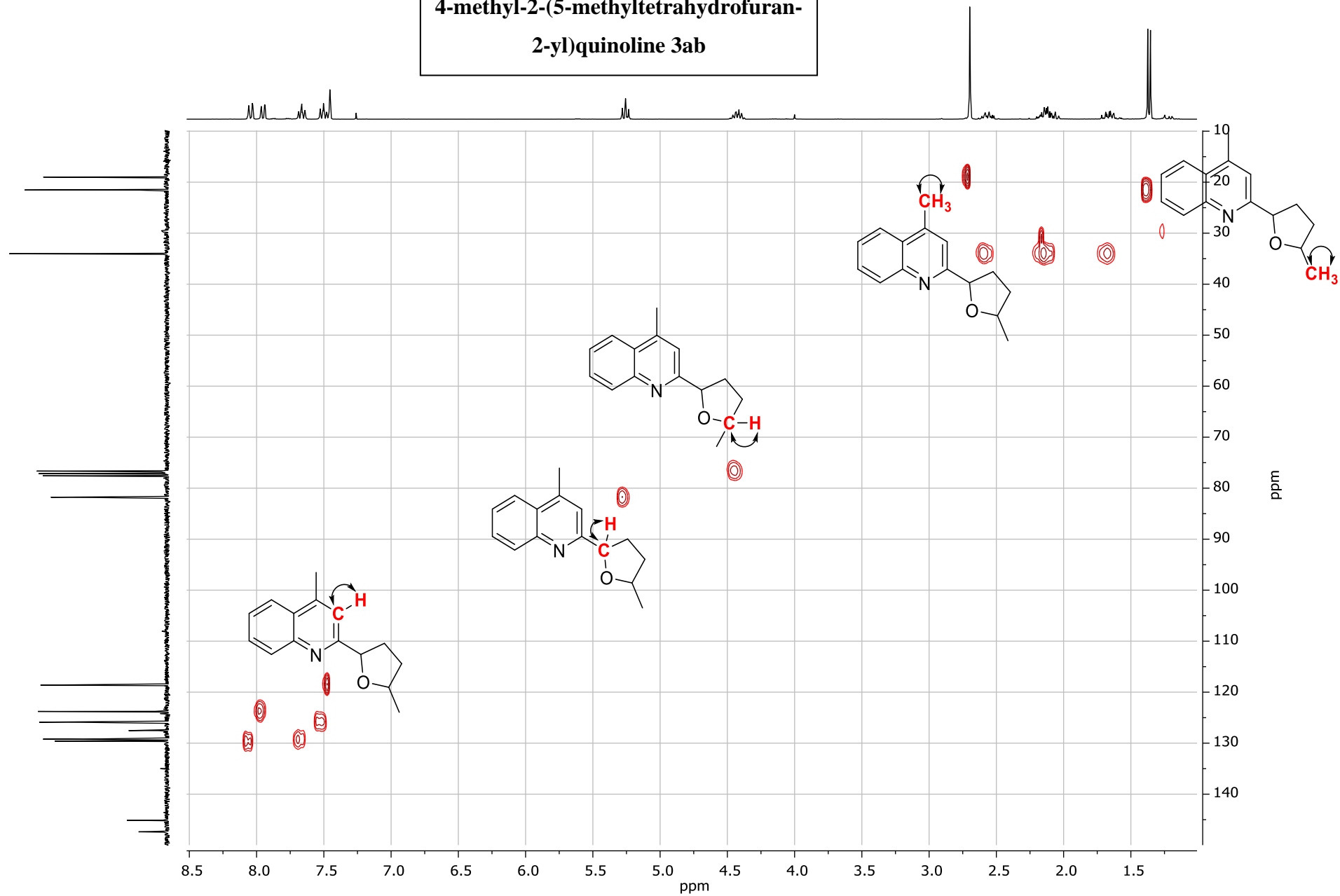
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**$^1\text{H}$ - $^{13}\text{C}$  HMBC**  
**4-methyl-2-(5-methyltetrahydrofuran-2-yl)quinoline 3ab**



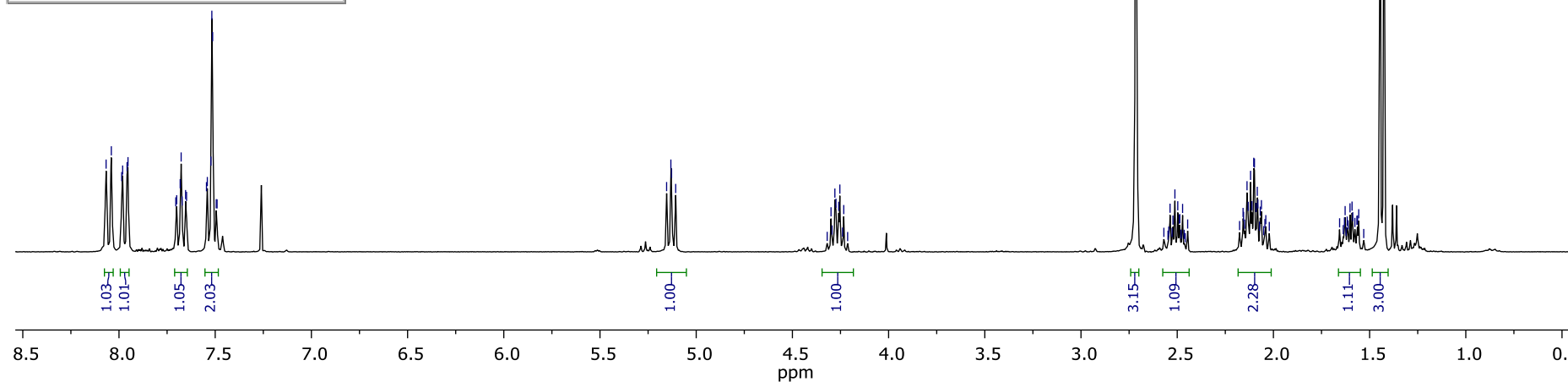
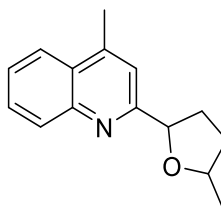
**$^1\text{H}$ - $^{13}\text{C}$  HSQC**  
**4-methyl-2-(5-methyltetrahydrofuran-**  
**2-yl)quinoline 3ab**



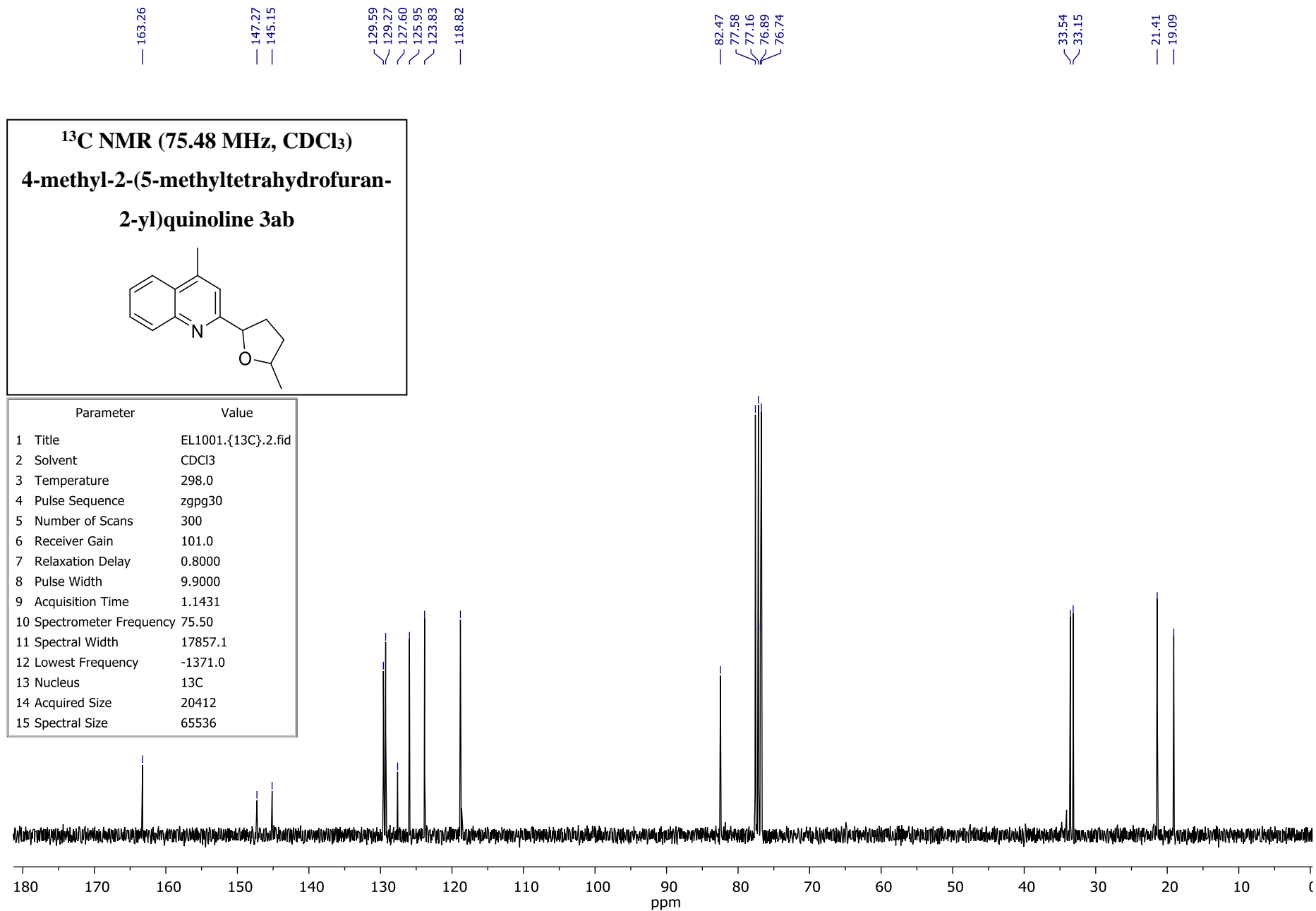


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|---------------------------|-------------------|
| 1 Title                   | EL1001.{1H}.1.fid |
| 2 Solvent                 | CDCl3             |
| 3 Temperature             | 298.0             |
| 4 Pulse Sequence          | zg                |
| 5 Number of Scans         | 1                 |
| 6 Receiver Gain           | 32.0              |
| 7 Relaxation Delay        | 0.1000            |
| 8 Pulse Width             | 14.7000           |
| 9 Acquisition Time        | 2.7724            |
| 10 Spectrometer Frequency | 300.23            |
| 11 Spectral Width         | 5882.4            |
| 12 Lowest Frequency       | -1094.5           |
| 13 Nucleus                | 1H                |
| 14 Acquired Size          | 16308             |
| 15 Spectral Size          | 65536             |

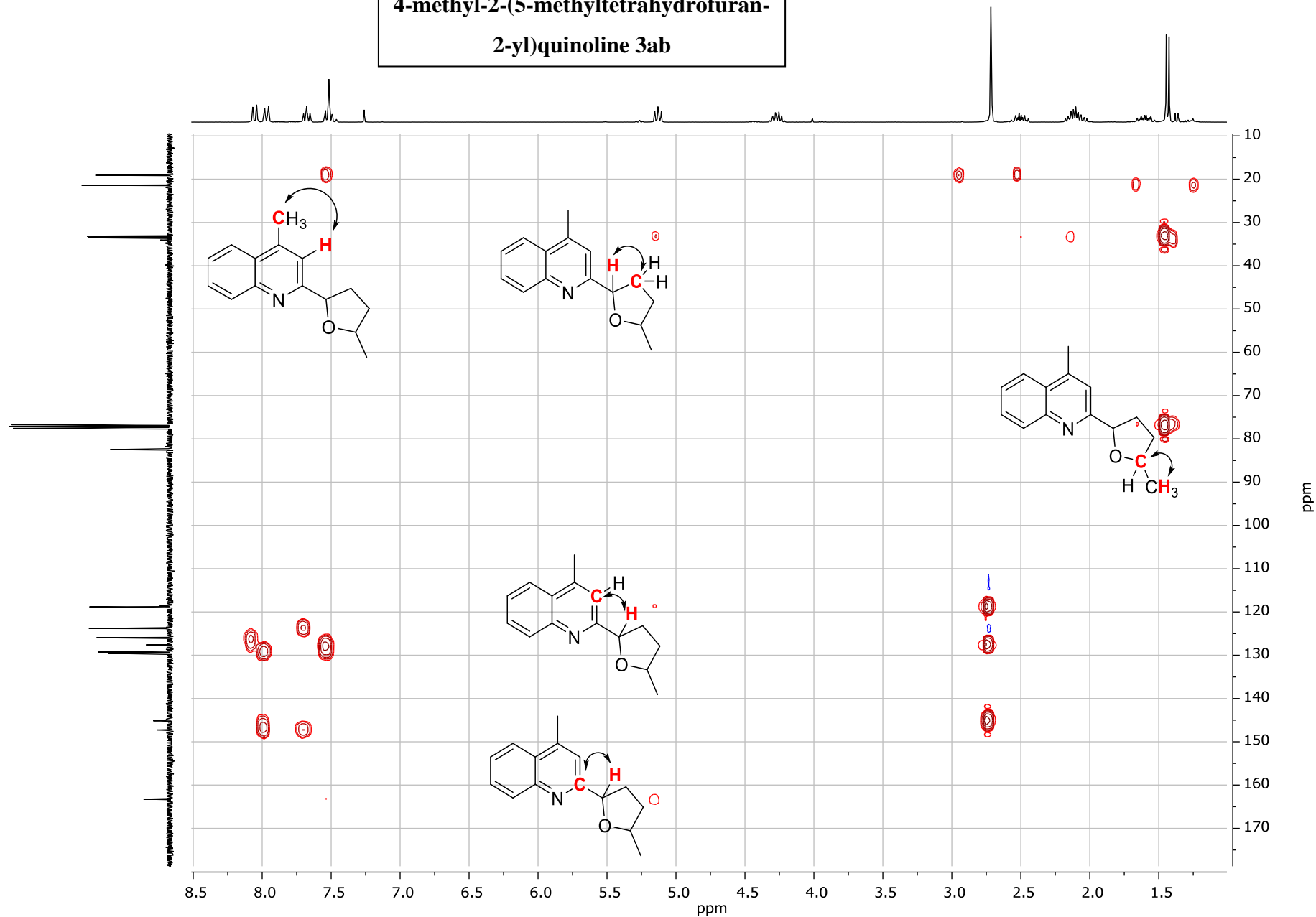
**<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)**  
**4-methyl-2-(5-methyltetrahydrofuran-2-yl)quinoline 3ab**



