

## Supporting Information

### New Journal of Chemistry

#### Metal-Free Annulative Hydrosulfonation of Propiolate Esters: Synthesis of 4-Sulfonates of Coumarins and Butenolides

Rodney A. Fernandes,\* Ashvin J. Gangani and Rupesh A. Kunkalkar

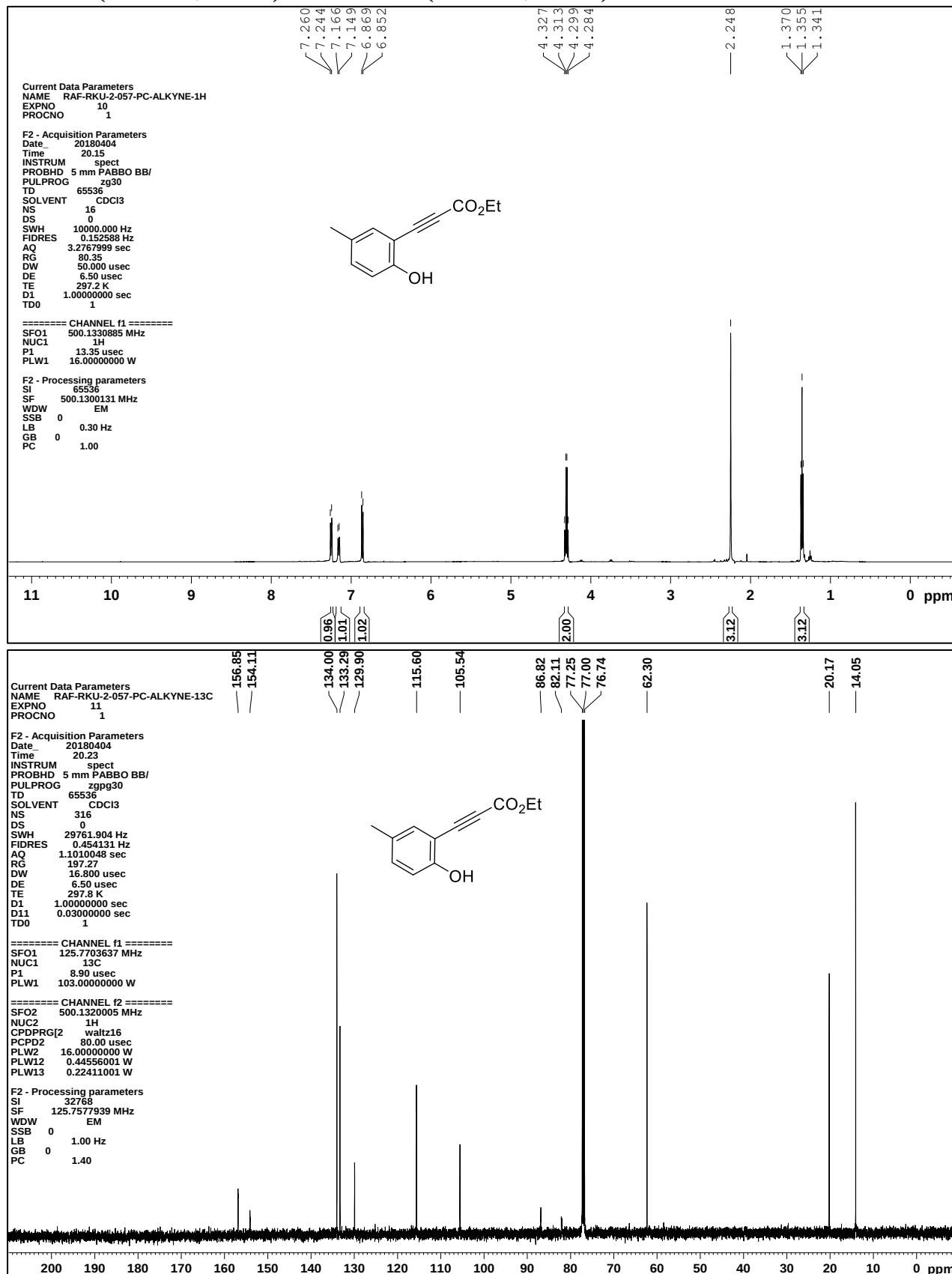
Department of Chemistry, Indian Institute of Technology Bombay, Powai

Mumbai 400076, Maharashtra, India

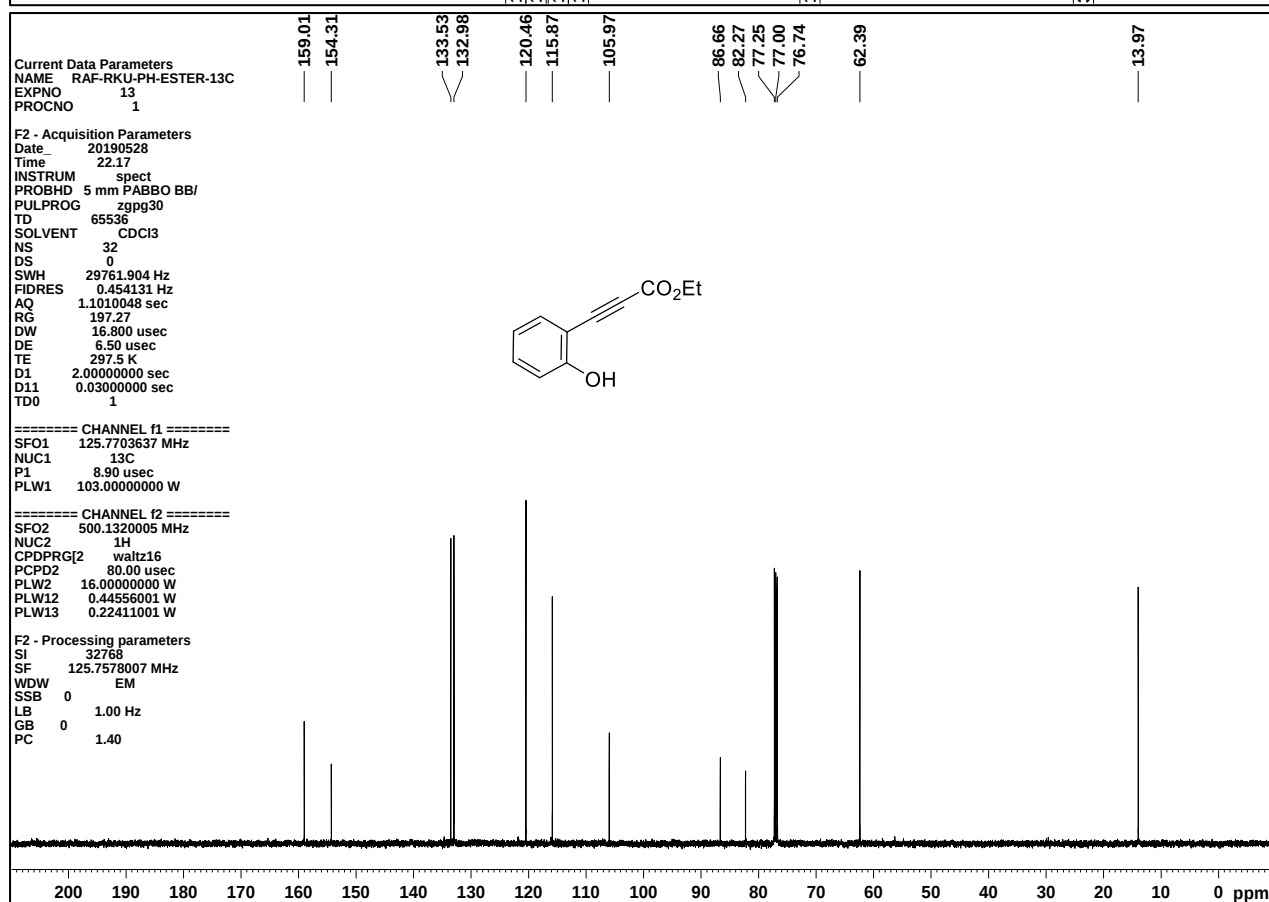
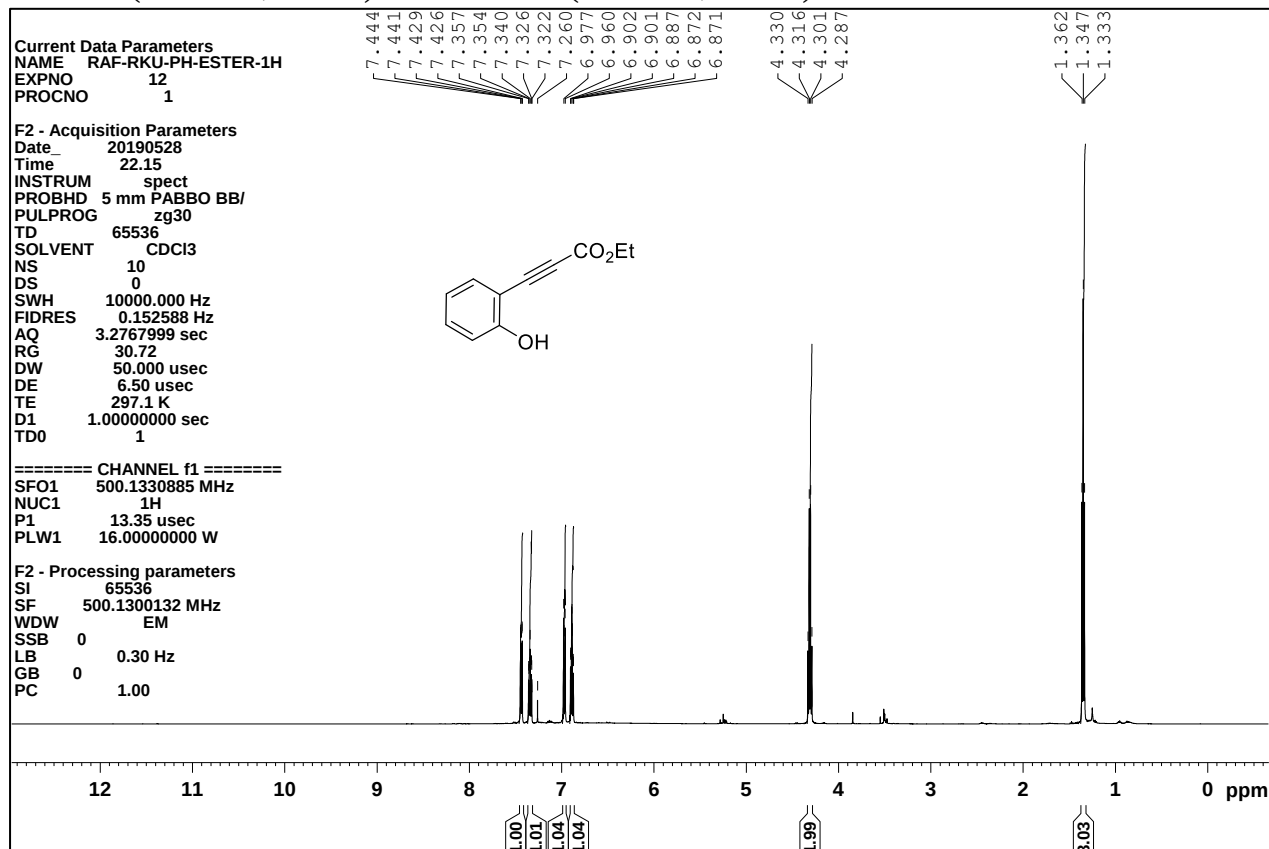
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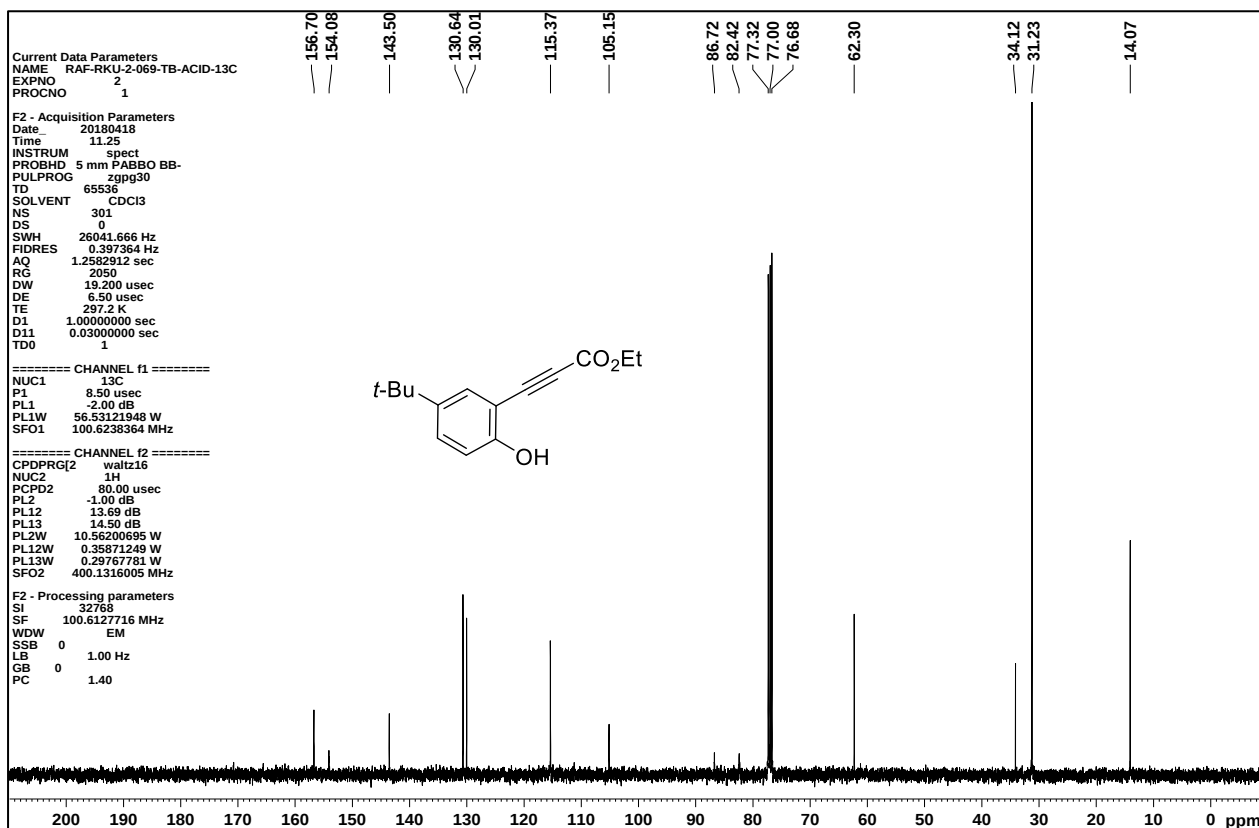
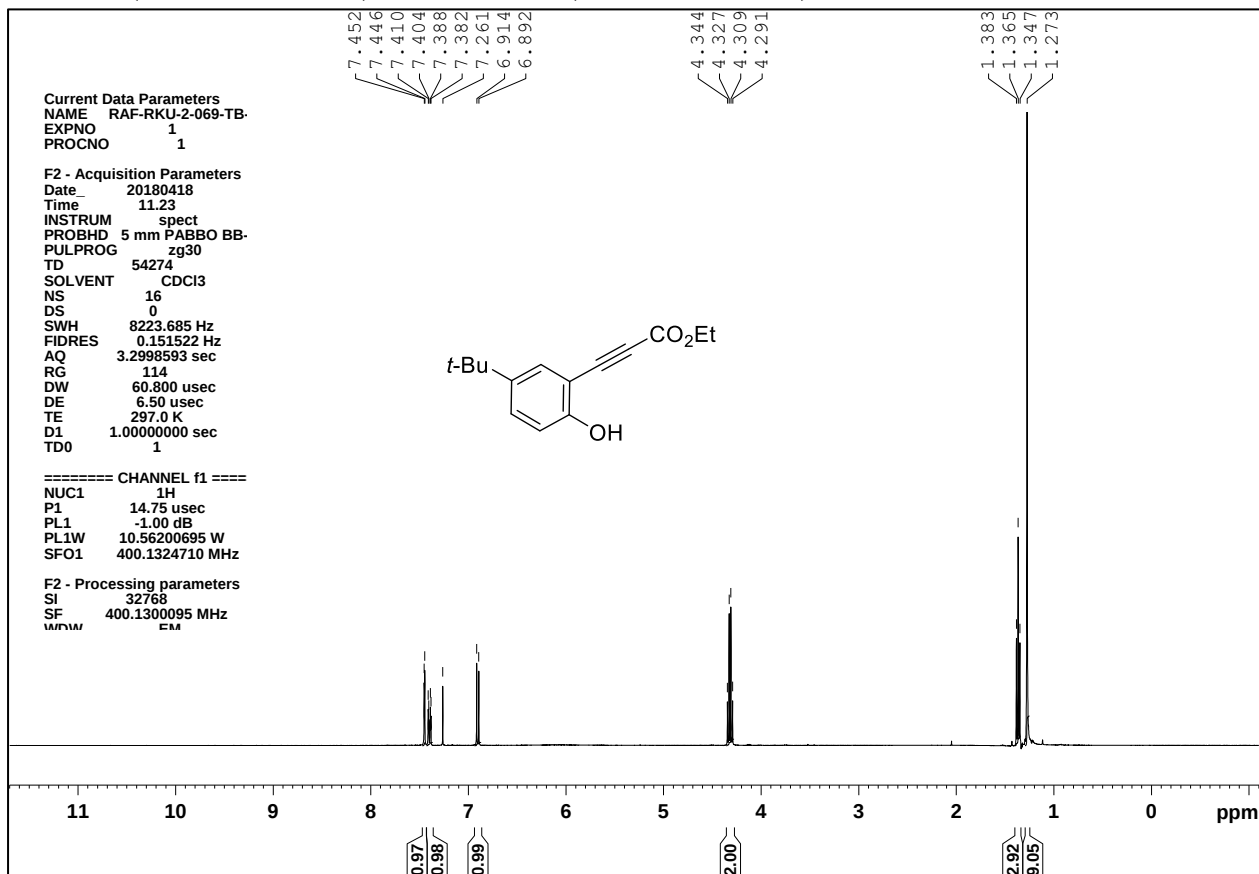
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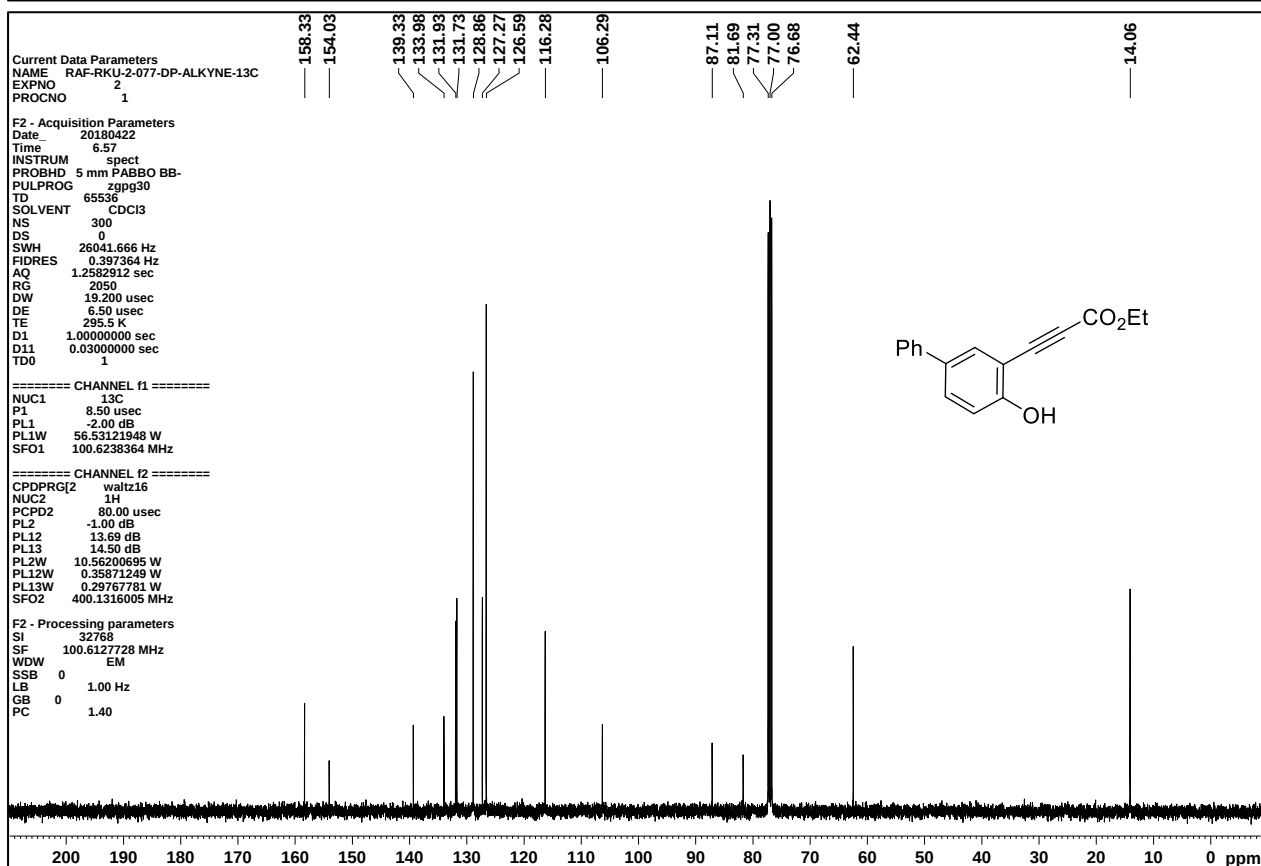
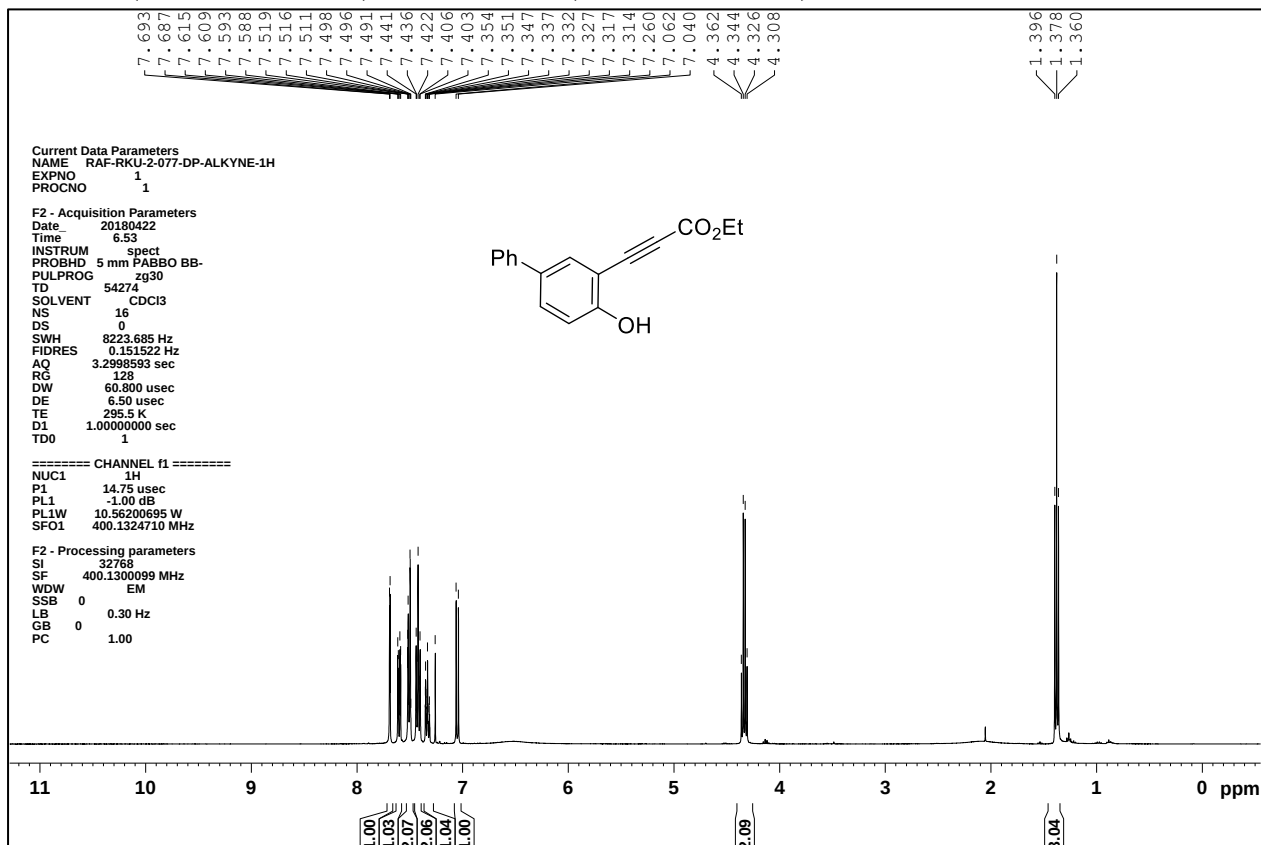
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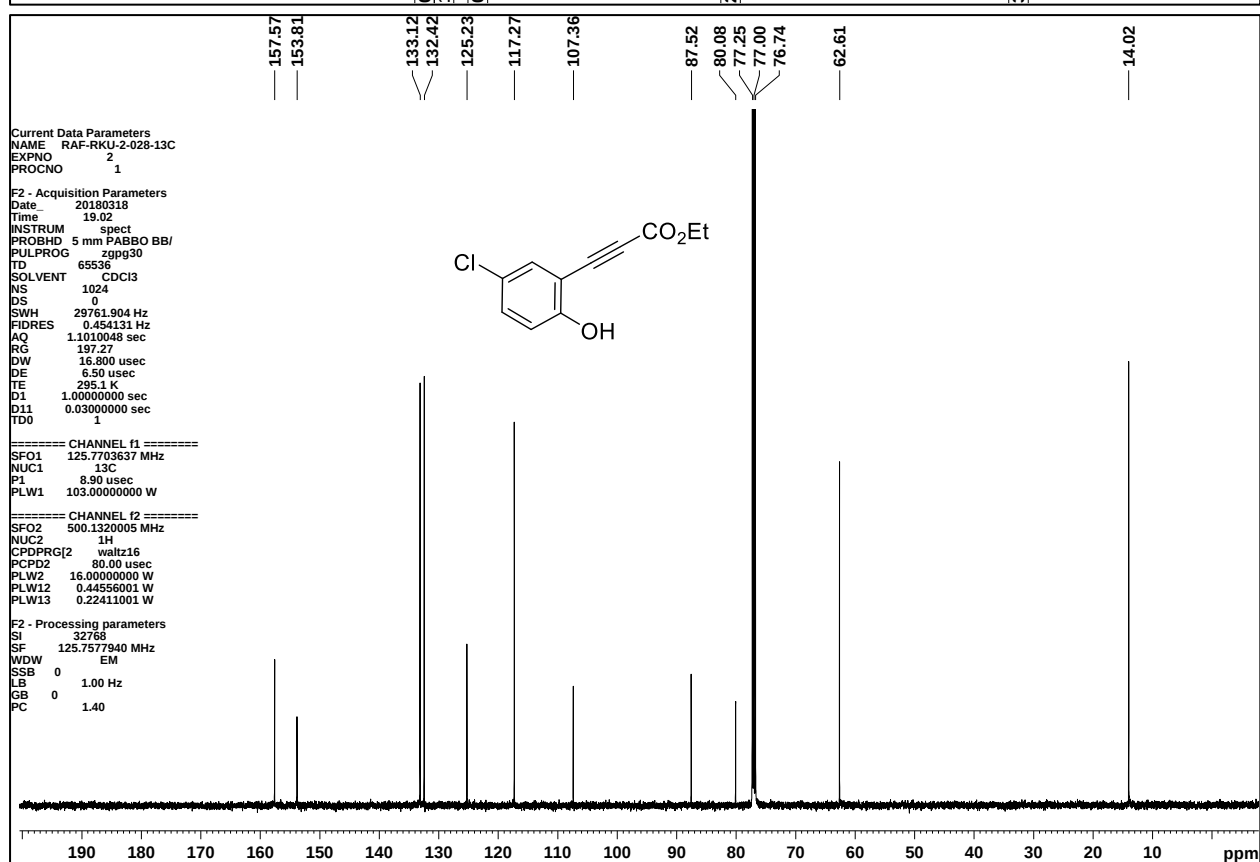
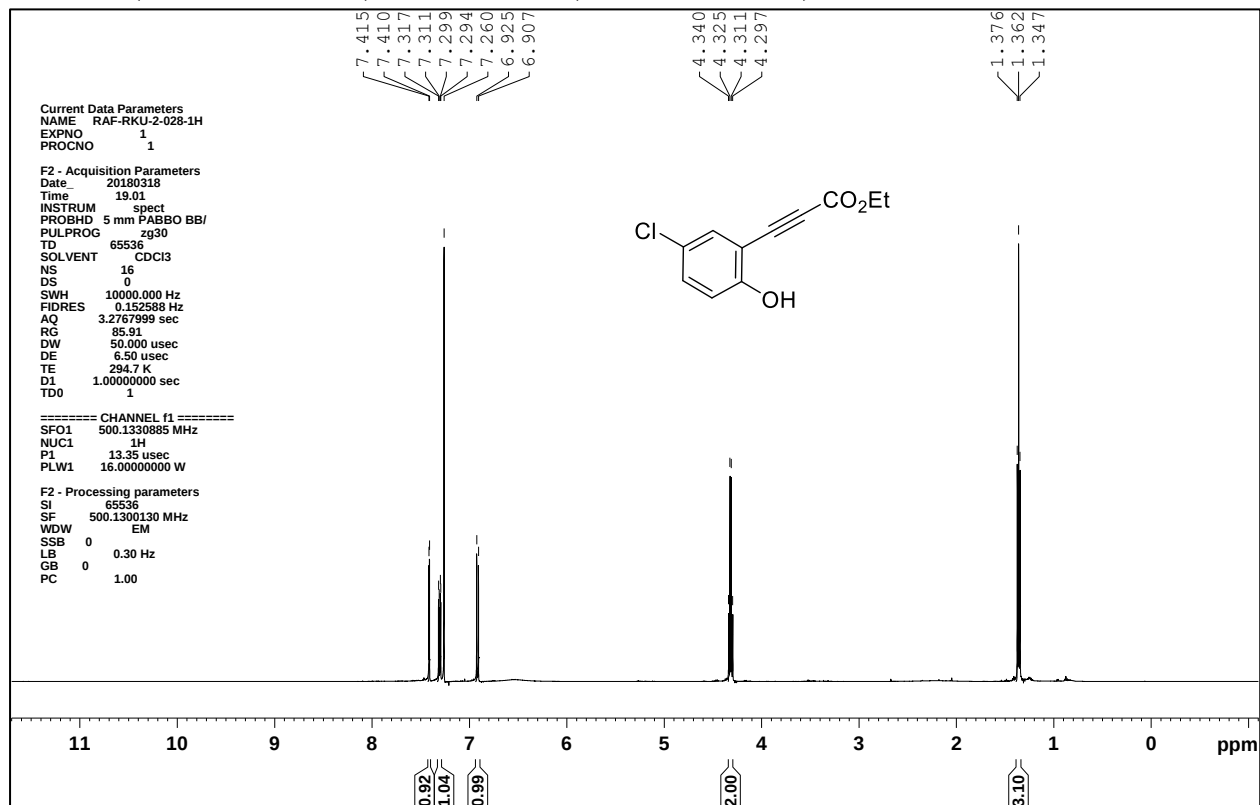
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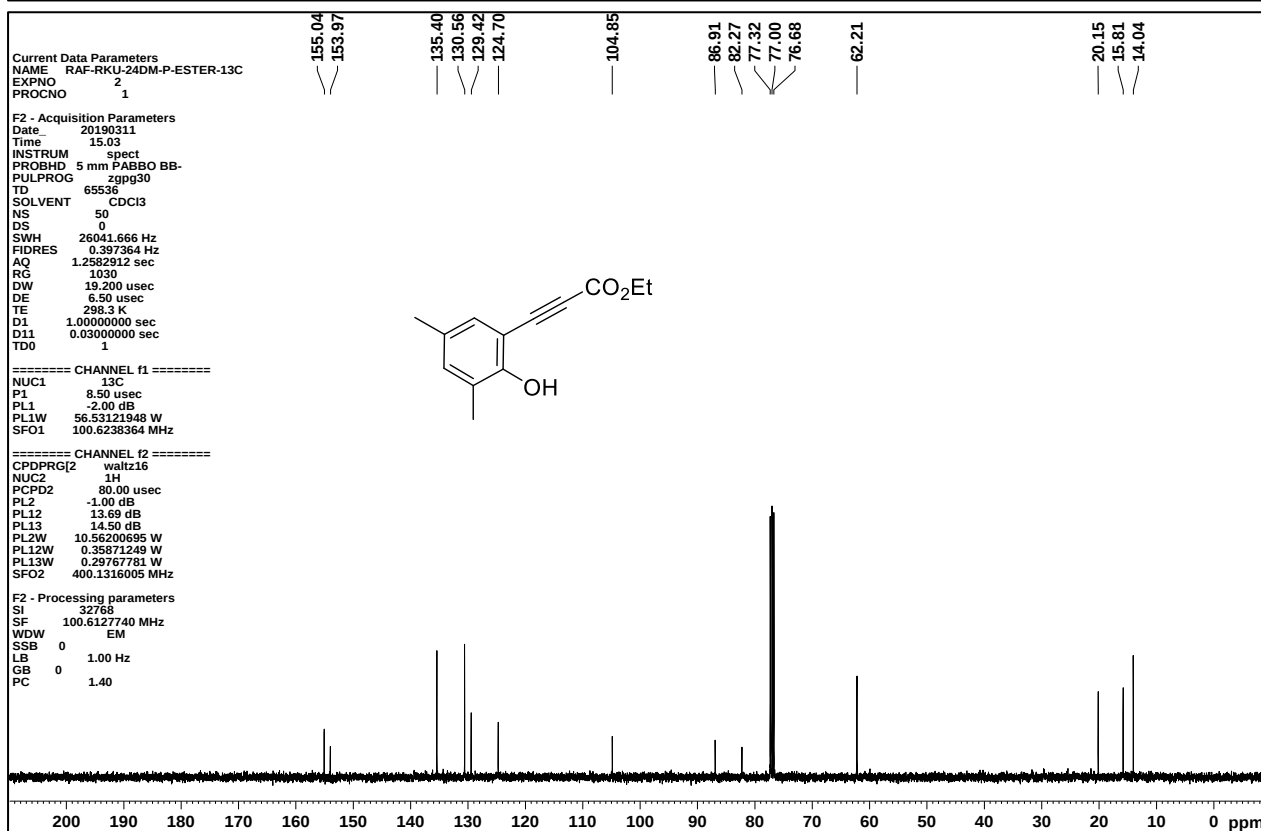
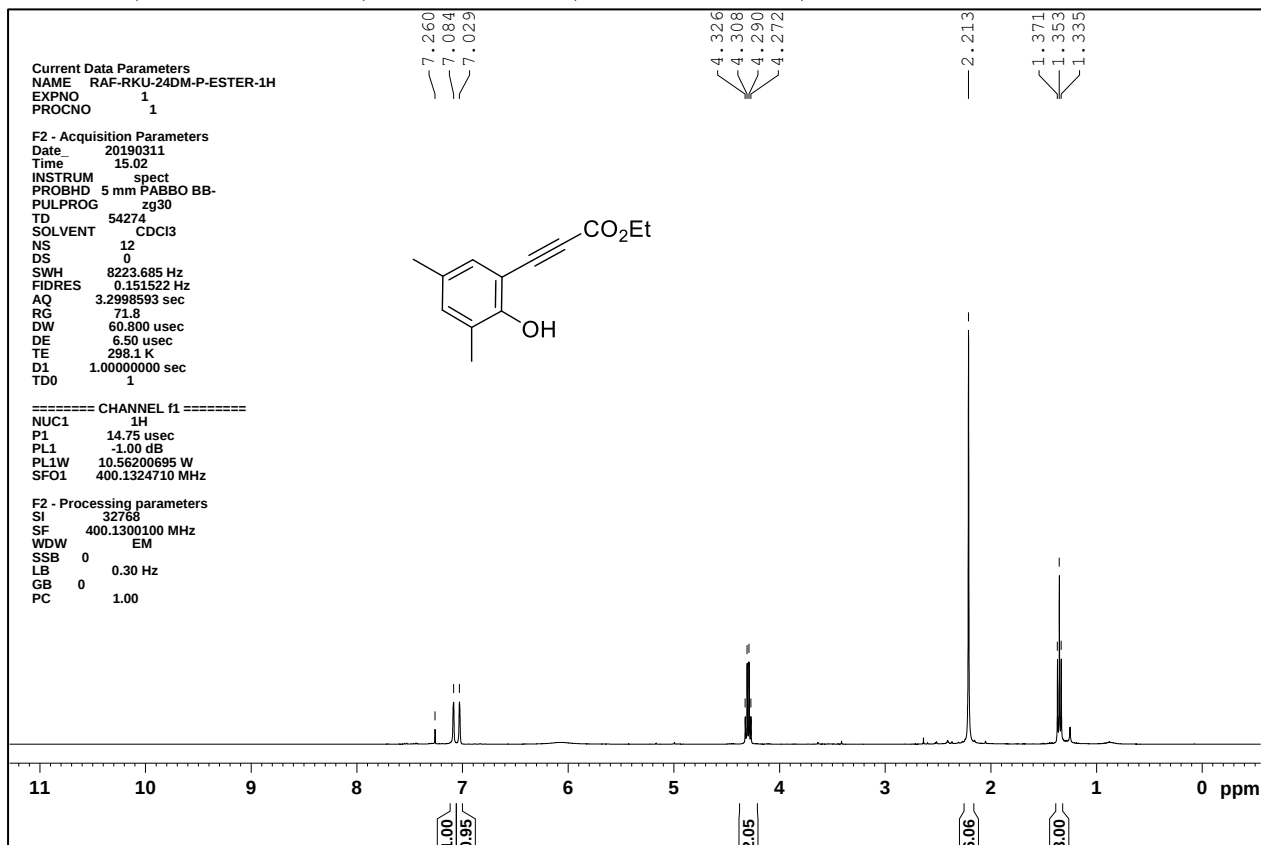
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **6d**