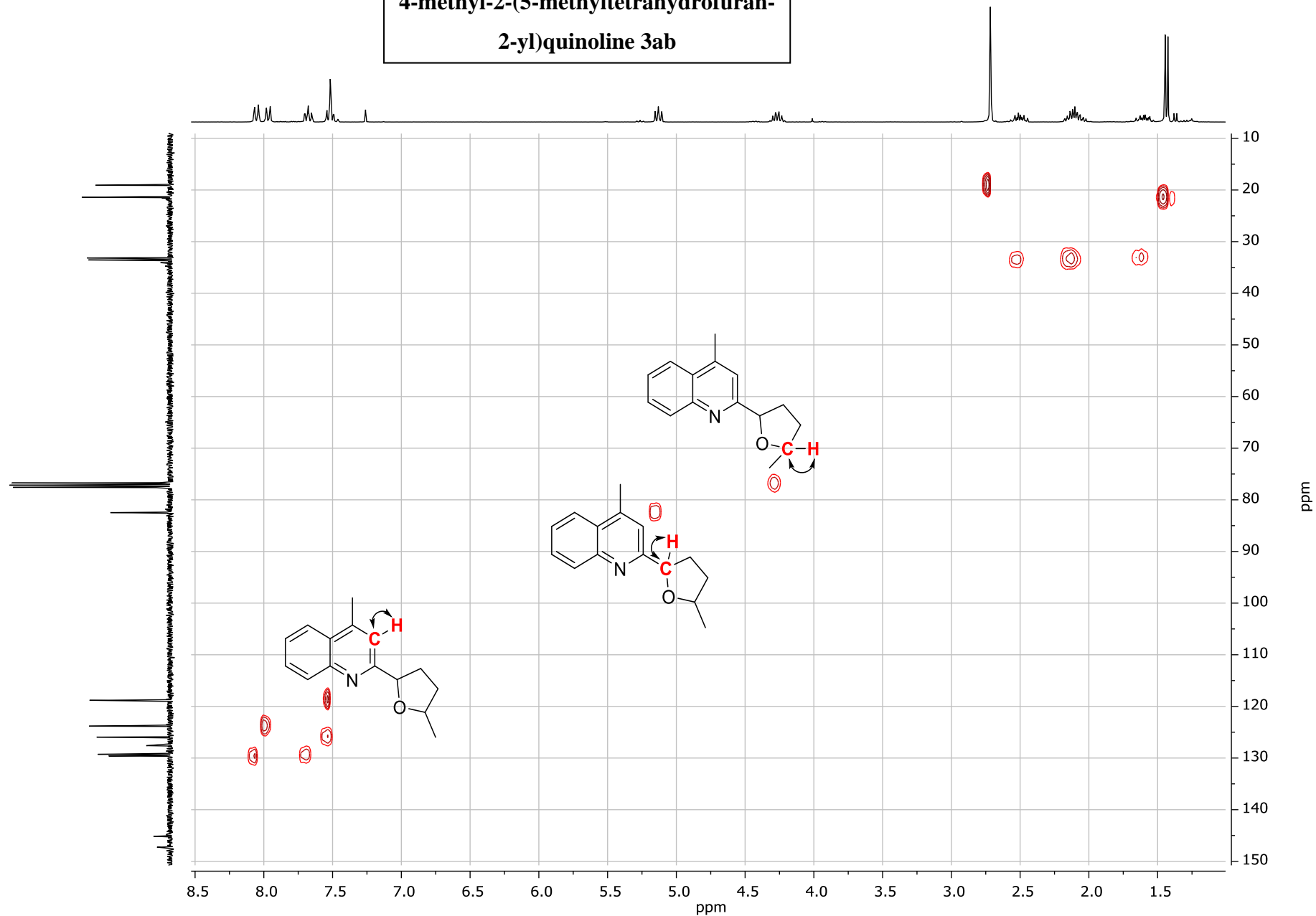


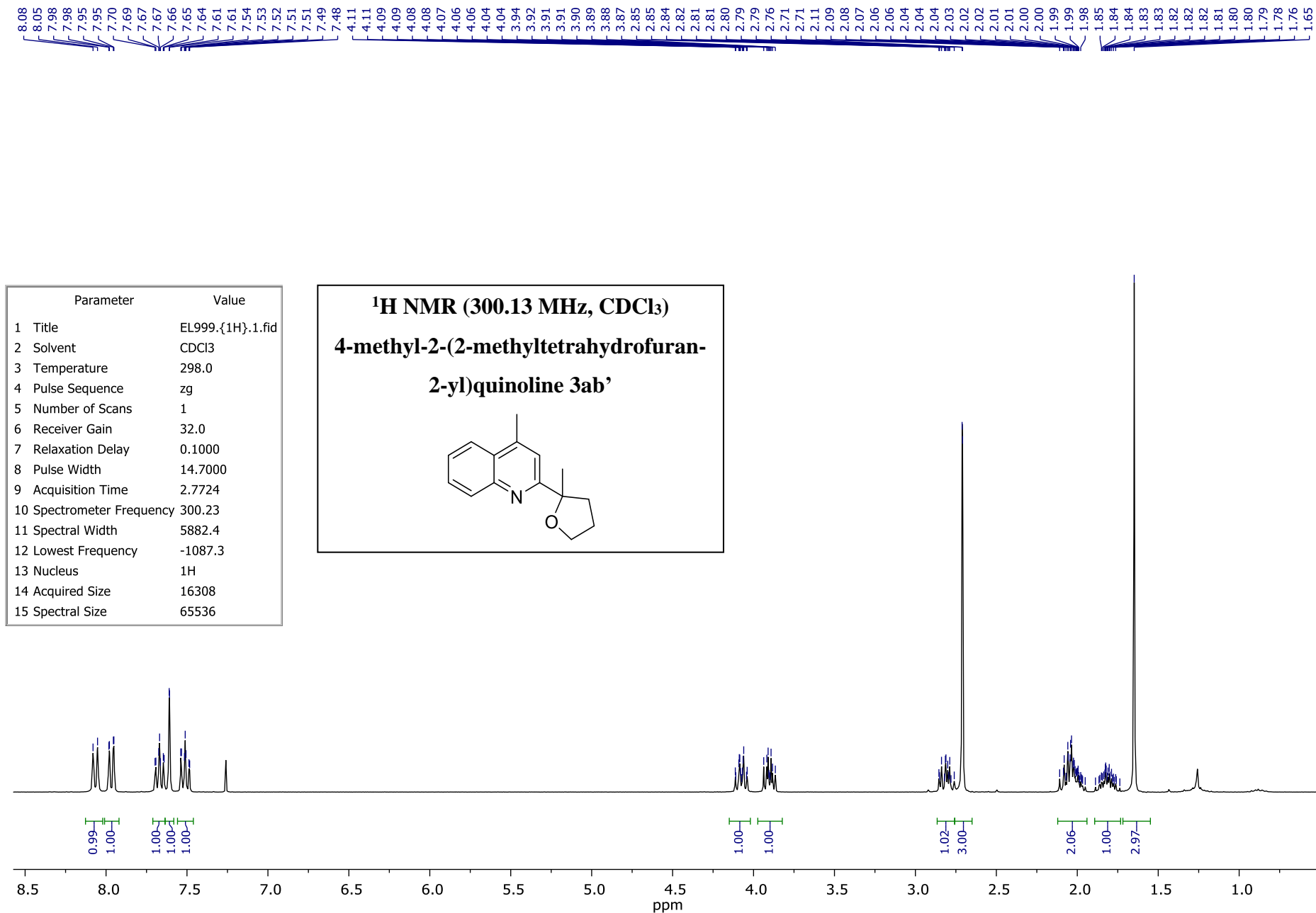


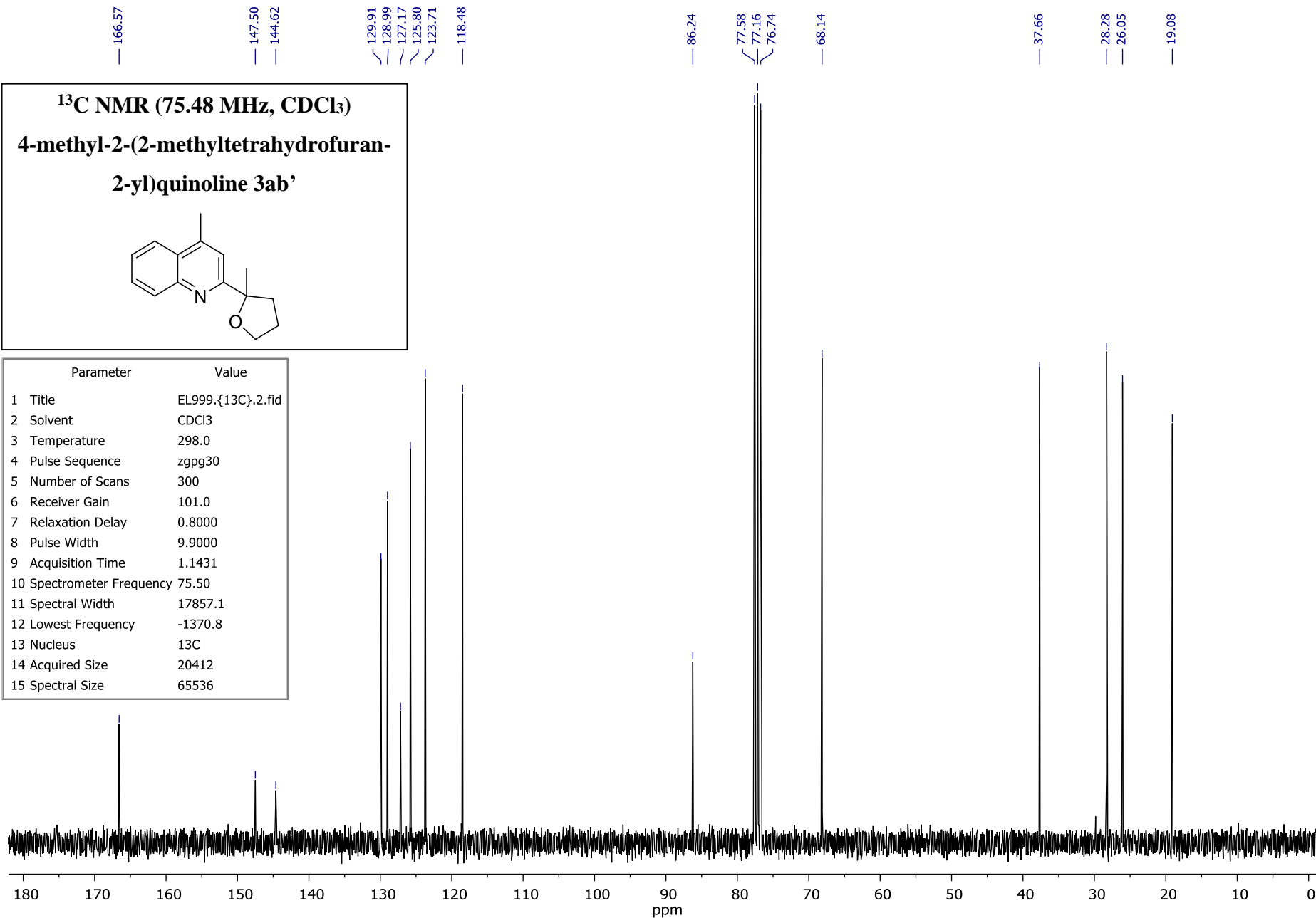
**$^1\text{H}$ - $^{13}\text{C}$  HMBC**  
**4-methyl-2-(5-methyltetrahydrofuran-2-yl)quinoline 3ab**



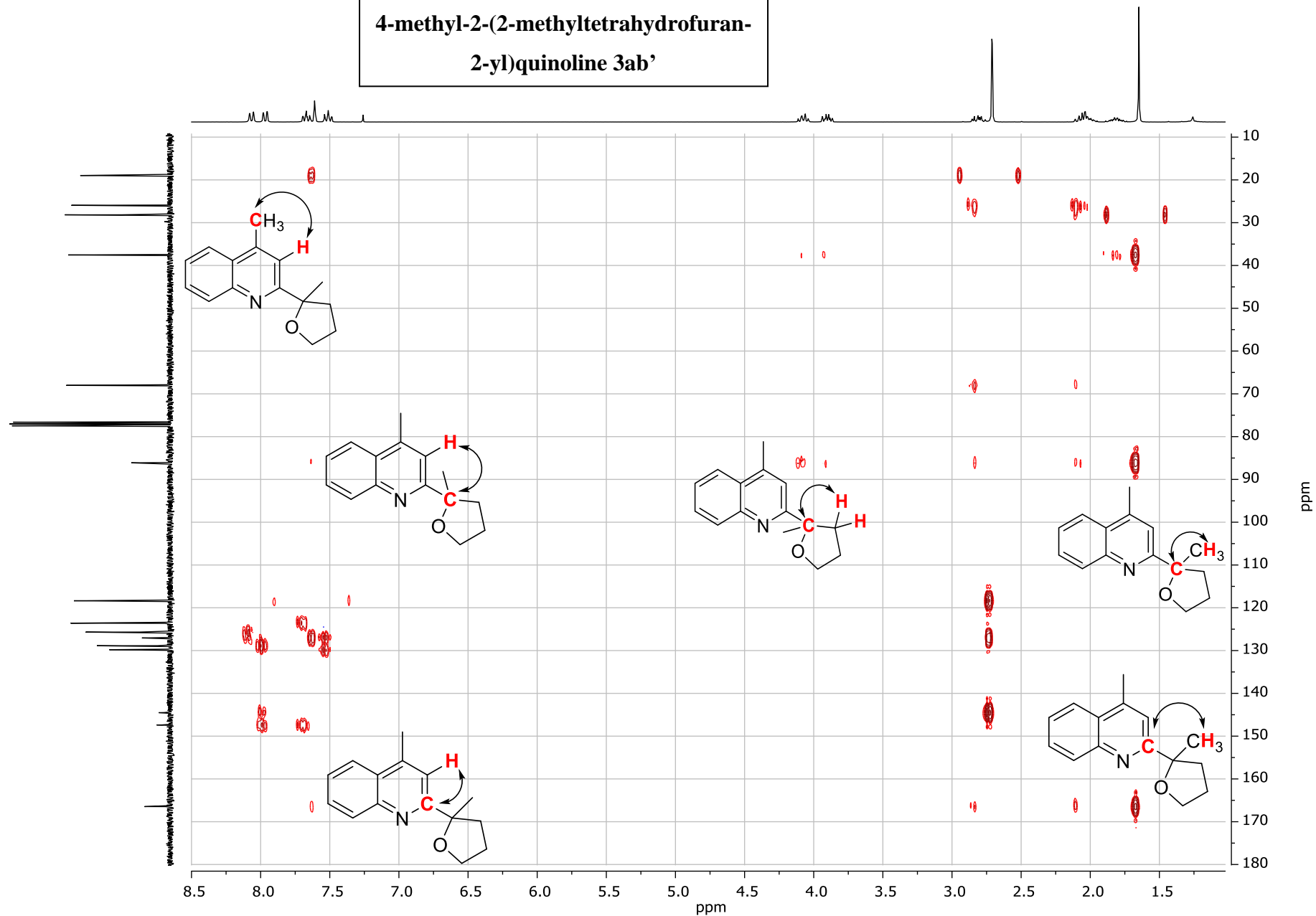
**$^1\text{H}$ - $^{13}\text{C}$  HSQC**  
**4-methyl-2-(5-methyltetrahydrofuran-2-yl)quinoline 3ab**

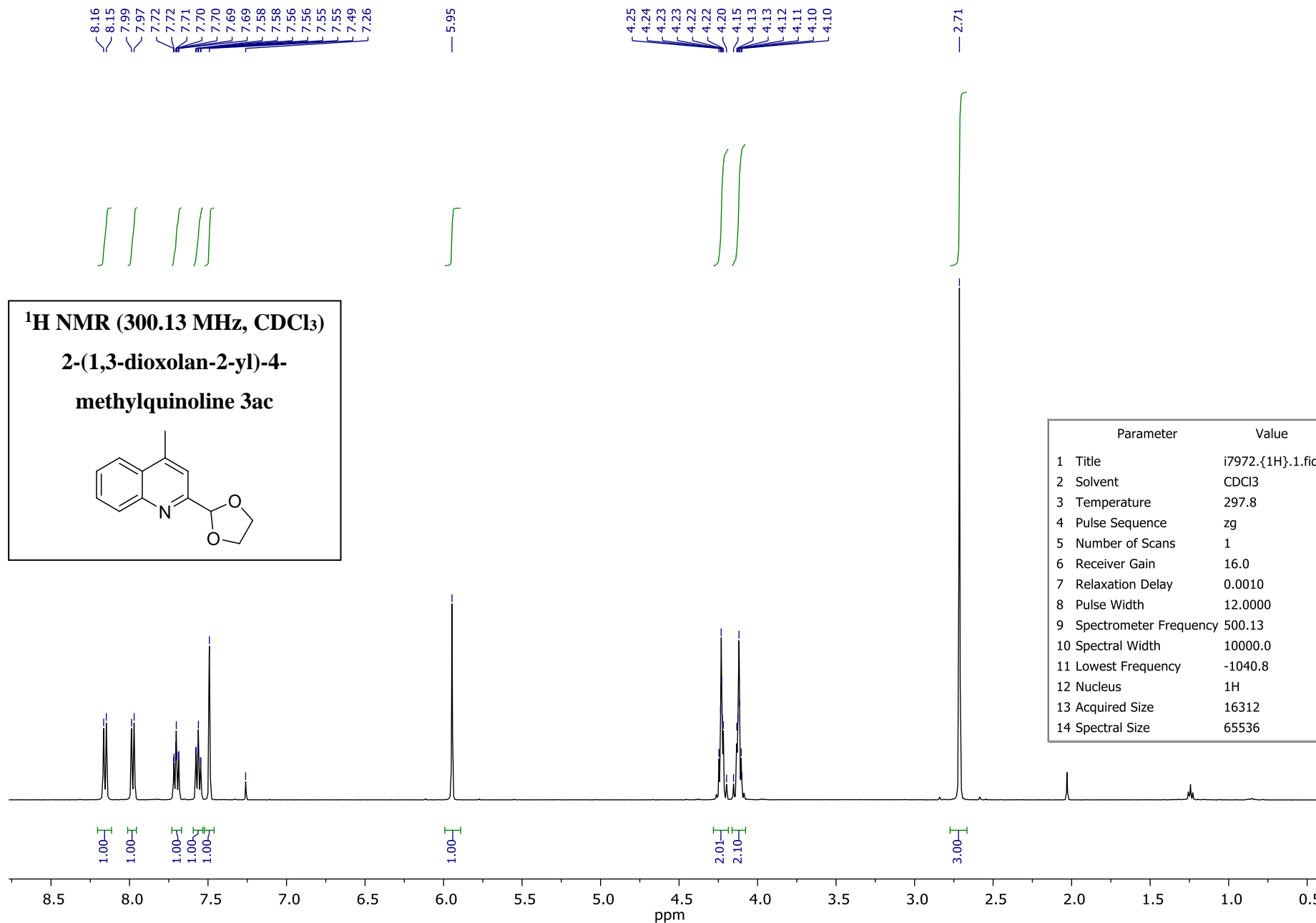






**$^1\text{H}$ - $^{13}\text{C}$  HMBC**  
**4-methyl-2-(2-methyltetrahydrofuran-2-yl)quinoline 3ab'**

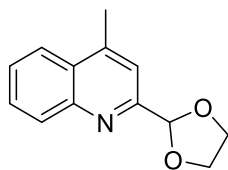




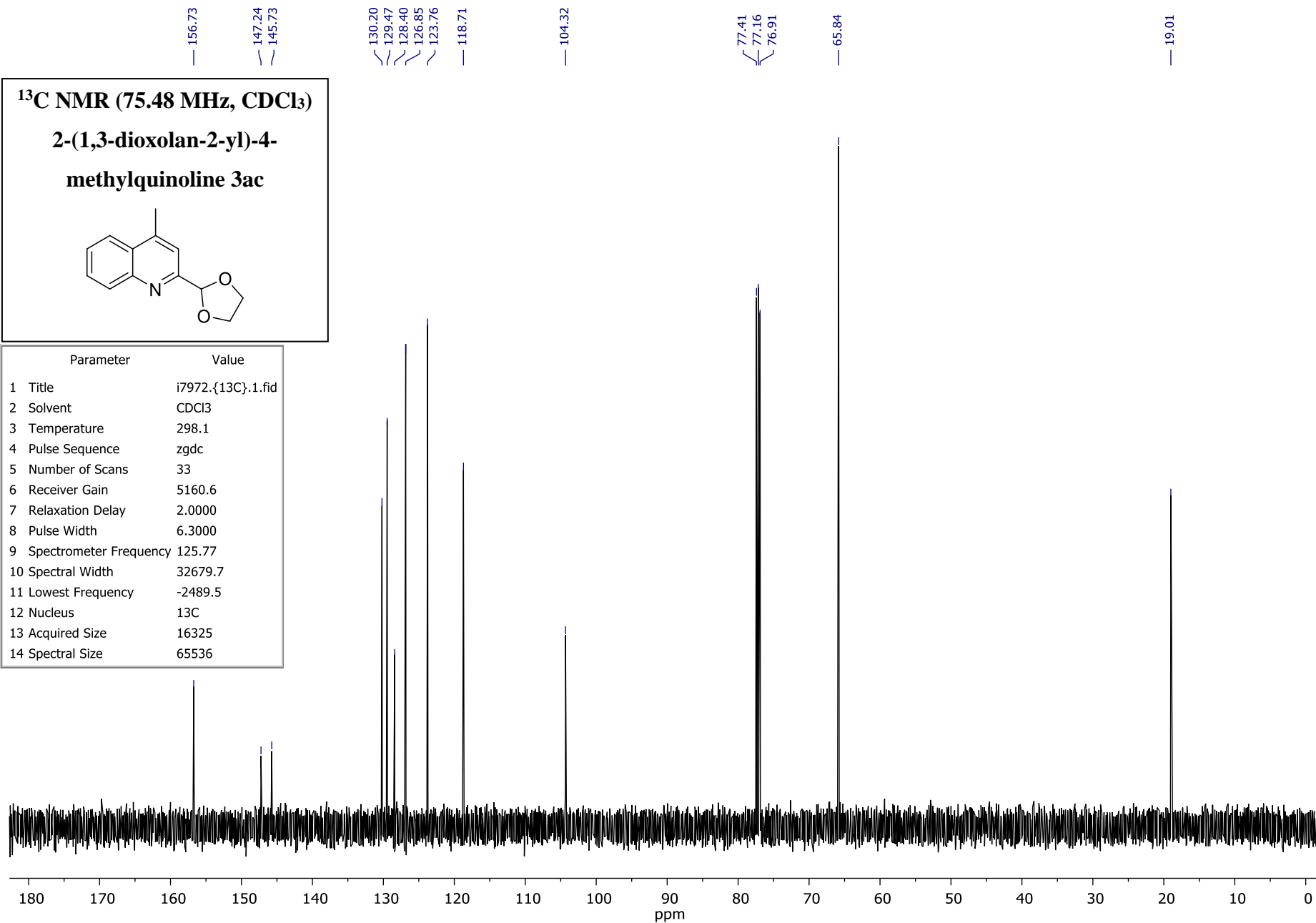
**$^{13}\text{C}$  NMR (75.48 MHz,  $\text{CDCl}_3$ )**

**2-(1,3-dioxolan-2-yl)-4-**

**methylquinoline 3ac**



| Parameter                | Value             |
|--------------------------|-------------------|
| 1 Title                  | i7972.{13C}.1.fid |
| 2 Solvent                | $\text{CDCl}_3$   |
| 3 Temperature            | 298.1             |
| 4 Pulse Sequence         | zgdc              |
| 5 Number of Scans        | 33                |
| 6 Receiver Gain          | 5160.6            |
| 7 Relaxation Delay       | 2.0000            |
| 8 Pulse Width            | 6.3000            |
| 9 Spectrometer Frequency | 125.77            |
| 10 Spectral Width        | 32679.7           |
| 11 Lowest Frequency      | -2489.5           |
| 12 Nucleus               | $^{13}\text{C}$   |
| 13 Acquired Size         | 16325             |
| 14 Spectral Size         | 65536             |





EL106.{1H}.1.fid  
/TERN EL106

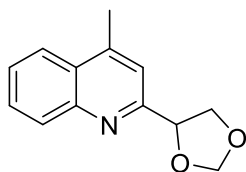
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7.53  
7.48  
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5.32  
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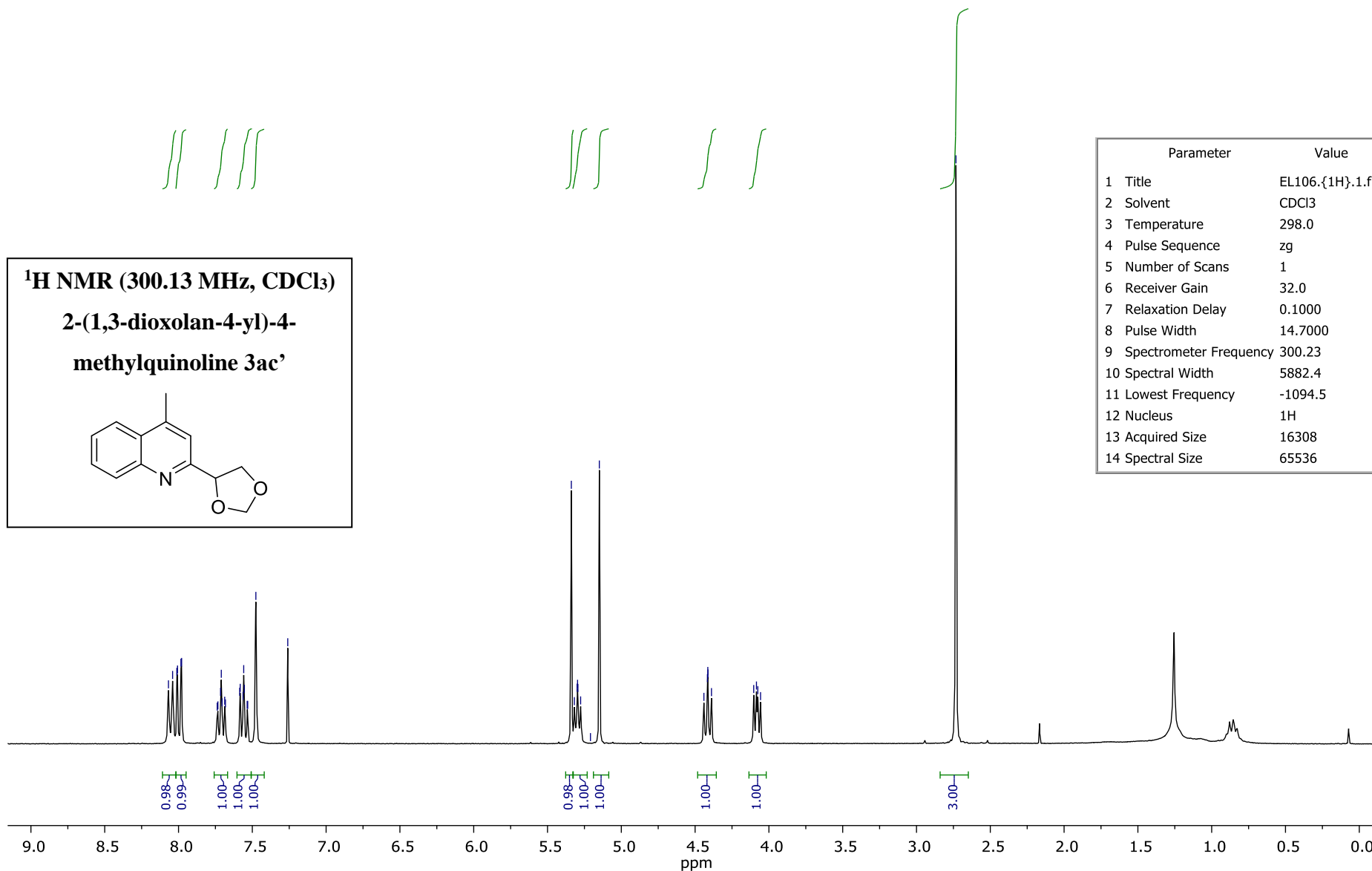
2.73

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

**2-(1,3-dioxolan-4-yl)-4-methylquinoline 3ac'**



| Parameter                | Value             |
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| 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      | -1094.5           |
| 12 Nucleus               | <sup>1</sup> H    |
| 13 Acquired Size         | 16308             |
| 14 Spectral Size         | 65536             |



EL106-1.{13C}.1.fid  
/TERN EL106-1

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123.94  
118.76

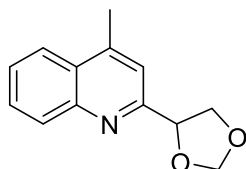
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76.74  
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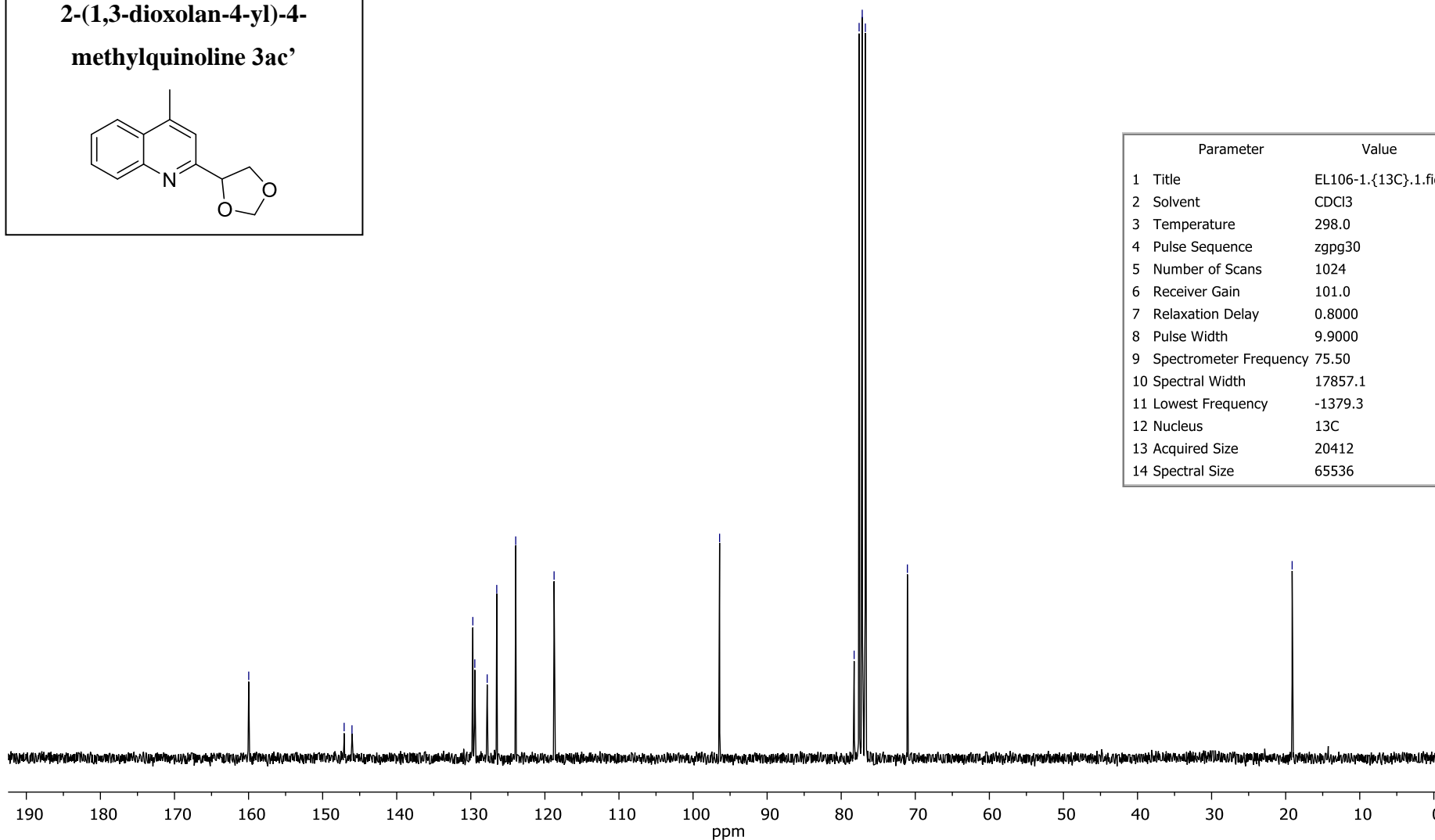
19.13

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

2-(1,3-dioxolan-4-yl)-4-  
methylquinoline 3ac'



| Parameter                | Value               |
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| 3 Temperature            | 298.0               |
| 4 Pulse Sequence         | zgpg30              |
| 5 Number of Scans        | 1024                |
| 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      | -1379.3             |
| 12 Nucleus               | 13C                 |
| 13 Acquired Size         | 20412               |
| 14 Spectral Size         | 65536               |



EL105.{1H}.1.fid  
Avance-300, CDCl<sub>3</sub>

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7.55  
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7.53  
7.47  
7.26

4.94  
4.93  
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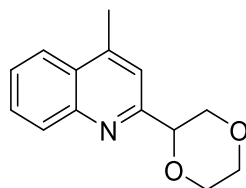
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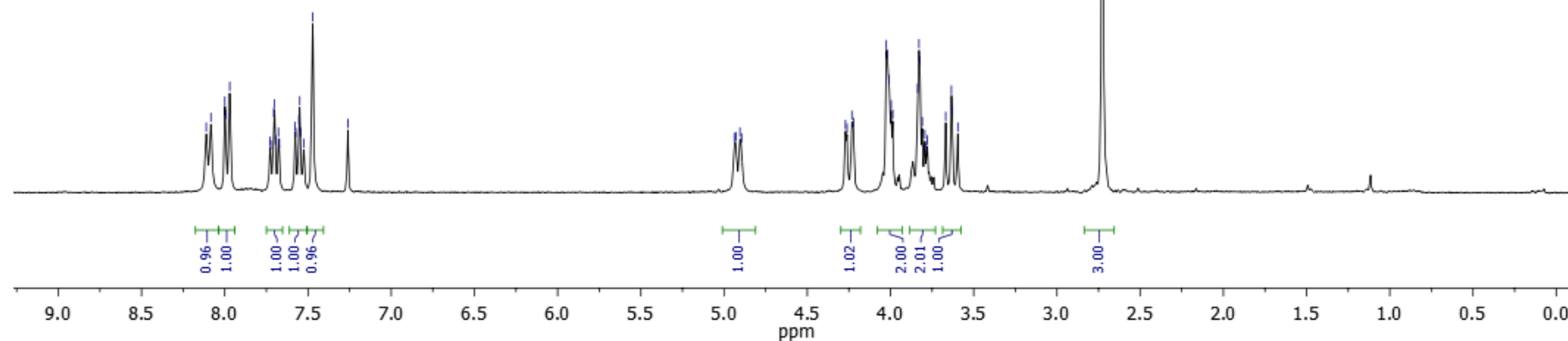
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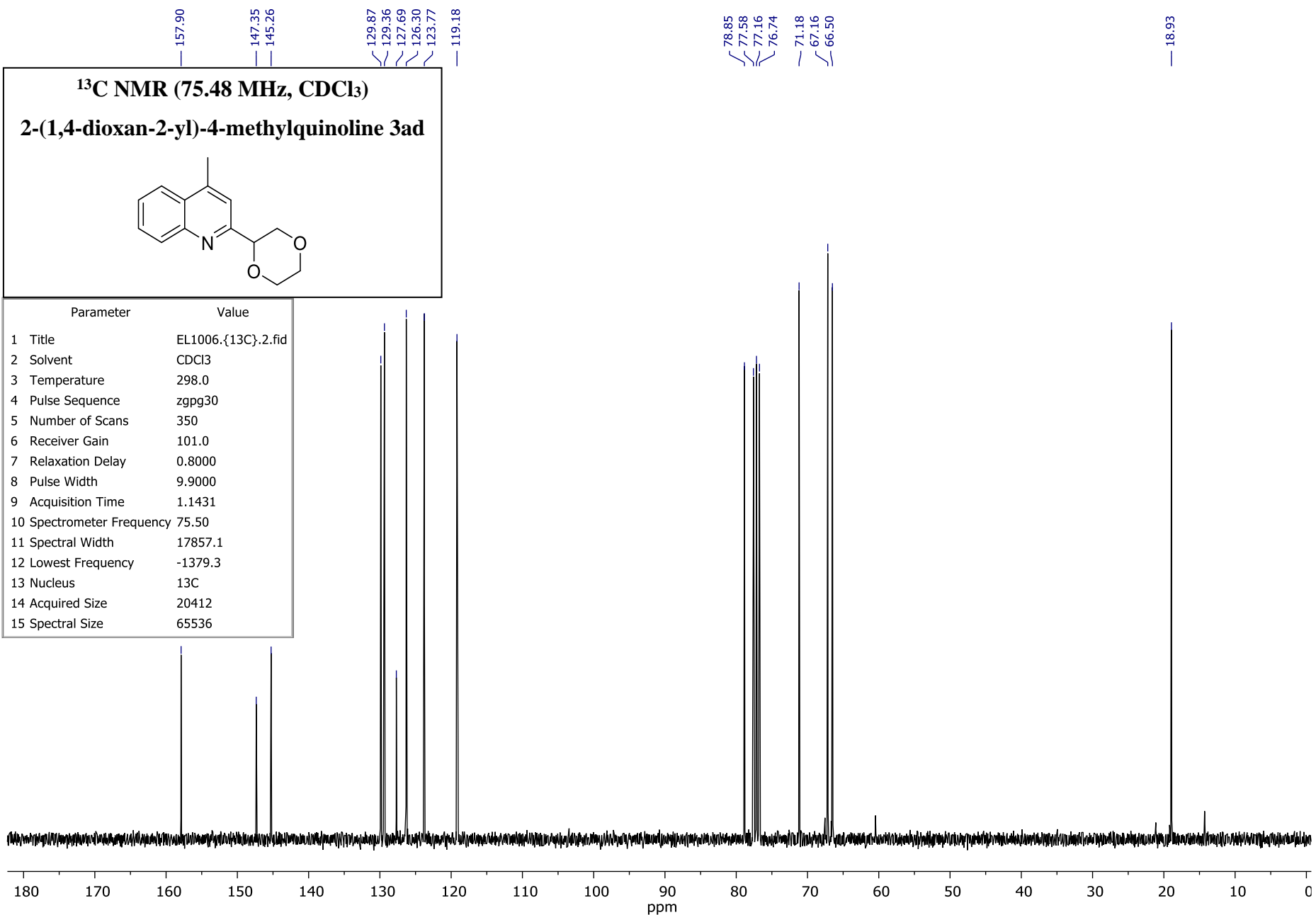
<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)