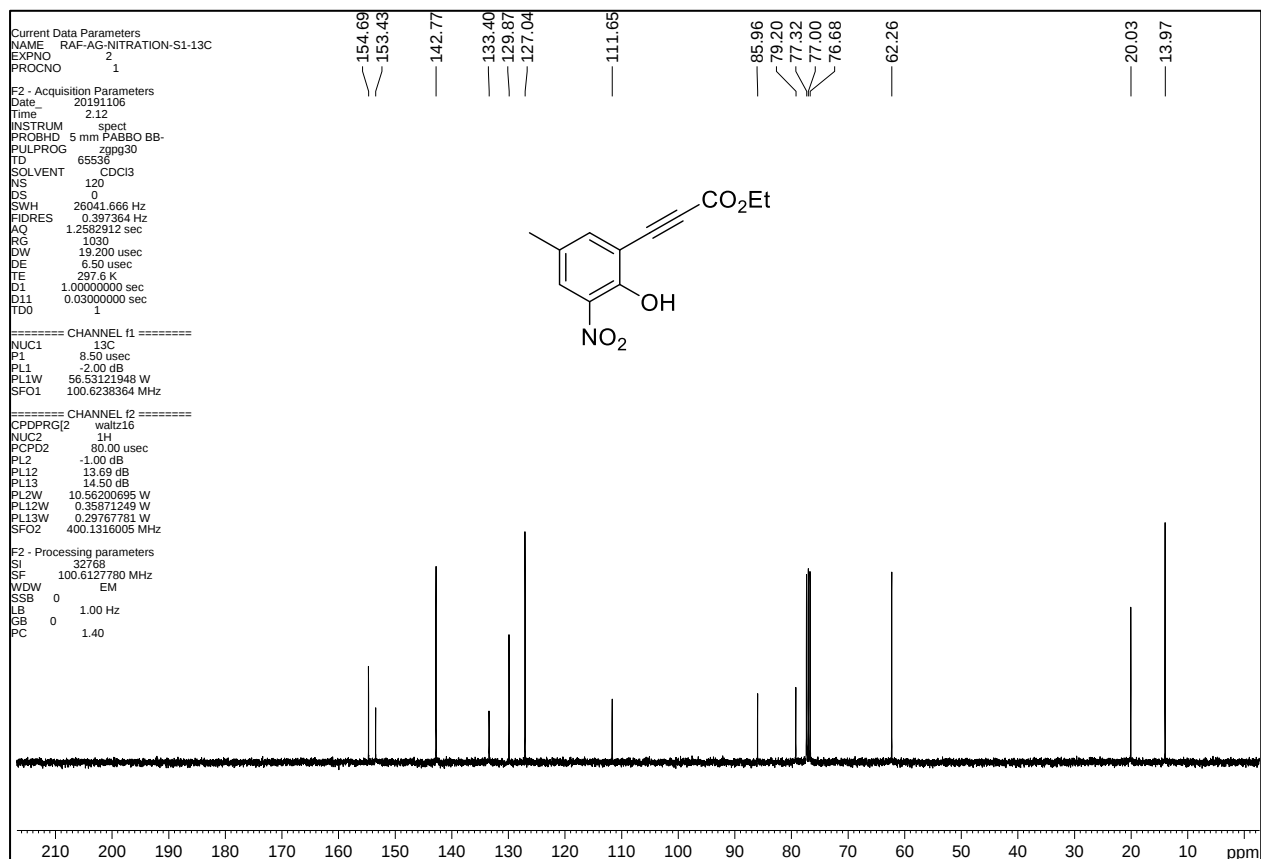
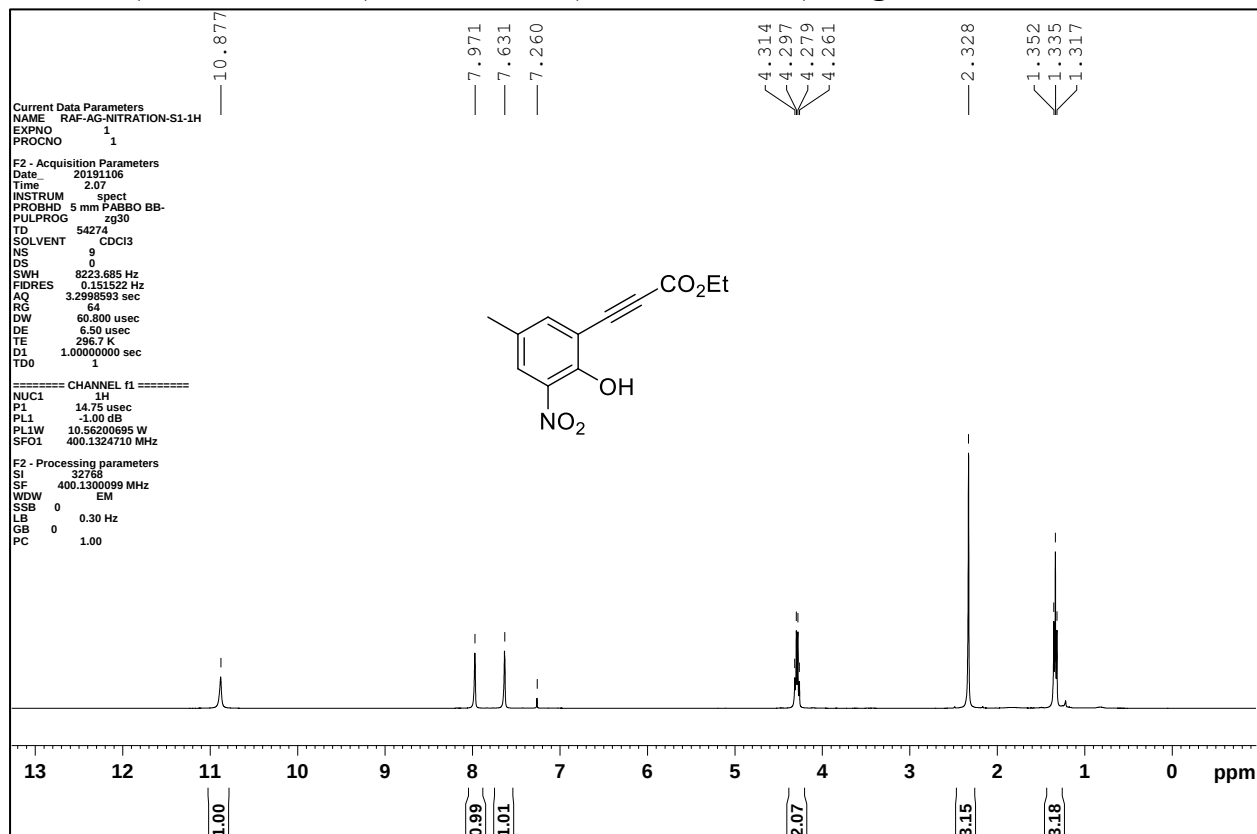
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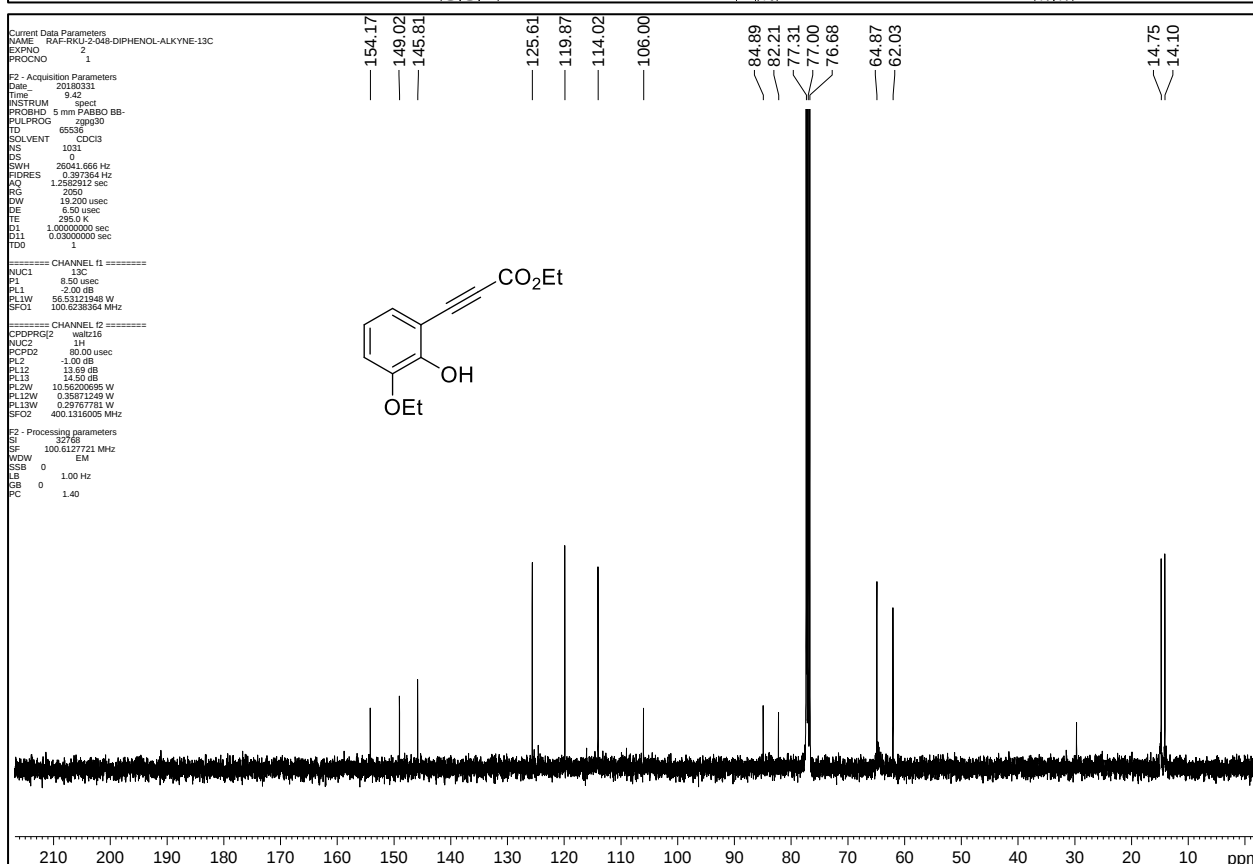
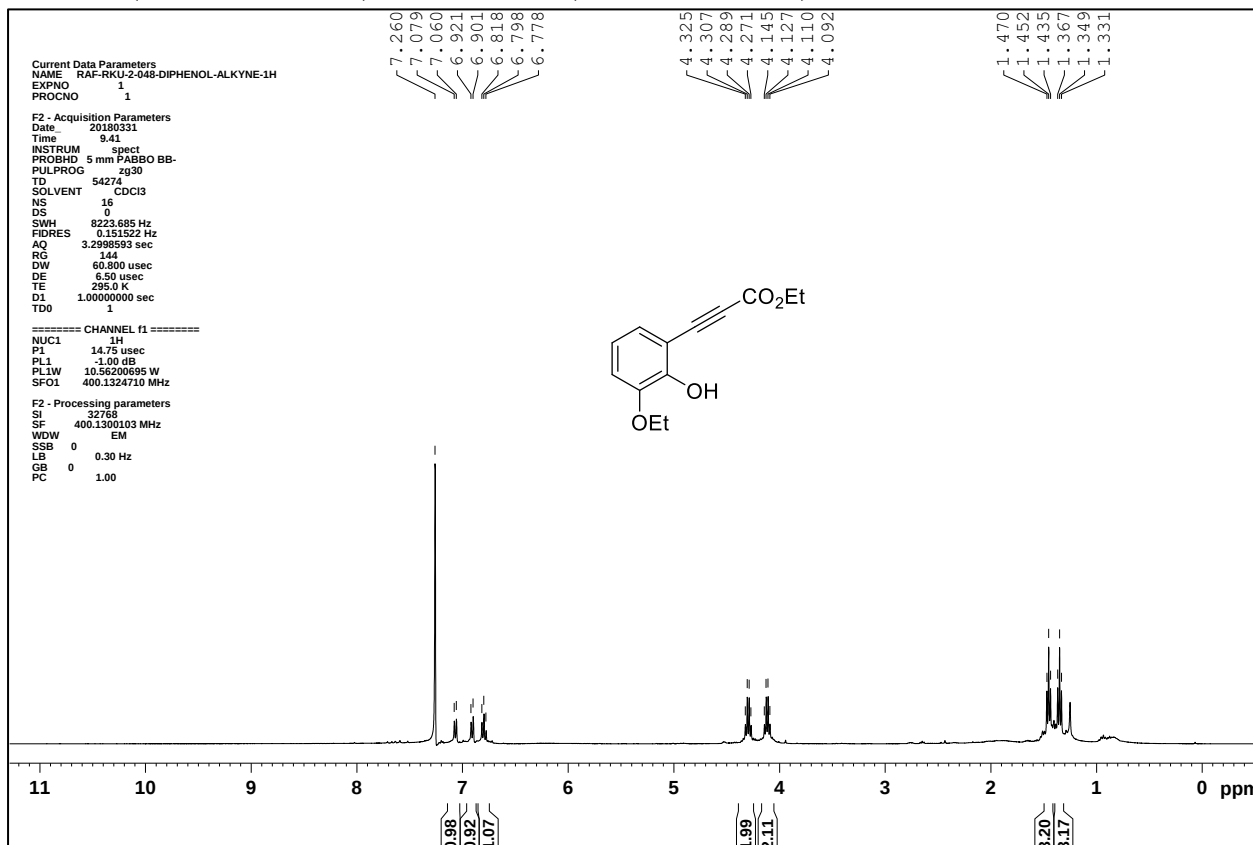
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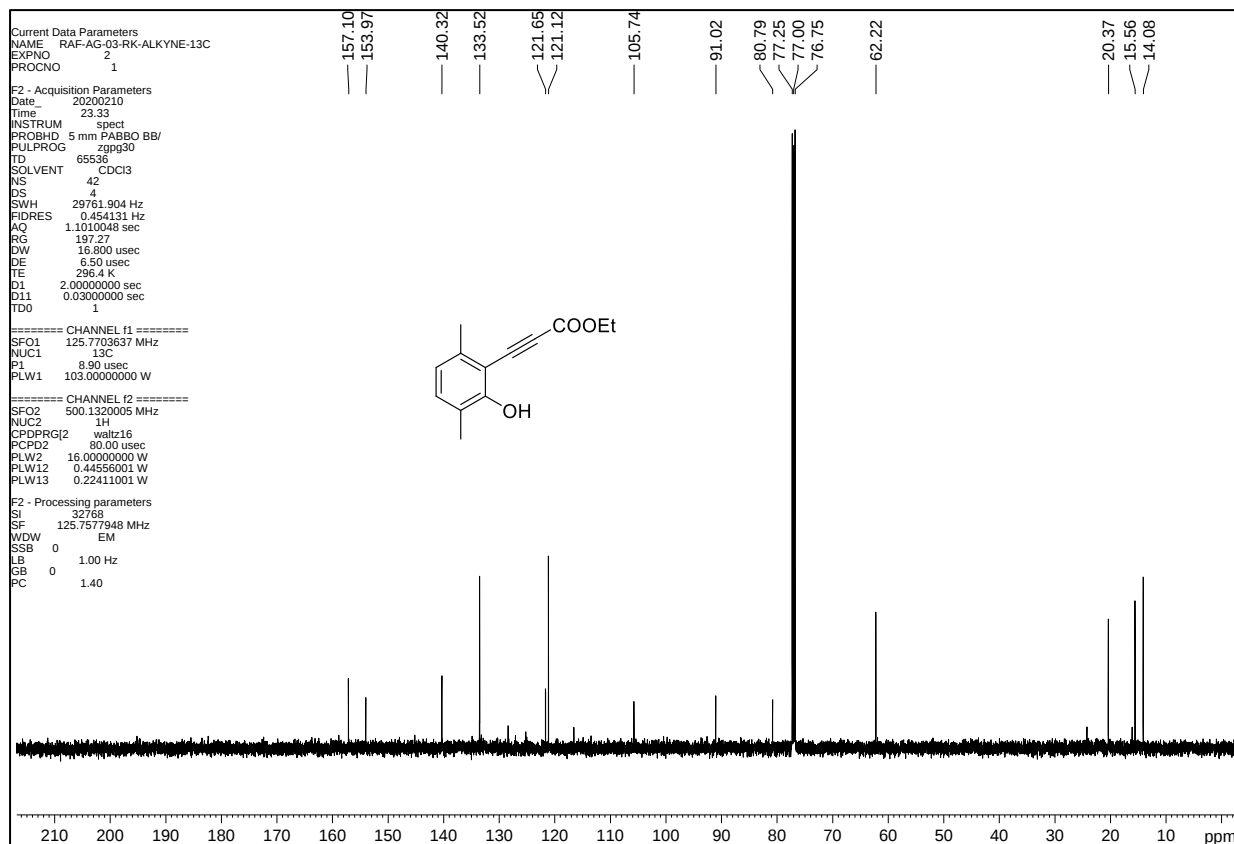
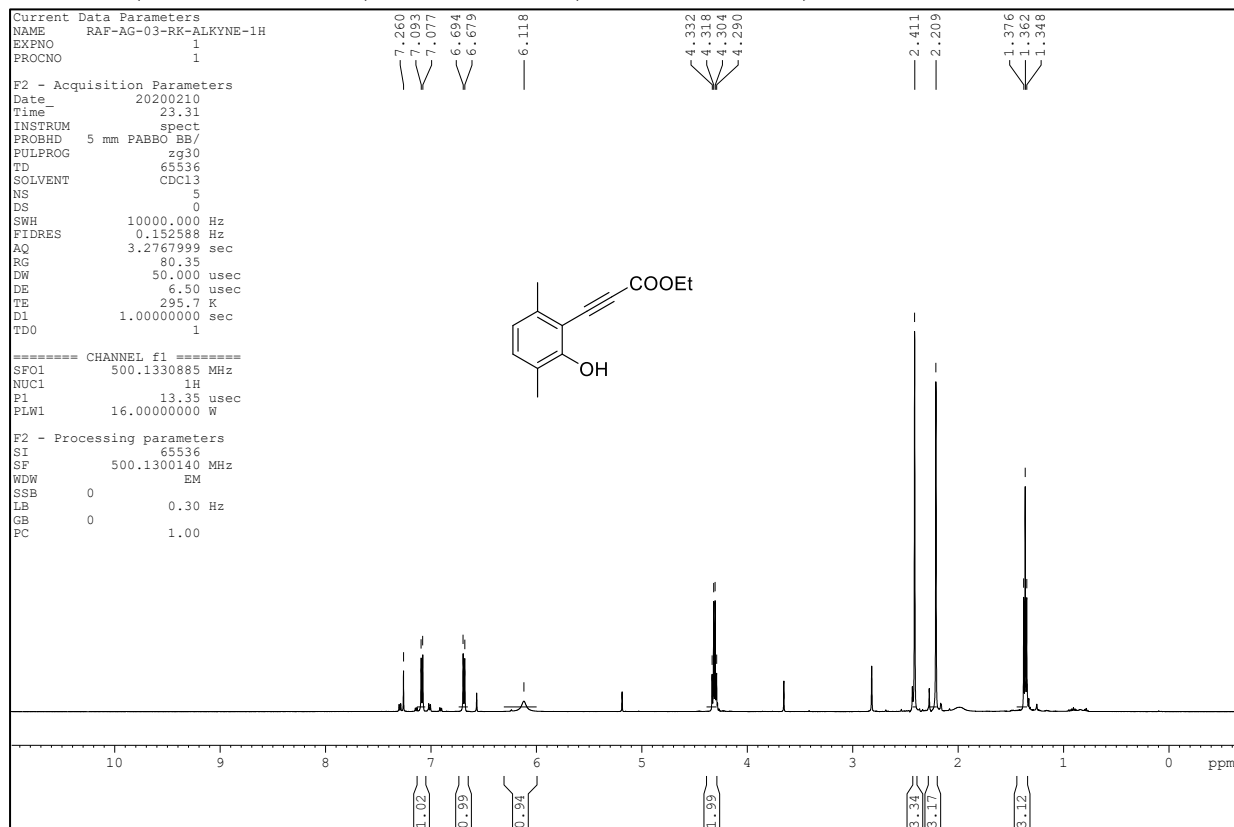
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **6g**



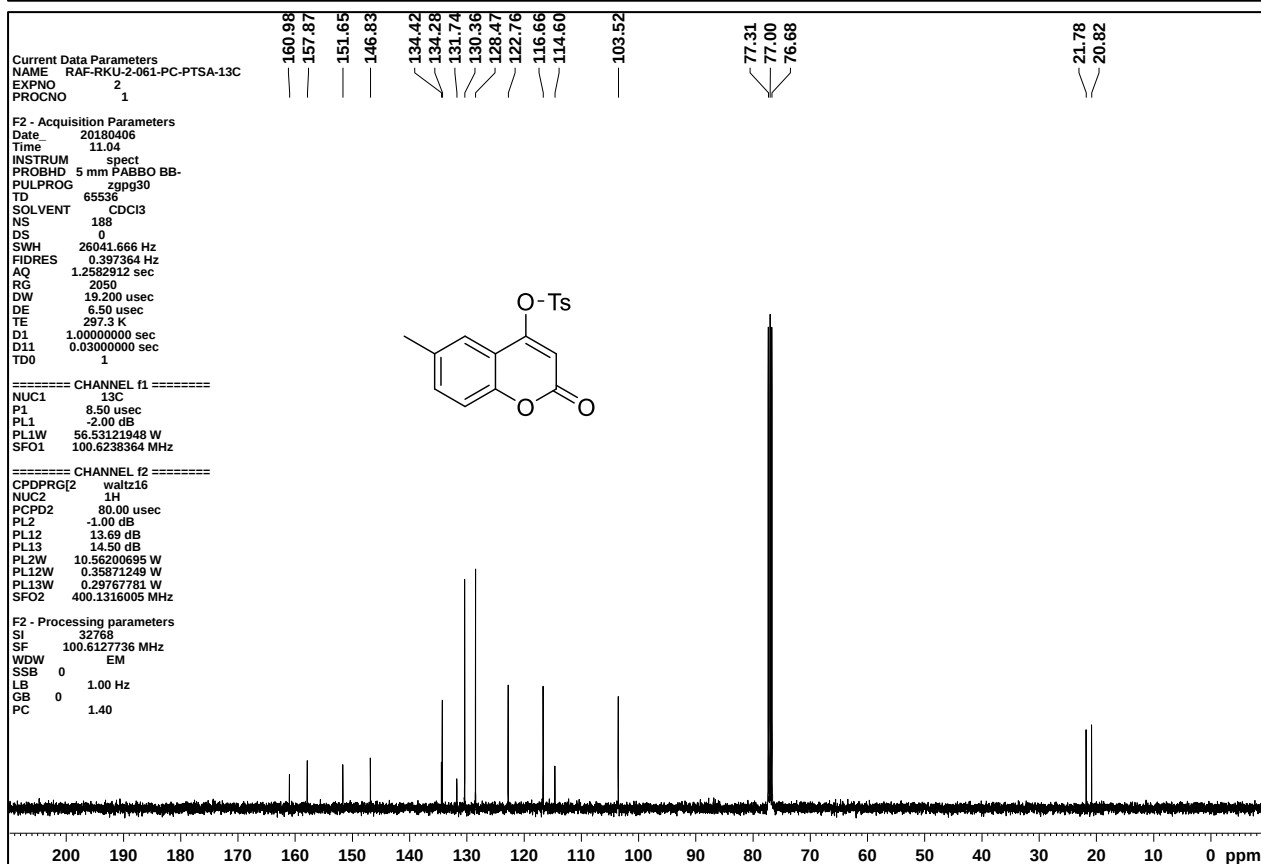
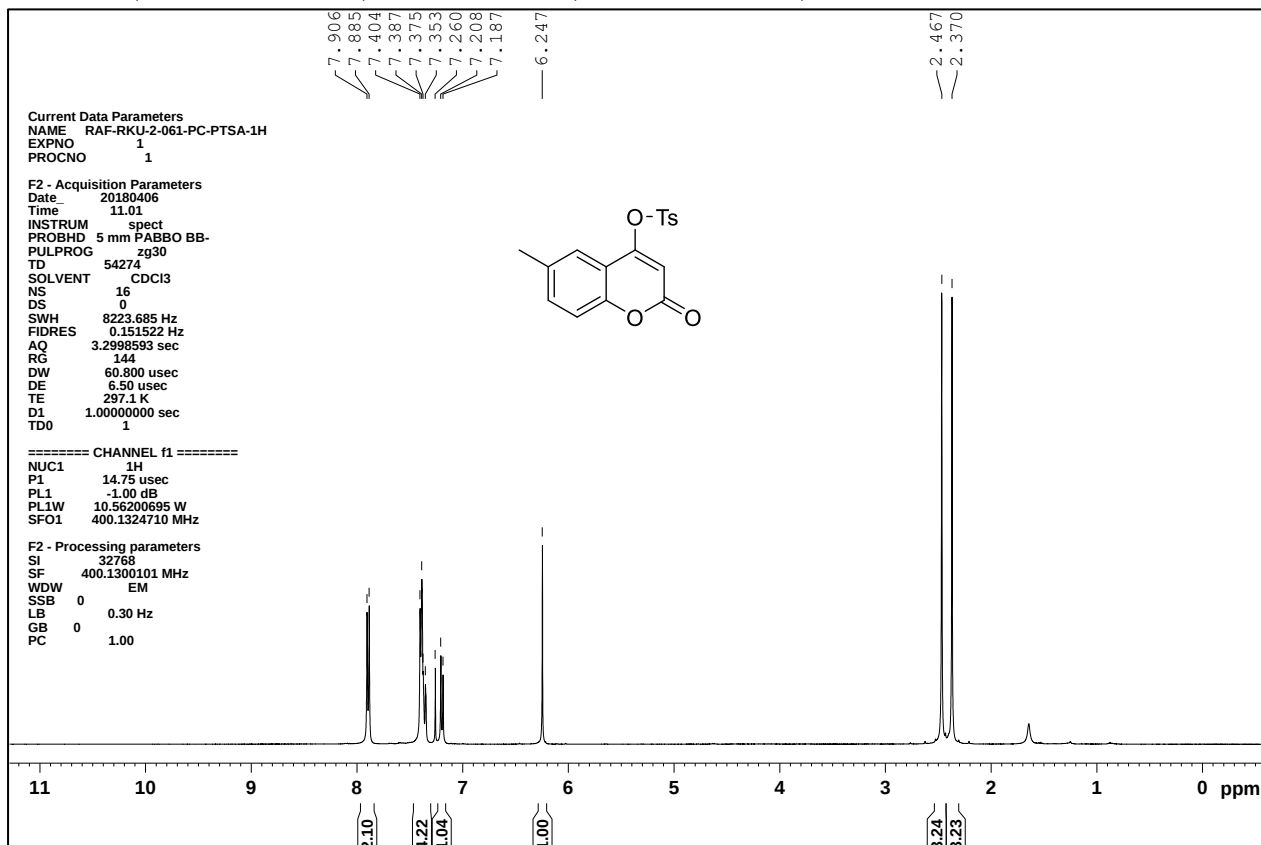
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **6h**



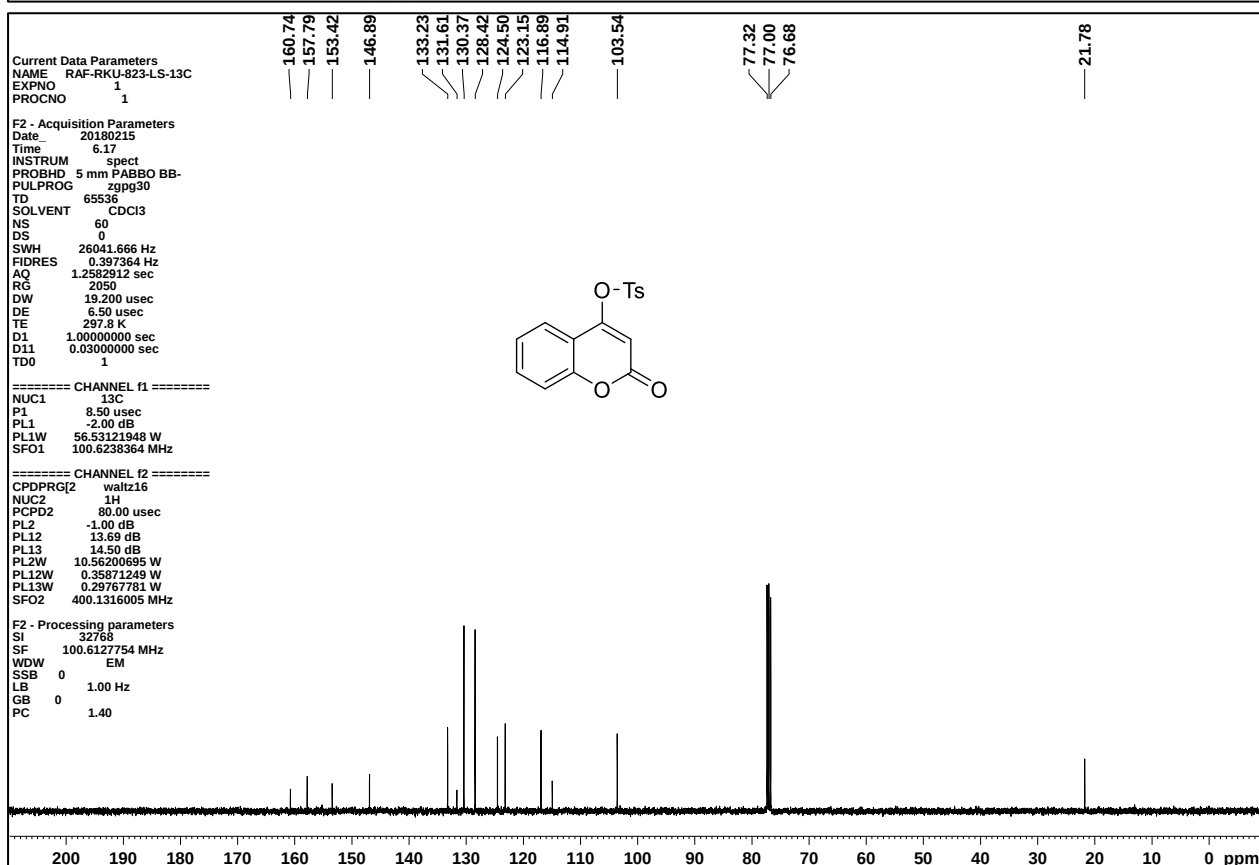
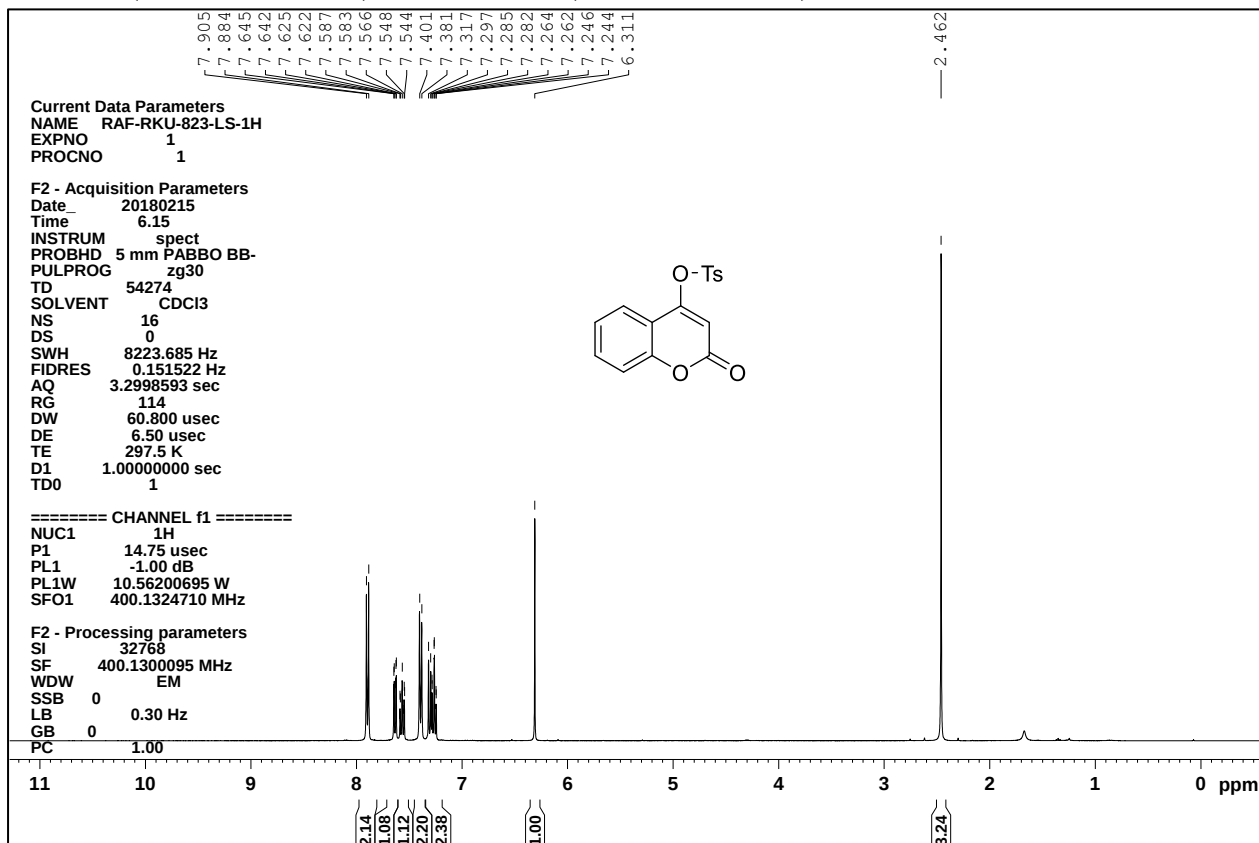
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **6i**