2-(1,4-dioxan-2-yl)-4-  
methylquinoline 3ad

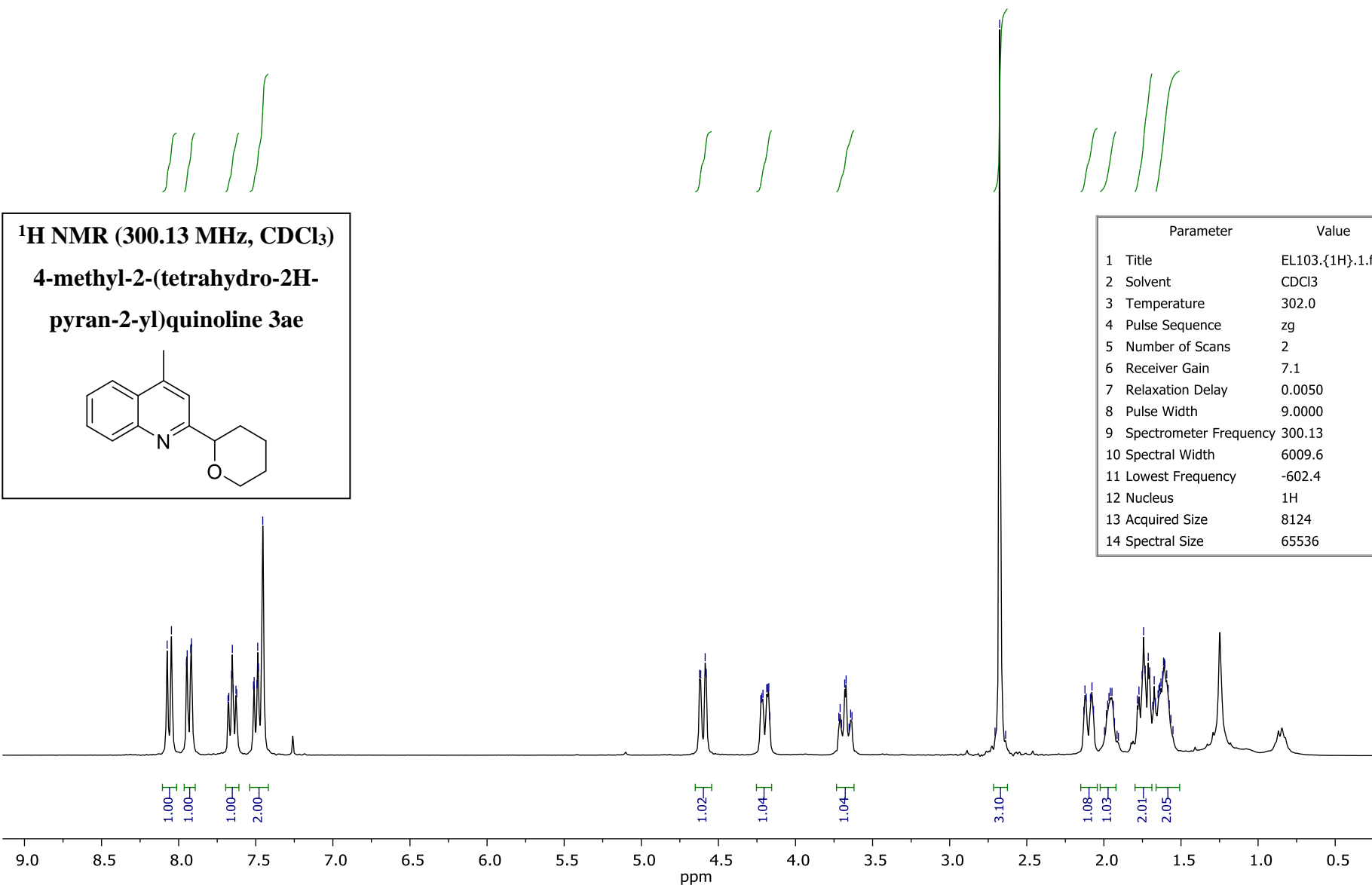


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| 3 Temperature            | 302.0             |
| 4 Pulse Sequence         | zg                |
| 5 Number of Scans        | 2                 |
| 6 Receiver Gain          | 50.8              |
| 7 Relaxation Delay       | 0.0050            |
| 8 Pulse Width            | 9.0000            |
| 9 Spectrometer Frequency | 300.13            |
| 10 Spectral Width        | 6009.6            |
| 11 Lowest Frequency      | -609.9            |
| 12 Nucleus               | <sup>1</sup> H    |
| 13 Acquired Size         | 8124              |
| 14 Spectral Size         | 65536             |





EL103.{1H}.1.fid  
Avance-300, CDCl<sub>3</sub>



| Parameter                | Value             |
|--------------------------|-------------------|
| 1 Title                  | EL103.{1H}.1.fid  |
| 2 Solvent                | CDCl <sub>3</sub> |
| 3 Temperature            | 302.0             |
| 4 Pulse Sequence         | zg                |
| 5 Number of Scans        | 2                 |
| 6 Receiver Gain          | 7.1               |
| 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             |

EL103.{13C}.1.fid  
EL103 Terentyev-04417

— 162.19

— 147.19  
— 145.05

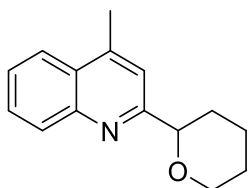
129.67  
129.11  
127.57  
125.89  
123.68  
118.87

81.64  
77.58  
77.16  
76.74  
— 68.94

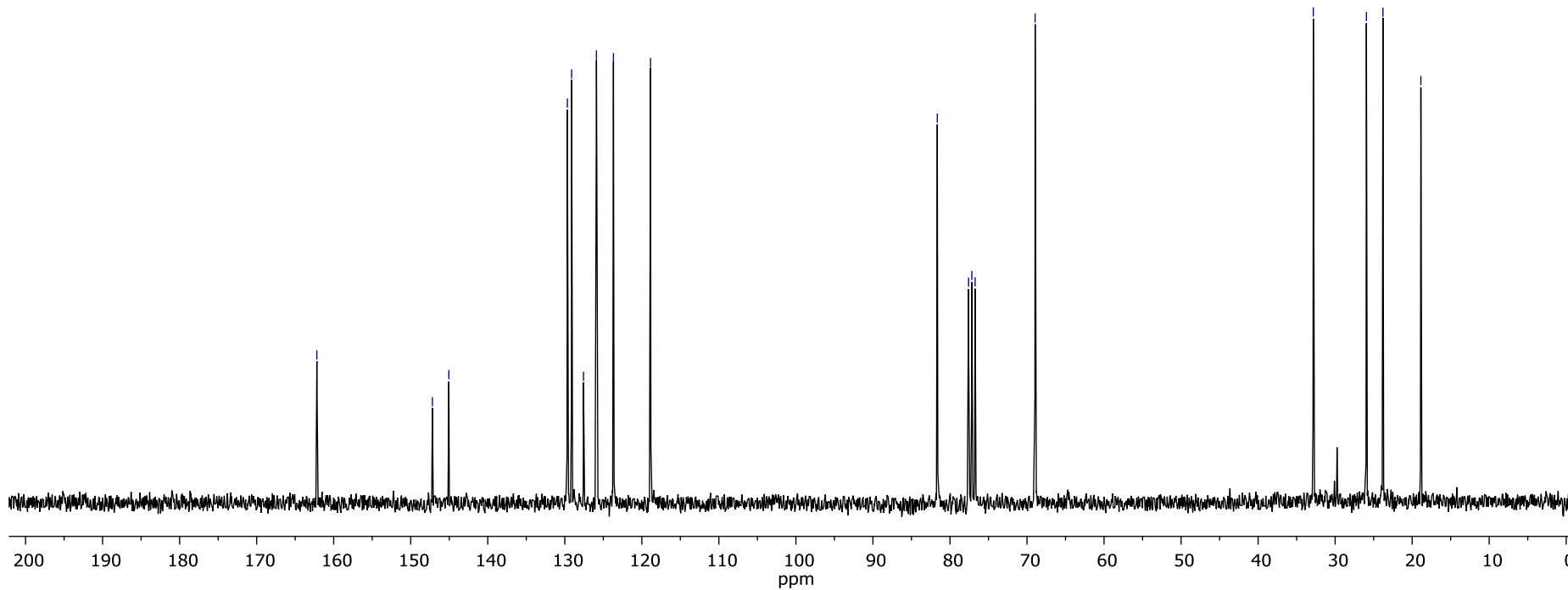
— 32.83  
— 25.94  
— 23.79  
— 18.87

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

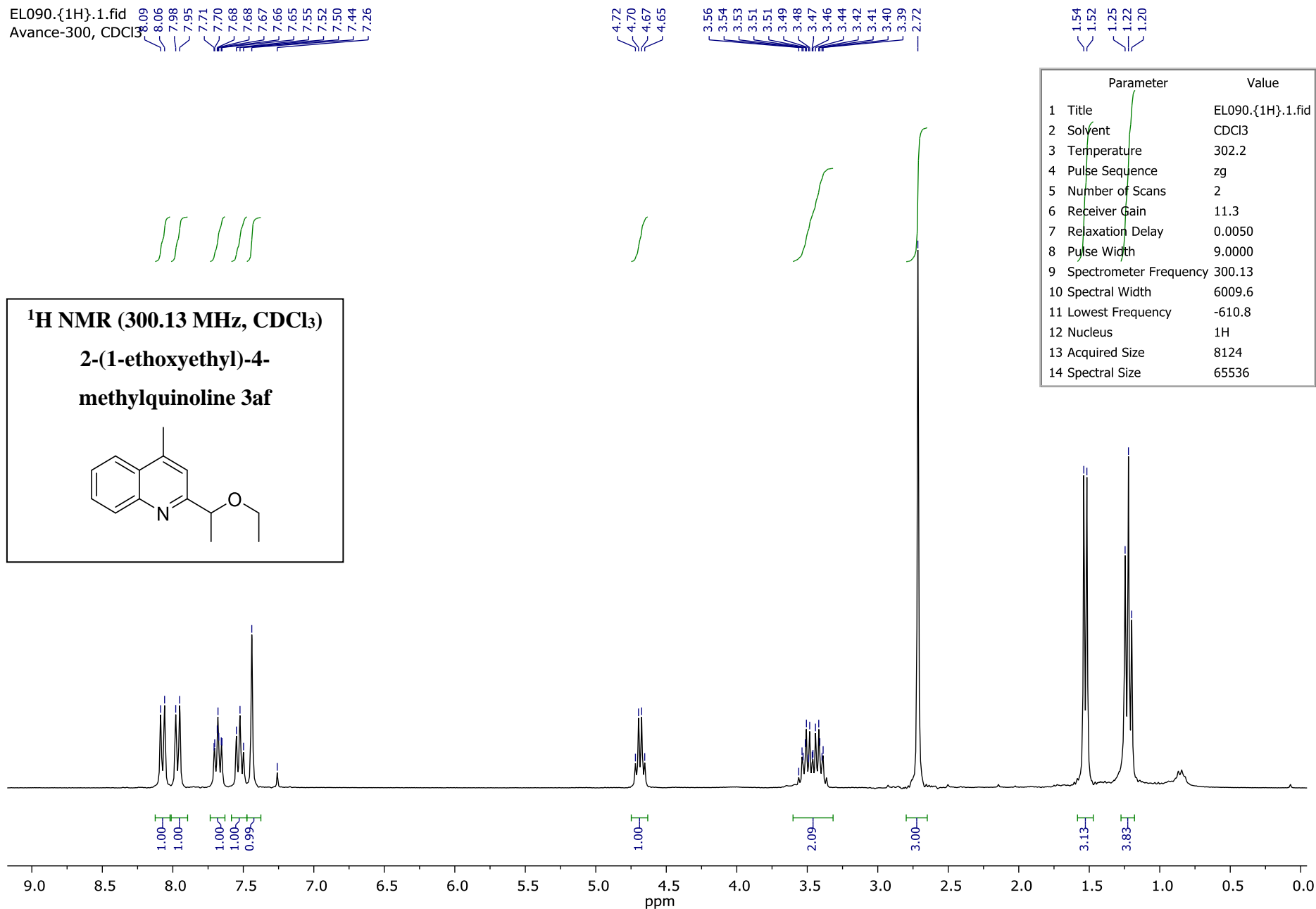
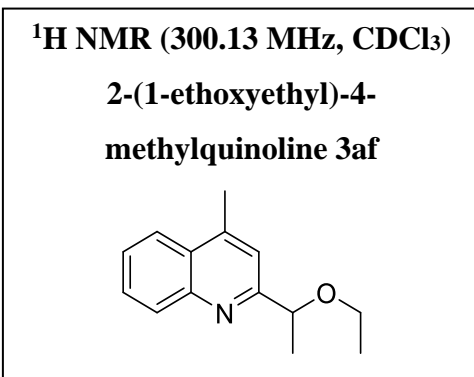
**4-methyl-2-(tetrahydro-2H-  
pyran-2-yl)quinoline 3ae**



|    | Parameter              | Value             |
|----|------------------------|-------------------|
| 1  | Title                  | EL103.{13C}.1.fid |
| 2  | Solvent                | CDCl3             |
| 3  | Temperature            | 300.0             |
| 4  | Pulse Sequence         | zgpg30            |
| 5  | Number of Scans        | 100               |
| 6  | Receiver Gain          | 214.2             |
| 7  | Relaxation Delay       | 0.8000            |
| 8  | Pulse Width            | 9.3000            |
| 9  | Spectrometer Frequency | 75.48             |
| 10 | Spectral Width         | 18028.8           |
| 11 | Lowest Frequency       | -1464.8           |
| 12 | Nucleus                | 13C               |
| 13 | Acquired Size          | 16316             |
| 14 | Spectral Size          | 65536             |



EL090.{1H}.1.fid  
 Avance-300, CDCl<sub>3</sub>

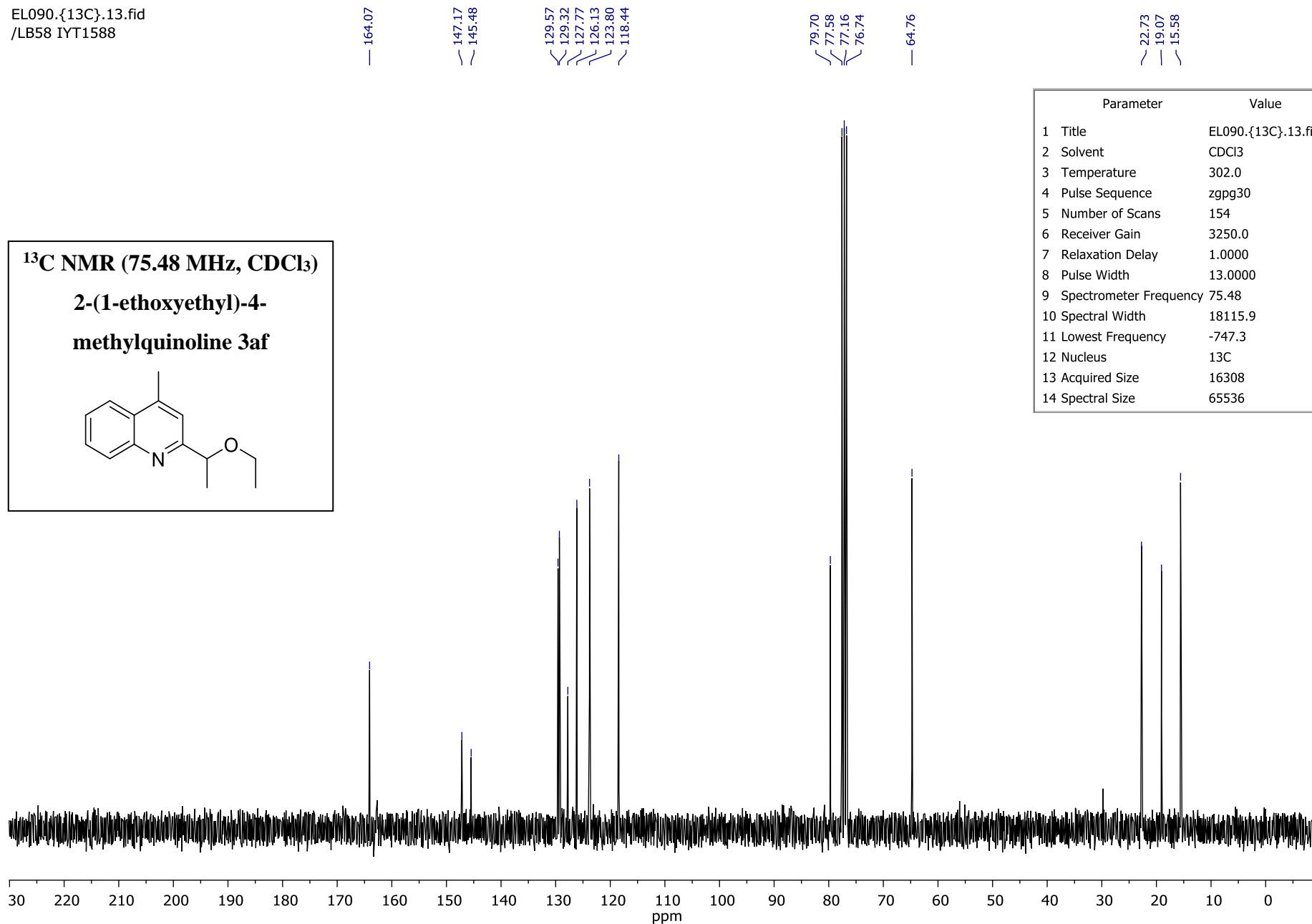
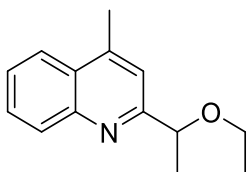


| Parameter                | Value             |
|--------------------------|-------------------|
| 1 Title                  | EL090.{1H}.1.fid  |
| 2 Solvent                | CDCl <sub>3</sub> |
| 3 Temperature            | 302.2             |
| 4 Pulse Sequence         | zg                |
| 5 Number of Scans        | 2                 |
| 6 Receiver Gain          | 11.3              |
| 7 Relaxation Delay       | 0.0050            |
| 8 Pulse Width            | 9.0000            |
| 9 Spectrometer Frequency | 300.13            |
| 10 Spectral Width        | 6009.6            |
| 11 Lowest Frequency      | -610.8            |
| 12 Nucleus               | <sup>1</sup> H    |
| 13 Acquired Size         | 8124              |
| 14 Spectral Size         | 65536             |