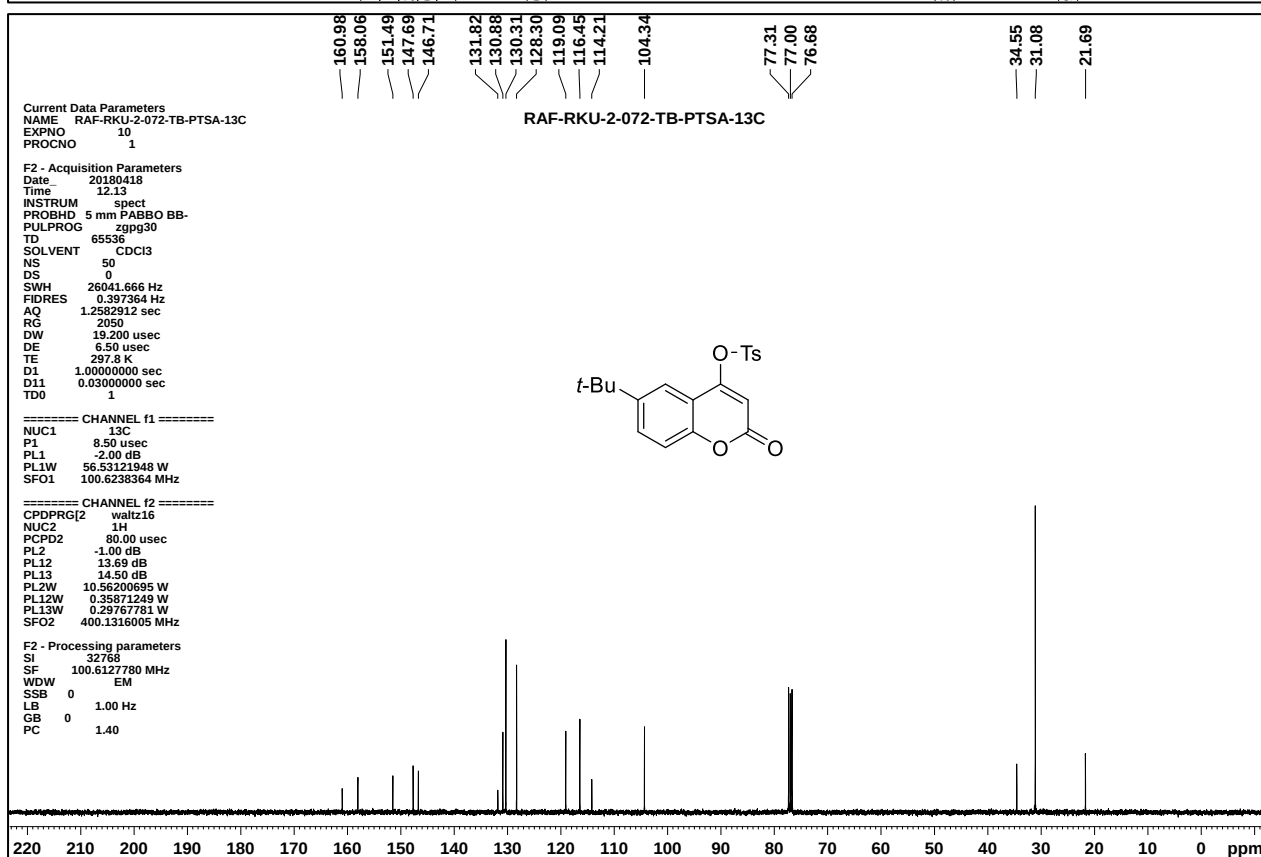
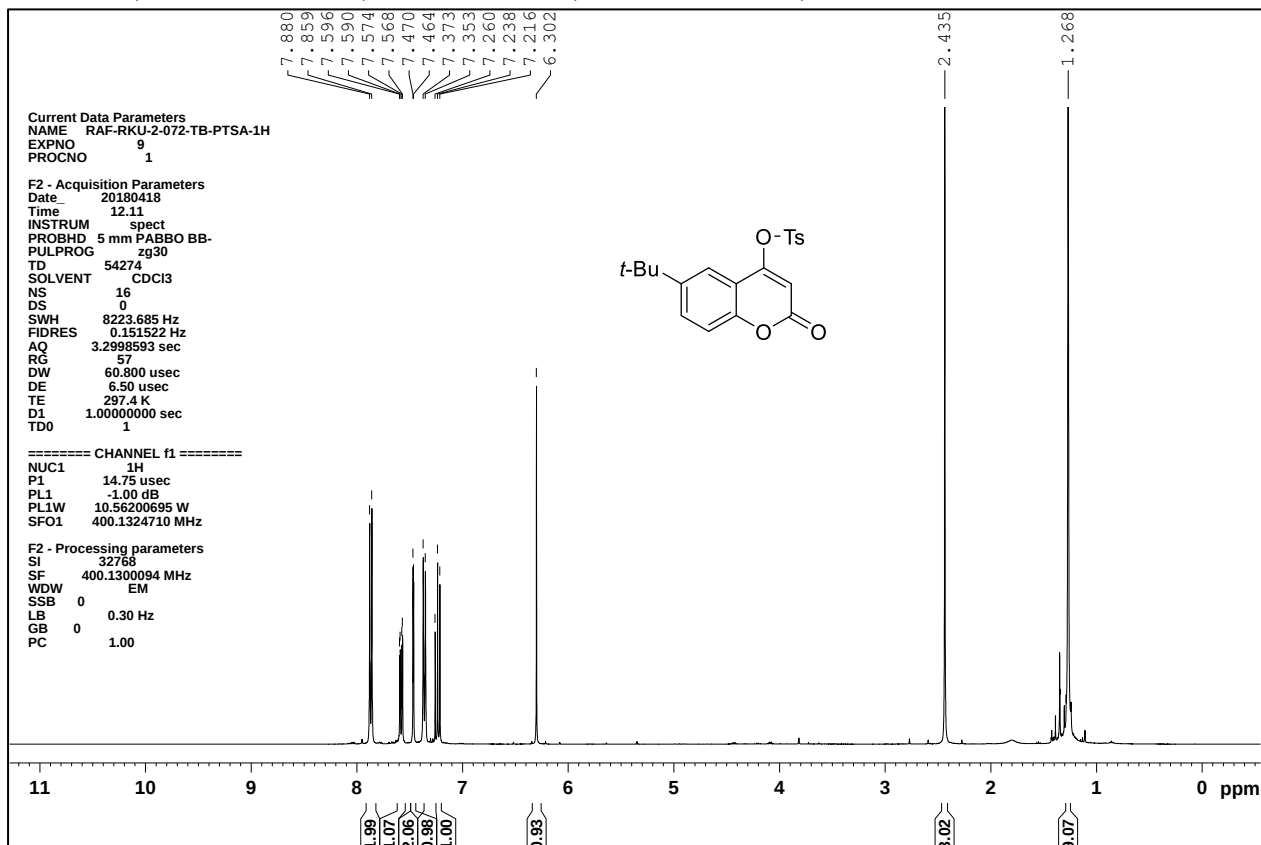
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **5a**



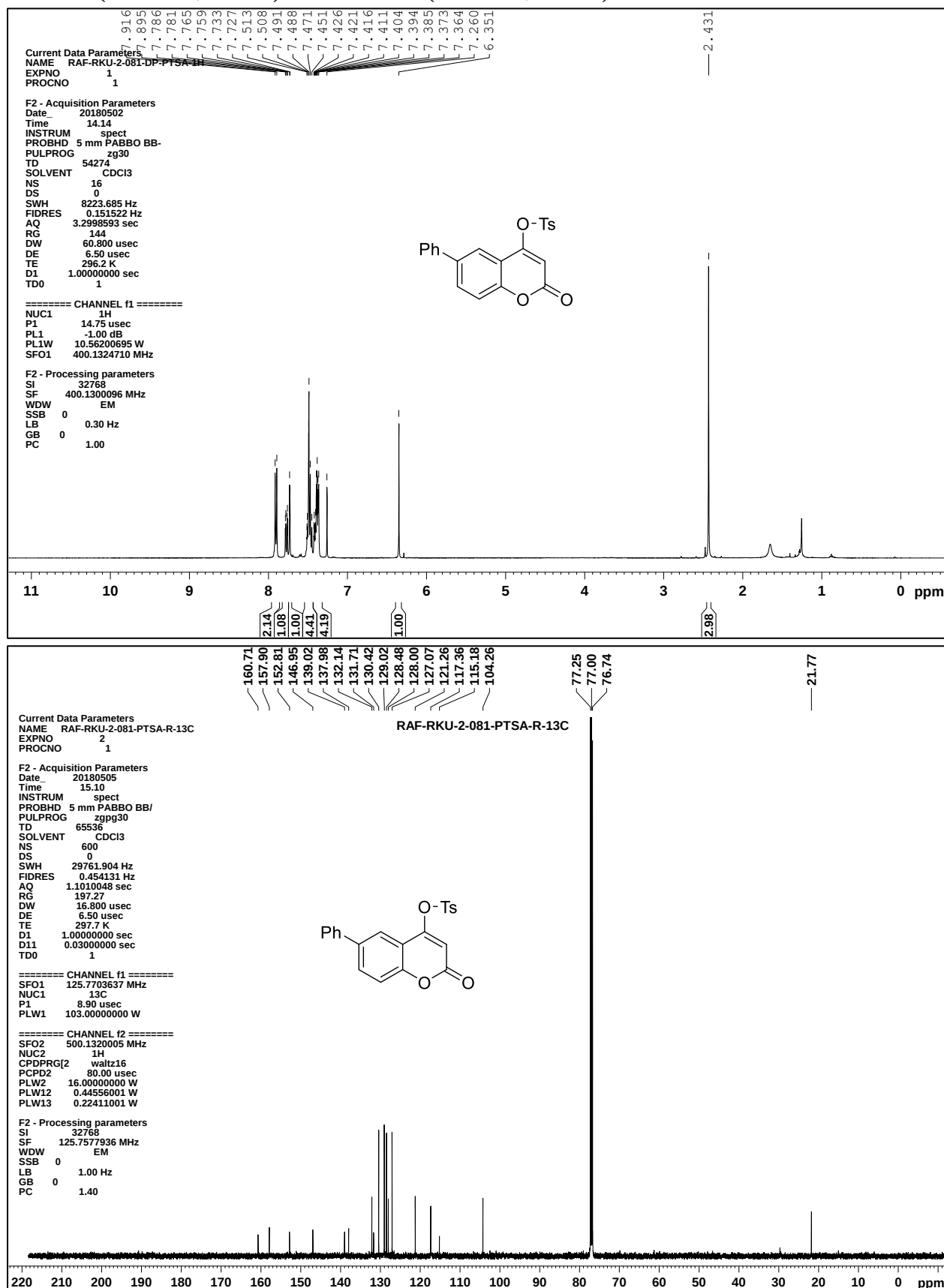
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **5b**



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of 5c



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **5d**



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PROCNO 1

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FIDRES 0.152588 Hz  
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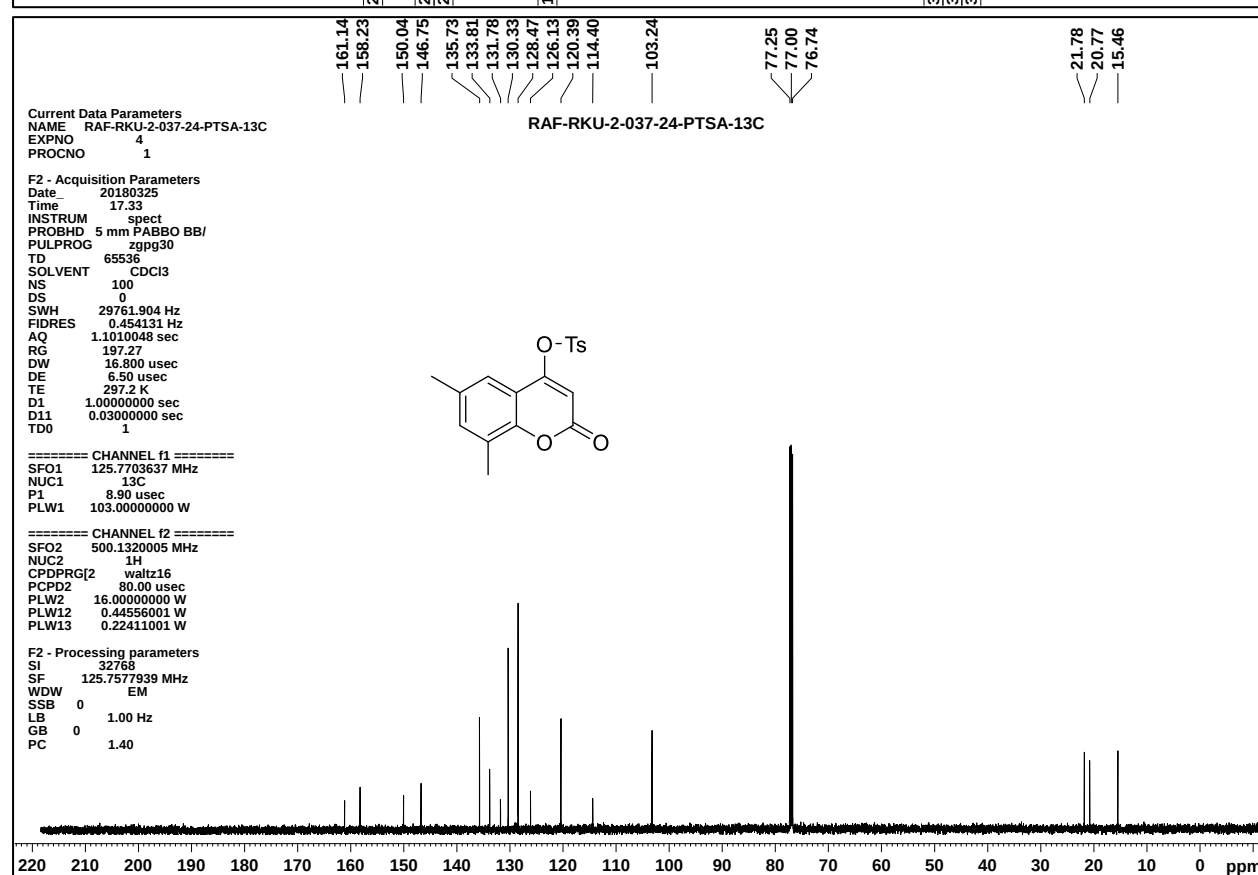
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7.883  
7.880  
7.394  
7.378  
7.260  
7.234  
7.225  
— 6.229

2.463  
2.373  
2.326

11 10 9 8 7 6 5 4 3 2 1 0 ppm

2.14  
2.20  
2.23  
1.00  
3.10  
3.10  
3.15

Cc1cc(C)c2c(c1)c(=O)oc2O[Si](C)(C)C

Current Data Parameters

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| PROCNO | 1                    |

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| PULPROG | zg30           |
| TD      | 54274          |
| SOLVENT | CDCl3          |
| NS      | 3              |
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| SWH     | 8223.685 Hz    |
| FIDRES  | 0.151522 Hz    |
| AQ      | 3.2998593 sec  |
| RG      | 57             |
| DW      | 60.800 usec    |
| DE      | 6.50 usec      |
| TE      | 295.7 K        |
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| TD0     | 1              |

Chemical structure of 4-methyl-2-(4-methylphenyl)-2H-chromene-3-carboxylic acid (1):

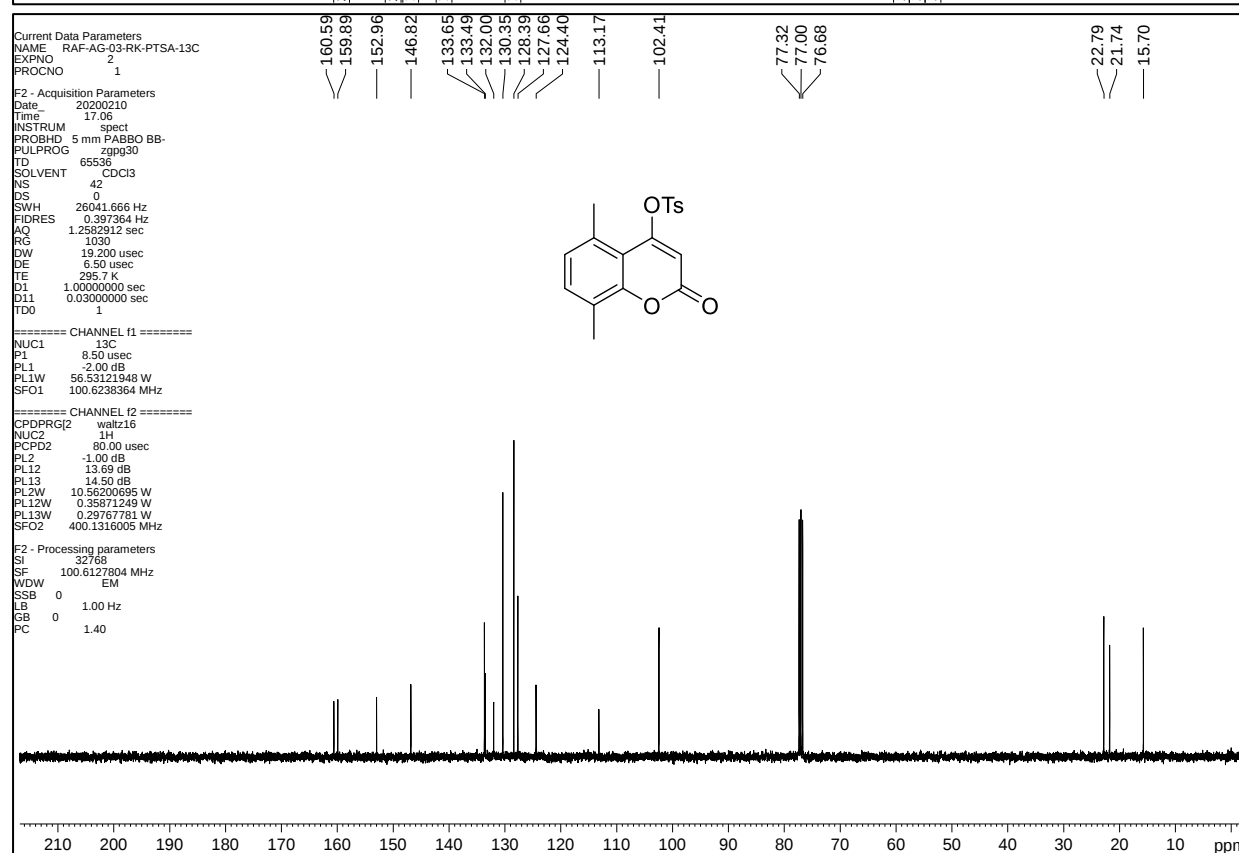
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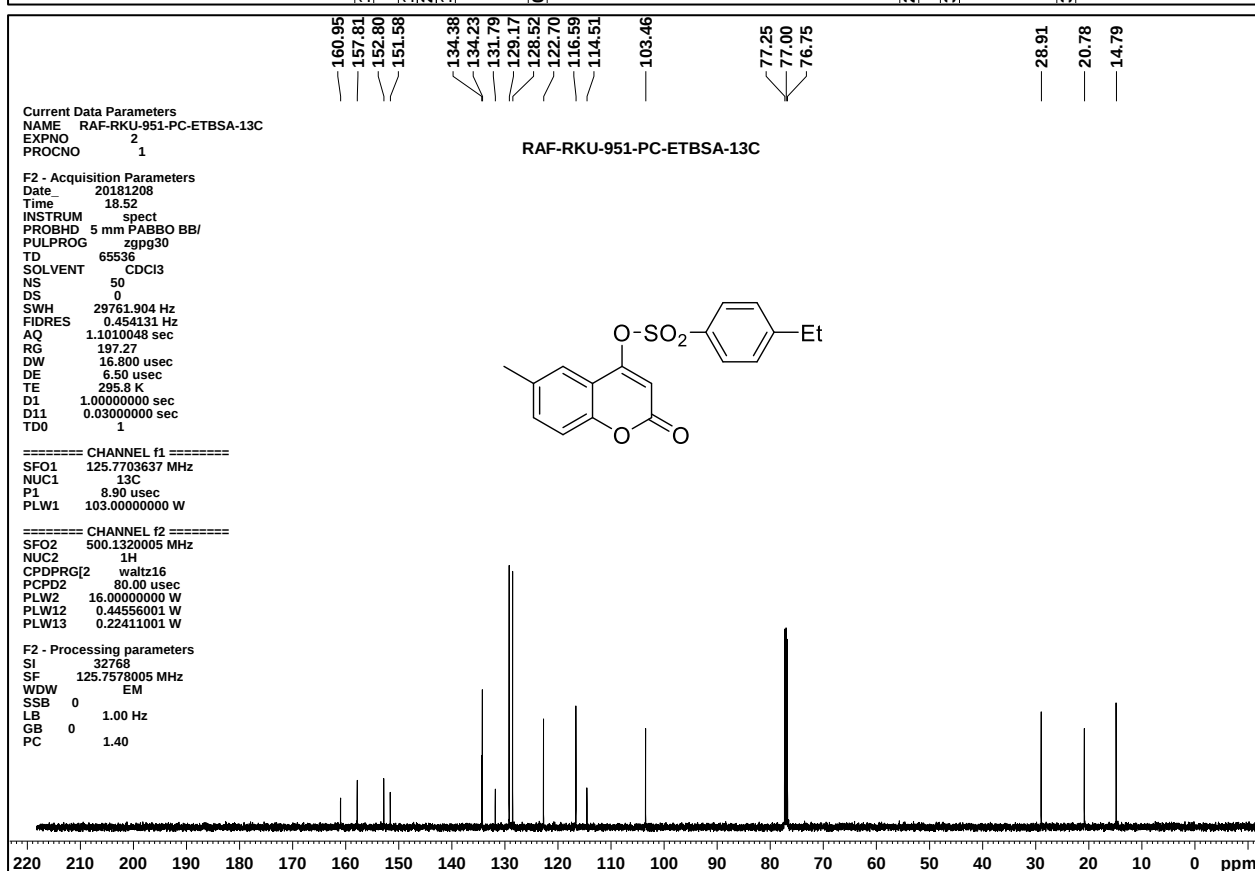
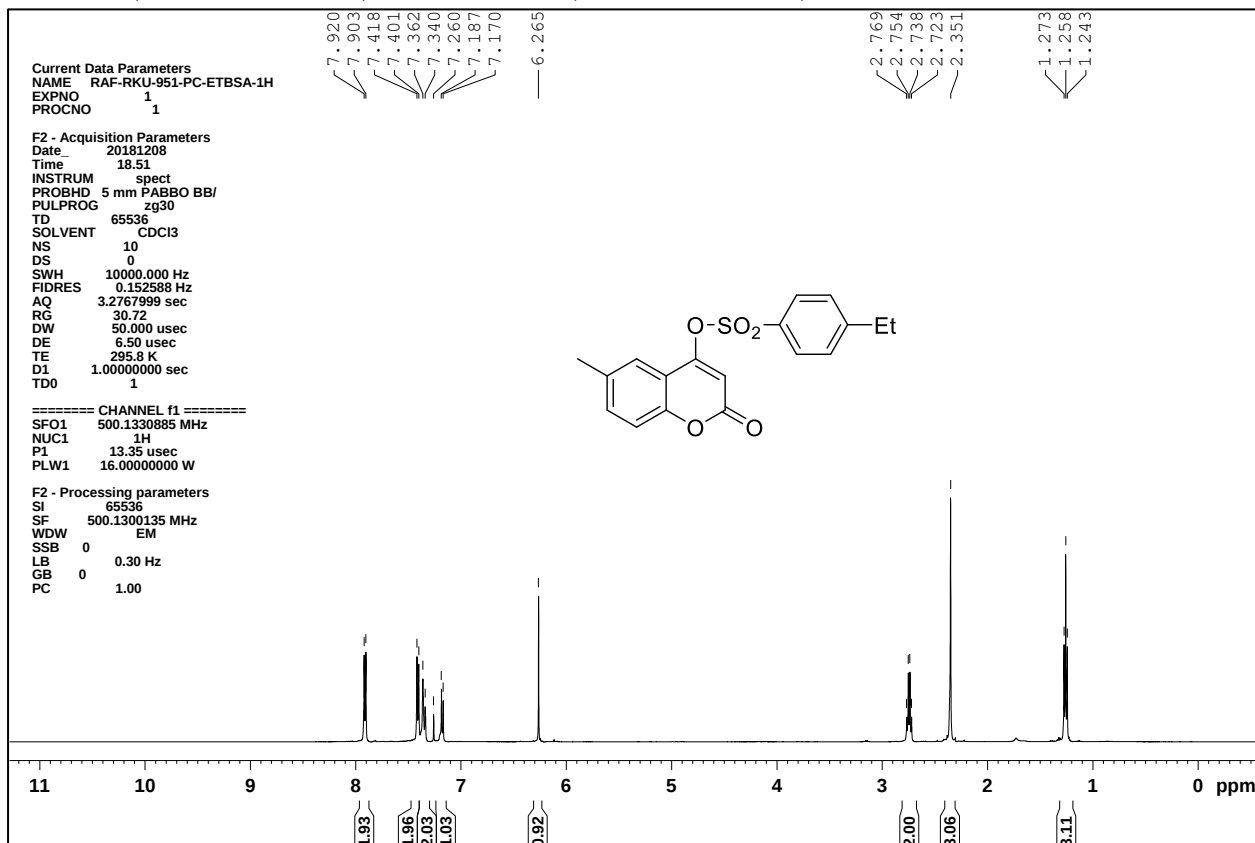
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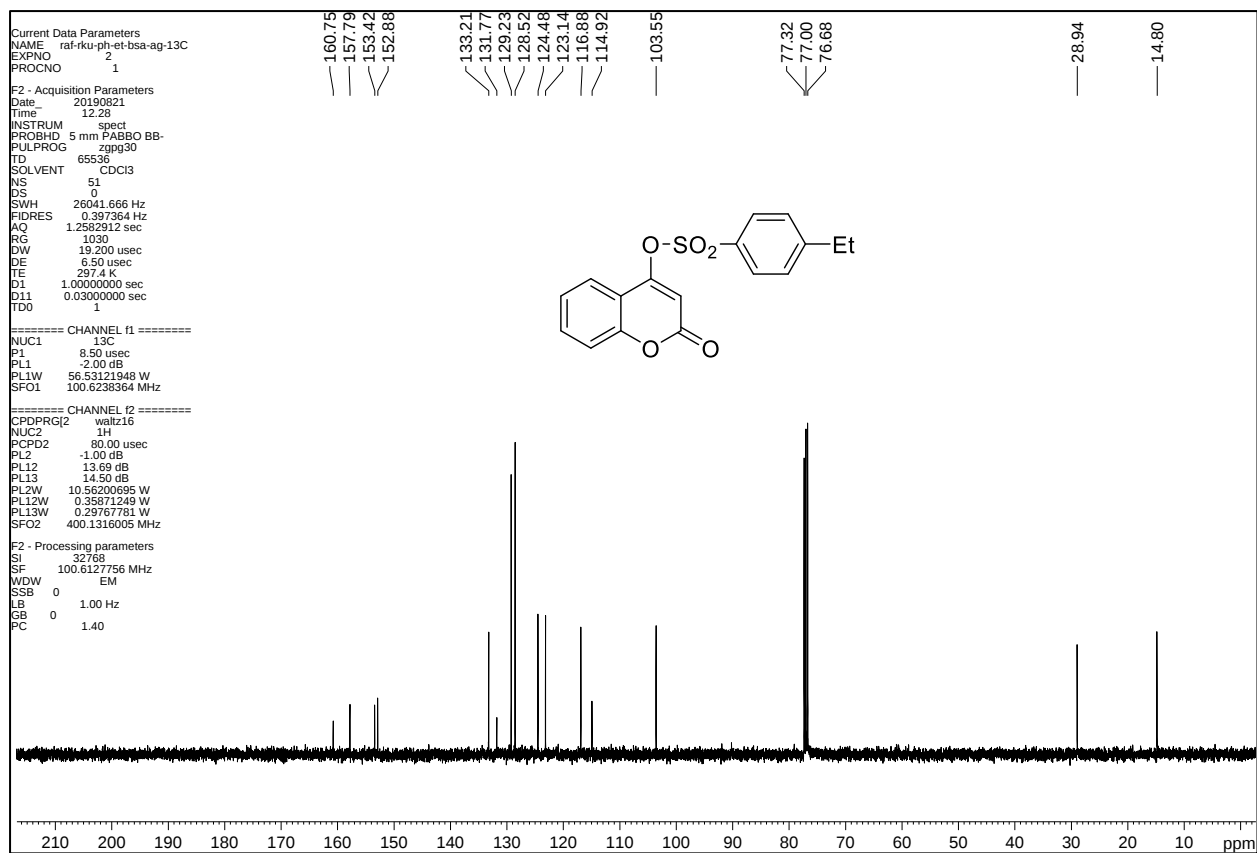
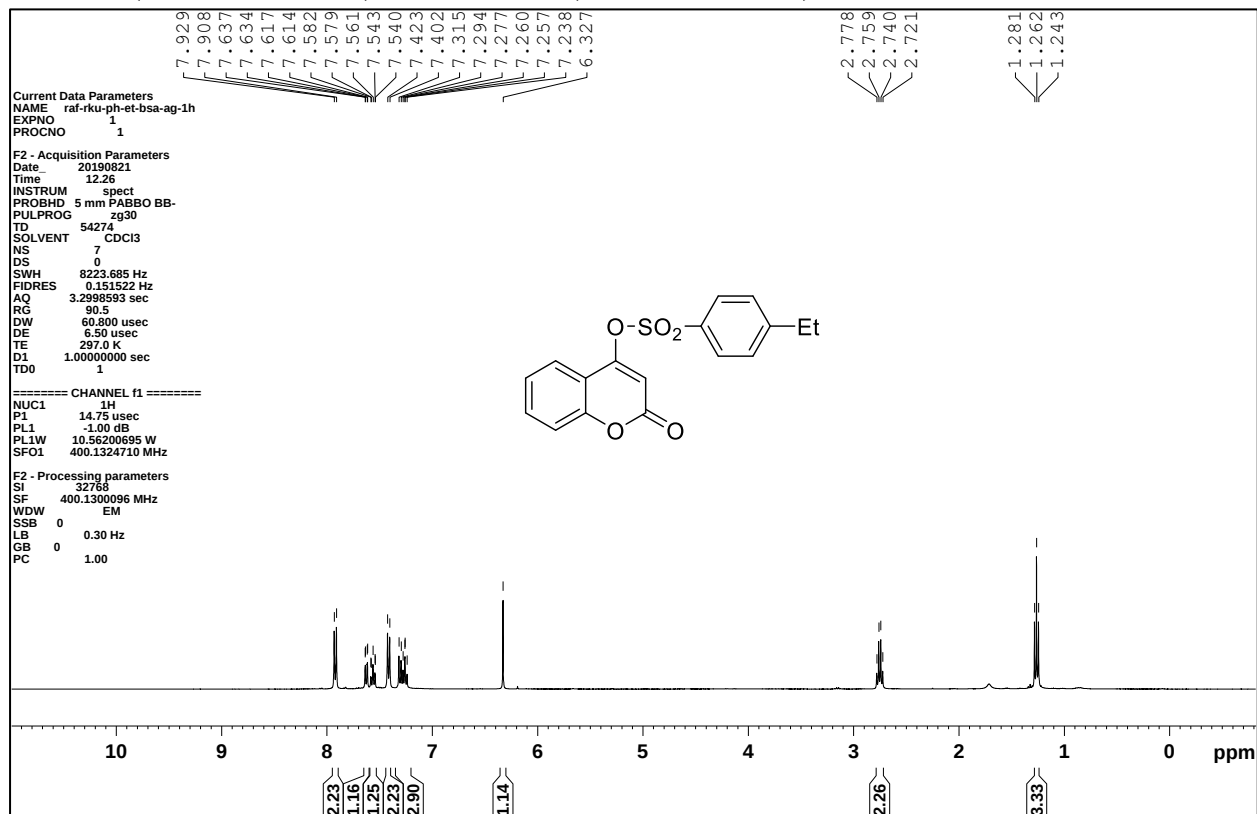
1H NMR spectrum (CDCl3) of compound 1. The x-axis represents chemical shift in ppm, ranging from 0 to 10. The spectrum shows several peaks: a singlet at ~7.8 ppm (integration 2.02), a multiplet between 7.0 and 7.5 ppm (integrations 2.04, 1.13, 1.03), a singlet at ~6.3 ppm (integration 1.00), and a doublet at ~2.4 ppm (integration 3.06, 3.16, 3.15). The solvent peak for CDCl3 is visible at ~7.26 ppm.



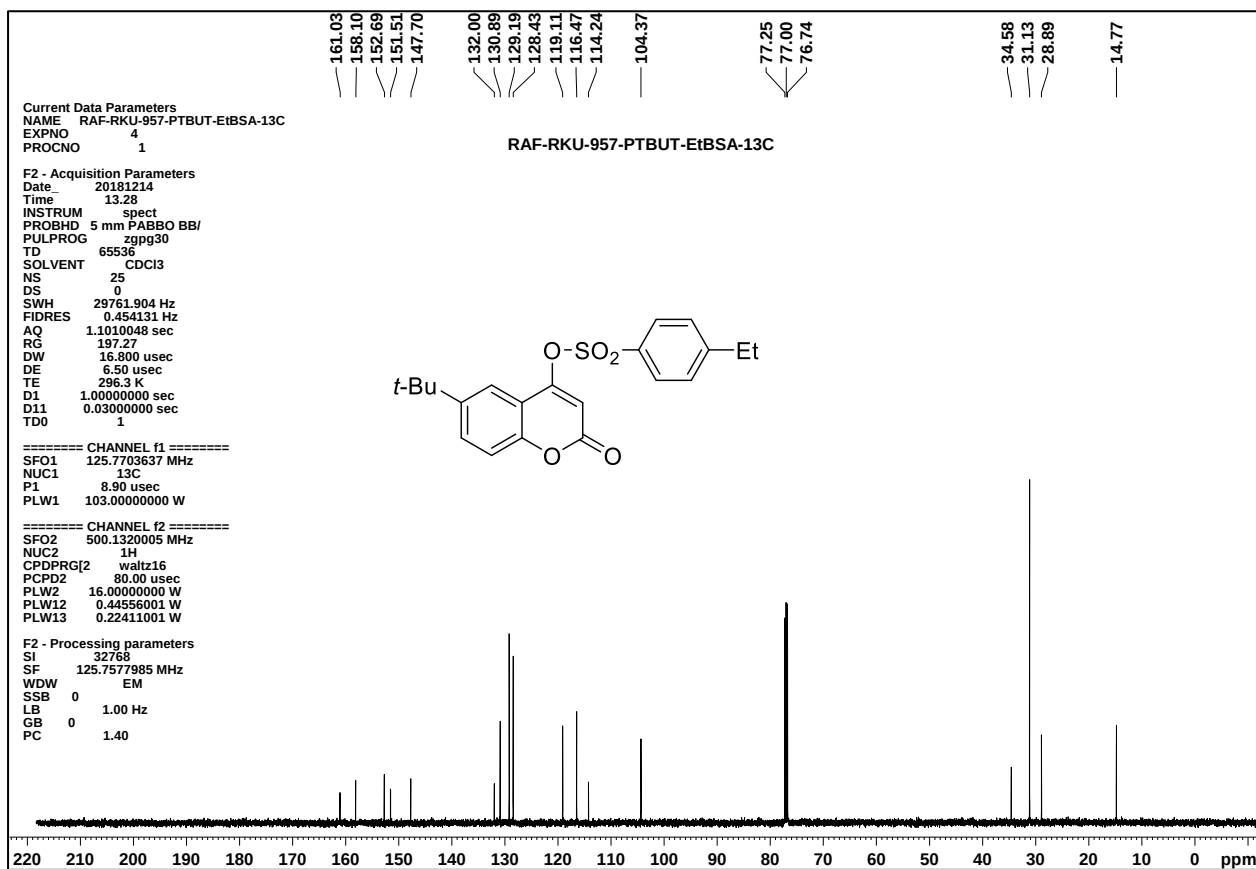
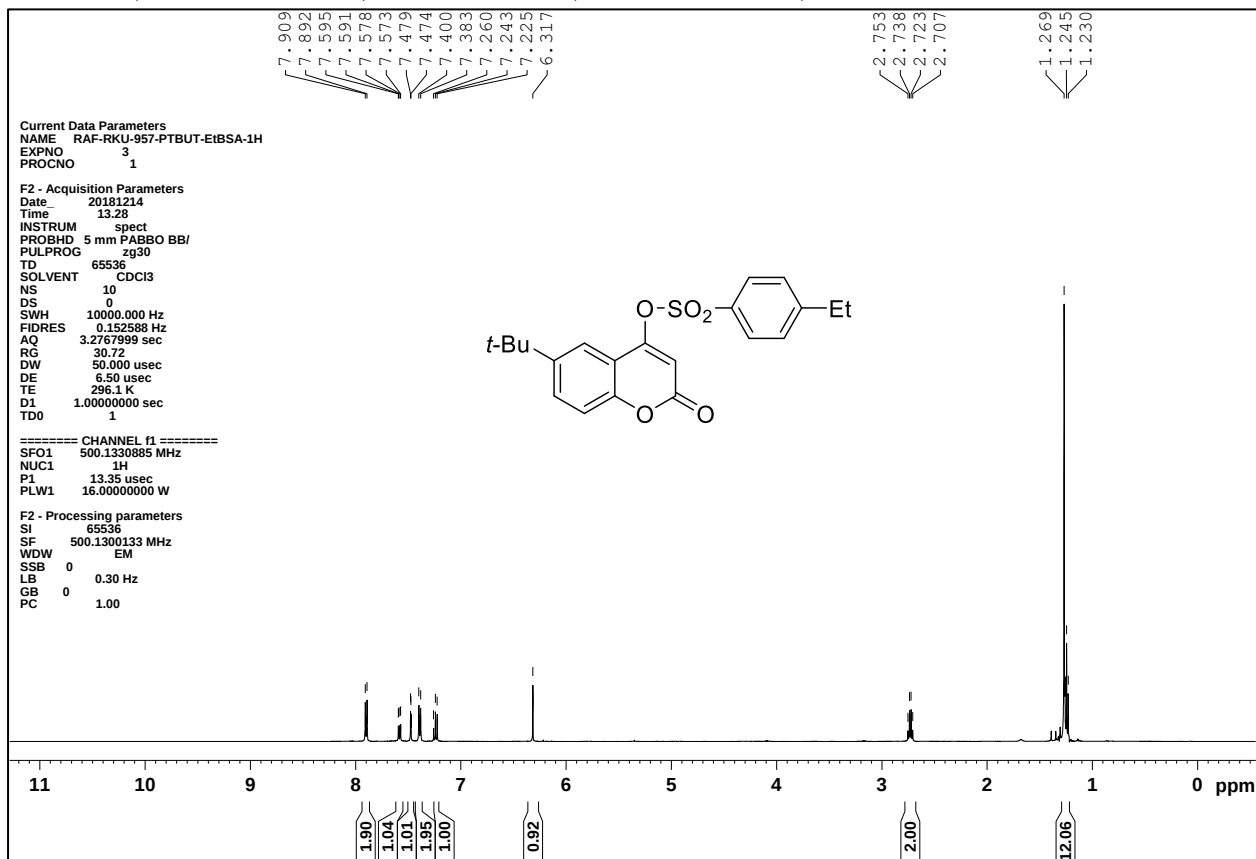
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **7a**