EL090.{13C}.13.fid  
/LB58 IYT1588

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

**2-(1-ethoxyethyl)-4-  
methylquinoline 3af**

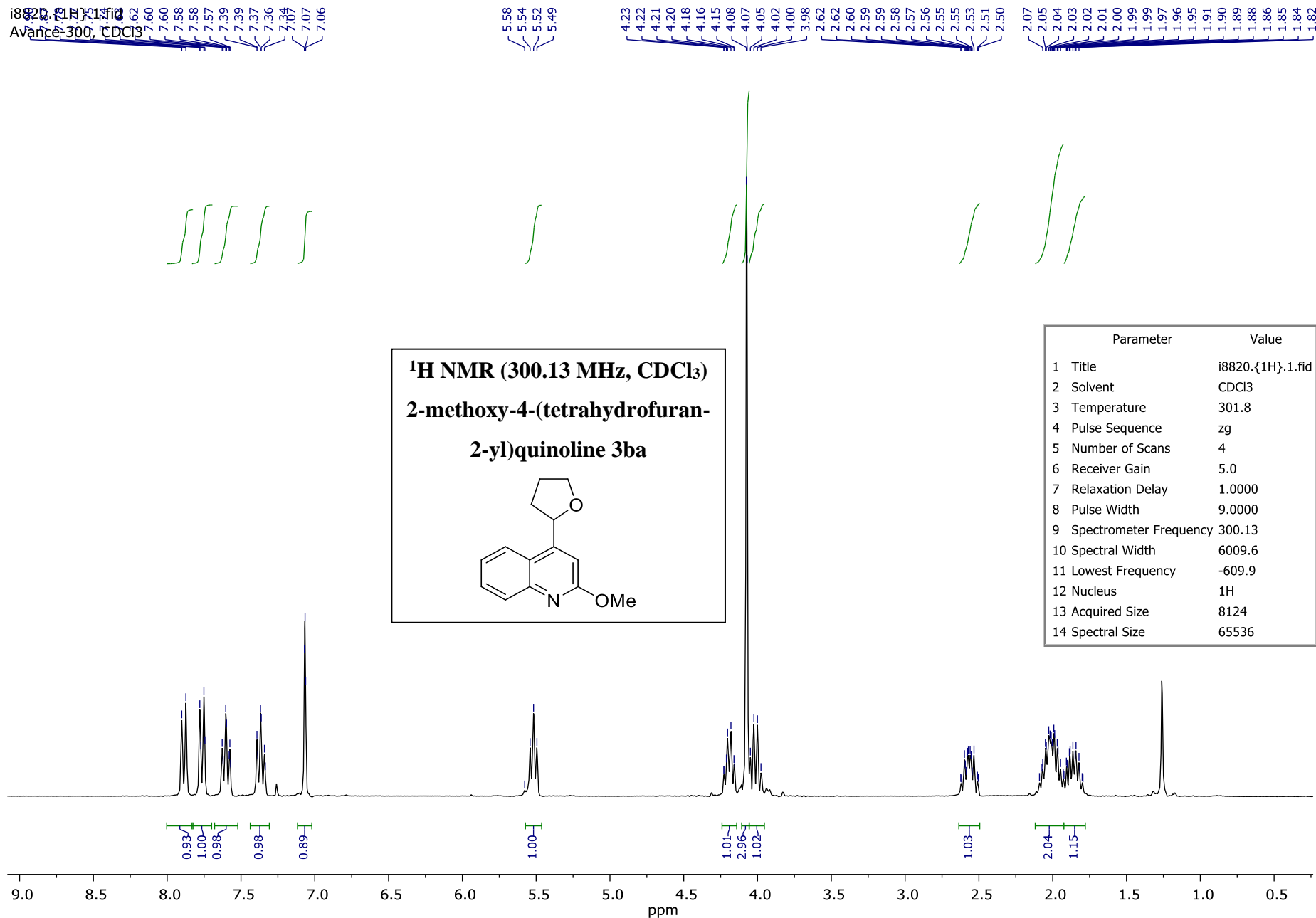
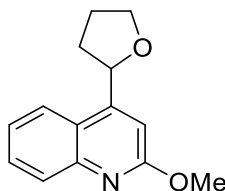


| Parameter                | Value              |
|--------------------------|--------------------|
| 1 Title                  | EL090.{13C}.13.fid |
| 2 Solvent                | CDCl3              |
| 3 Temperature            | 302.0              |
| 4 Pulse Sequence         | zgpg30             |
| 5 Number of Scans        | 154                |
| 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      | -747.3             |
| 12 Nucleus               | 13C                |
| 13 Acquired Size         | 16308              |
| 14 Spectral Size         | 65536              |



i8820.1f1  
 300.13 MHz  
 Avance-300, CDCl<sub>3</sub>

**<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)**  
**2-methoxy-4-(tetrahydrofuran-2-yl)quinoline 3ba**



| Parameter                | Value             |
|--------------------------|-------------------|
| 1 Title                  | i8820.{1H}.1.fid  |
| 2 Solvent                | CDCl <sub>3</sub> |
| 3 Temperature            | 301.8             |
| 4 Pulse Sequence         | zg                |
| 5 Number of Scans        | 4                 |
| 6 Receiver Gain          | 5.0               |
| 7 Relaxation Delay       | 1.0000            |
| 8 Pulse Width            | 9.0000            |
| 9 Spectrometer Frequency | 300.13            |
| 10 Spectral Width        | 6009.6            |
| 11 Lowest Frequency      | -609.9            |
| 12 Nucleus               | 1H                |
| 13 Acquired Size         | 8124              |
| 14 Spectral Size         | 65536             |

i8820.{13C}.13.fid  
/LB58 IYT1588

— 162.78

— 152.27

— 147.14

— 129.16

— 128.12

— 123.83

— 123.30

— 122.86

— 108.45

— 77.58

— 77.16

— 76.83

— 76.74

— 69.06

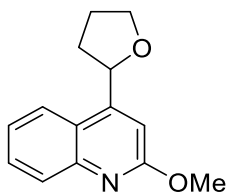
— 53.40

— 33.73

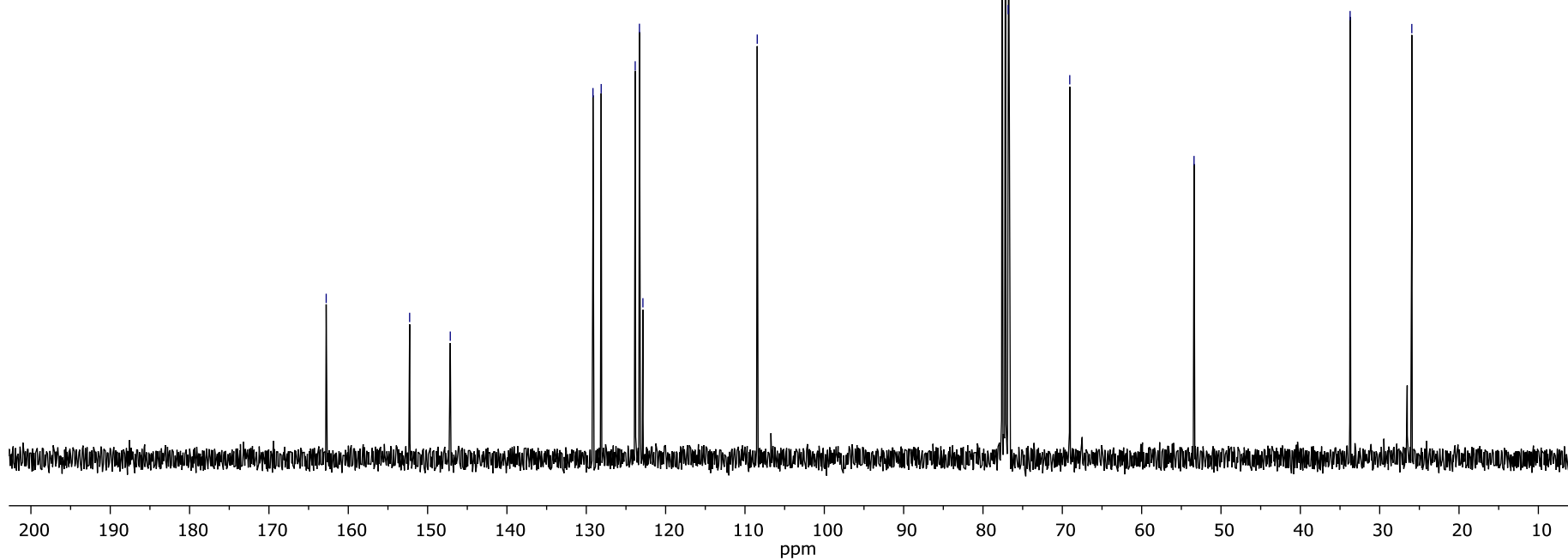
— 25.95

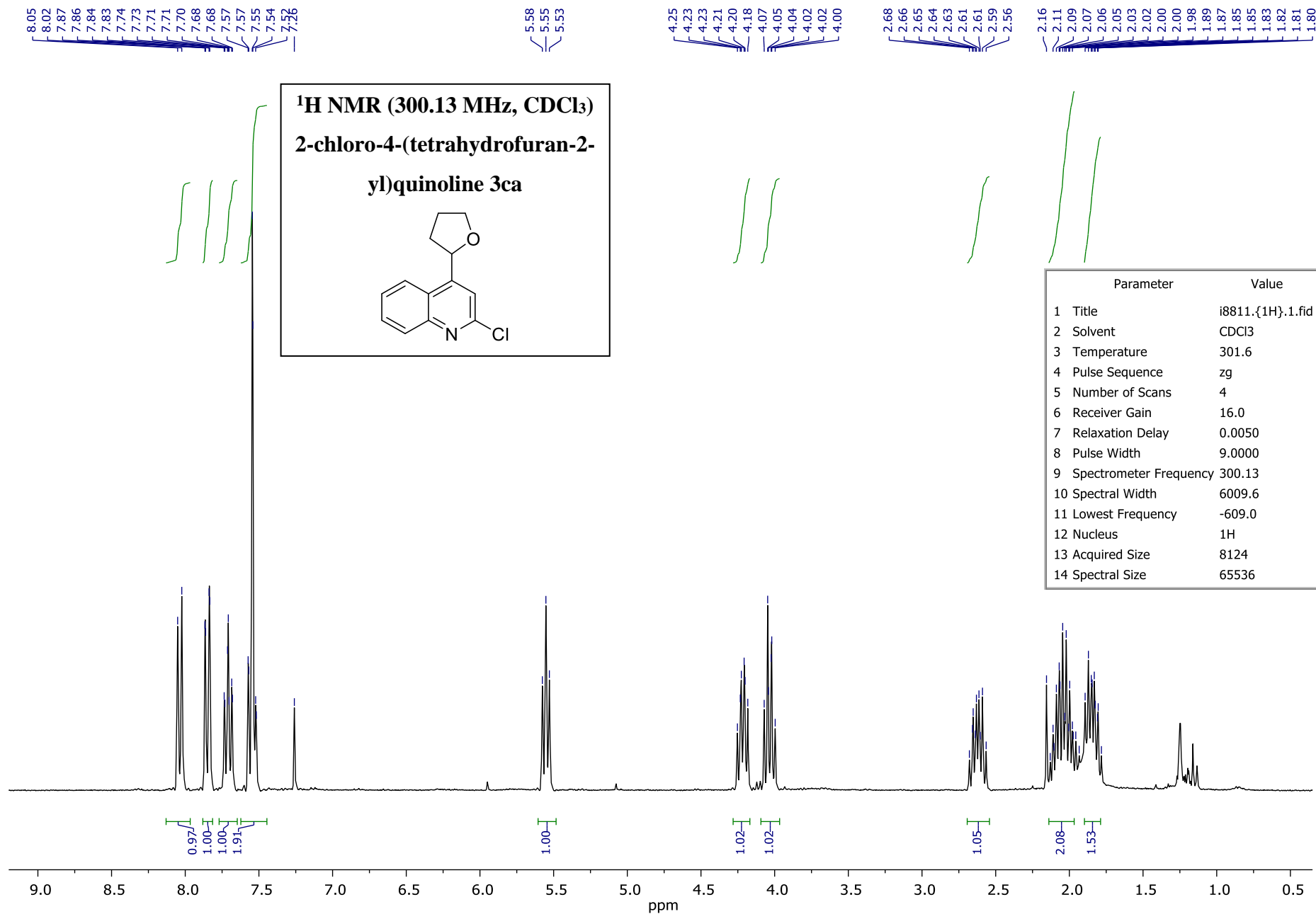
**<sup>13</sup>C NMR (75.48 MHz, CDCl<sub>3</sub>)**

**2-methoxy-4-(tetrahydrofuran-  
2-yl)quinoline 3ba**



| Parameter                | Value              |
|--------------------------|--------------------|
| 1 Title                  | i8820.{13C}.13.fid |
| 2 Solvent                | CDCl3              |
| 3 Temperature            | 302.0              |
| 4 Pulse Sequence         | zgpg30             |
| 5 Number of Scans        | 230                |
| 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      | -747.7             |
| 12 Nucleus               | 13C                |
| 13 Acquired Size         | 16308              |
| 14 Spectral Size         | 65536              |





i8811.{13C}.13.fid  
/LB58 IYT1588

153.13  
151.39  
148.13

130.18  
129.53  
126.83  
124.45  
123.38  
117.92

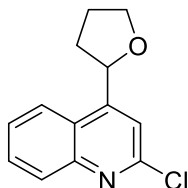
77.58  
77.16  
76.74  
76.65  
69.22

34.04

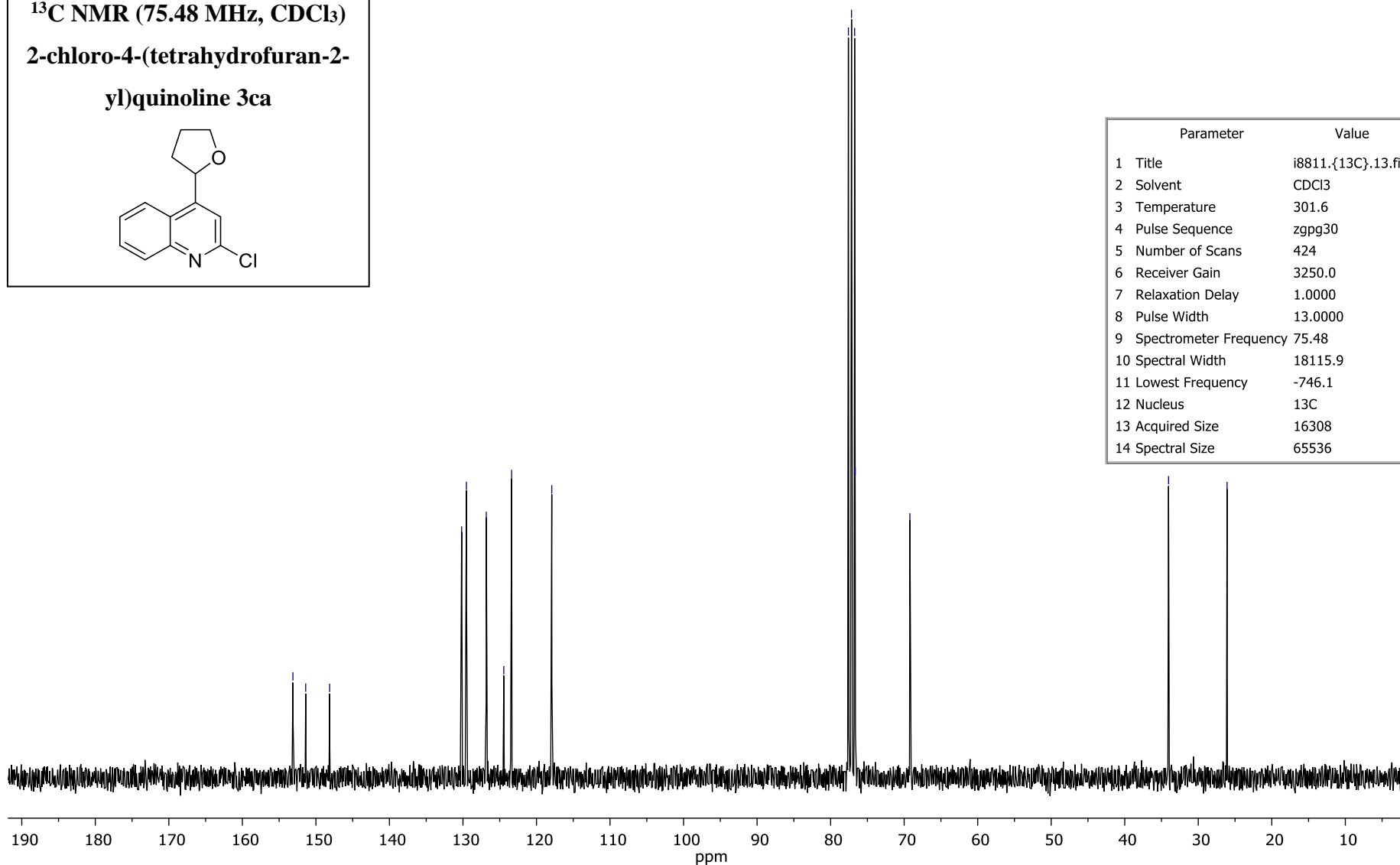
26.08

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

**2-chloro-4-(tetrahydrofuran-2-yl)quinoline 3ca**

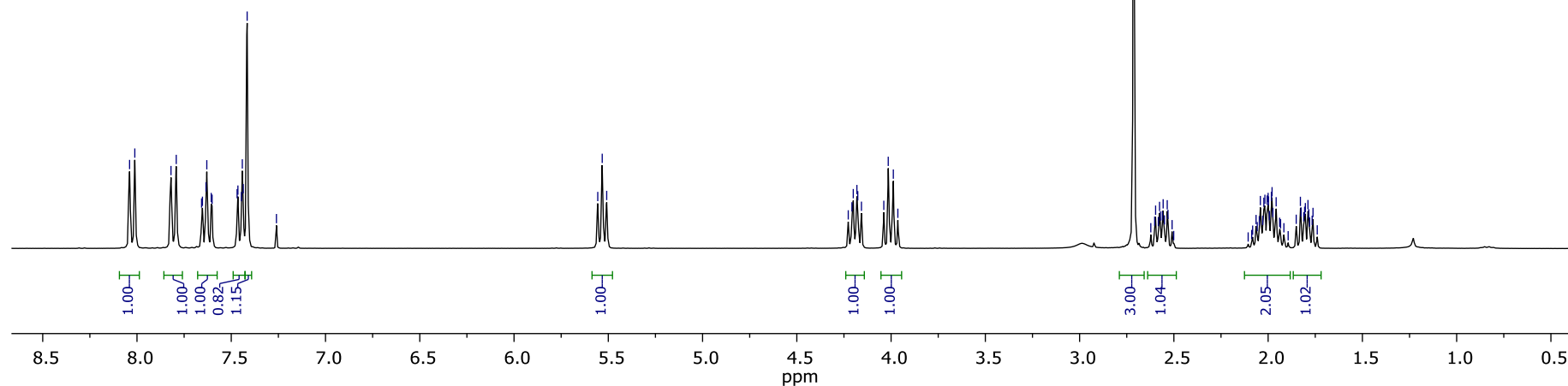
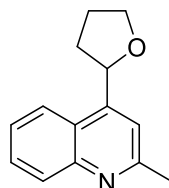


| Parameter                | Value              |
|--------------------------|--------------------|
| 1 Title                  | i8811.{13C}.13.fid |
| 2 Solvent                | CDCl3              |
| 3 Temperature            | 301.6              |
| 4 Pulse Sequence         | zgpg30             |
| 5 Number of Scans        | 424                |
| 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      | -746.1             |
| 12 Nucleus               | 13C                |
| 13 Acquired Size         | 16308              |
| 14 Spectral Size         | 65536              |

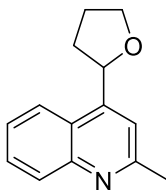


| Parameter                | Value            |
|--------------------------|------------------|
| 1 Title                  | EL583.{1H}.1.fid |
| 2 Solvent                | CDCl3            |
| 3 Temperature            | 298.0            |
| 4 Pulse Sequence         | zg               |
| 5 Number of 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      | -1094.5          |
| 12 Nucleus               | 1H               |
| 13 Acquired Size         | 16308            |
| 14 Spectral Size         | 65536            |

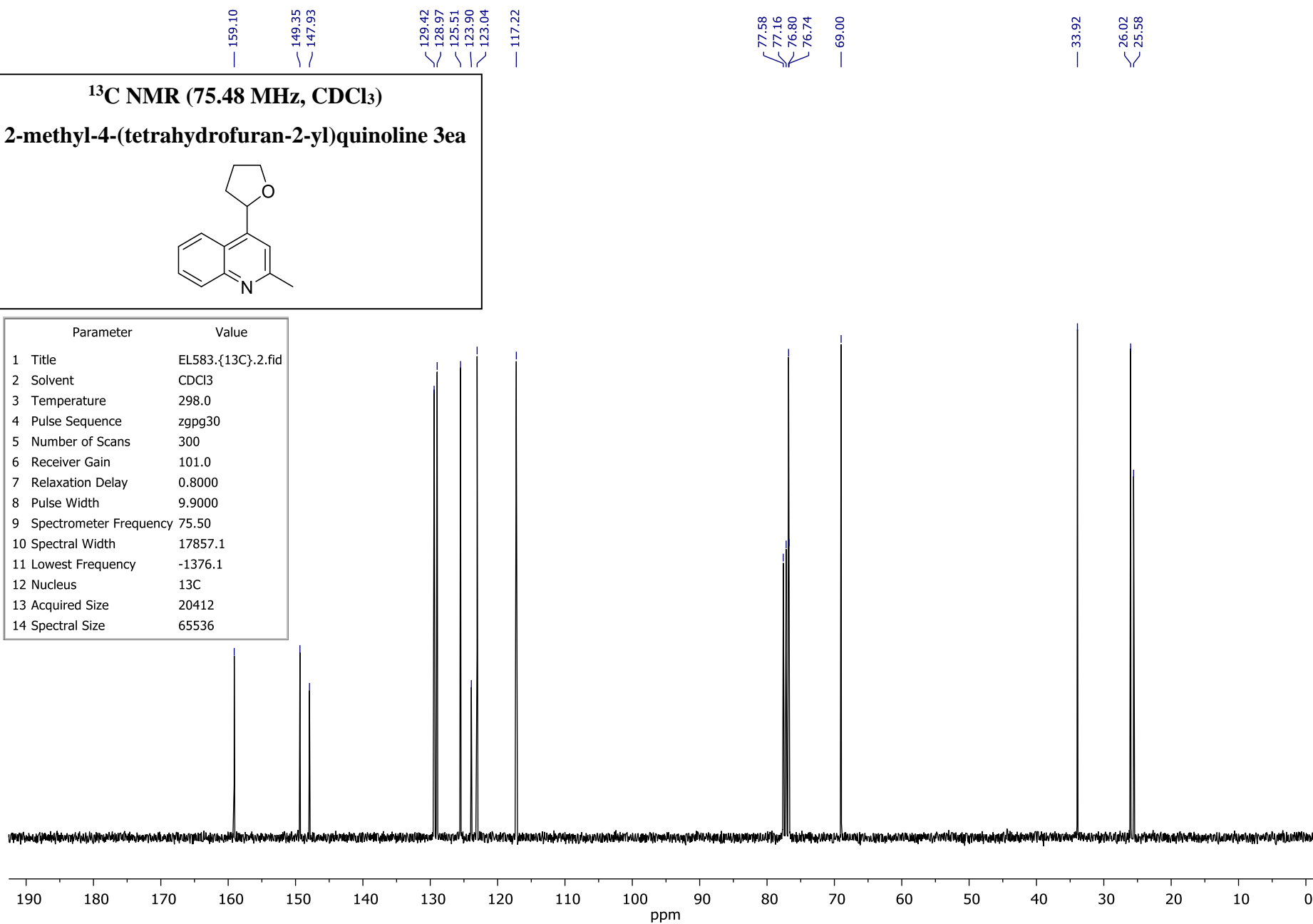
**<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)**  
**2-methyl-4-(tetrahydrofuran-2-yl)quinoline 3ea**

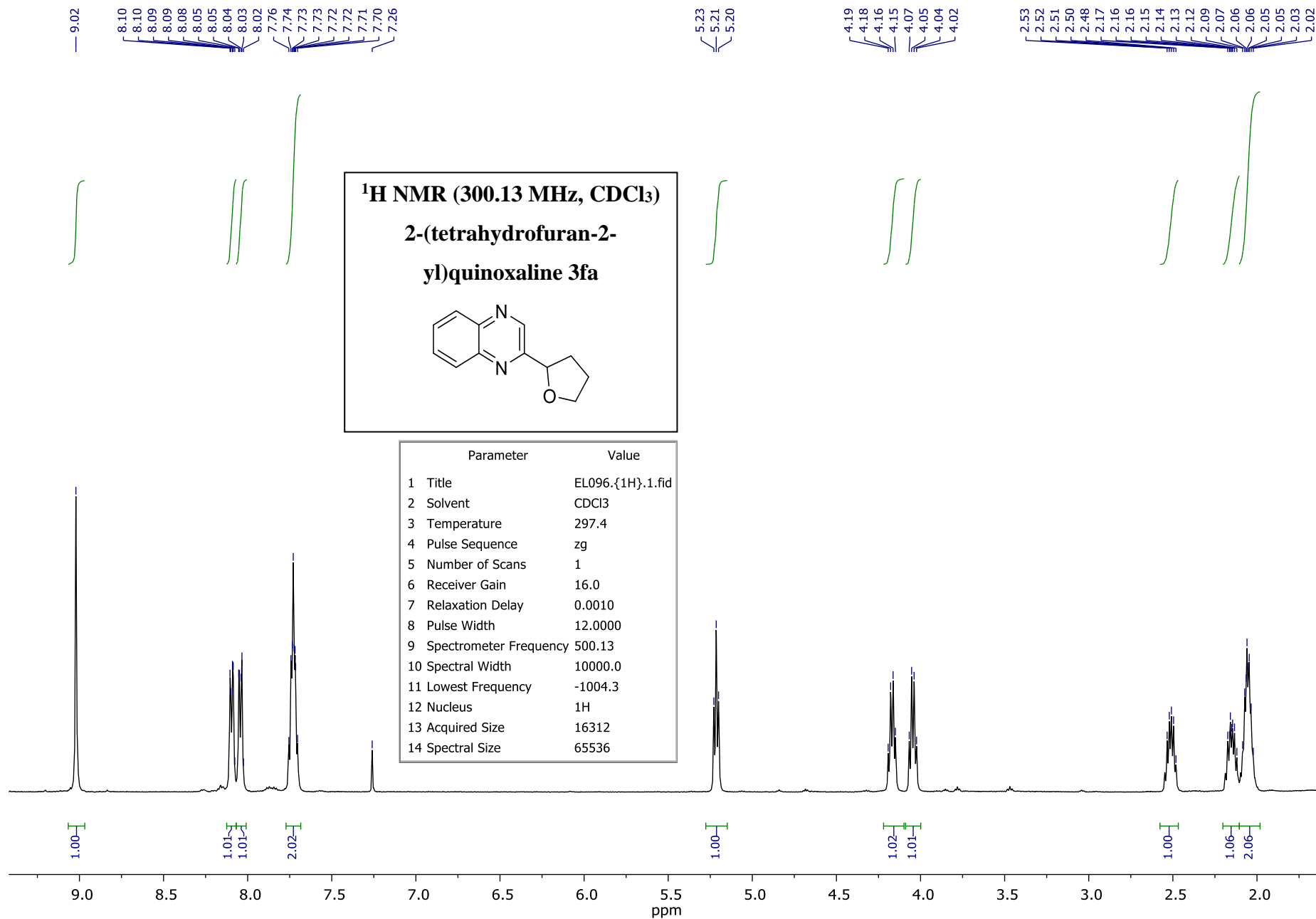


**$^{13}\text{C}$  NMR (75.48 MHz,  $\text{CDCl}_3$ )**  
**2-methyl-4-(tetrahydrofuran-2-yl)quinoline 3ea**



| Parameter                | Value                           |
|--------------------------|---------------------------------|
| 1 Title                  | EL583.{ $^{13}\text{C}$ }.2.fid |
| 2 Solvent                | $\text{CDCl}_3$                 |
| 3 Temperature            | 298.0                           |
| 4 Pulse Sequence         | zgpg30                          |
| 5 Number of Scans        | 300                             |
| 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      | -1376.1                         |
| 12 Nucleus               | $^{13}\text{C}$                 |
| 13 Acquired Size         | 20412                           |
| 14 Spectral Size         | 65536                           |





EL096-<sup>13</sup>C}.1.fid  
/TERN EL096

— 157.71

143.61  
141.97  
141.67

130.18  
129.58  
129.31  
129.16

80.61  
77.41  
77.16  
76.91

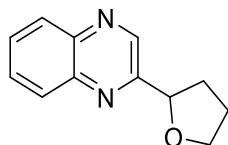
— 69.52

— 33.04

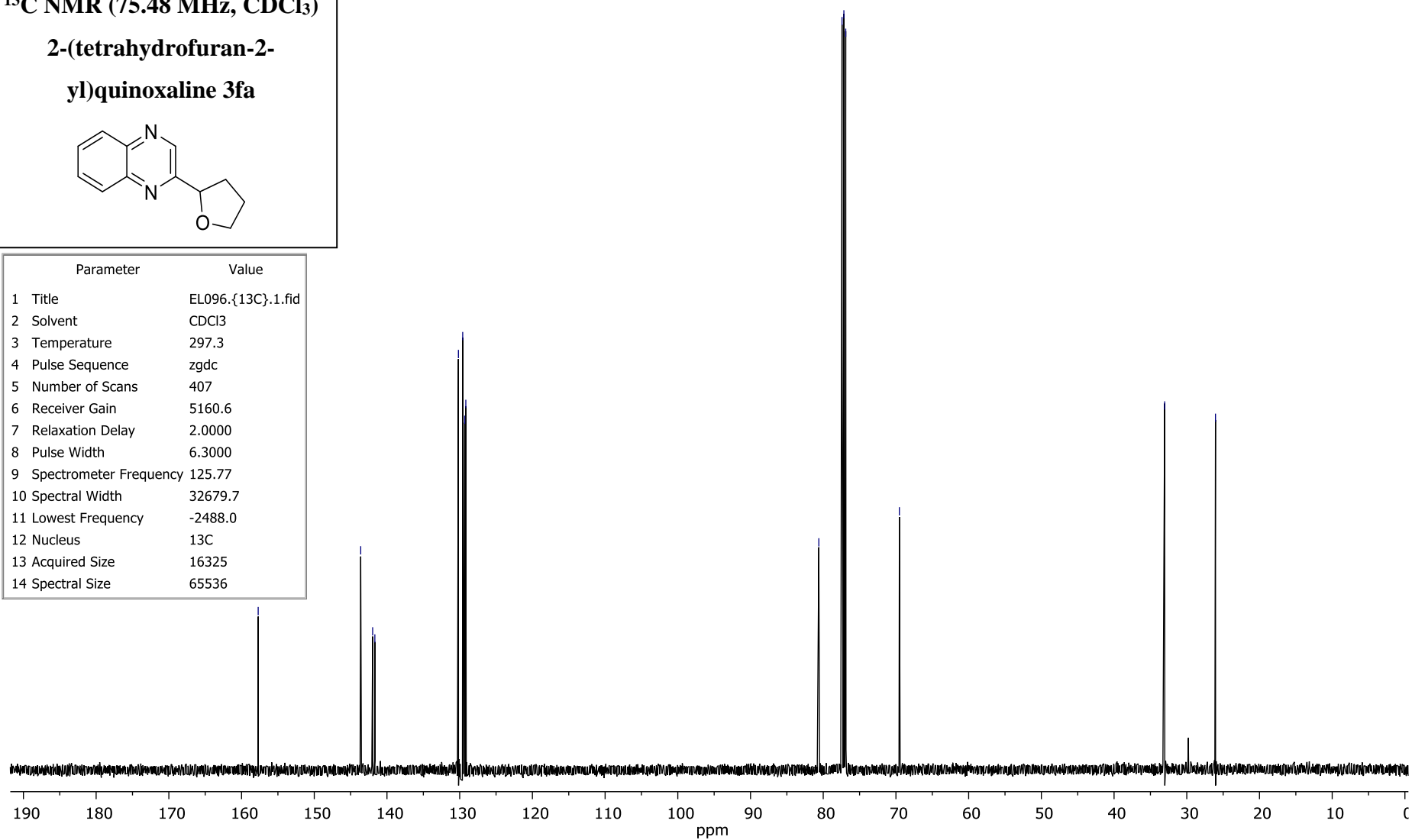
— 26.05

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

**2-(tetrahydrofuran-2-  
yl)quinoxaline 3fa**



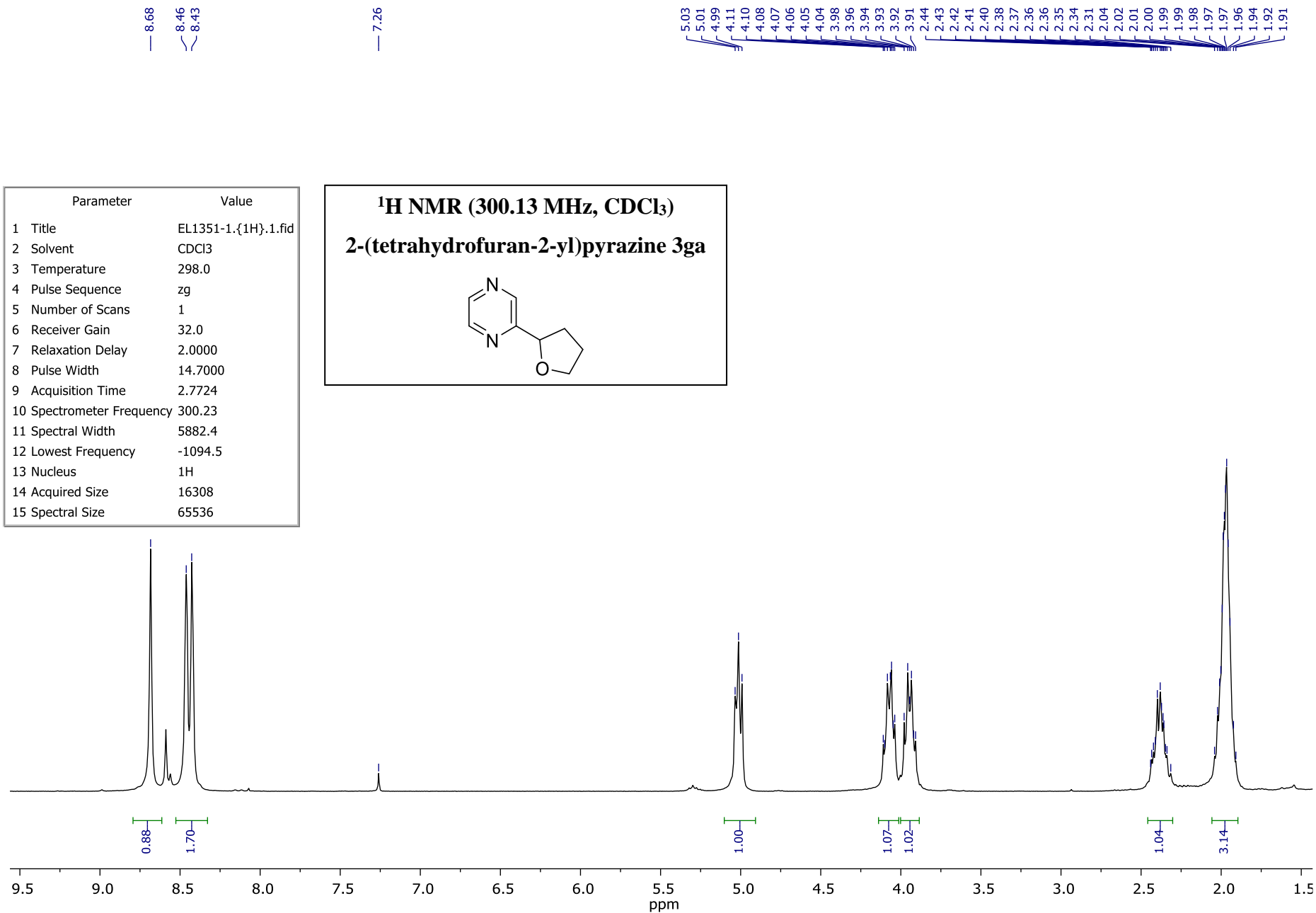
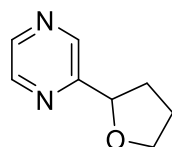
| Parameter                | Value                         |
|--------------------------|-------------------------------|
| 1 Title                  | EL096- <sup>13</sup> C}.1.fid |
| 2 Solvent                | CDCl <sub>3</sub>             |
| 3 Temperature            | 297.3                         |
| 4 Pulse Sequence         | zgdc                          |
| 5 Number of Scans        | 407                           |
| 6 Receiver Gain          | 5160.6                        |
| 7 Relaxation Delay       | 2.0000                        |
| 8 Pulse Width            | 6.3000                        |
| 9 Spectrometer Frequency | 125.77                        |
| 10 Spectral Width        | 32679.7                       |
| 11 Lowest Frequency      | -2488.0                       |
| 12 Nucleus               | <sup>13</sup> C               |
| 13 Acquired Size         | 16325                         |
| 14 Spectral Size         | 65536                         |





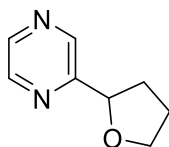
| Parameter                 | Value               |
|---------------------------|---------------------|
| 1 Title                   | EL1351-1-{1H}.1.fid |
| 2 Solvent                 | CDCl3               |
| 3 Temperature             | 298.0               |
| 4 Pulse Sequence          | zg                  |
| 5 Number of Scans         | 1                   |
| 6 Receiver Gain           | 32.0                |
| 7 Relaxation Delay        | 2.0000              |
| 8 Pulse Width             | 14.7000             |
| 9 Acquisition Time        | 2.7724              |
| 10 Spectrometer Frequency | 300.23              |
| 11 Spectral Width         | 5882.4              |
| 12 Lowest Frequency       | -1094.5             |
| 13 Nucleus                | 1H                  |
| 14 Acquired Size          | 16308               |
| 15 Spectral Size          | 65536               |