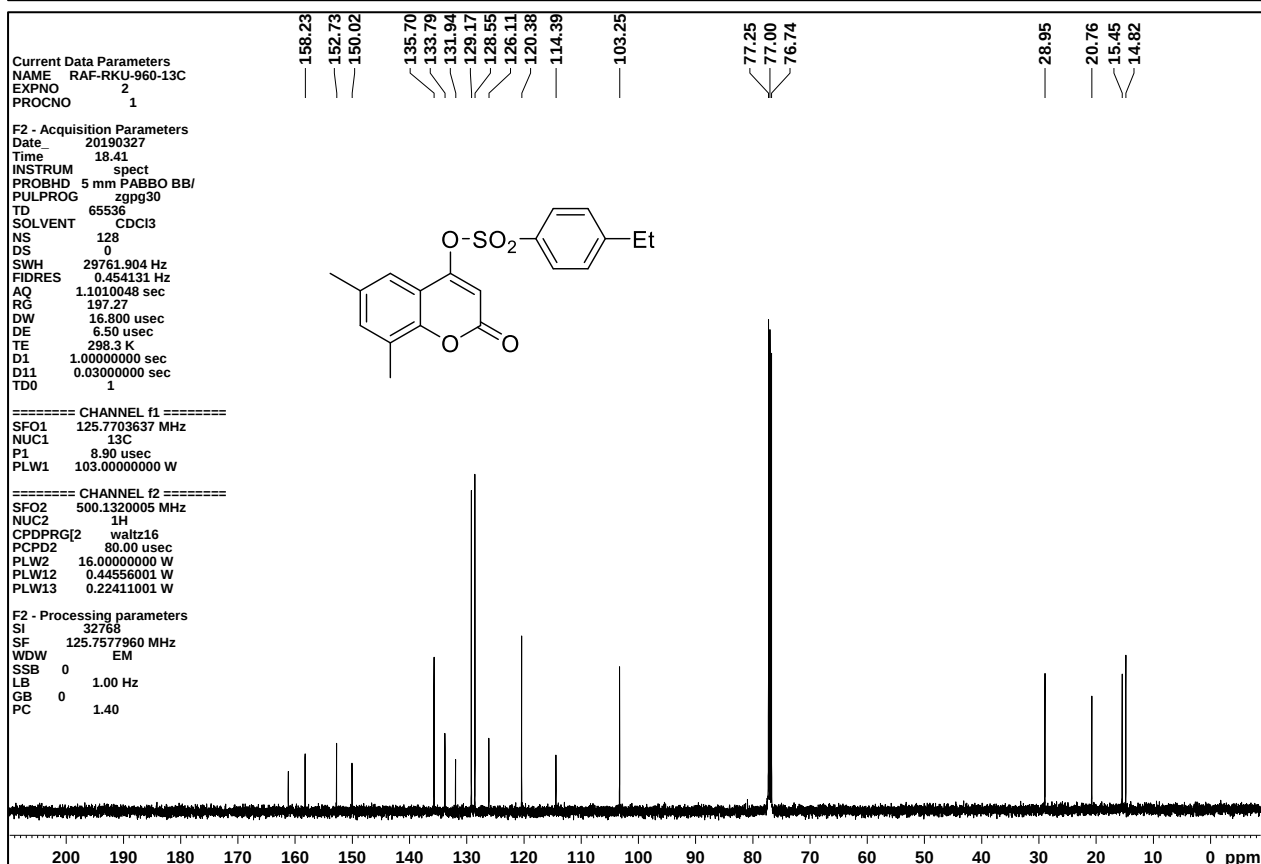
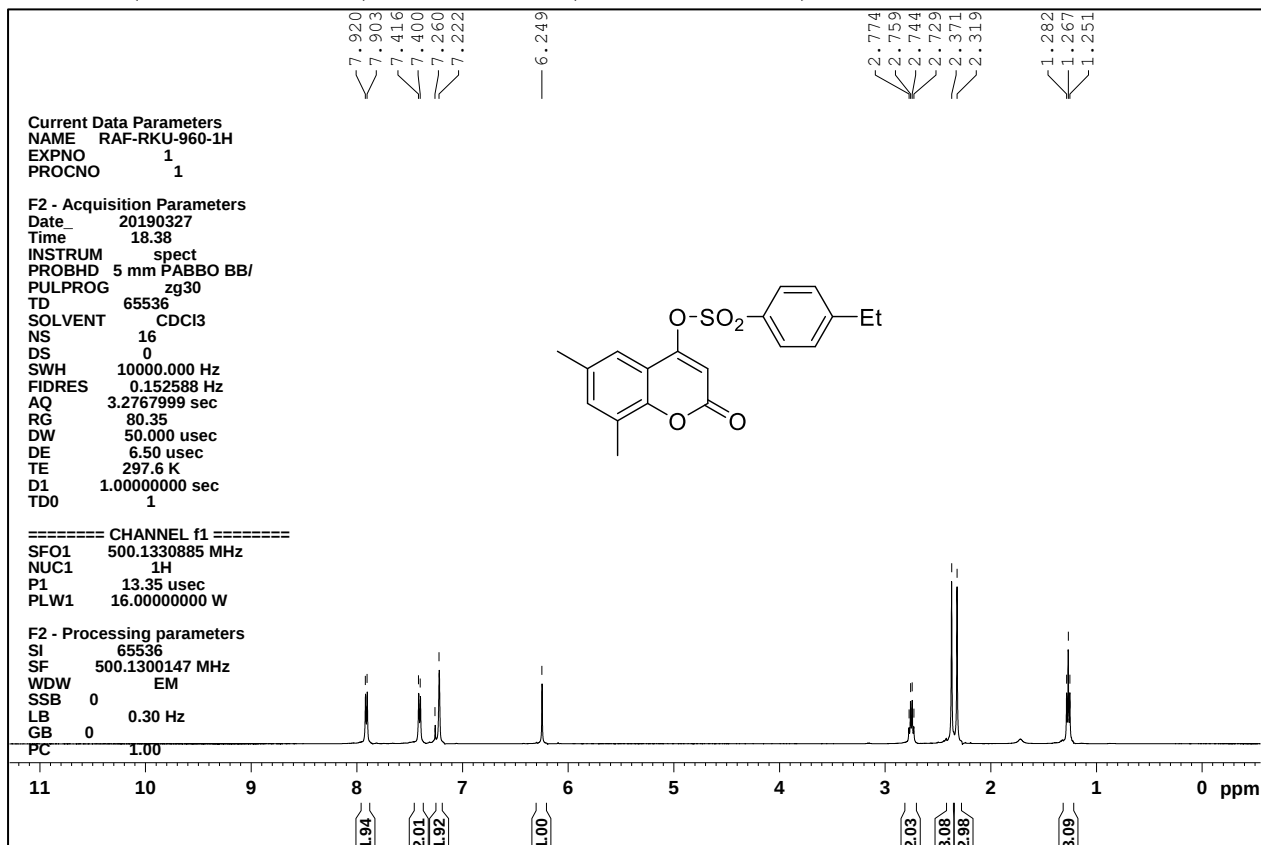
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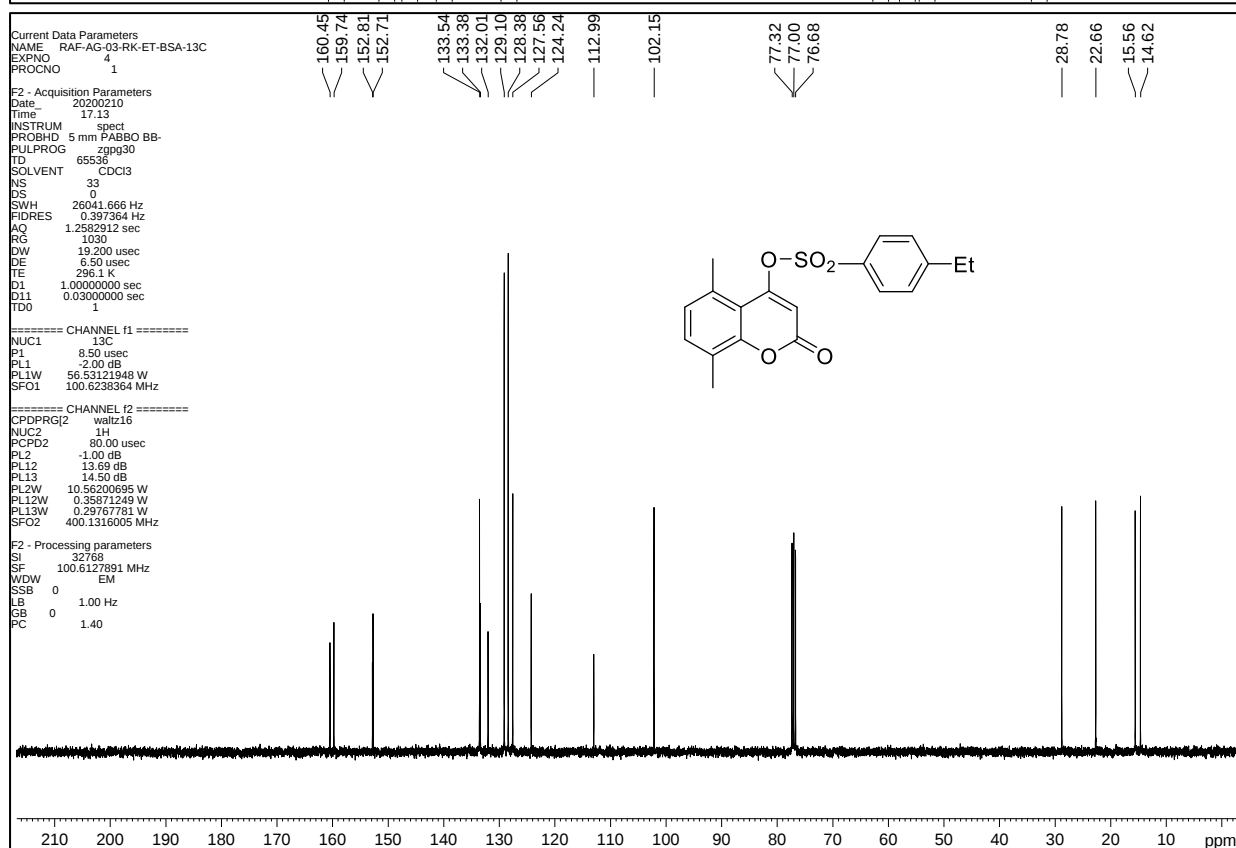
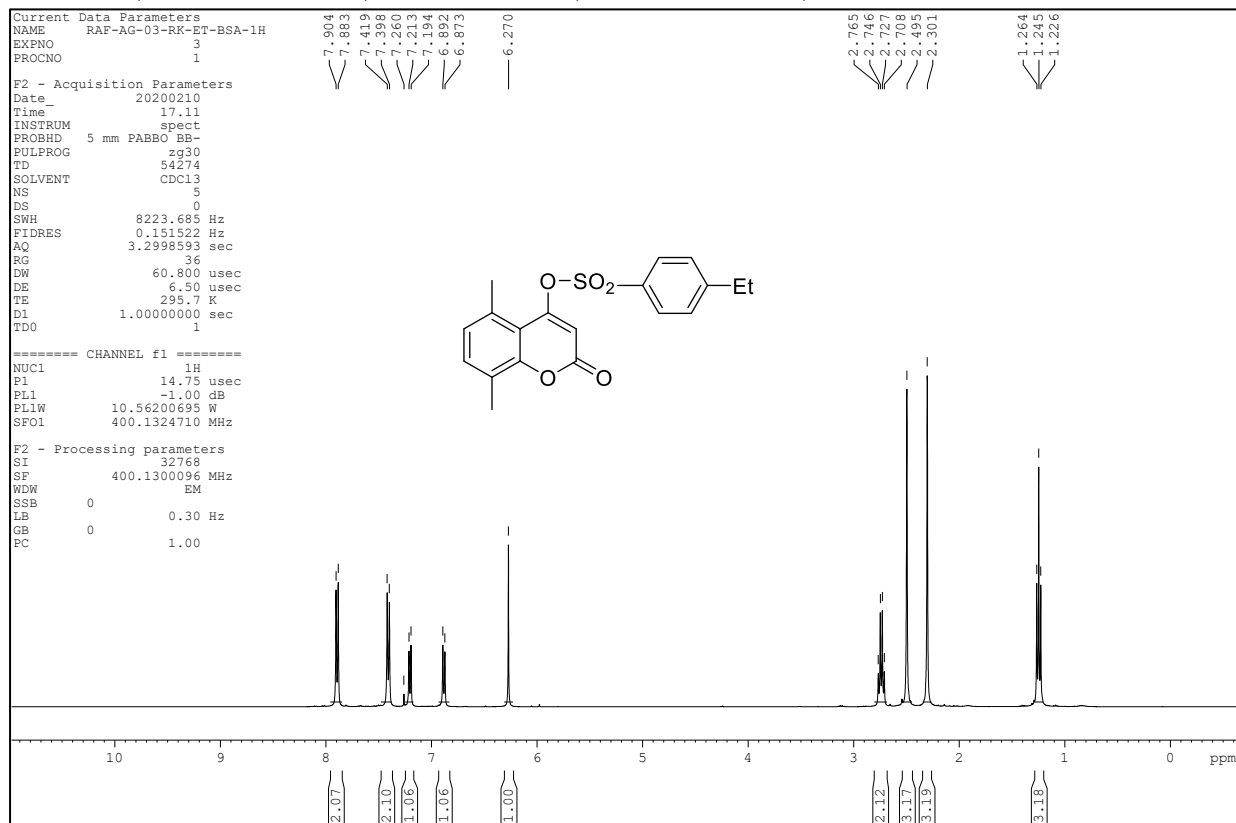
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **7c**



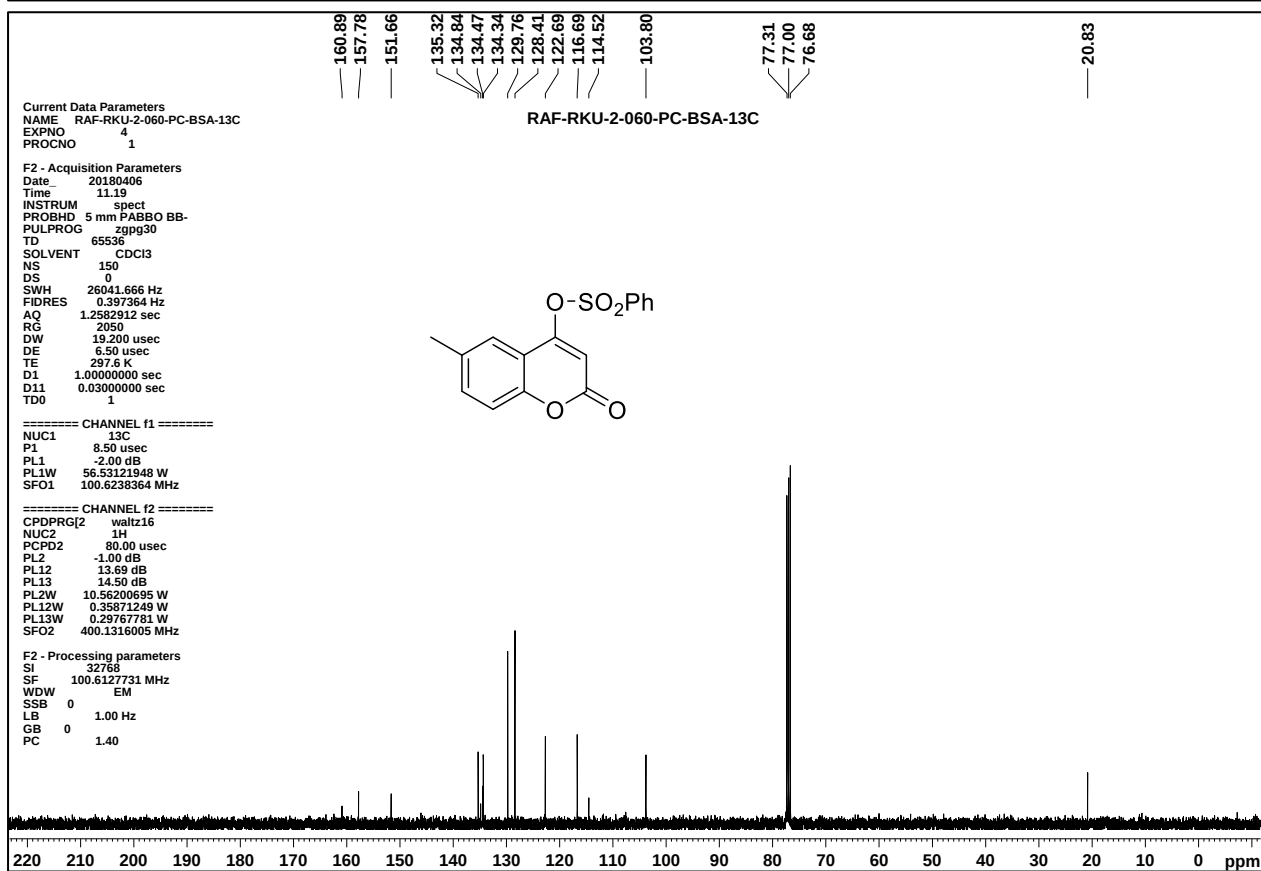
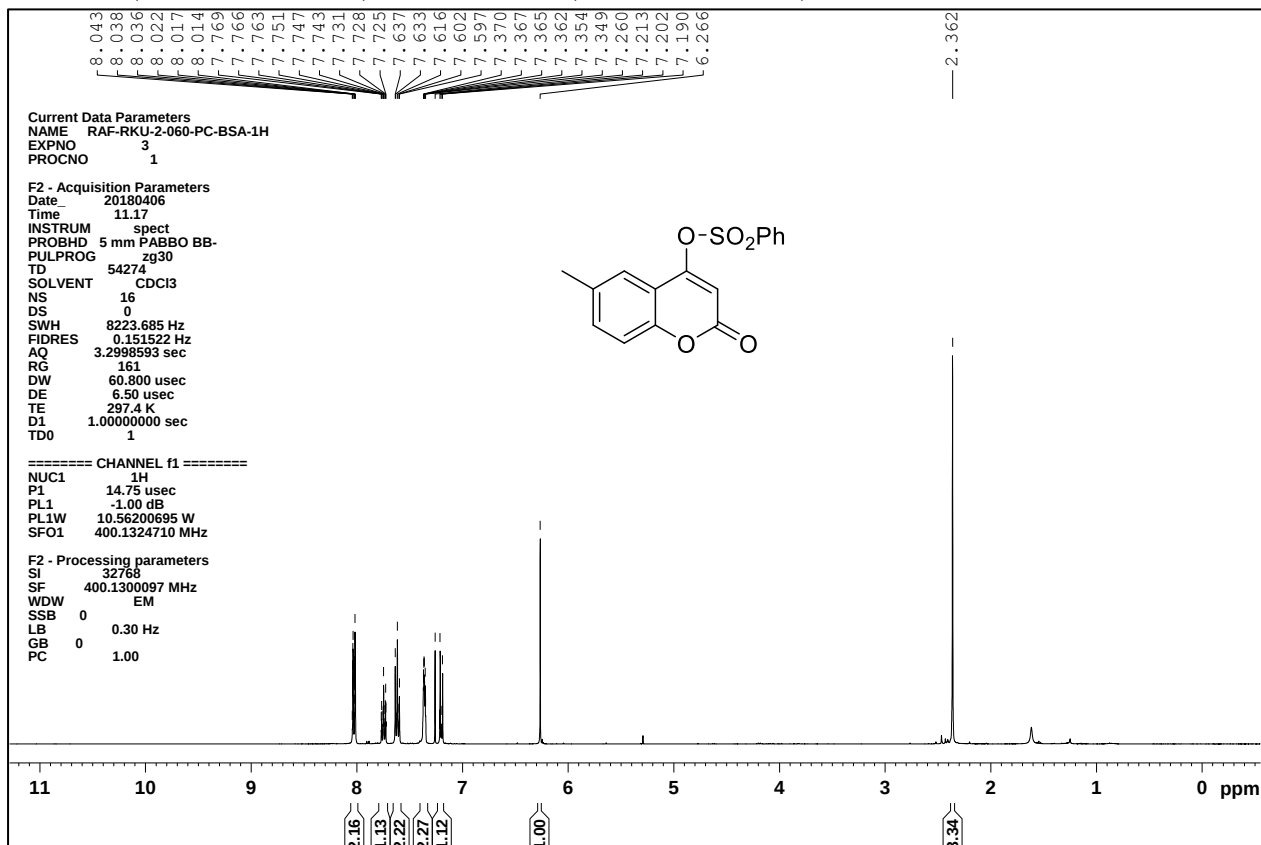
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **7f**



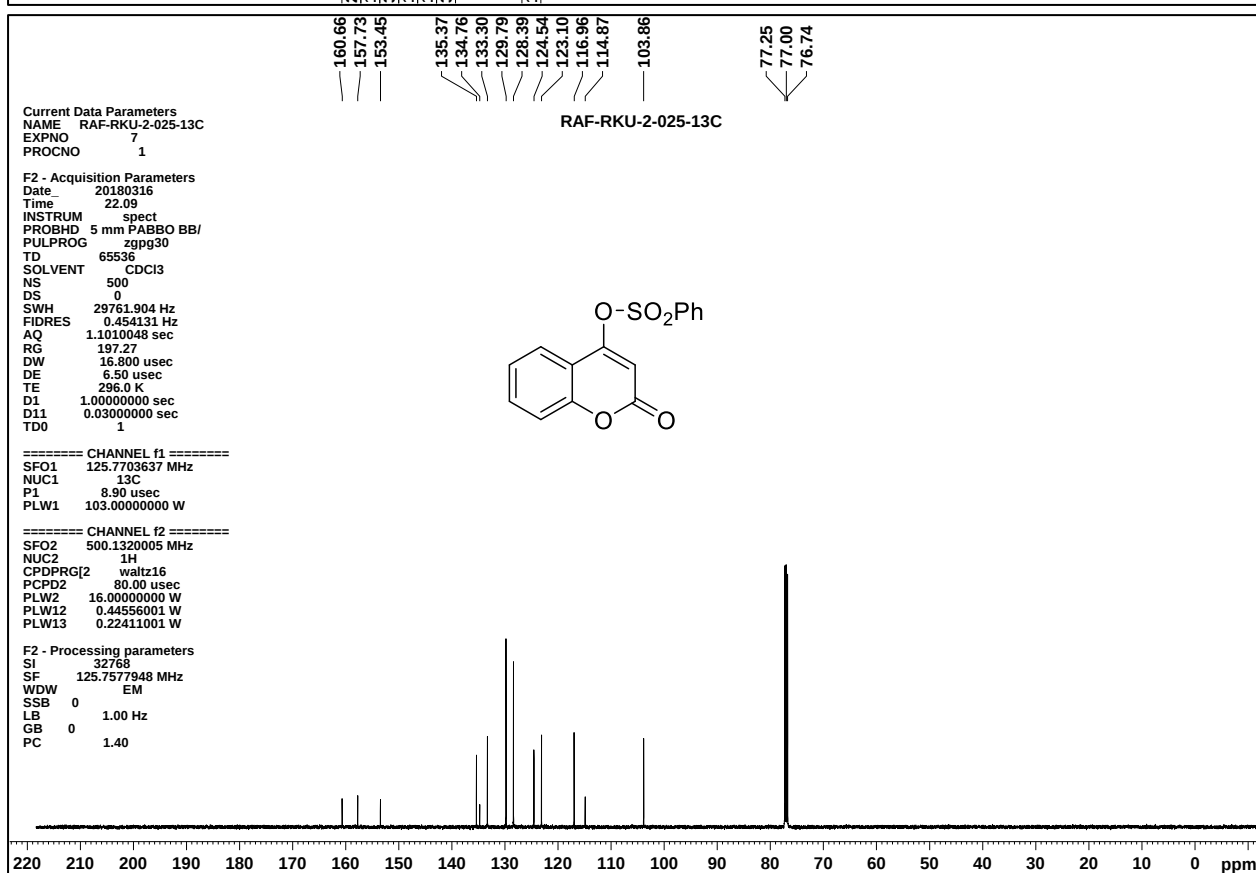
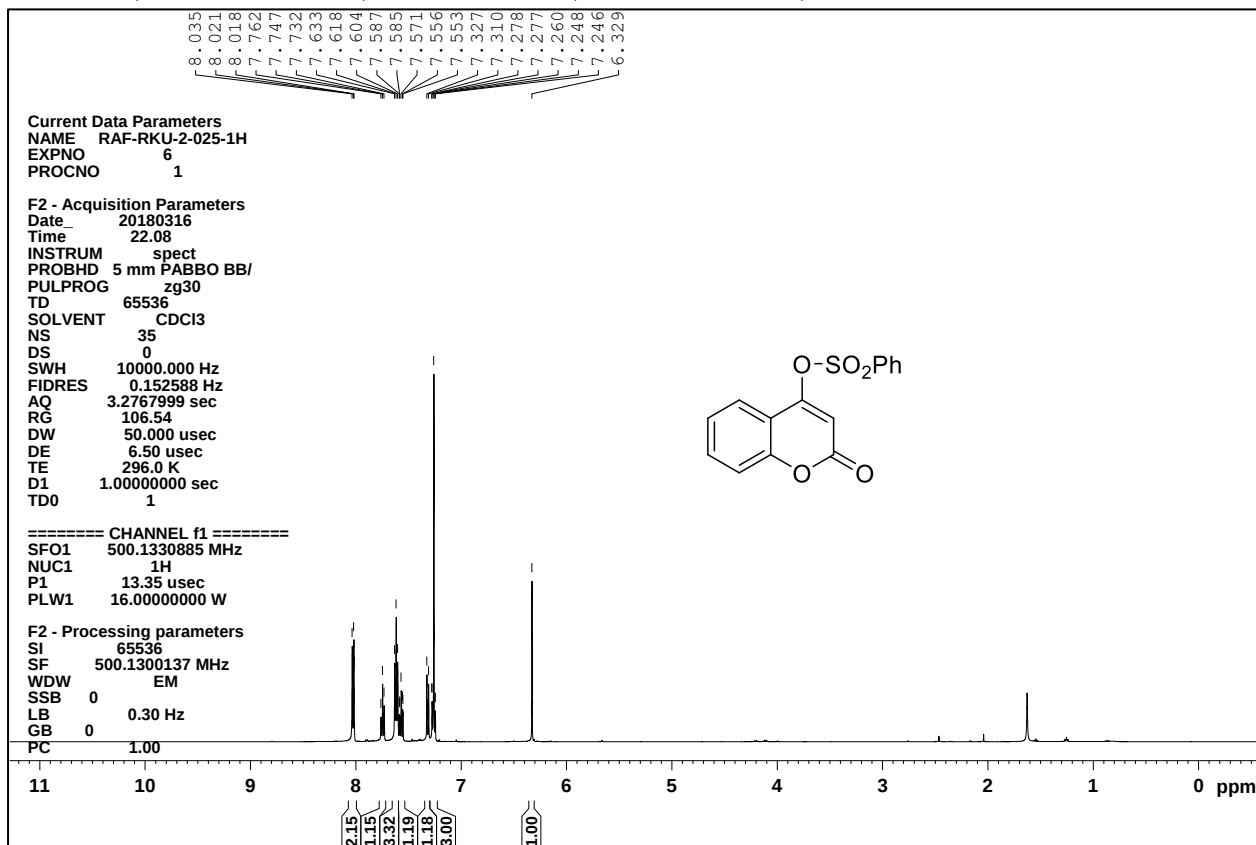
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **7i**



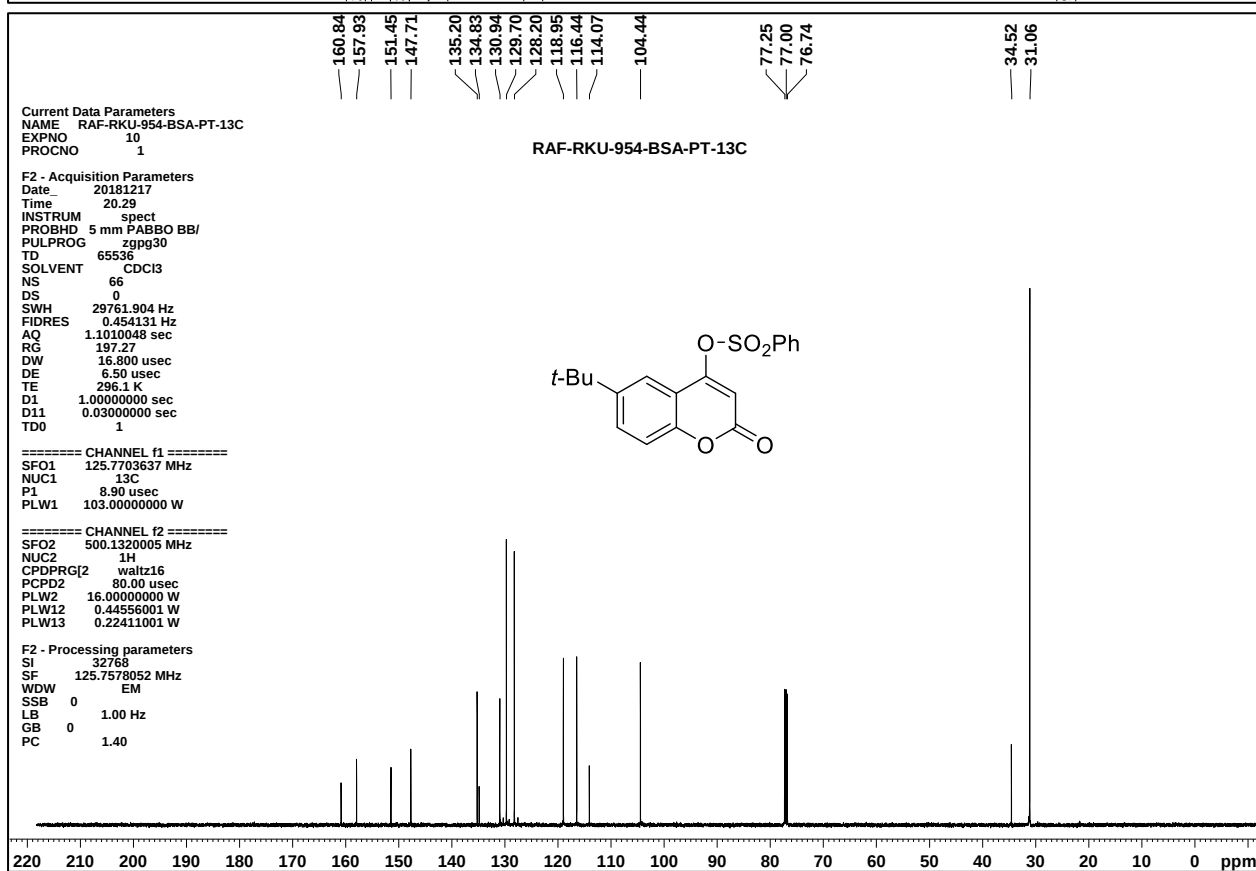
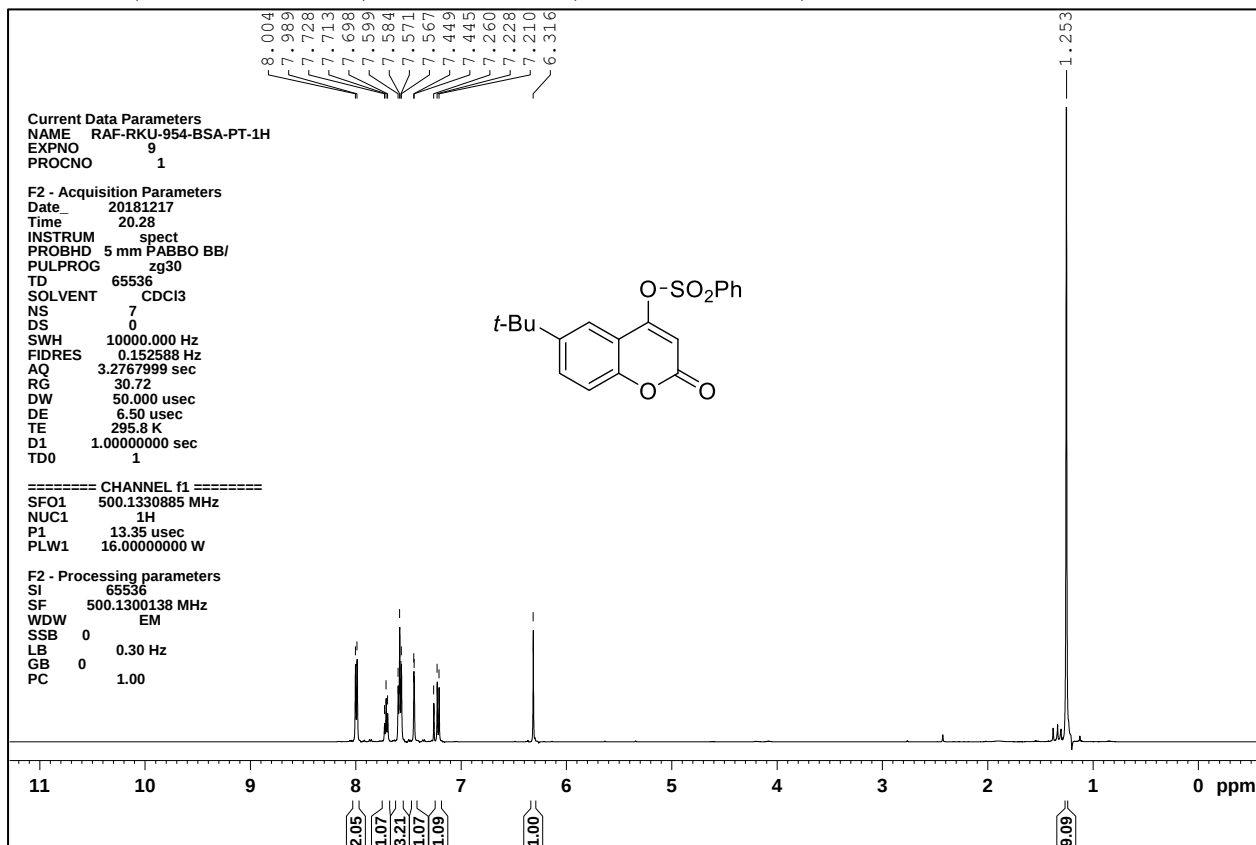
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **8a**



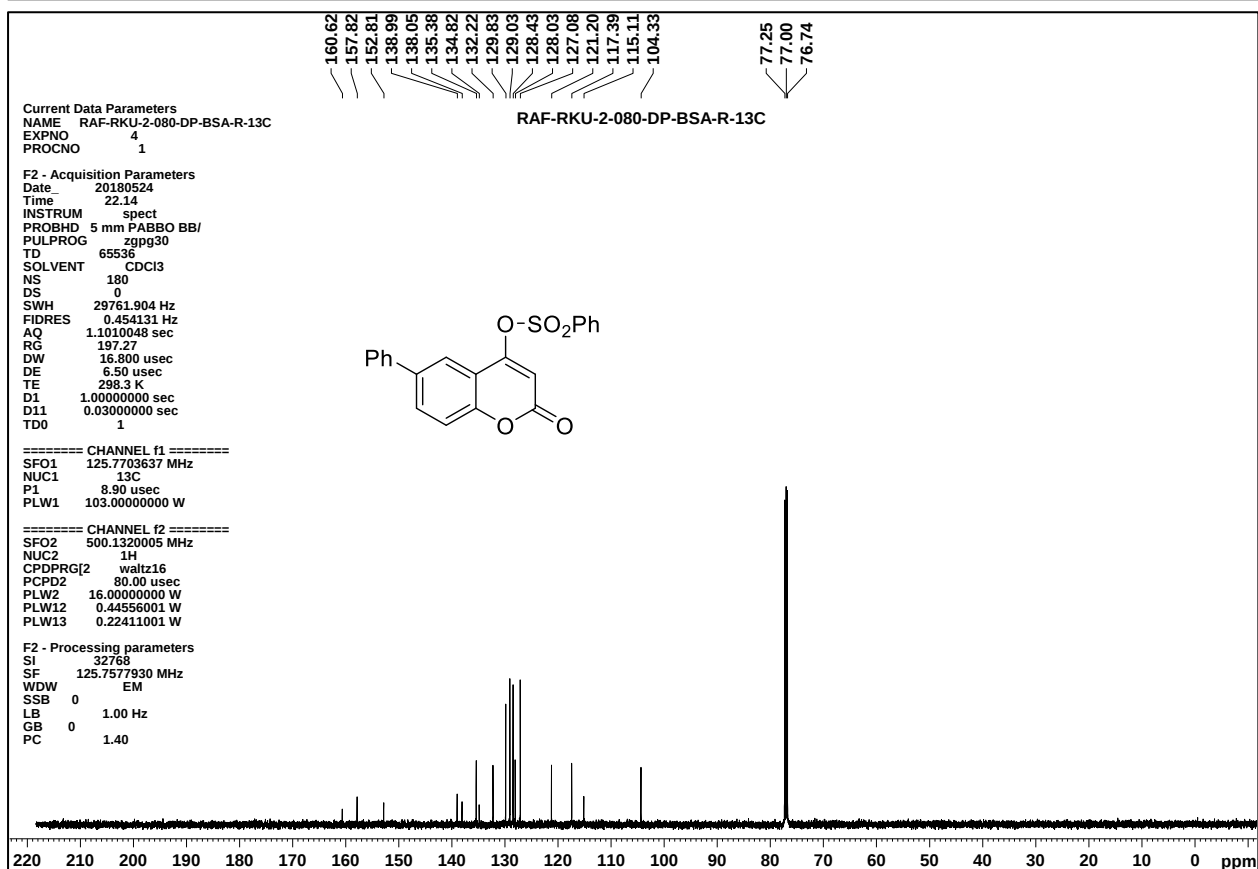
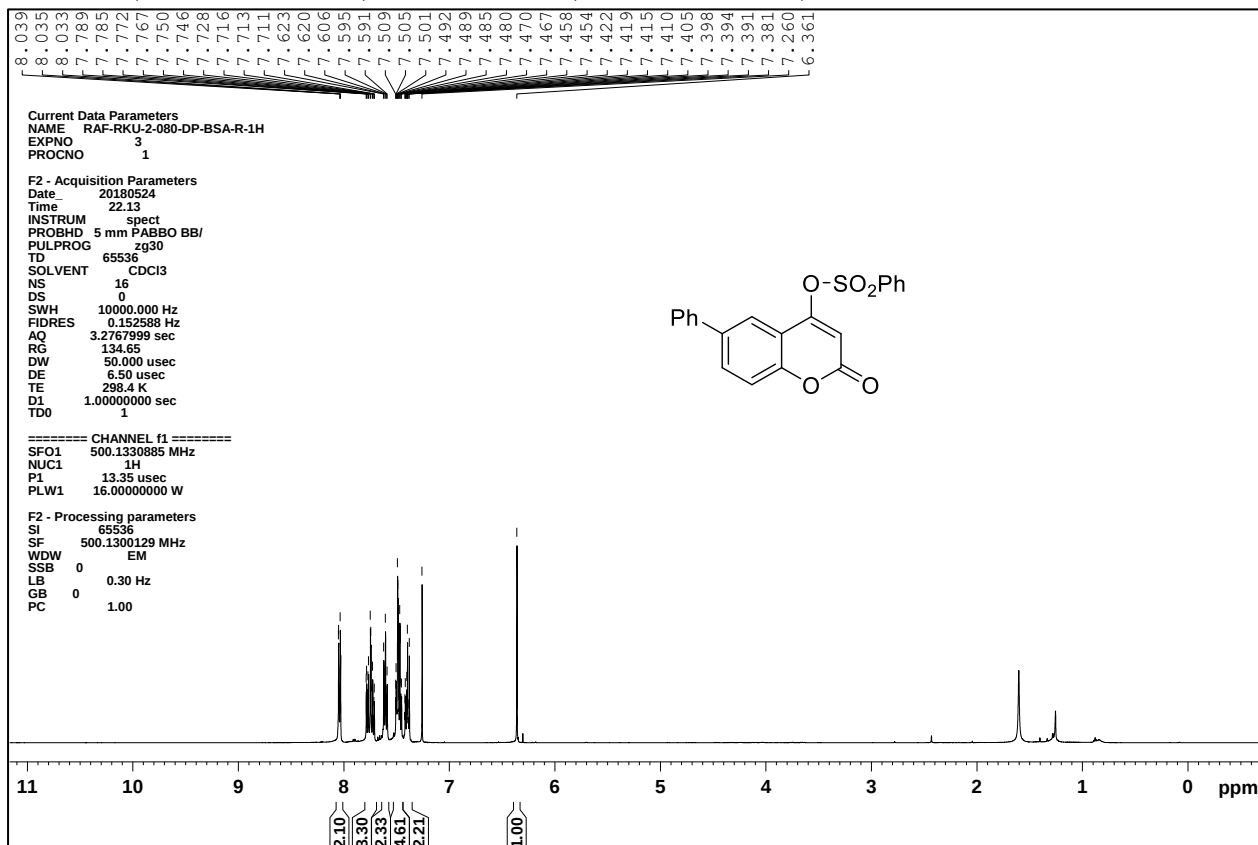
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **8b**



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **8c**



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **8d**



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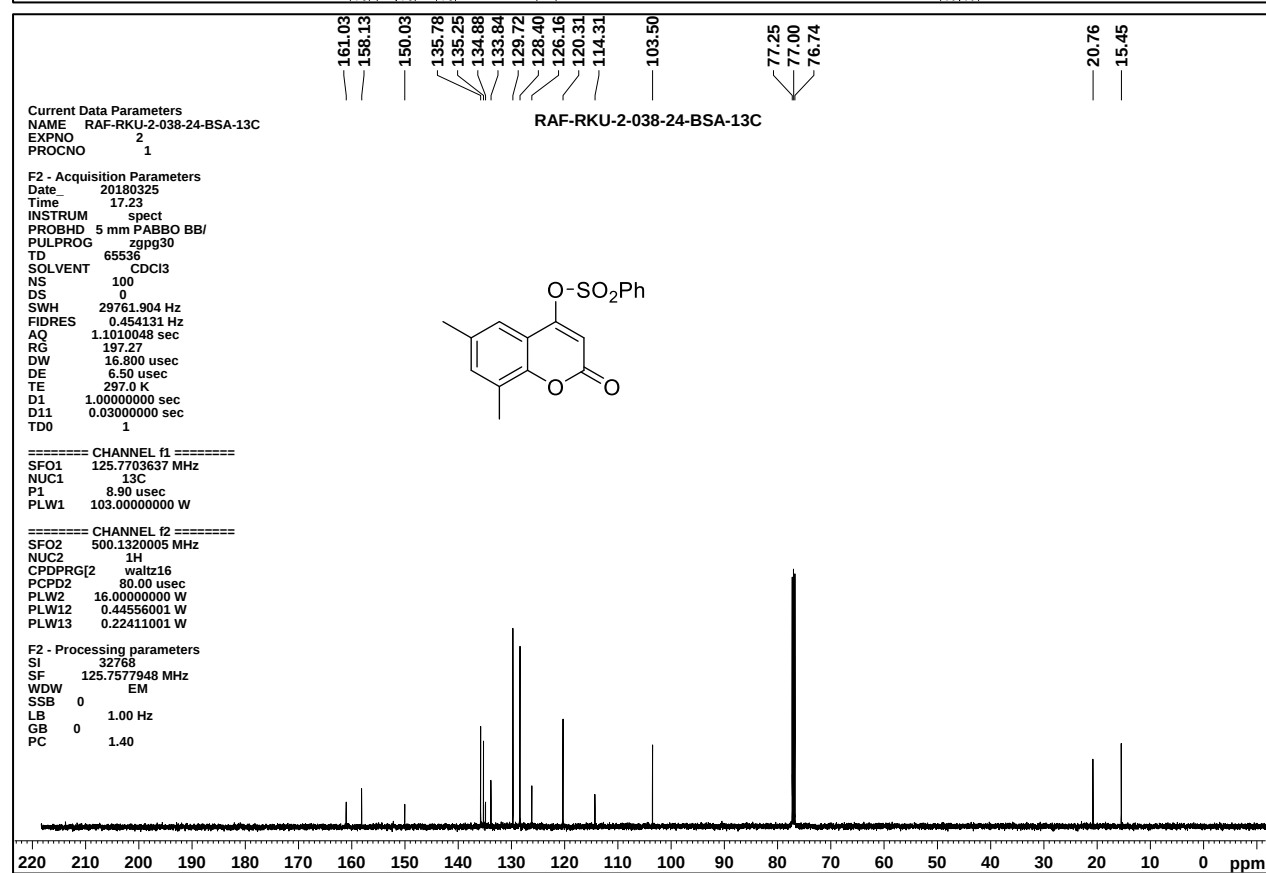
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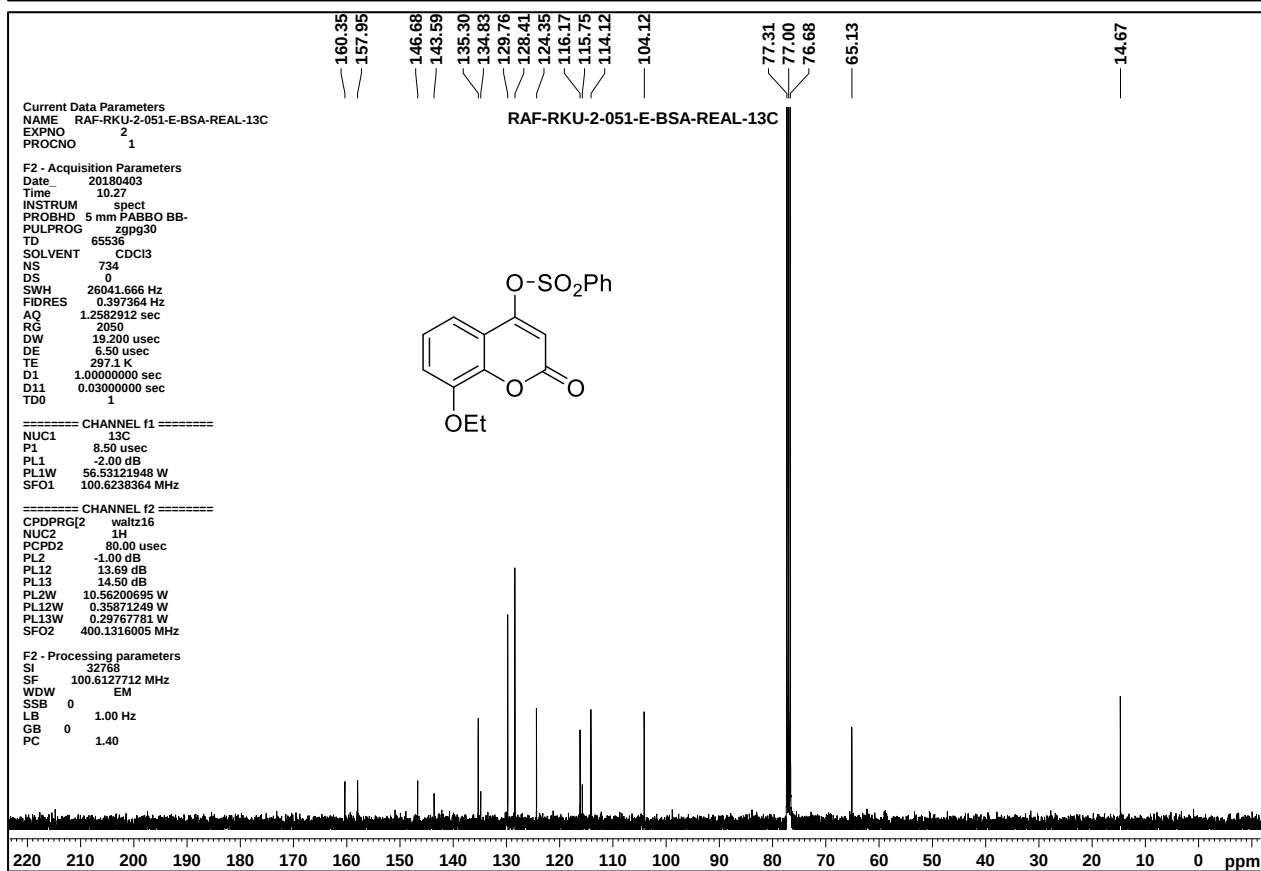
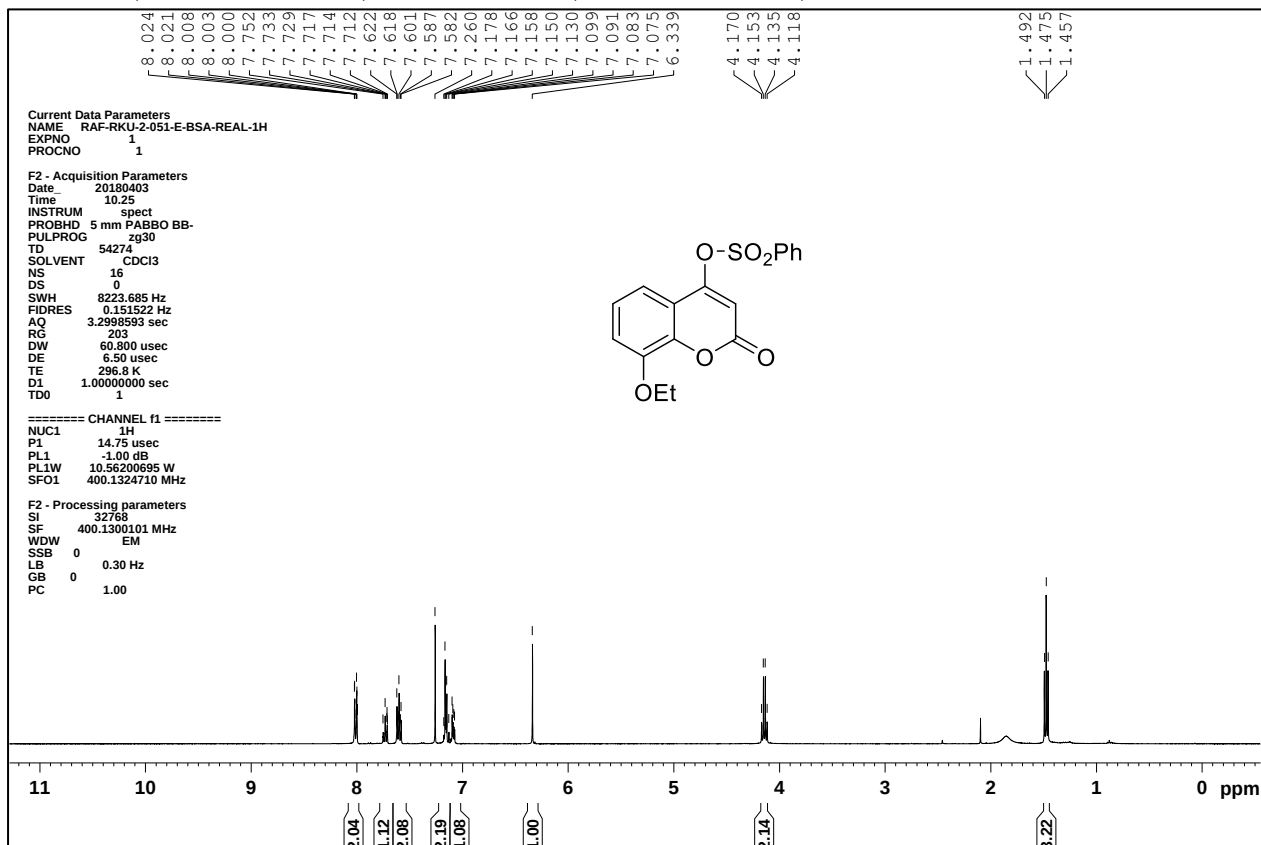
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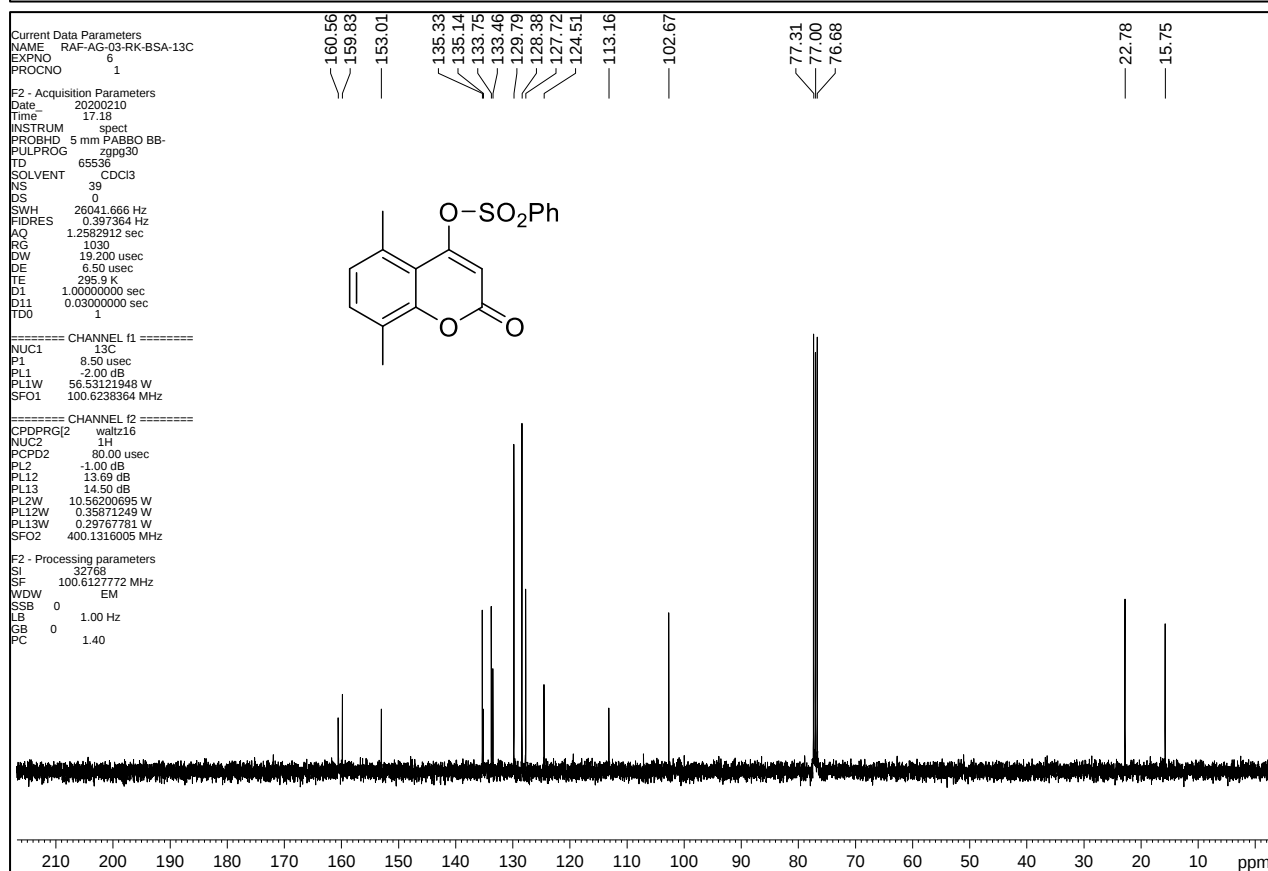
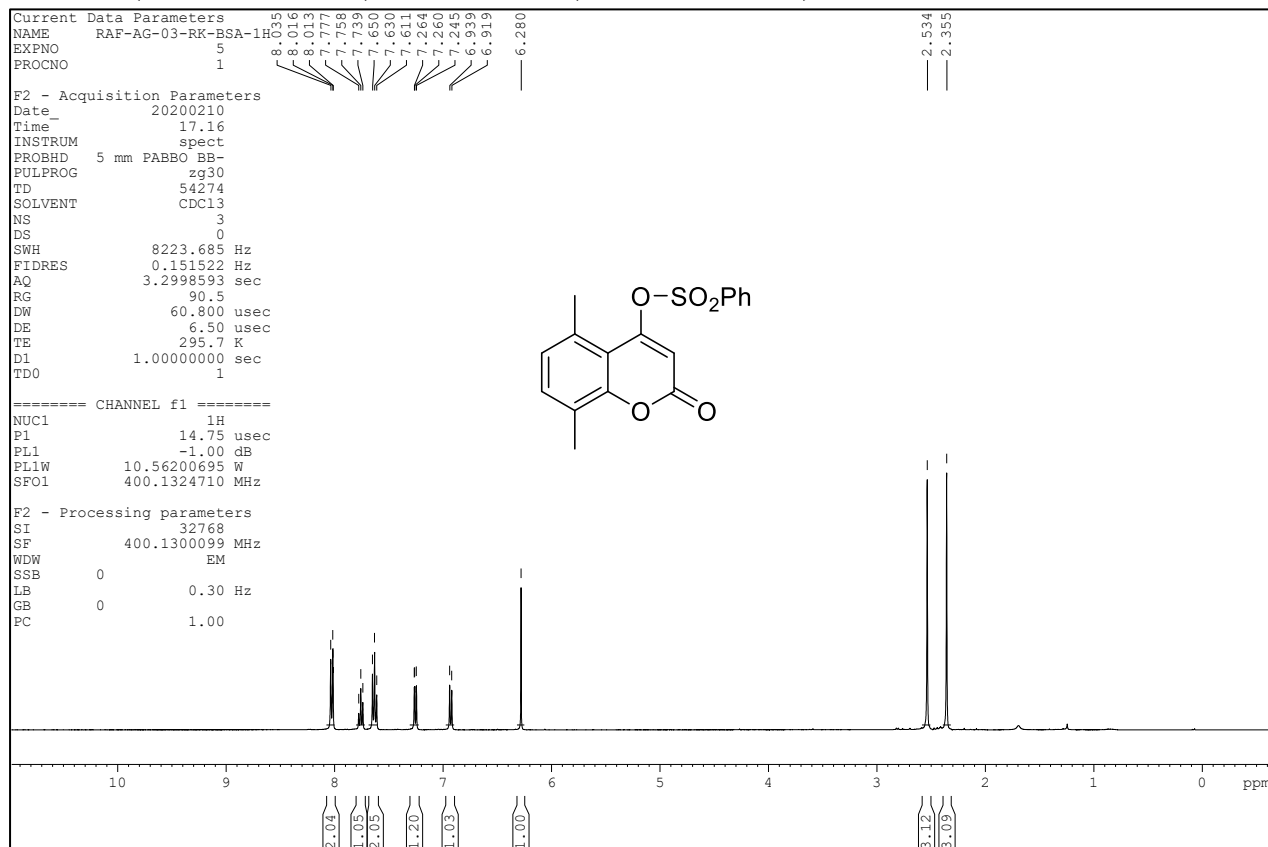
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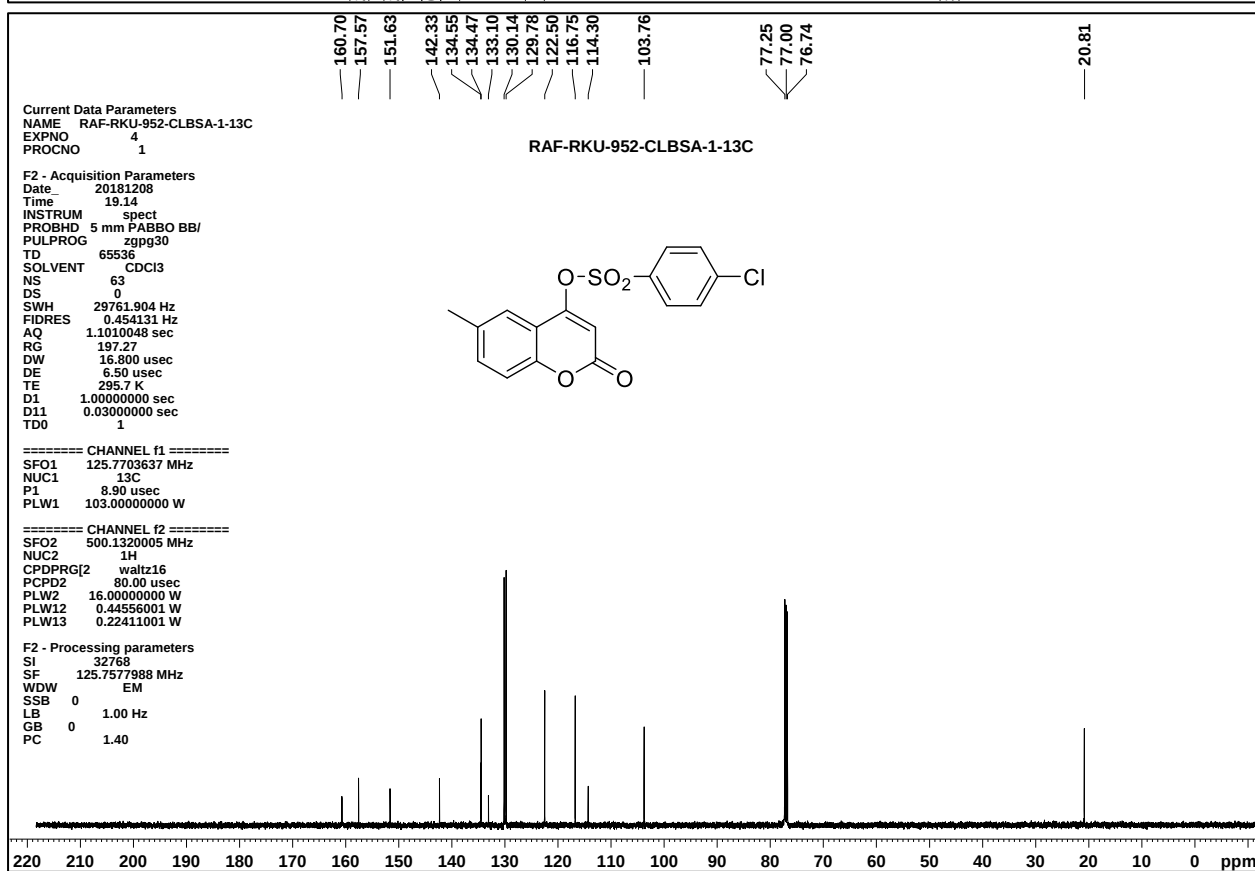
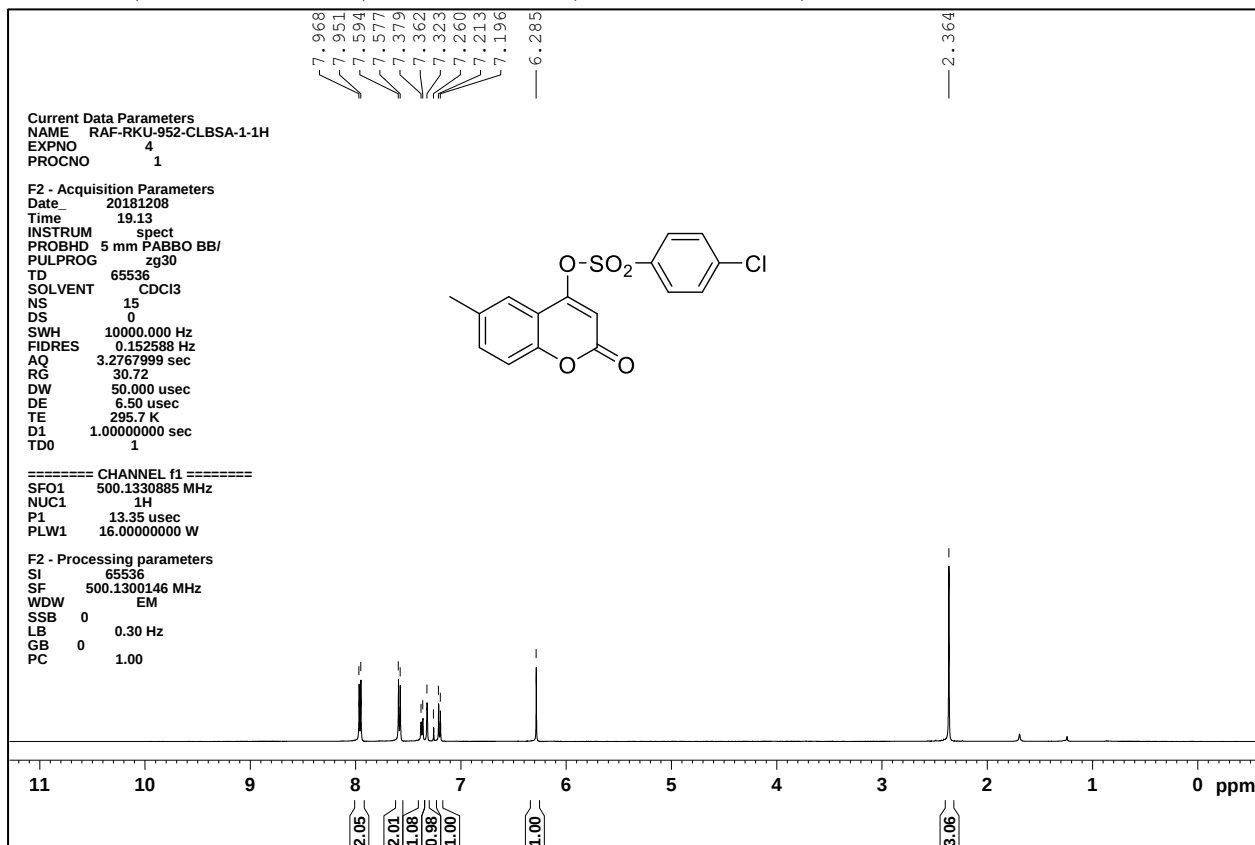
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **8h**



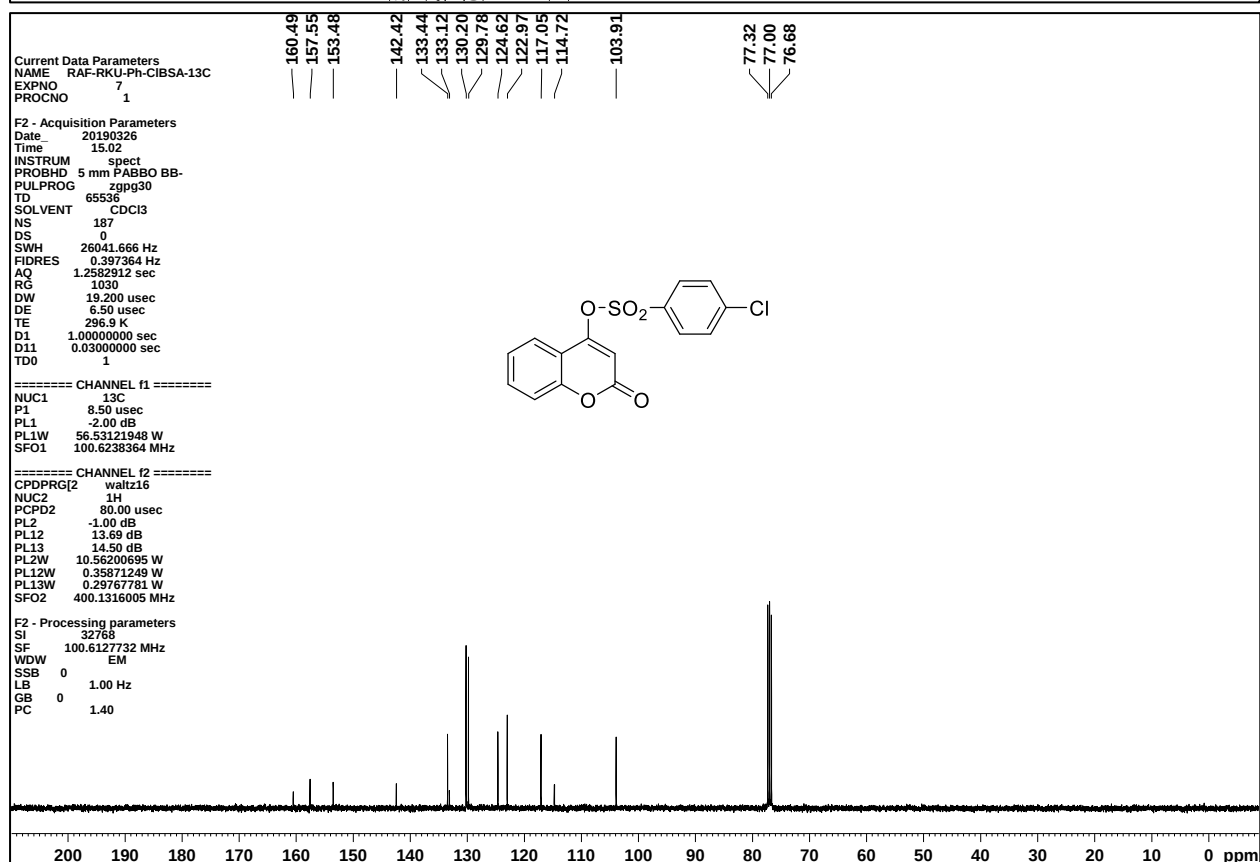
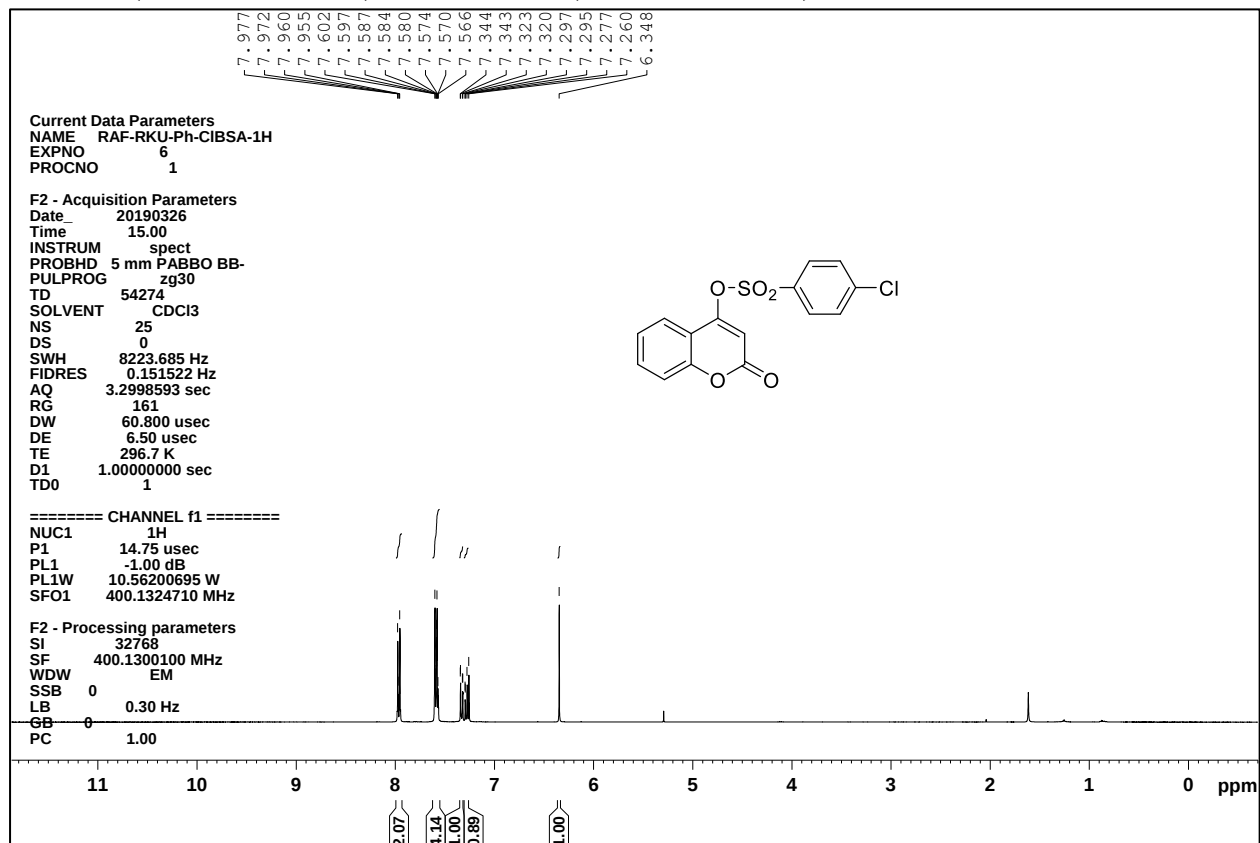
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **8i**