**<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)**  
**2-(tetrahydrofuran-2-yl)pyrazine 3ga**

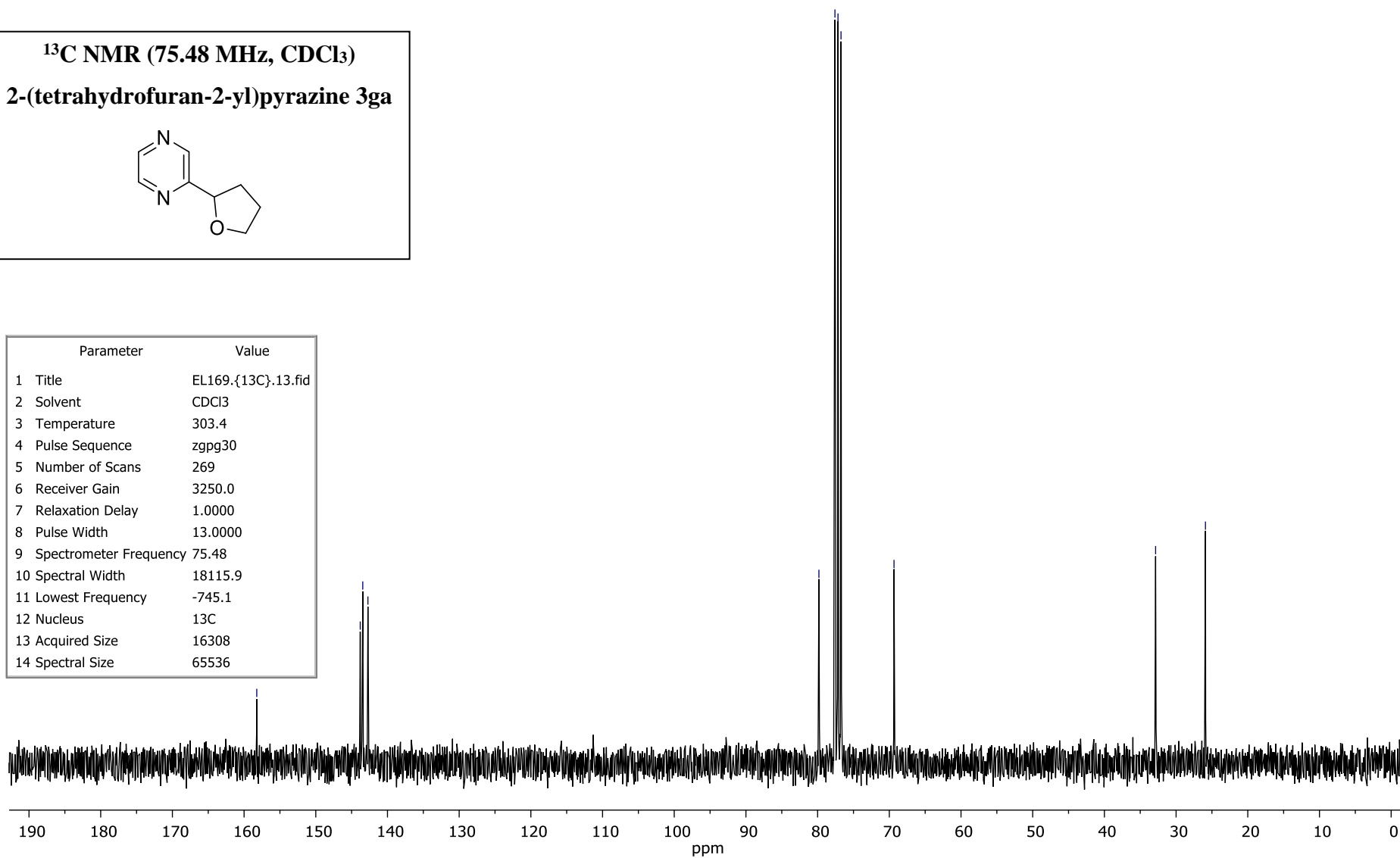


**$^{13}\text{C}$  NMR (75.48 MHz,  $\text{CDCl}_3$ )**

**2-(tetrahydrofuran-2-yl)pyrazine 3ga**

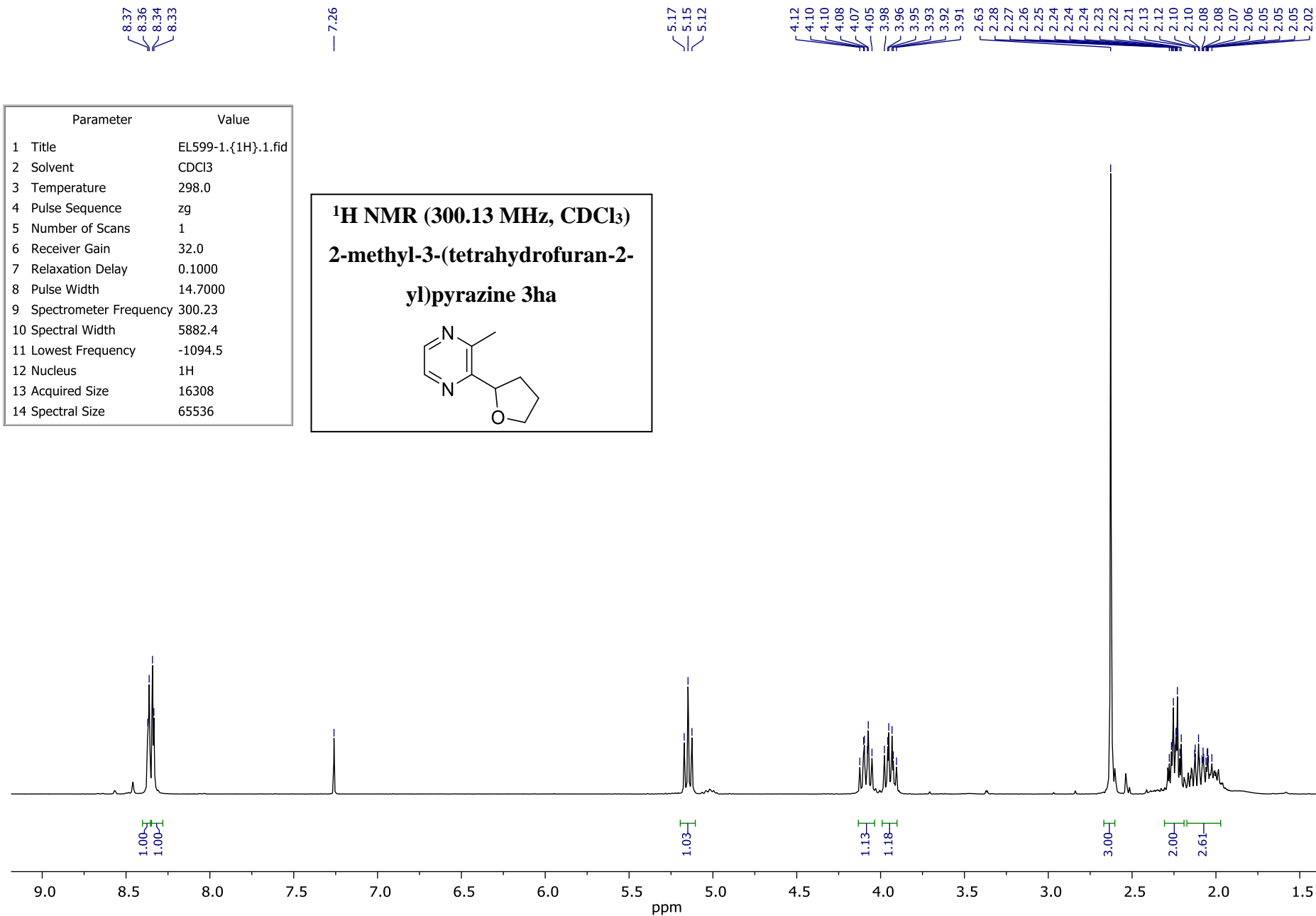
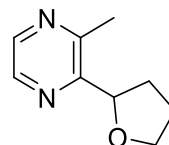


| Parameter                | Value              |
|--------------------------|--------------------|
| 1 Title                  | EL169.{13C}.13.fid |
| 2 Solvent                | $\text{CDCl}_3$    |
| 3 Temperature            | 303.4              |
| 4 Pulse Sequence         | zgpg30             |
| 5 Number of Scans        | 269                |
| 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      | -745.1             |
| 12 Nucleus               | $^{13}\text{C}$    |
| 13 Acquired Size         | 16308              |
| 14 Spectral Size         | 65536              |



| Parameter                | Value              |
|--------------------------|--------------------|
| 1 Title                  | EL599-1-{1H}.1.fid |
| 2 Solvent                | CDCl3              |
| 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      | -1094.5            |
| 12 Nucleus               | 1H                 |
| 13 Acquired Size         | 16308              |
| 14 Spectral Size         | 65536              |

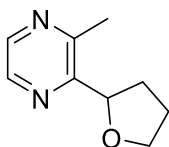
**<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)**  
**2-methyl-3-(tetrahydrofuran-2-yl)pyrazine 3ha**



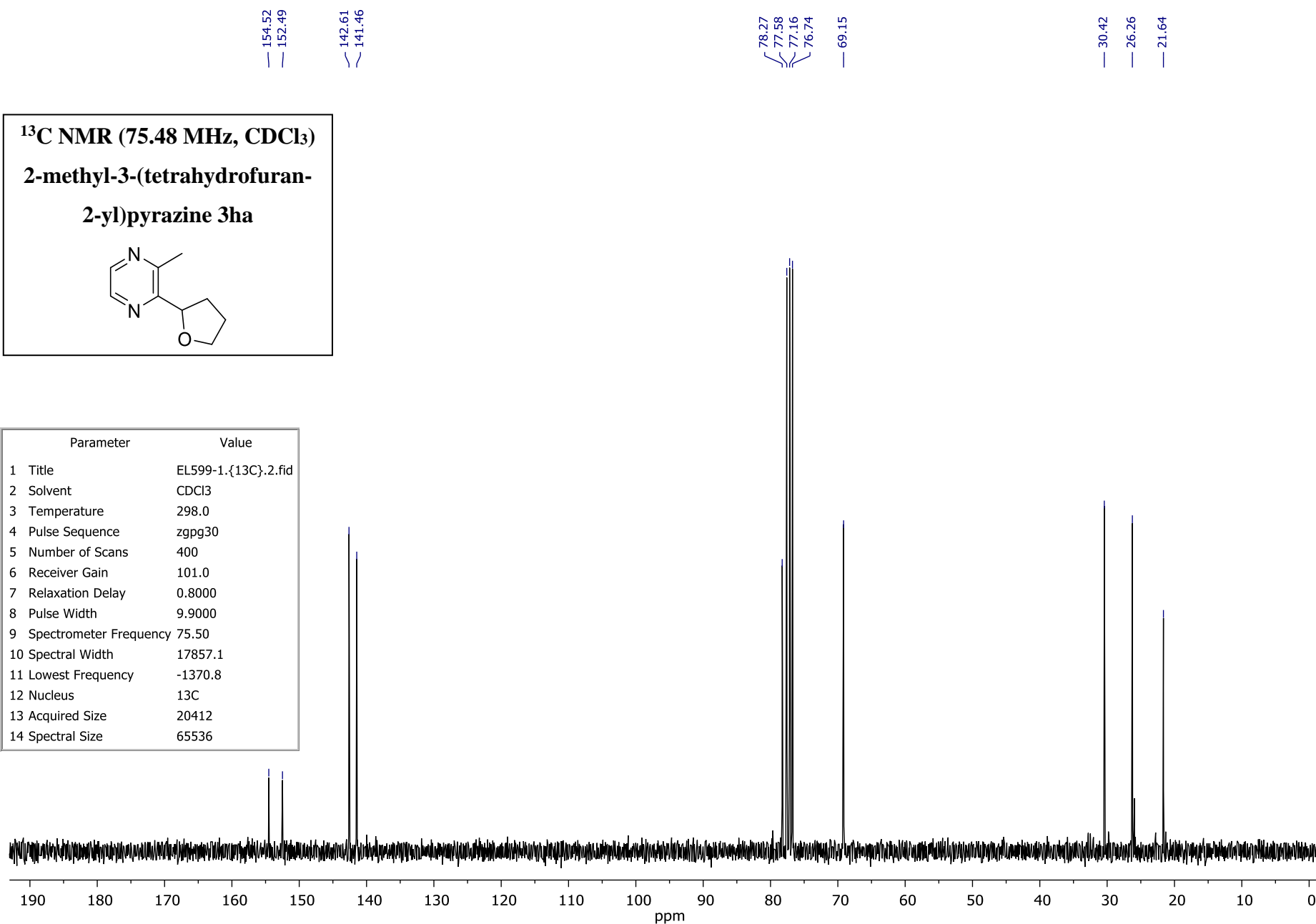
**$^{13}\text{C}$  NMR (75.48 MHz,  $\text{CDCl}_3$ )**

**2-methyl-3-(tetrahydrofuran-**

**2-yl)pyrazine 3ha**



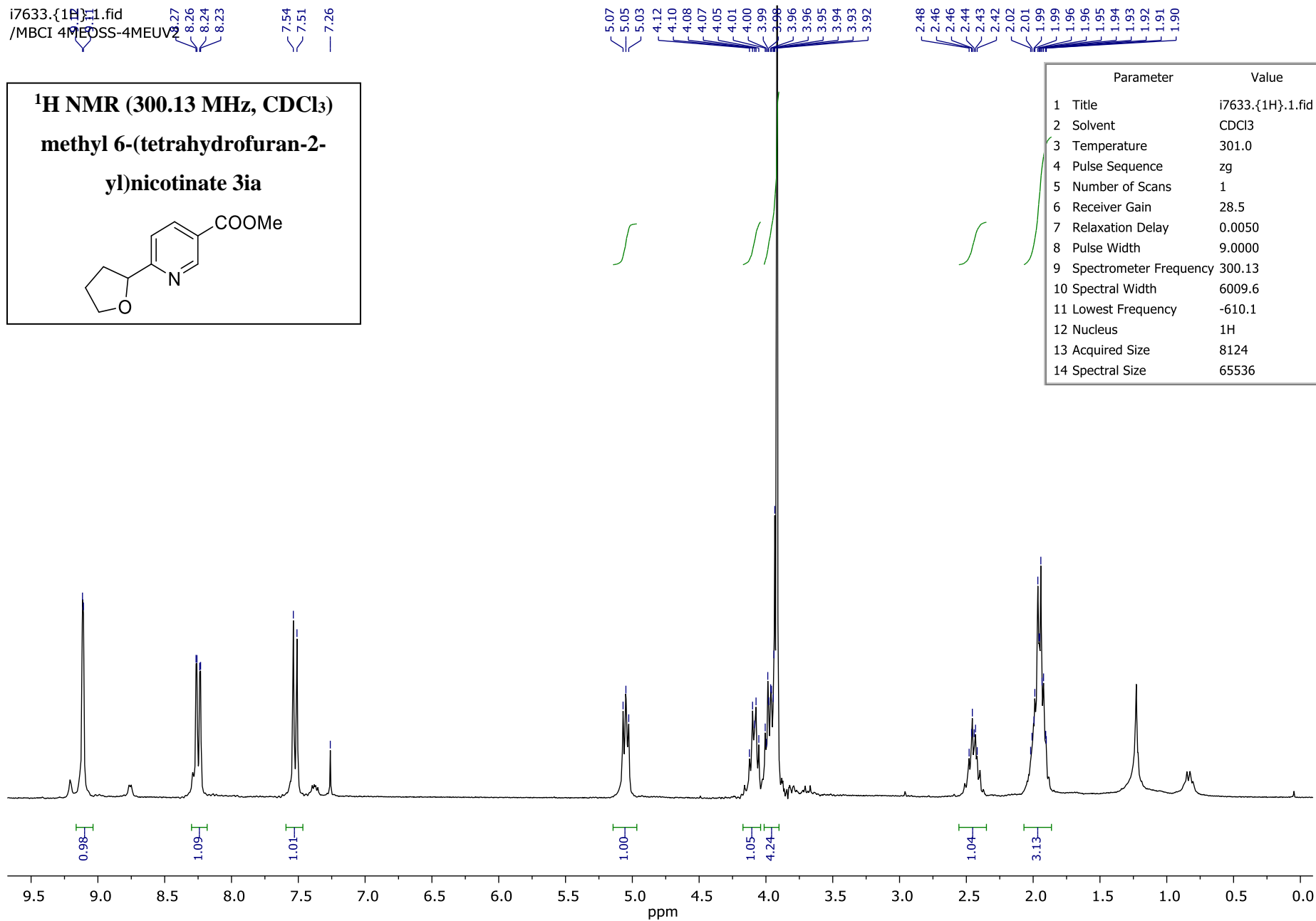
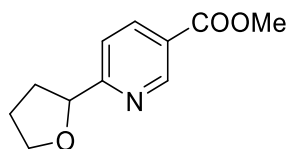
| Parameter                | Value                               |
|--------------------------|-------------------------------------|
| 1 Title                  | EL599-1- $\{^{13}\text{C}\}$ .2.fid |
| 2 Solvent                | $\text{CDCl}_3$                     |
| 3 Temperature            | 298.0                               |
| 4 Pulse Sequence         | zgpg30                              |
| 5 Number of Scans        | 400                                 |
| 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      | -1370.8                             |
| 12 Nucleus               | $^{13}\text{C}$                     |
| 13 Acquired Size         | 20412                               |
| 14 Spectral Size         | 65536                               |



i7633.{1H}.1.fid  
/MBCI 4MEOS-4MEUV2

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

**methyl 6-(tetrahydrofuran-2-yl)nicotinate 3ia**



i7633.{13C}.13.fid  
/KANI NKA013a

167.80  
165.87

150.36

137.87

124.56

119.37

81.23  
77.59  
77.16  
76.74

69.31

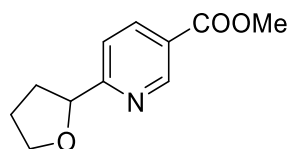
52.38

33.22

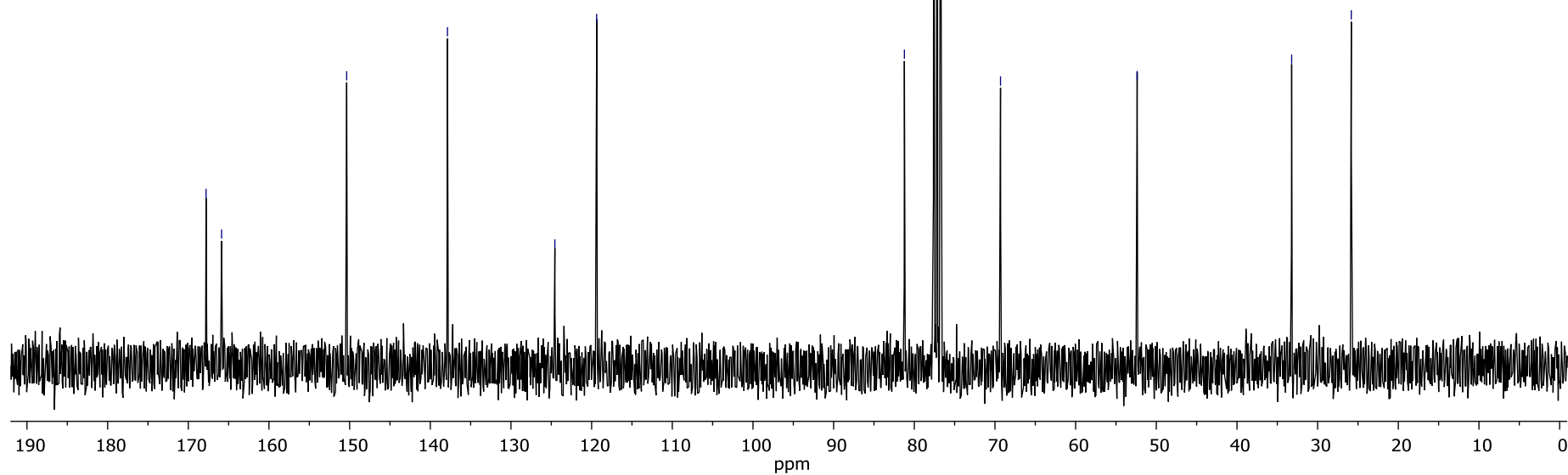
25.83

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

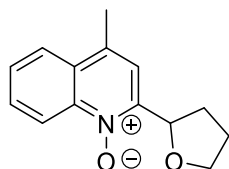
**methyl 6-(tetrahydrofuran-2-  
yl)nicotinate 3ia**