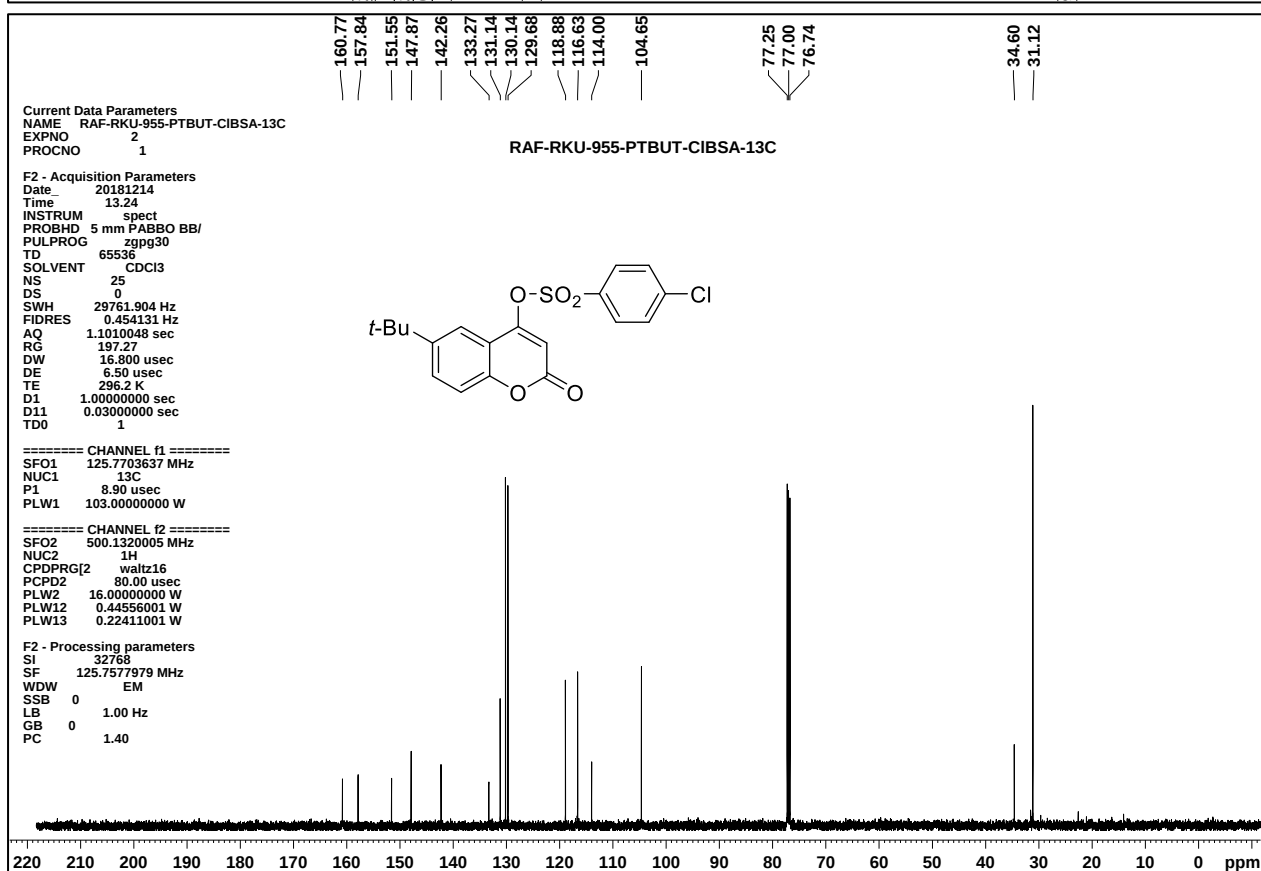
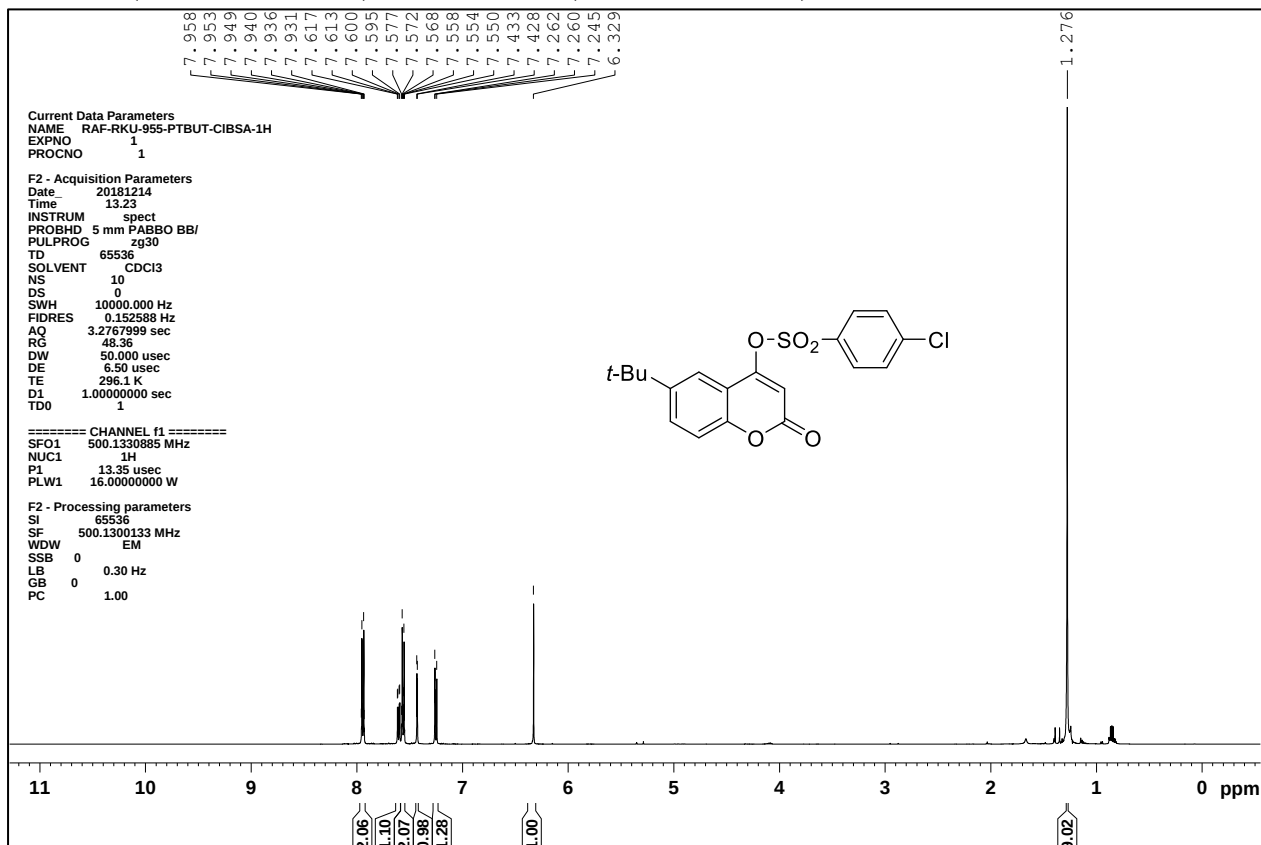
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **9a**



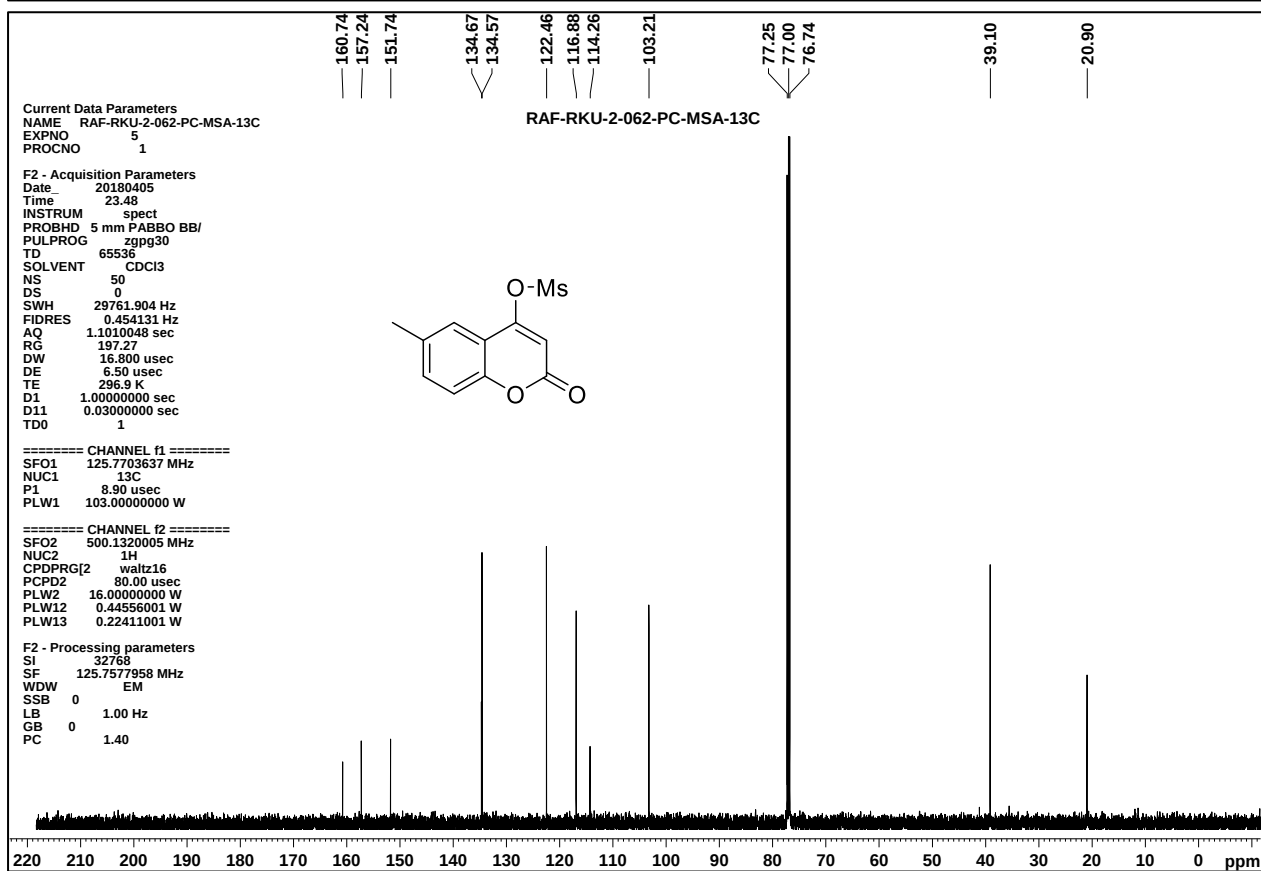
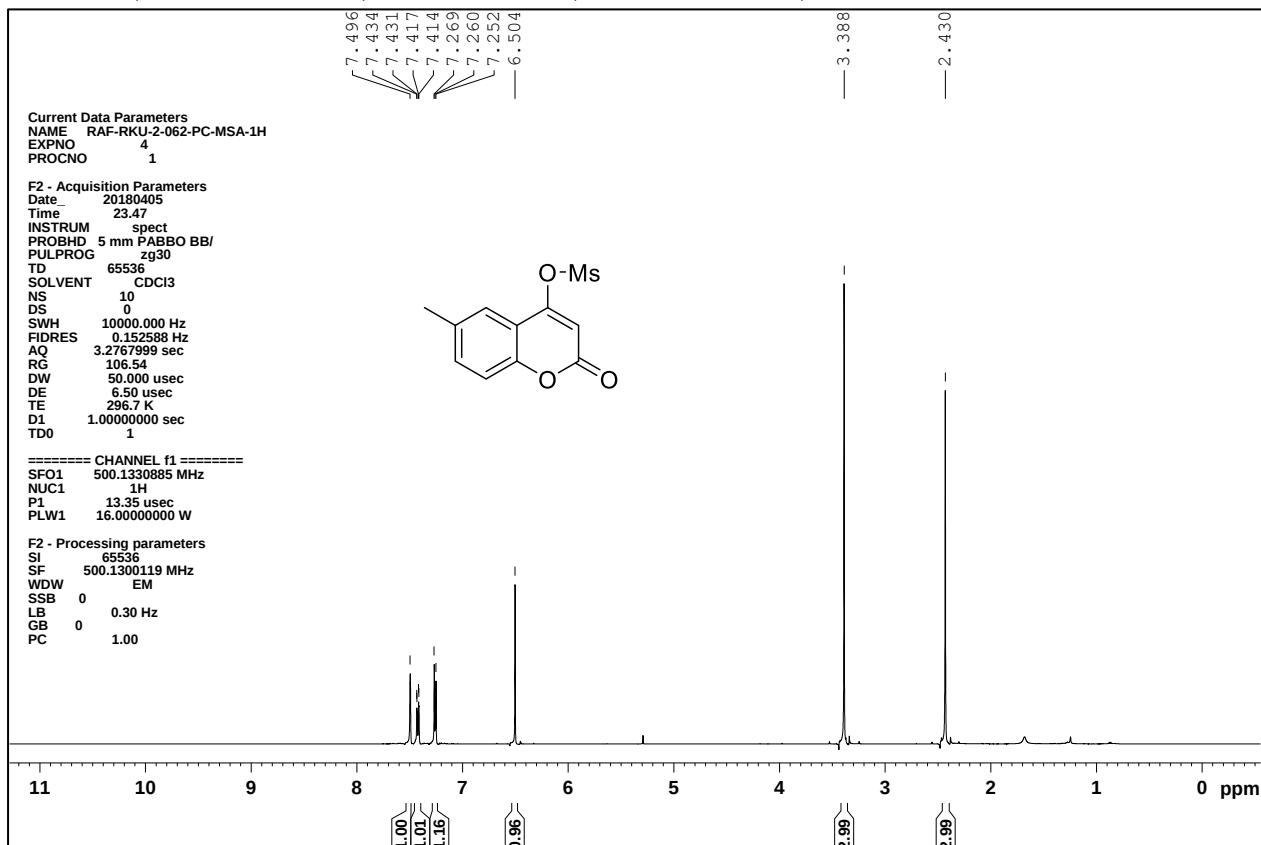
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **9b**



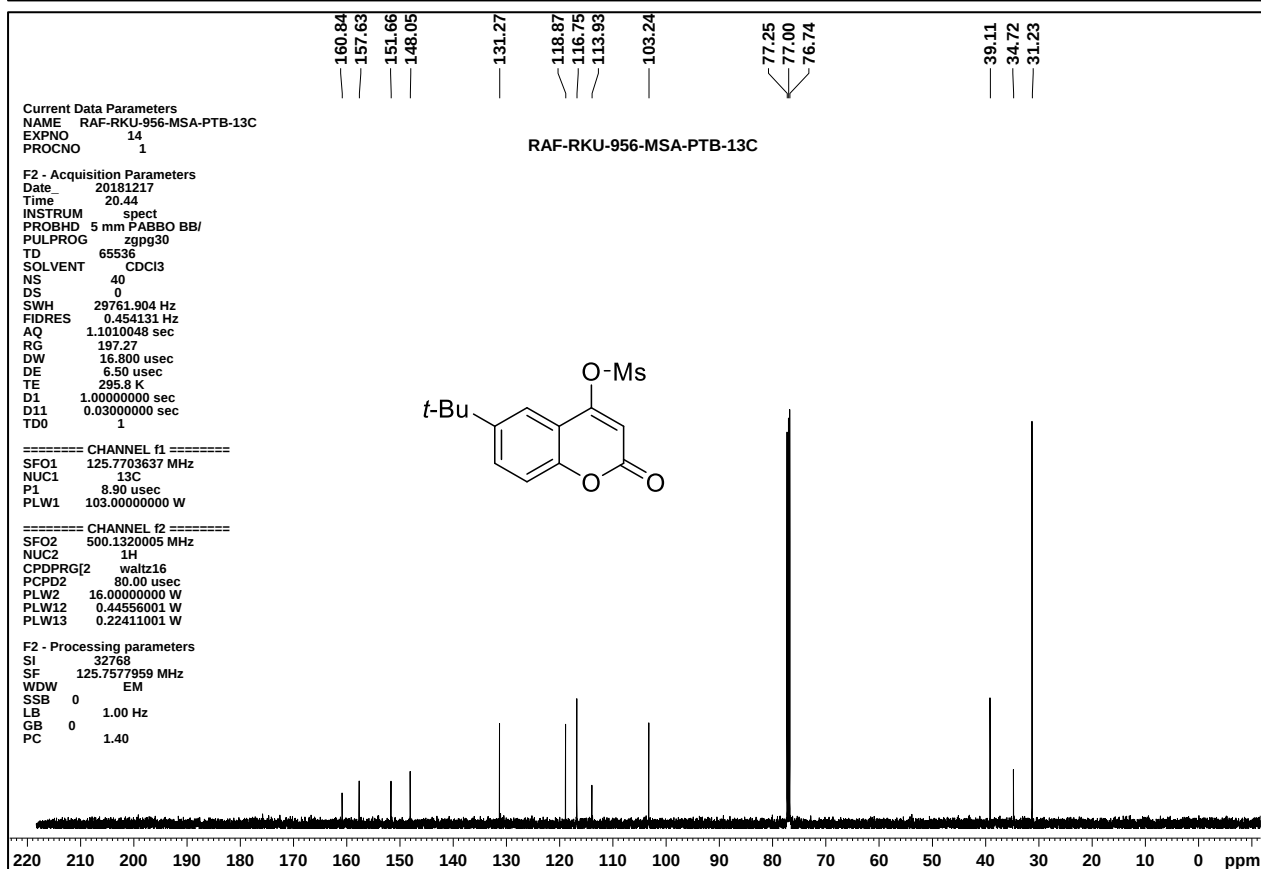
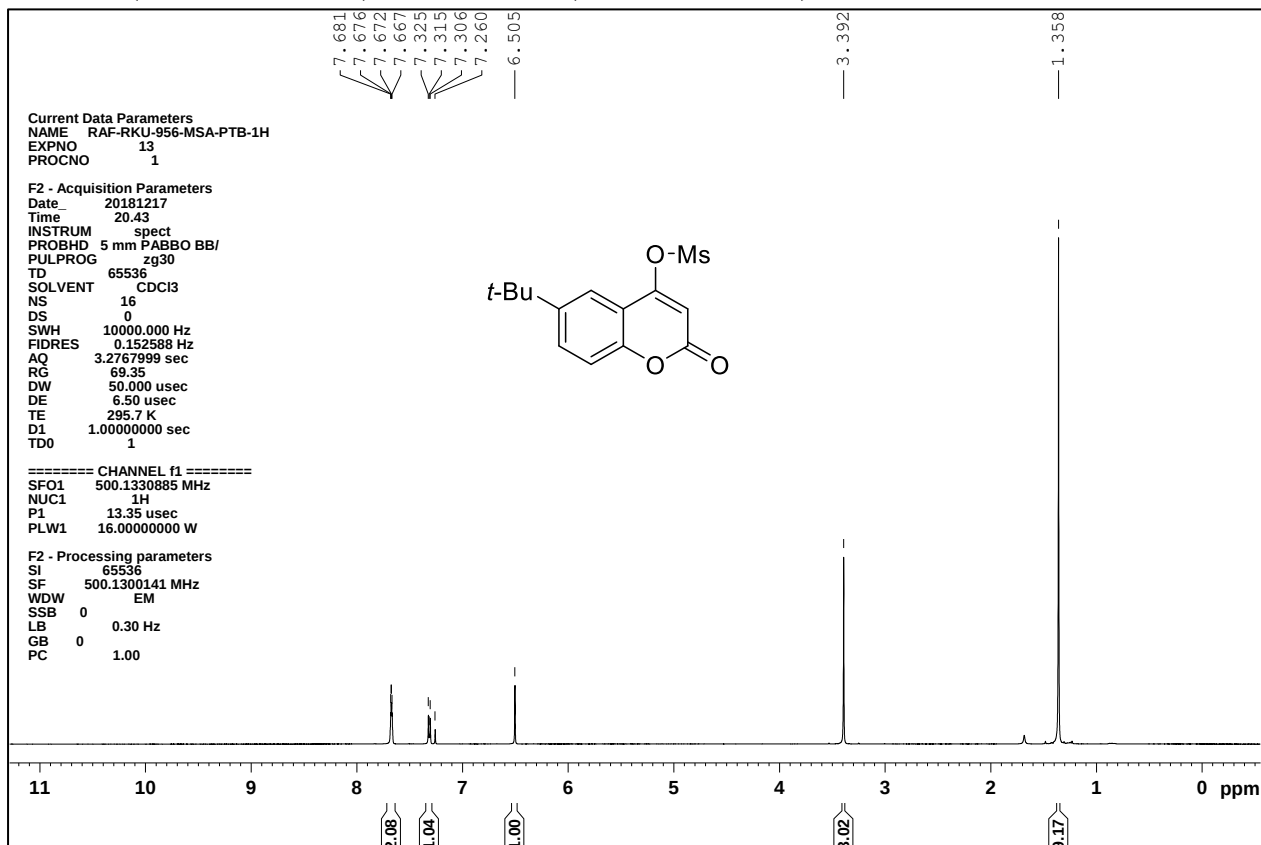
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of **9c**



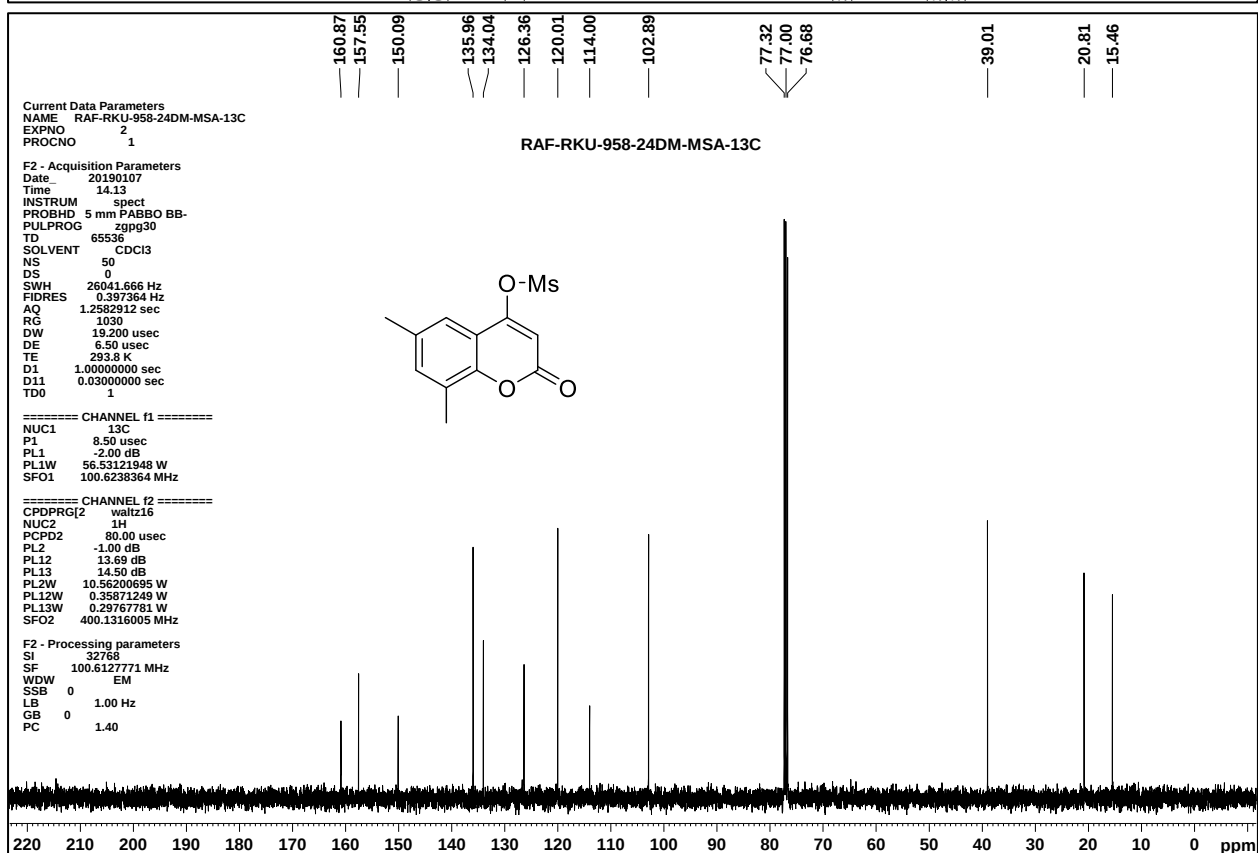
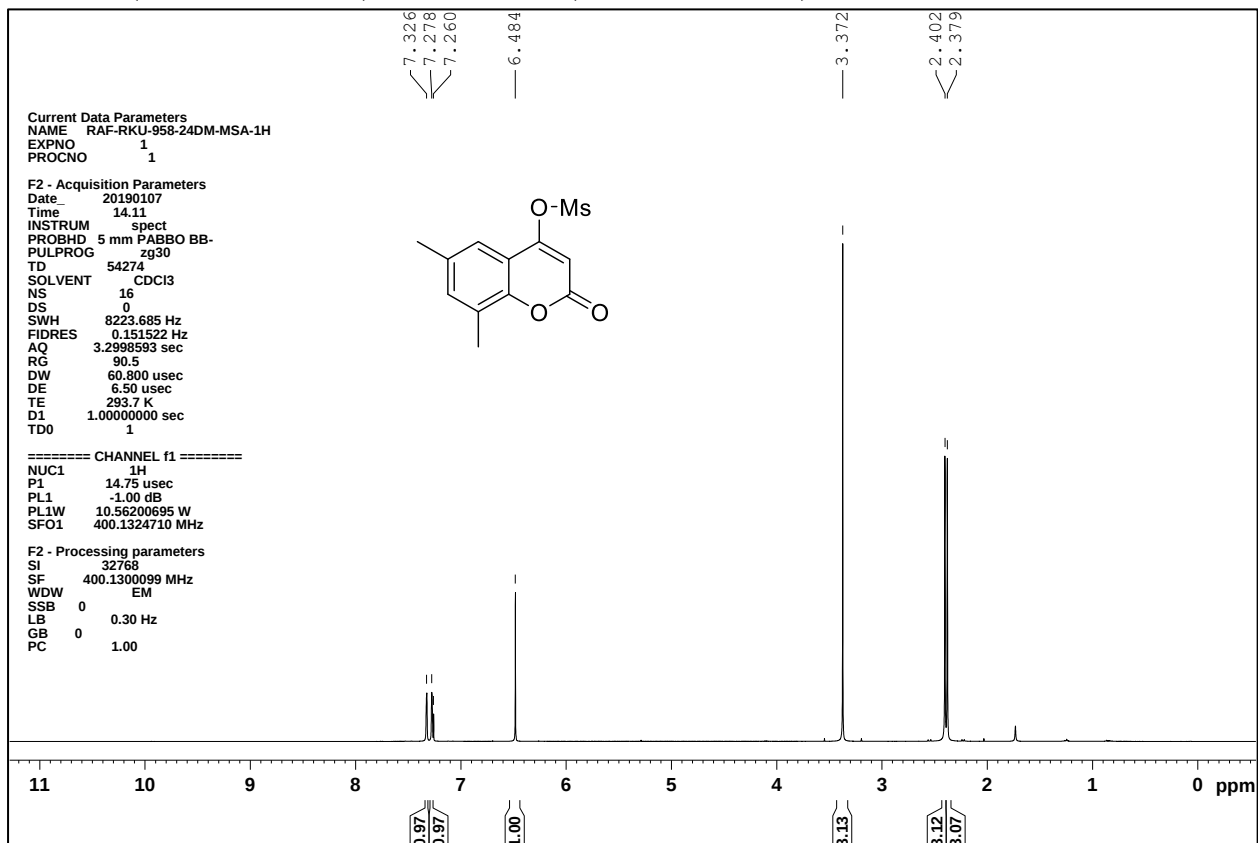
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of **10a**



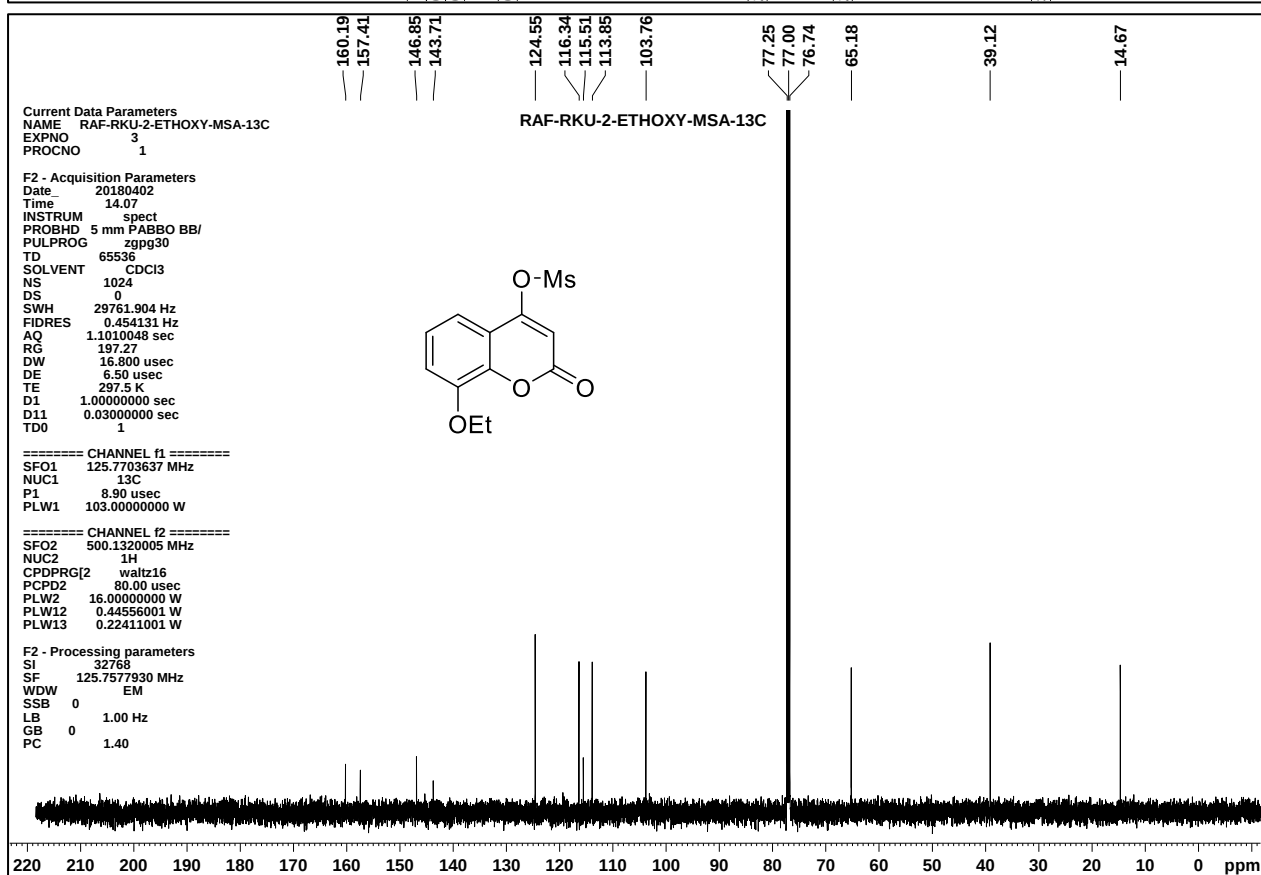
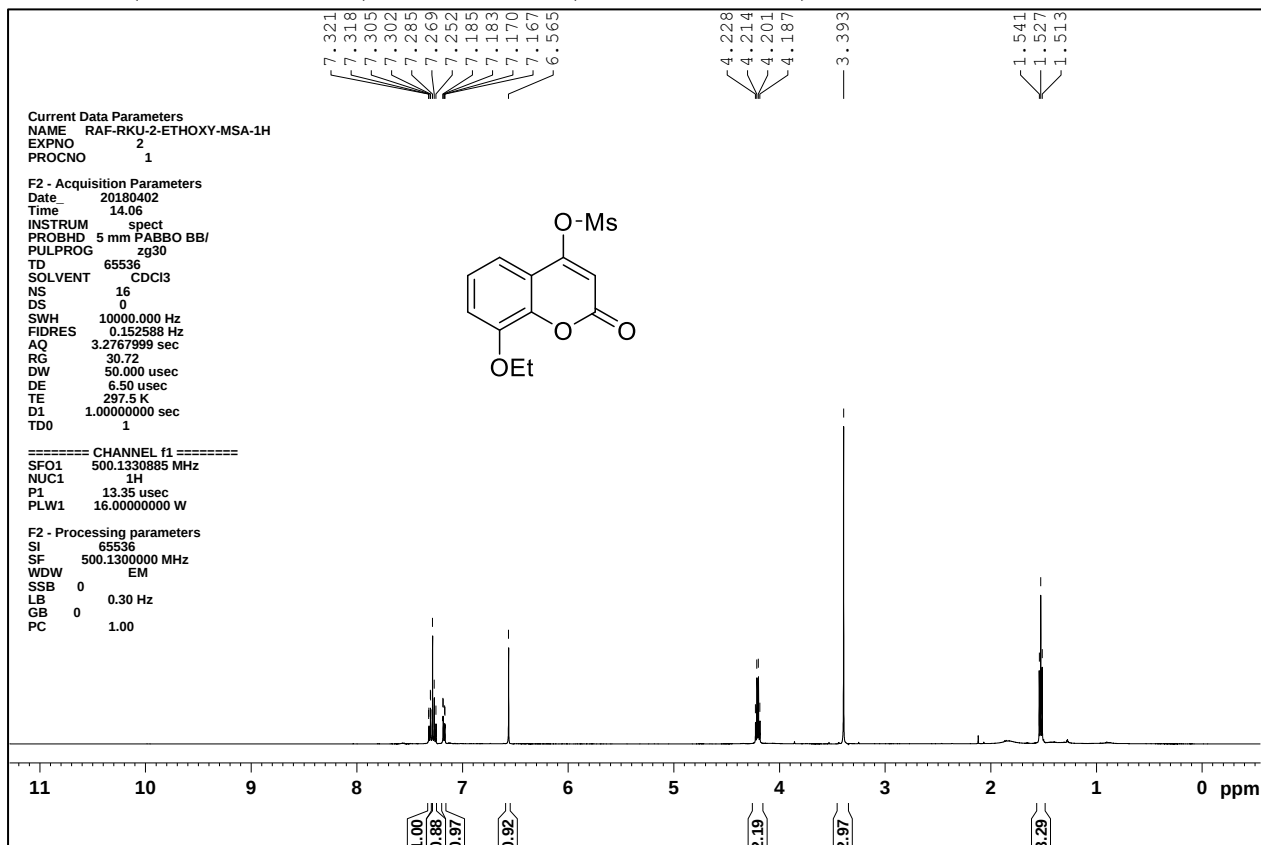
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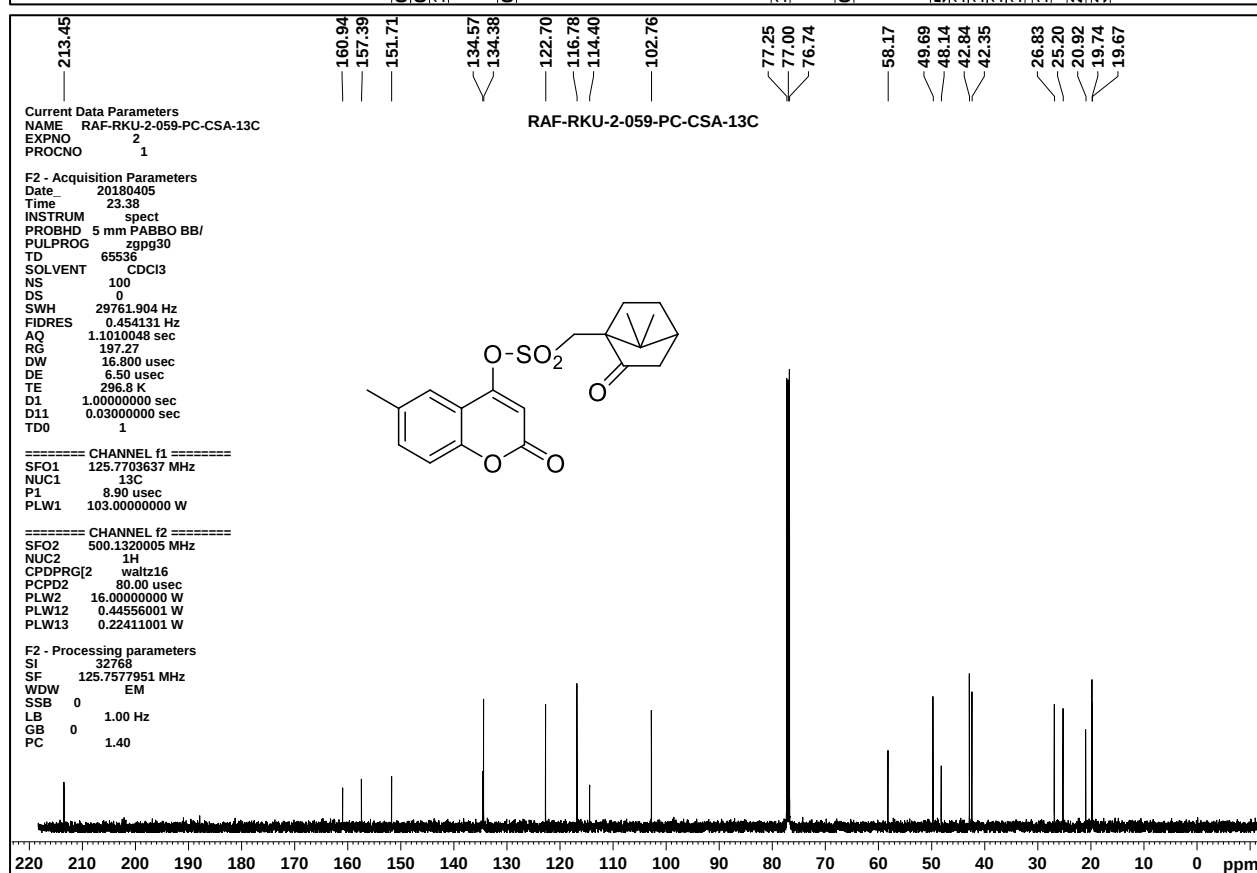
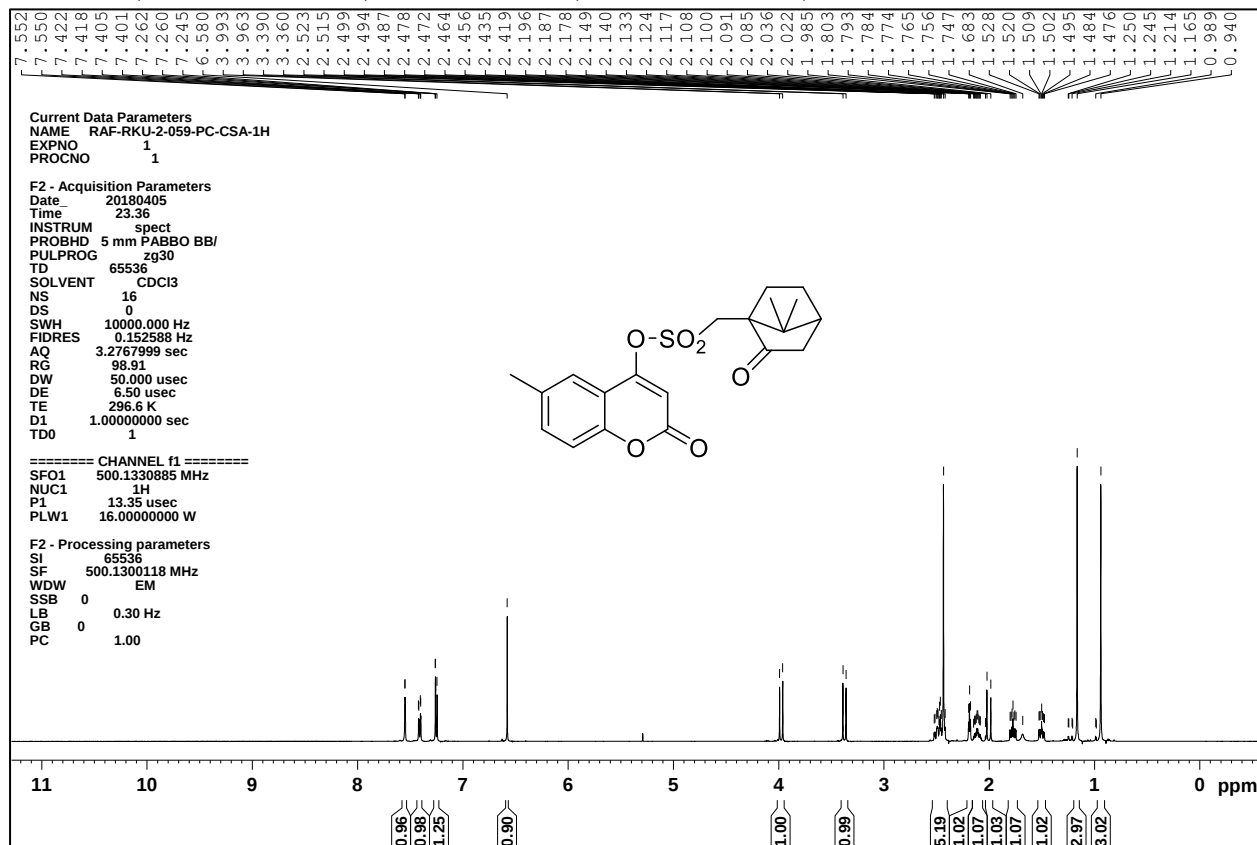
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of **10f**



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **10h**



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **11a**



Current Data Parameters  
NAME RAF-RKU-Ph-CSA-1H  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20190530  
Time 18.01  
INSTRUM spect  
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PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.298593 sec  
RG 90.5  
DW 60.800 usec  
DE 6.50 usec  
TE 297.5 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
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P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
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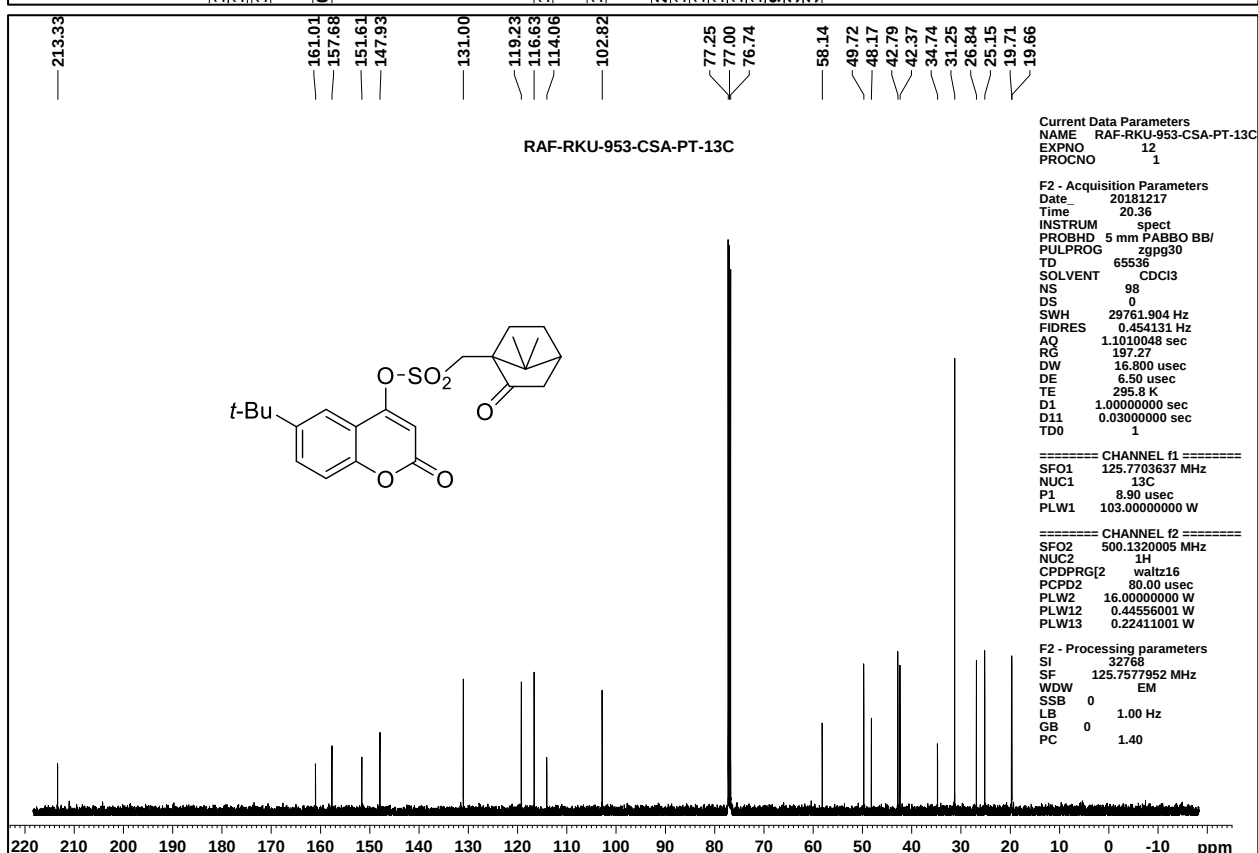
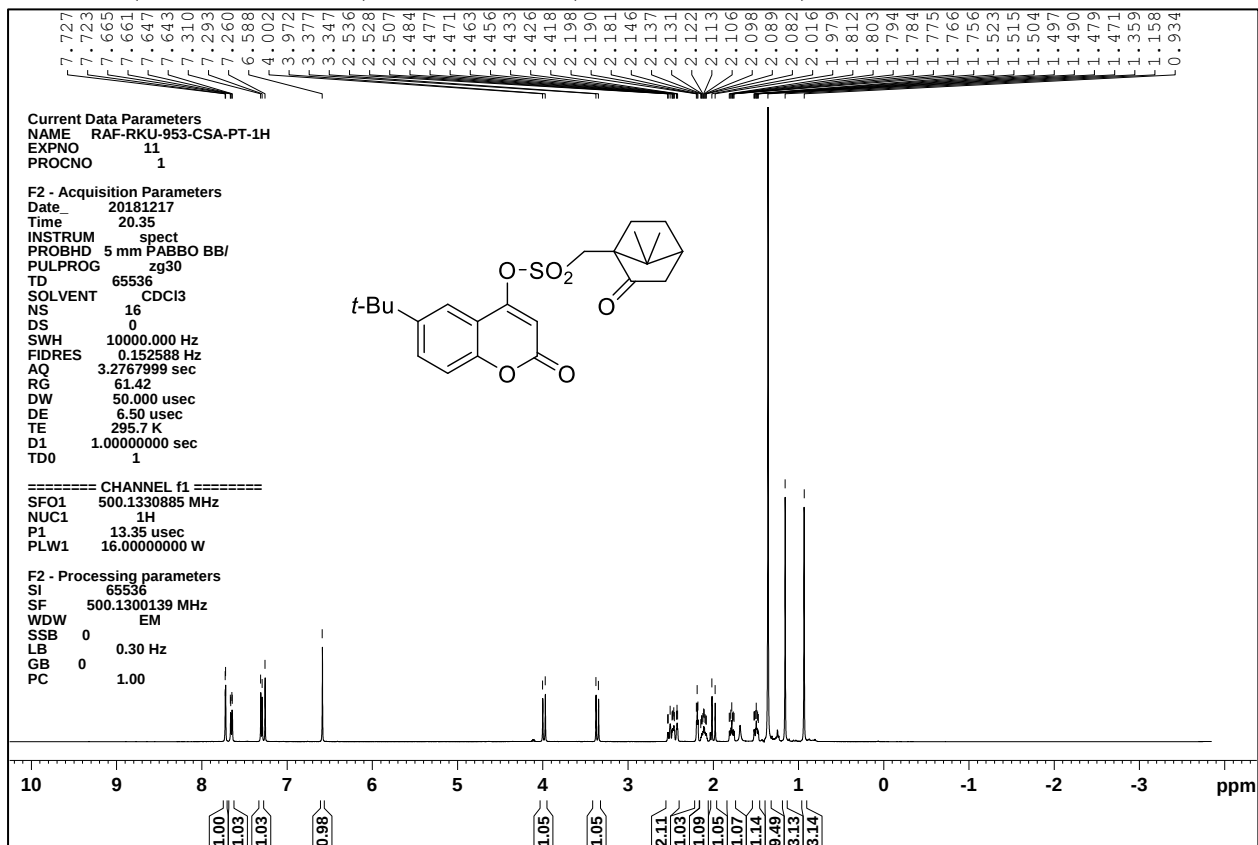
Chemical structure: O=C1C(=C2C(=C1)C=CC=C2)OS(=O)CC34C5C6C7C8C9C(C=C5)C(=O)C6(C=C8)C7(C=C9)C34

Peak list (ppm): 7.770, 7.632, 7.629, 7.611, 7.593, 7.590, 7.371, 7.362, 7.349, 7.343, 7.324, 7.260, 6.603, 3.999, 3.962, 3.393, 3.356, 2.517, 2.507, 2.479, 2.472, 2.452, 2.423, 2.413, 2.404, 2.194, 2.183, 2.172, 2.151, 2.141, 2.132, 2.121, 2.110, 2.101, 2.091, 2.080, 2.071, 2.061, 2.021, 1.974, 1.809, 1.797, 1.785, 1.774, 1.763, 1.751, 1.739, 1.529, 1.520, 1.506, 1.497, 1.489, 1.475, 1.465, 1.154, 0.932.

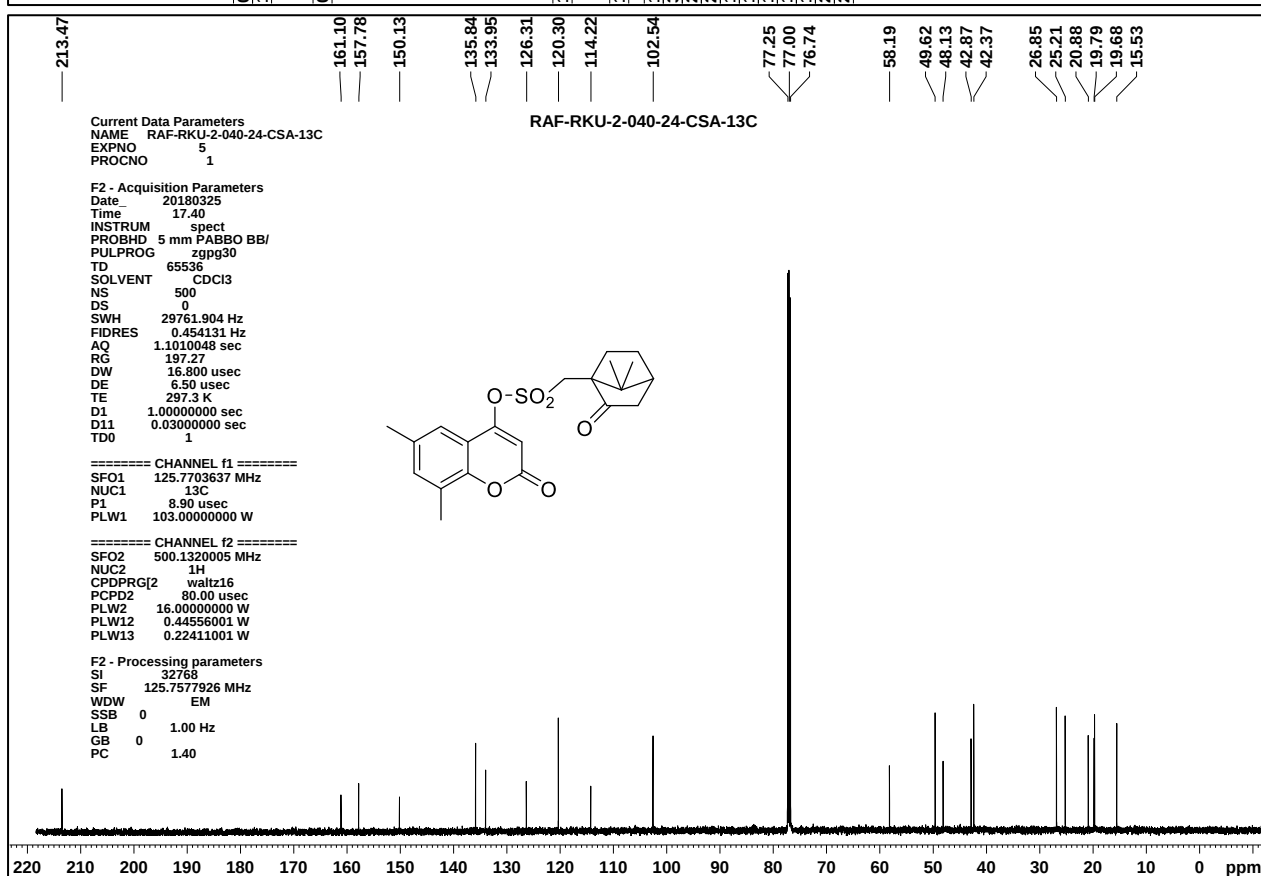
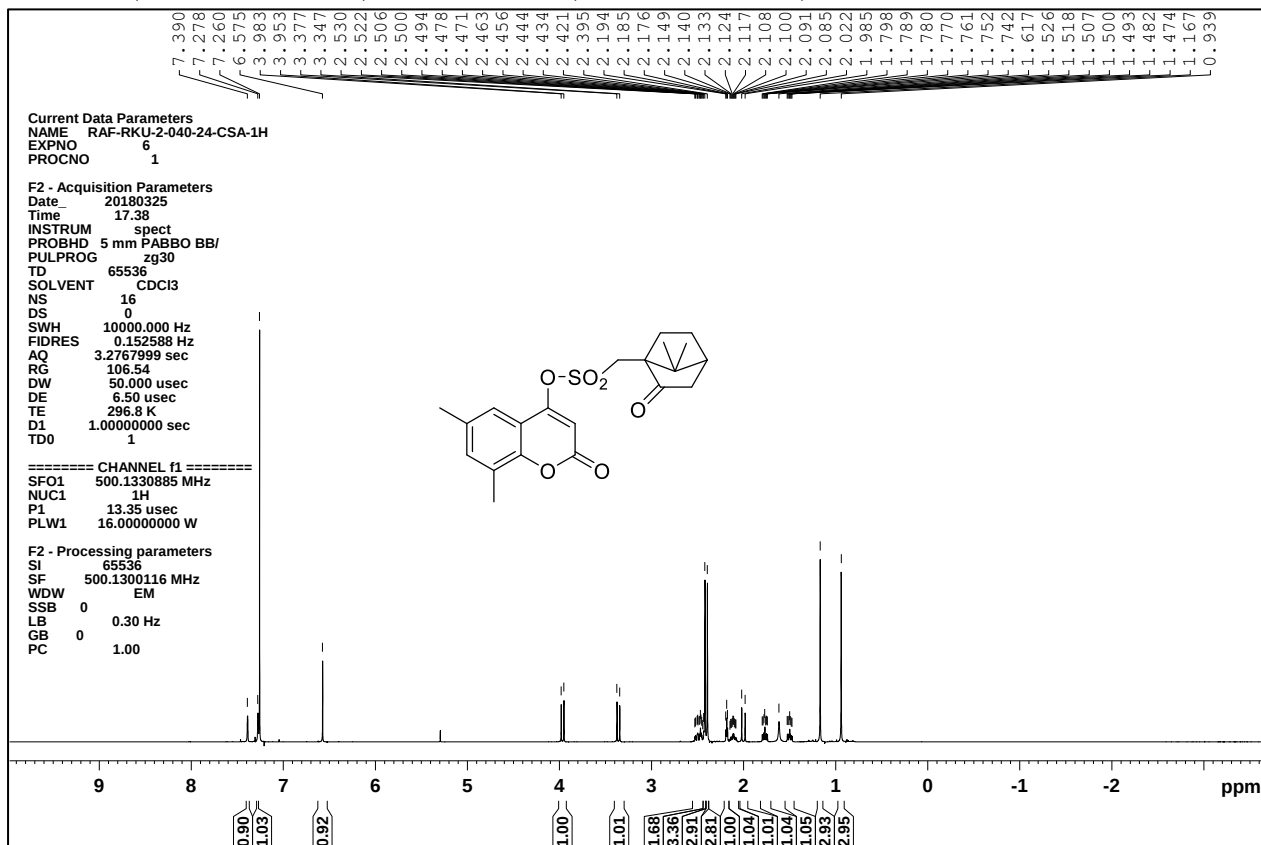
Integration values: 1.00, 1.01, 1.02, 0.98, 1.03, 1.02, 1.09, 0.97, 1.13, 1.08, 1.27, 1.02, 3.00, 2.98.



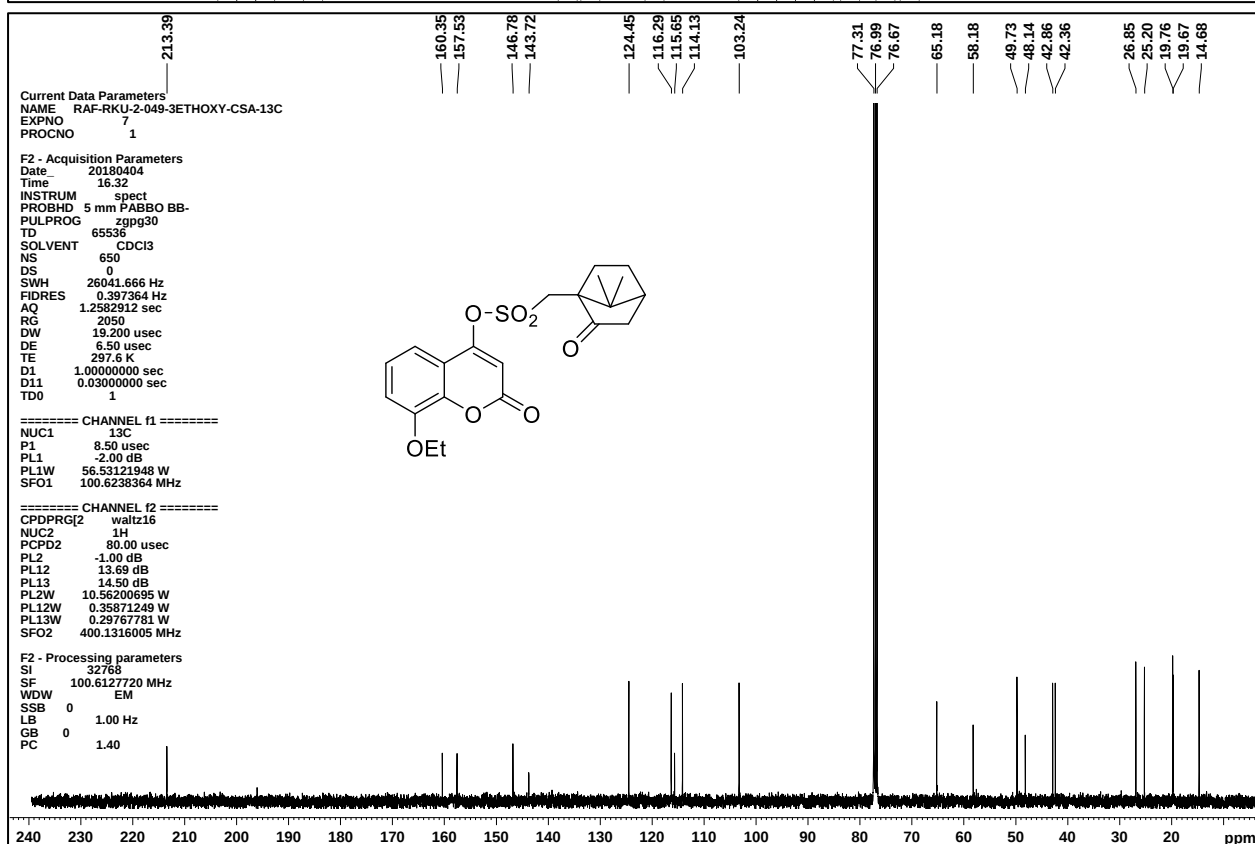
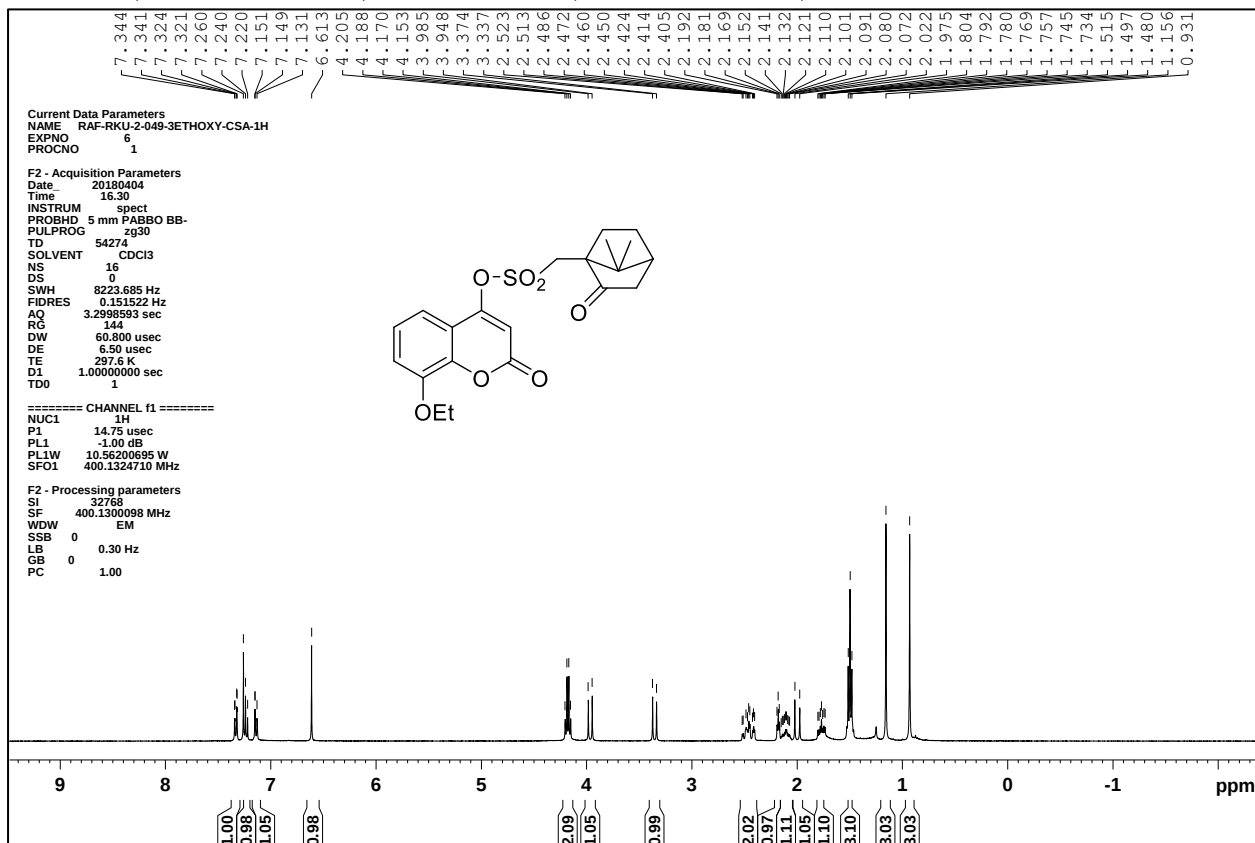
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of **11c**



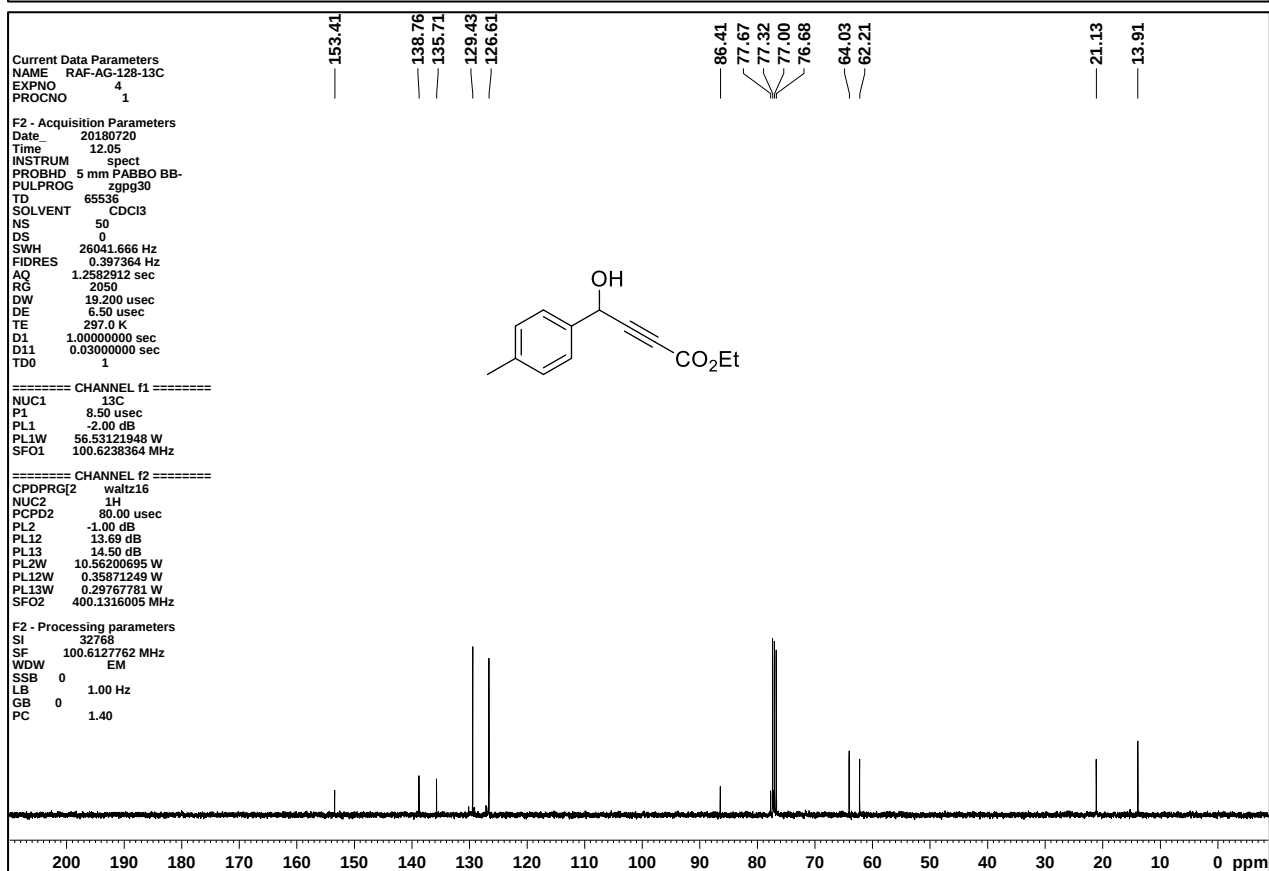
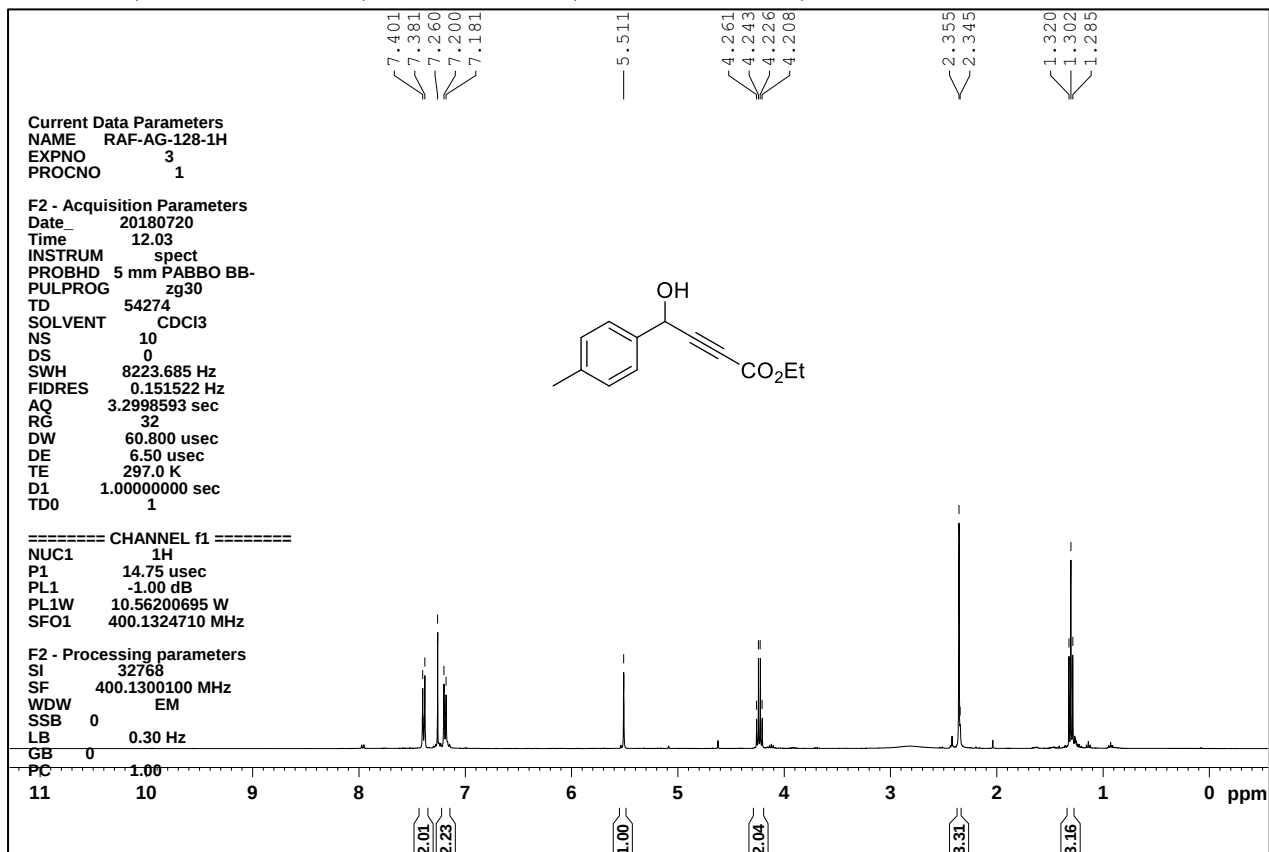
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of **11f**



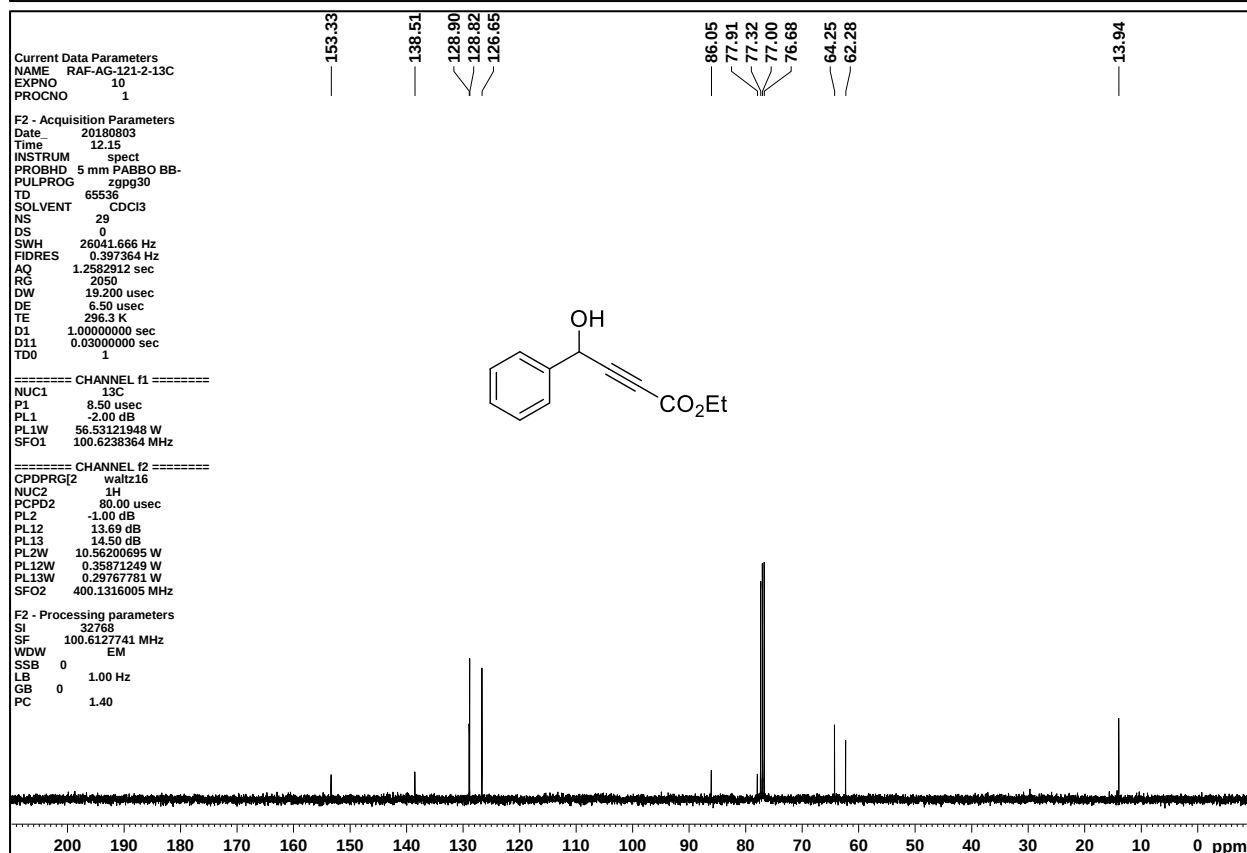
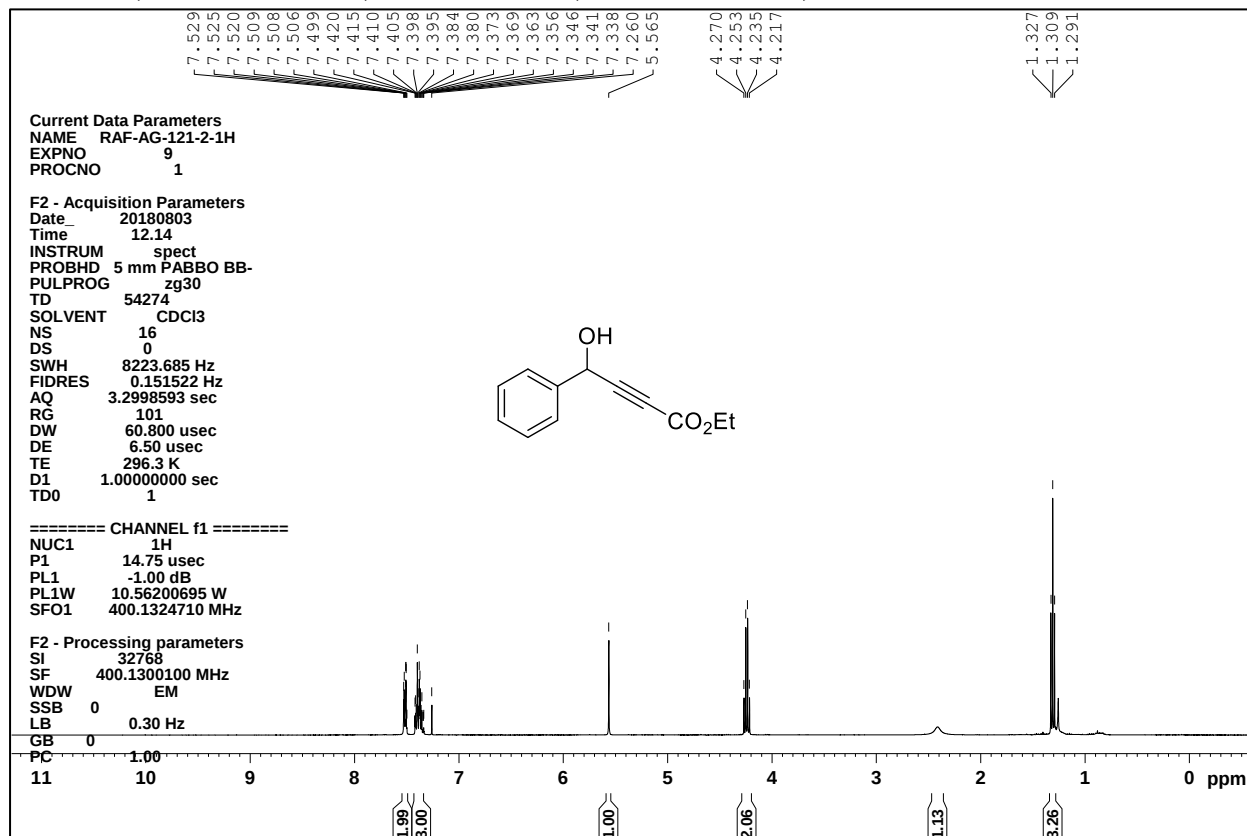
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **11h**