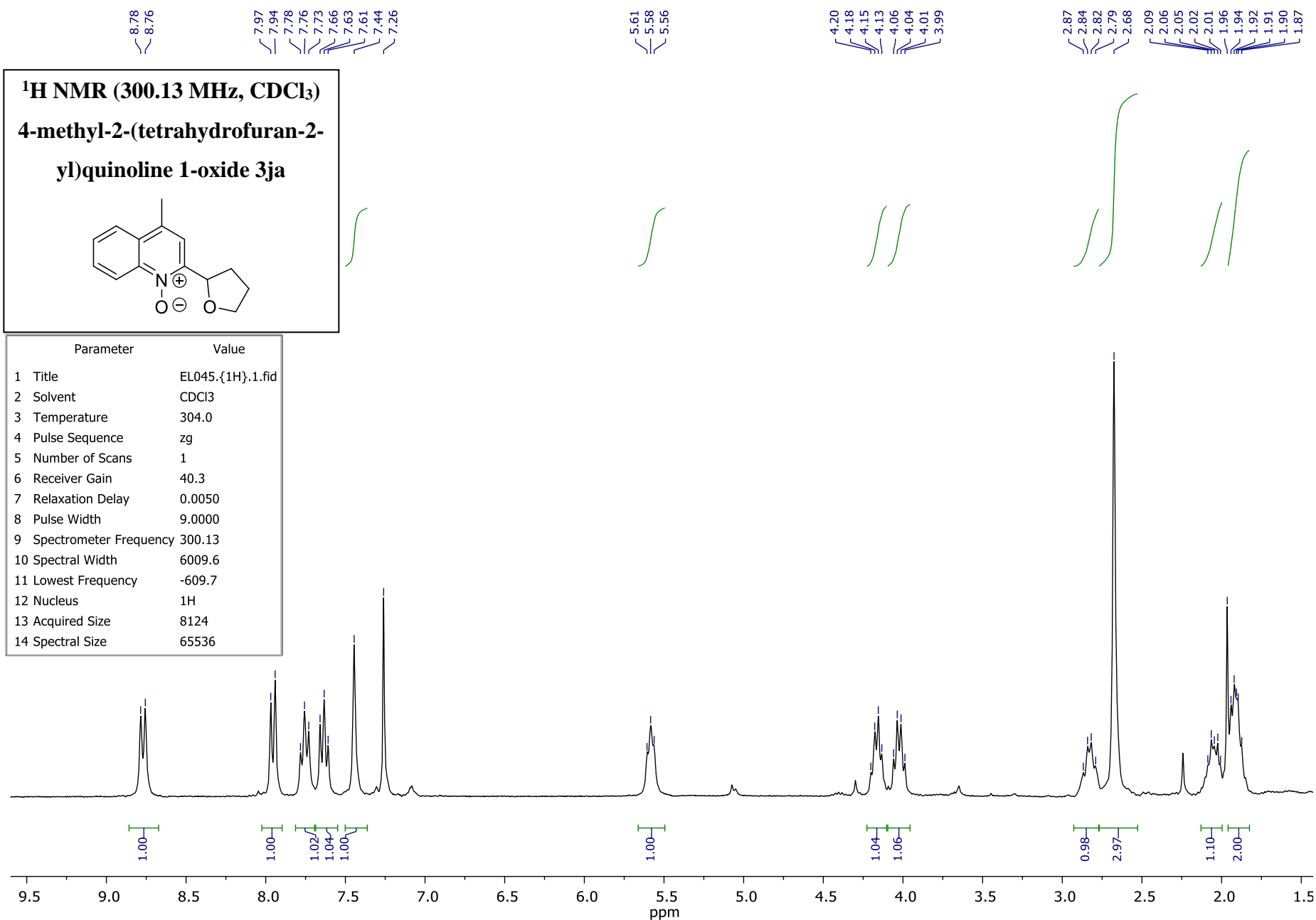
| Parameter                | Value              |
|--------------------------|--------------------|
| 1 Title                  | i7633.{13C}.13.fid |
| 2 Solvent                | CDCl3              |
| 3 Temperature            | 301.1              |
| 4 Pulse Sequence         | zgpg30             |
| 5 Number of Scans        | 113                |
| 6 Receiver Gain          | 40.3               |
| 7 Relaxation Delay       | 1.0000             |
| 8 Pulse Width            | 13.0000            |
| 9 Spectrometer Frequency | 75.48              |
| 10 Spectral Width        | 18115.9            |
| 11 Lowest Frequency      | -747.4             |
| 12 Nucleus               | 13C                |
| 13 Acquired Size         | 16308              |
| 14 Spectral Size         | 65536              |

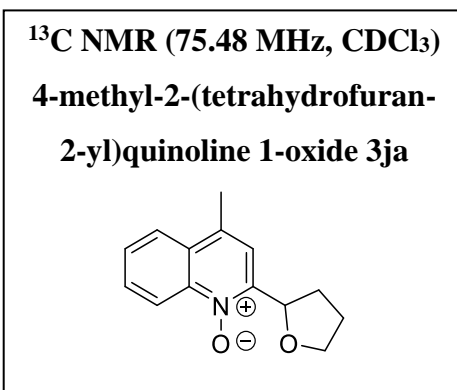


**<sup>1</sup>H NMR (300.13 MHz, CDCl<sub>3</sub>)**  
**4-methyl-2-(tetrahydrofuran-2-yl)quinoline 1-oxide 3ja**

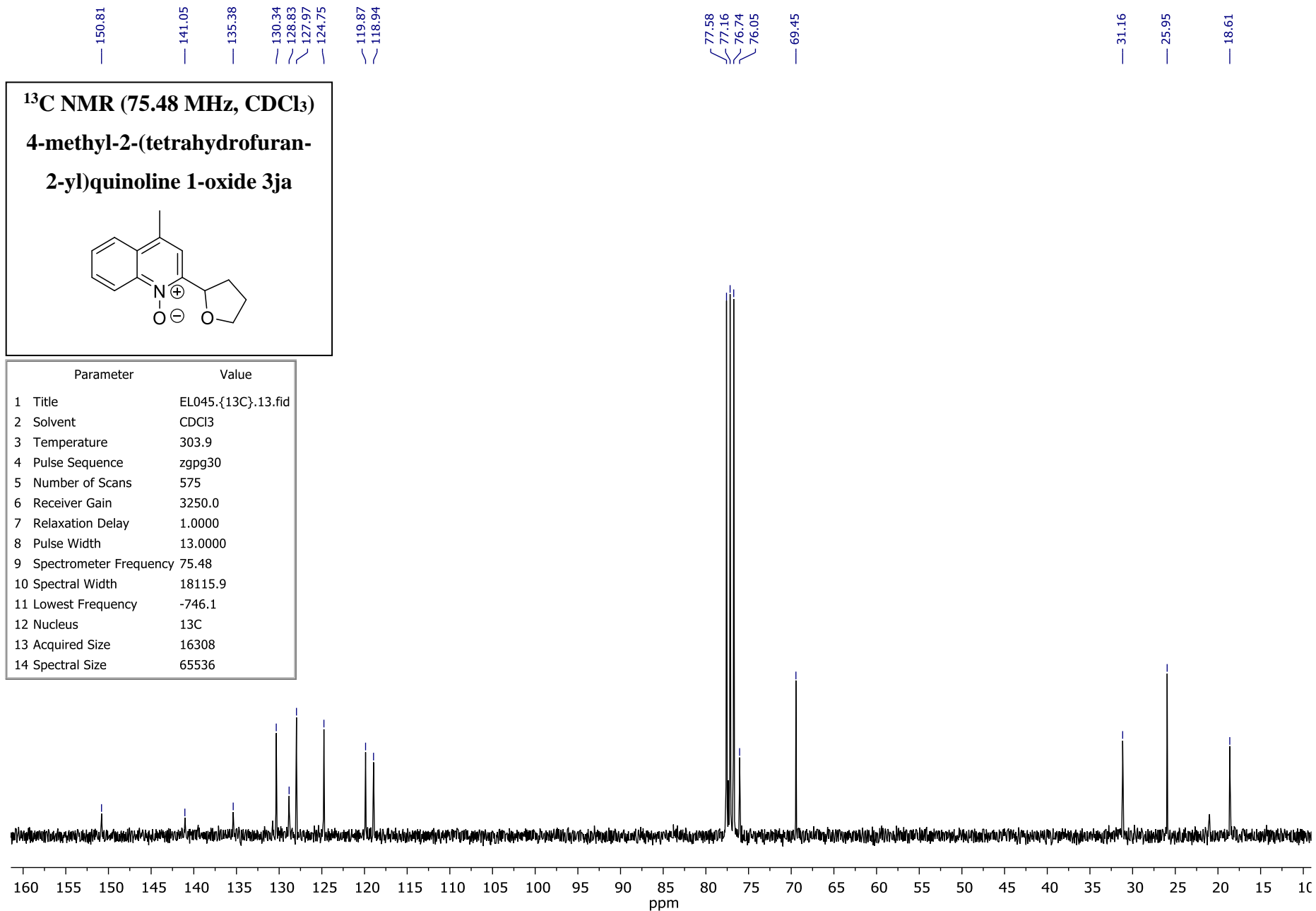


| Parameter                | Value             |
|--------------------------|-------------------|
| 1 Title                  | EL045.{1H}.1.fid  |
| 2 Solvent                | CDCl <sub>3</sub> |
| 3 Temperature            | 304.0             |
| 4 Pulse Sequence         | zg                |
| 5 Number of Scans        | 1                 |
| 6 Receiver Gain          | 40.3              |
| 7 Relaxation Delay       | 0.0050            |
| 8 Pulse Width            | 9.0000            |
| 9 Spectrometer Frequency | 300.13            |
| 10 Spectral Width        | 6009.6            |
| 11 Lowest Frequency      | -609.7            |
| 12 Nucleus               | <sup>1</sup> H    |
| 13 Acquired Size         | 8124              |
| 14 Spectral Size         | 65536             |





| Parameter                | Value                            |
|--------------------------|----------------------------------|
| 1 Title                  | EL045.{ $^{13}\text{C}$ }.13.fid |
| 2 Solvent                | $\text{CDCl}_3$                  |
| 3 Temperature            | 303.9                            |
| 4 Pulse Sequence         | zgpg30                           |
| 5 Number of Scans        | 575                              |
| 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      | -746.1                           |
| 12 Nucleus               | $^{13}\text{C}$                  |
| 13 Acquired Size         | 16308                            |
| 14 Spectral Size         | 65536                            |





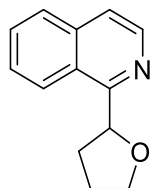
i7453.{1H}.1.fid  
/MBCI 4MEOS-4MEUV2

5.74  
5.72  
5.69

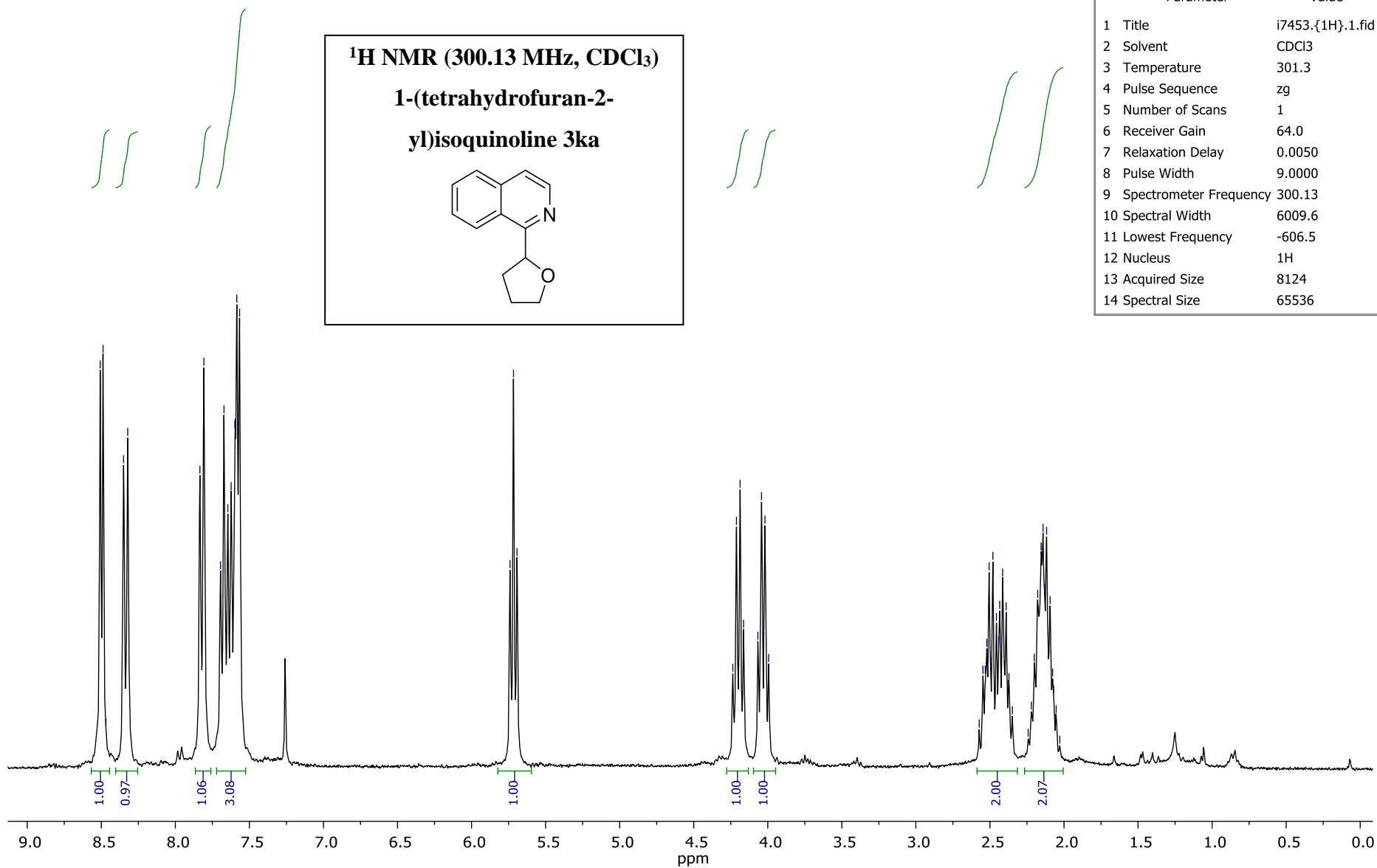
4.24  
4.21  
4.19  
4.16  
4.07  
4.04  
4.02  
3.99  
2.57  
2.55  
2.53  
2.52  
2.51  
2.50  
2.48  
2.46  
2.44  
2.43  
2.41  
2.39  
2.37  
2.35  
2.24  
2.22  
2.20  
2.18  
2.15  
2.14  
2.13  
2.12  
2.09  
2.08  
2.07  
2.05  
2.03

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

**1-(tetrahydrofuran-2-yl)isoquinoline 3ka**



| Parameter                | Value             |
|--------------------------|-------------------|
| 1 Title                  | i7453.{1H}.1.fid  |
| 2 Solvent                | CDCl <sub>3</sub> |
| 3 Temperature            | 301.3             |
| 4 Pulse Sequence         | zg                |
| 5 Number of Scans        | 1                 |
| 6 Receiver Gain          | 64.0              |
| 7 Relaxation Delay       | 0.0050            |
| 8 Pulse Width            | 9.0000            |
| 9 Spectrometer Frequency | 300.13            |
| 10 Spectral Width        | 6009.6            |
| 11 Lowest Frequency      | -606.5            |
| 12 Nucleus               | <sup>1</sup> H    |
| 13 Acquired Size         | 8124              |
| 14 Spectral Size         | 65536             |



i7535.{13C}.13.fid  
/KANI NKA013a

— 159.73

— 141.36

— 136.74

— 130.11

— 127.45

— 127.31

— 126.70

— 125.48

— 120.71

— 79.16

— 77.58

— 77.16

— 76.74

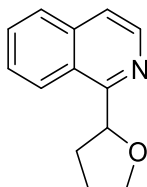
— 69.14

— 30.94

— 26.27

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

**1-(tetrahydrofuran-2-  
yl)isoquinoline 3ka**



| Parameter                | Value              |
|--------------------------|--------------------|
| 1 Title                  | i7535.{13C}.13.fid |
| 2 Solvent                | CDCl3              |
| 3 Temperature            | 301.5              |
| 4 Pulse Sequence         | zgpg30             |
| 5 Number of Scans        | 384                |
| 6 Receiver Gain          | 40.3               |
| 7 Relaxation Delay       | 1.0000             |
| 8 Pulse Width            | 13.0000            |
| 9 Spectrometer Frequency | 75.48              |
| 10 Spectral Width        | 18115.9            |
| 11 Lowest Frequency      | -746.8             |
| 12 Nucleus               | 13C                |
| 13 Acquired Size         | 16308              |
| 14 Spectral Size         | 65536              |