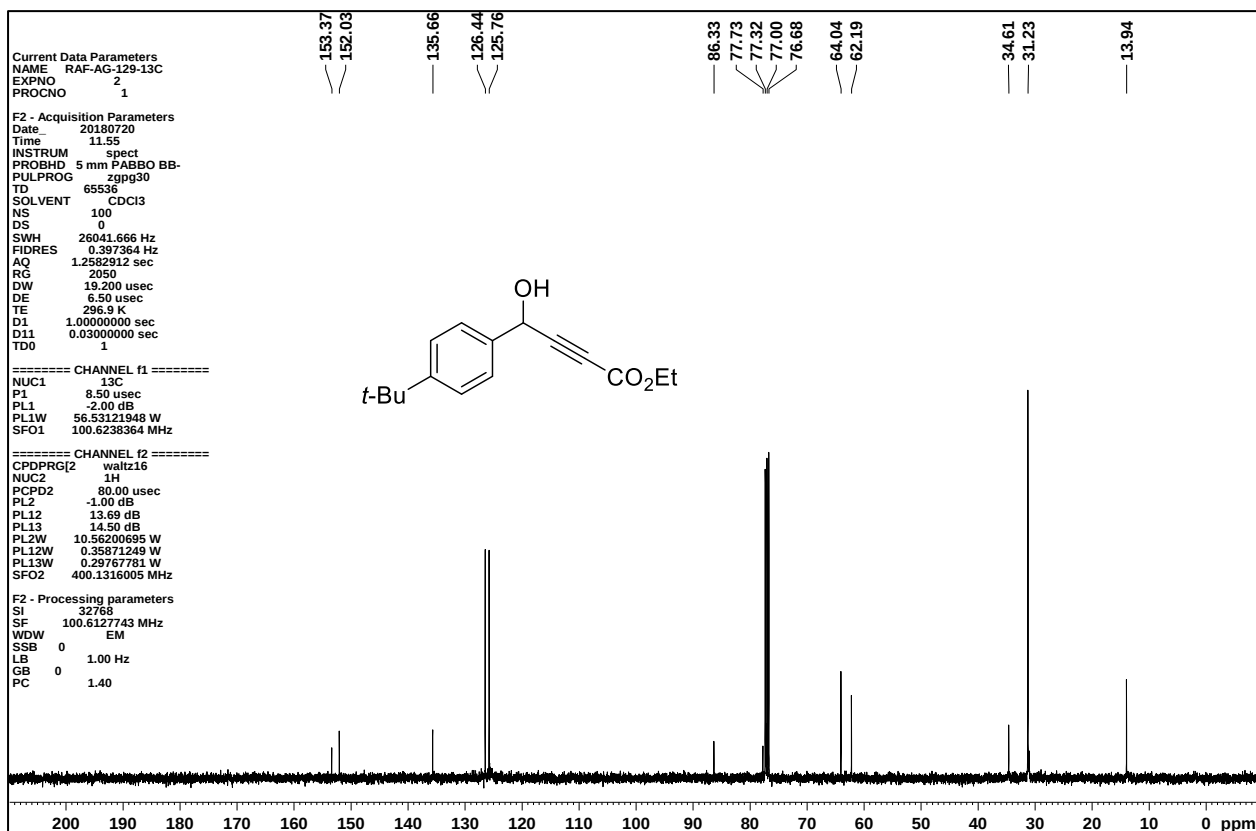
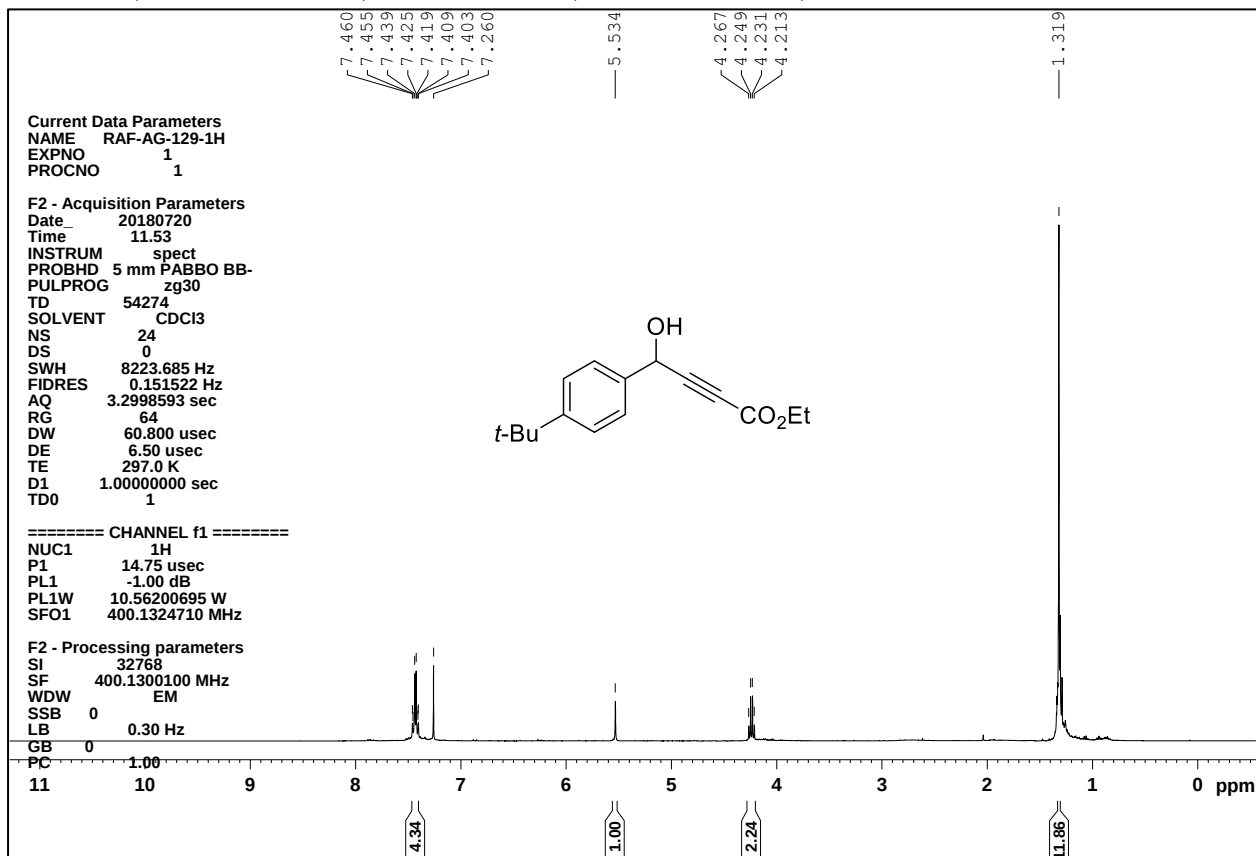
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of **12a**



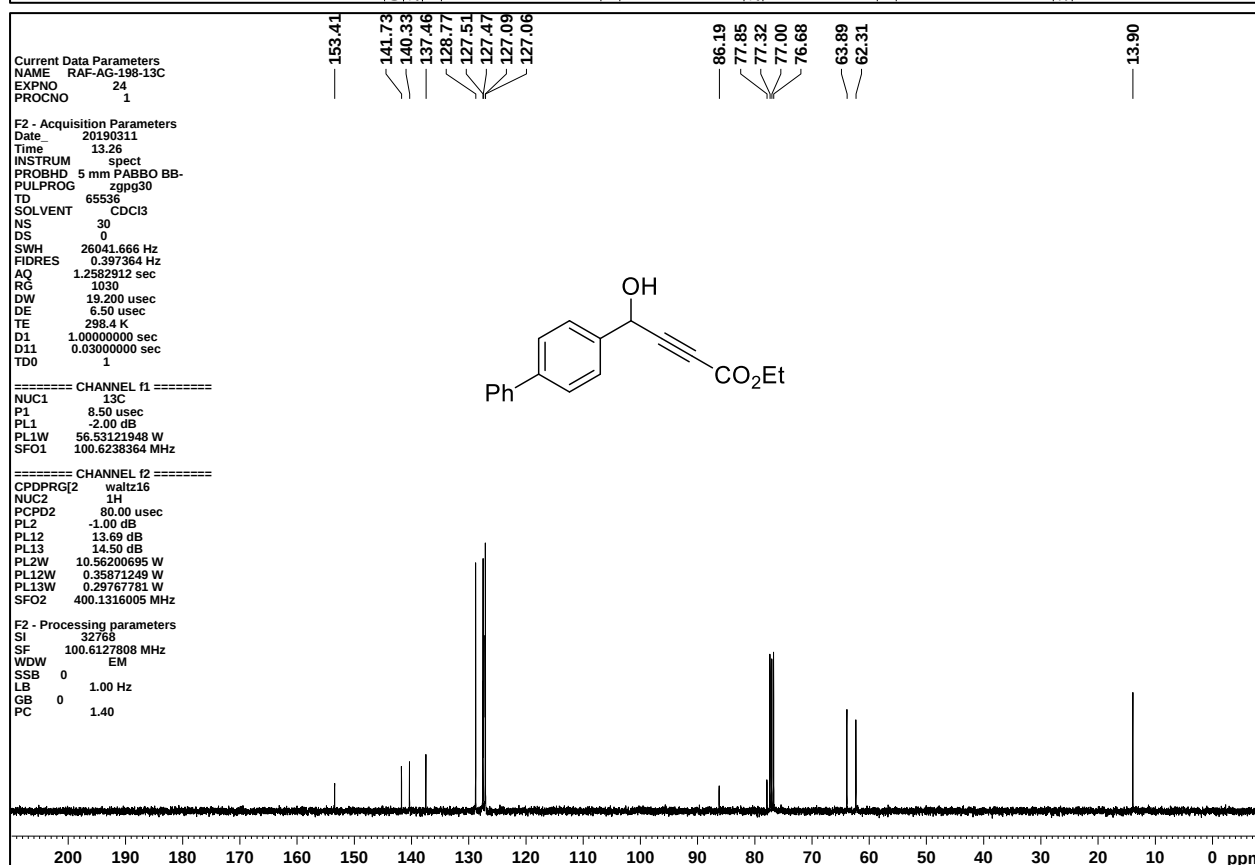
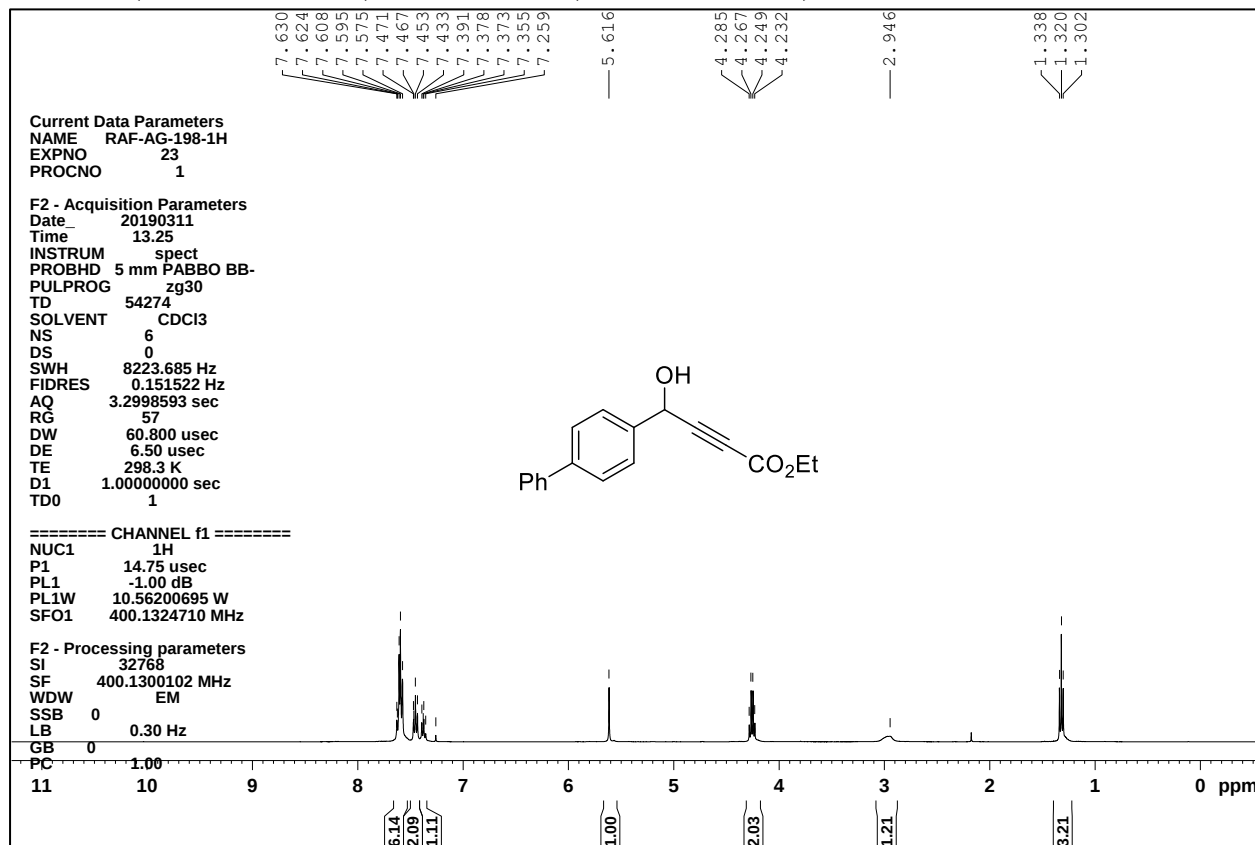
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **12b**



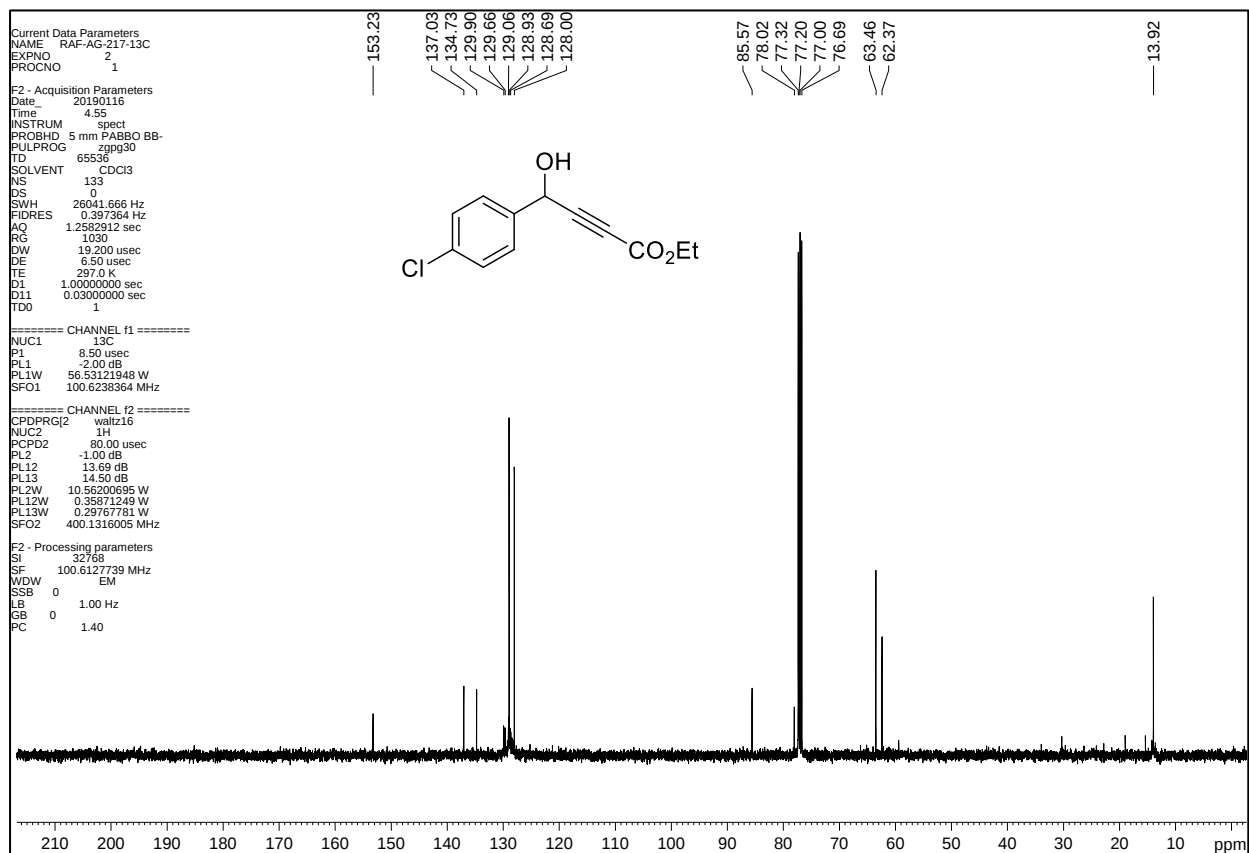
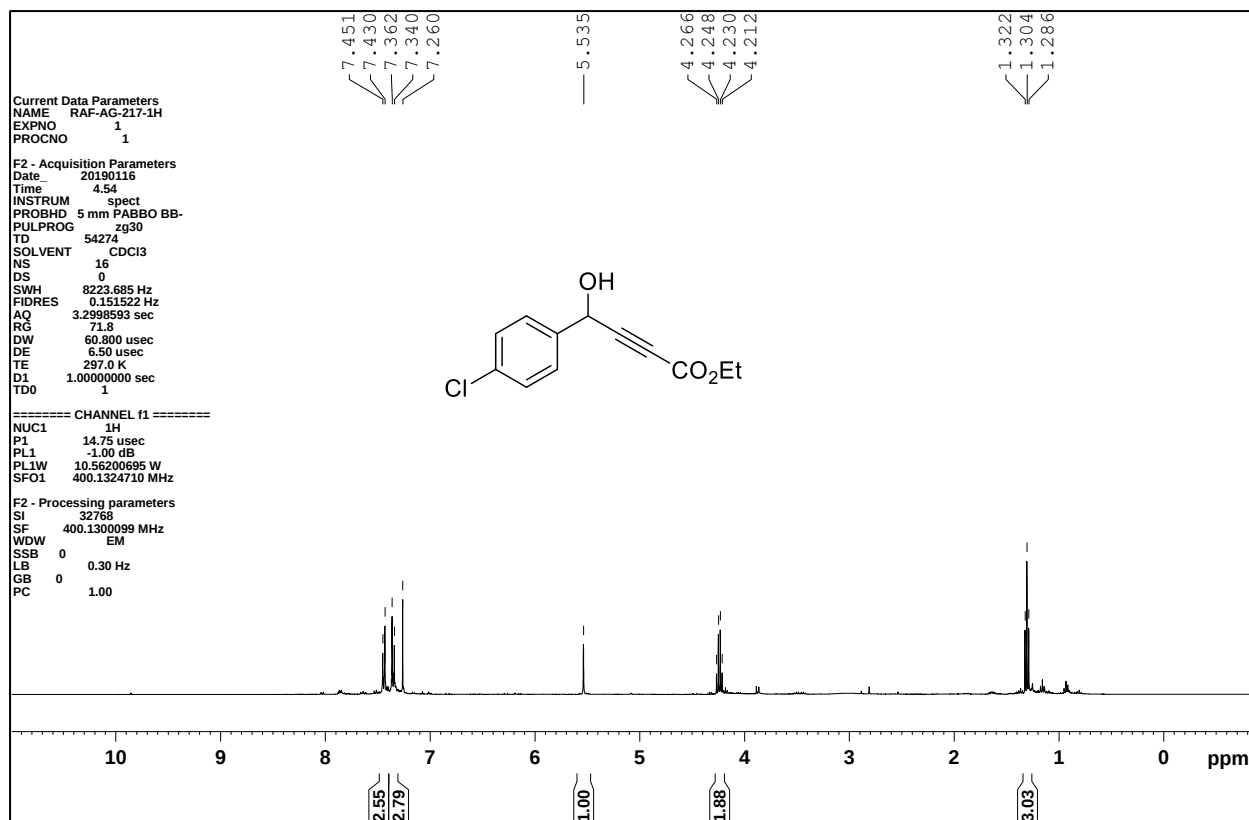
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of of **12c**



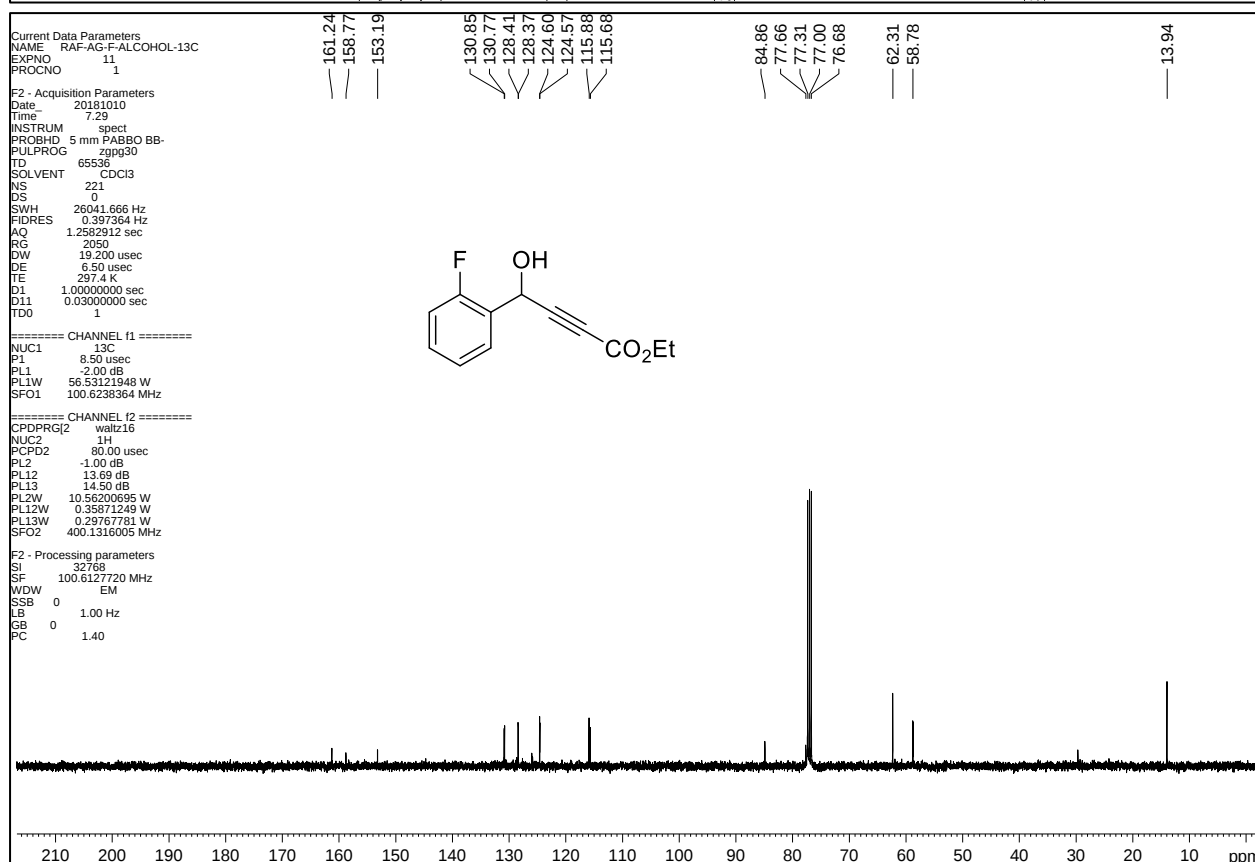
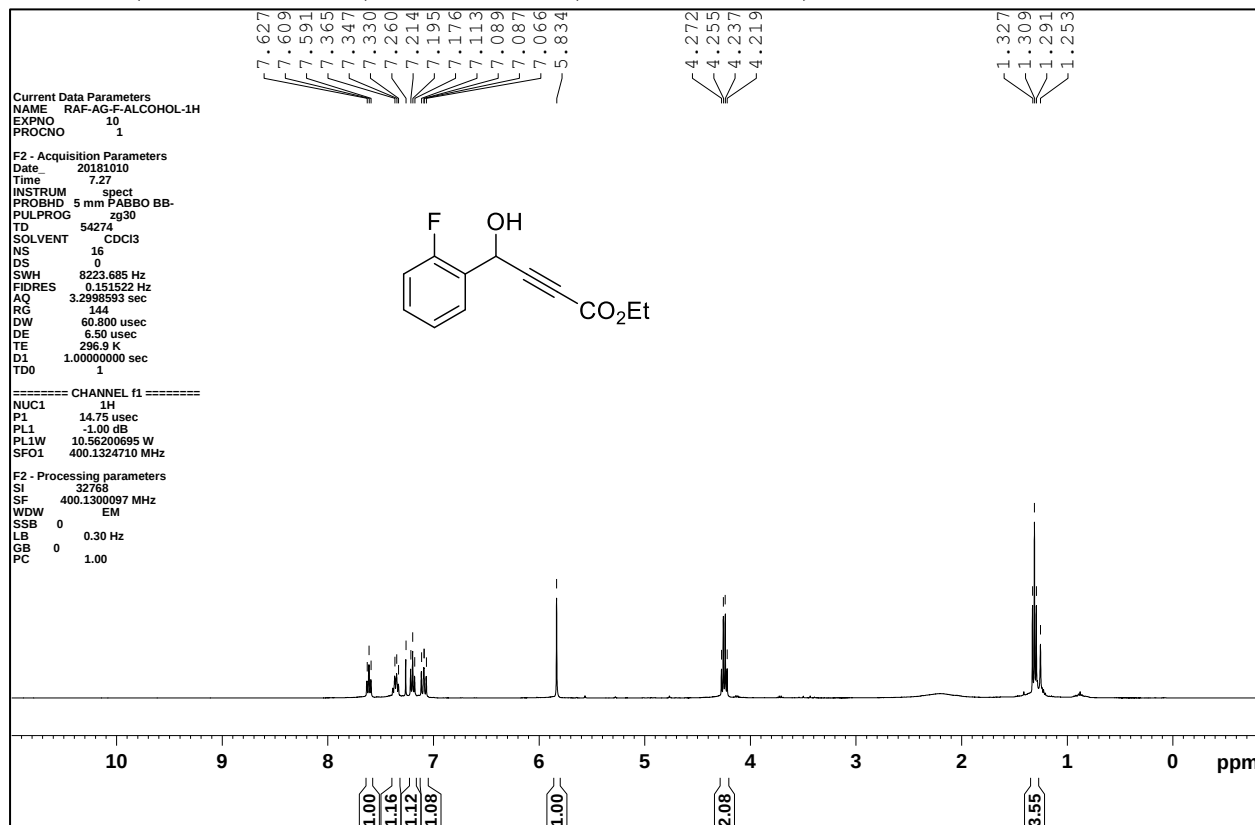
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of **12d**



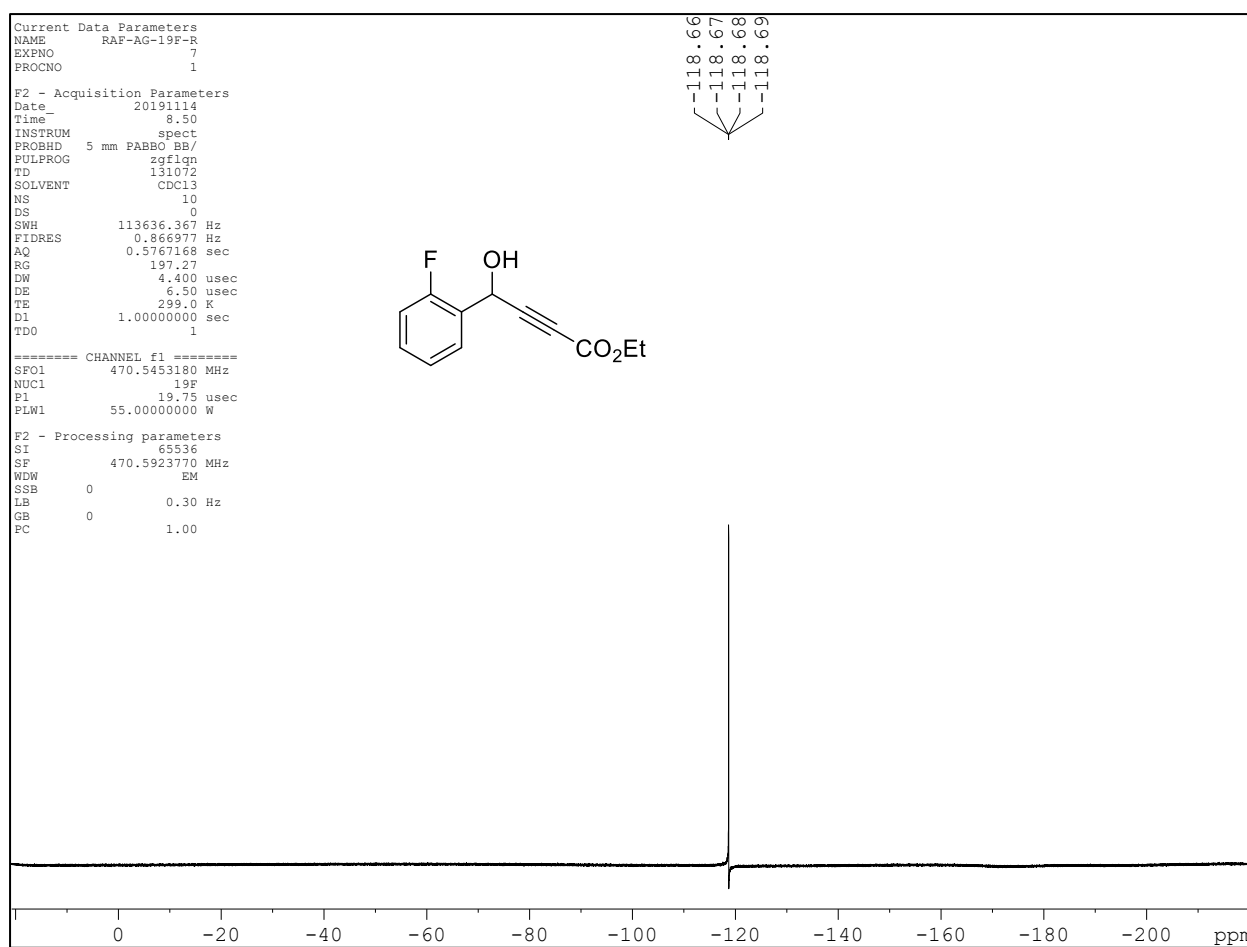
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **12e**



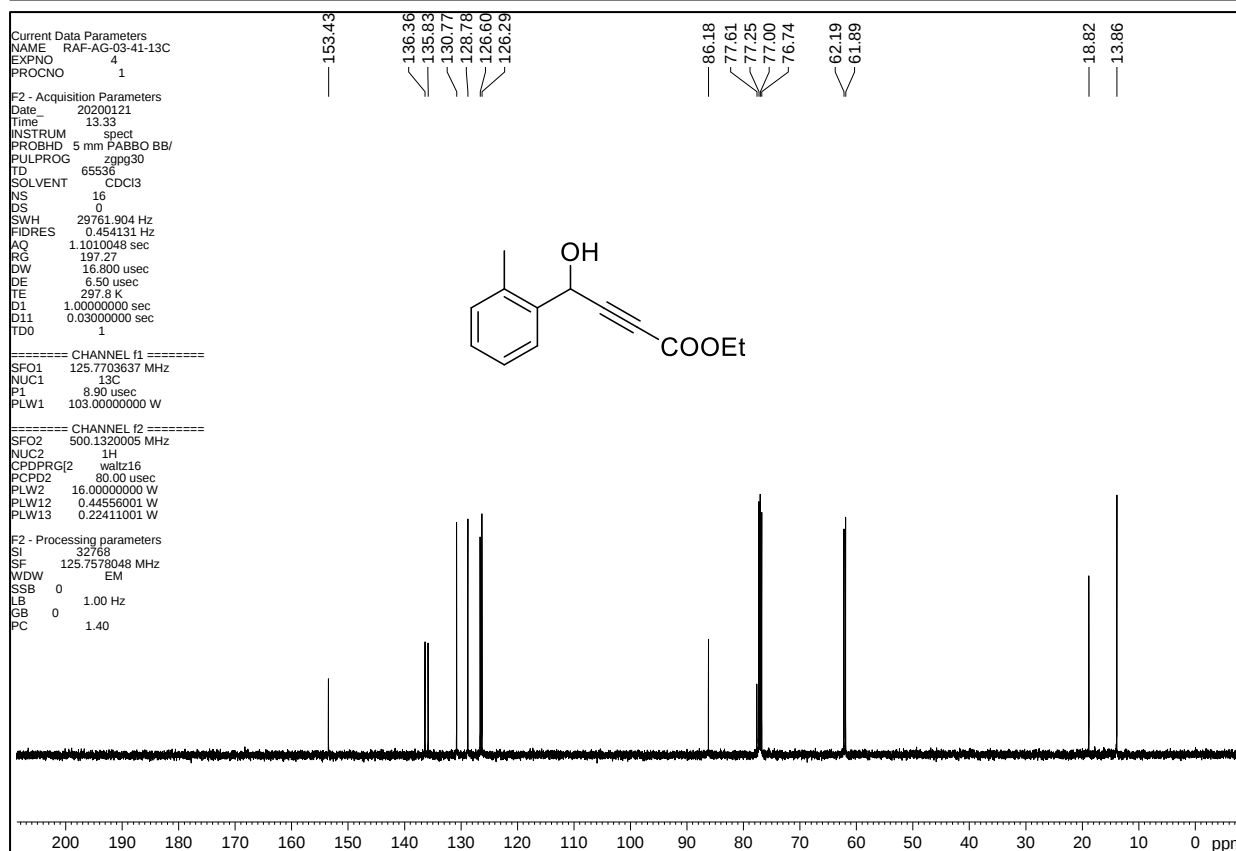
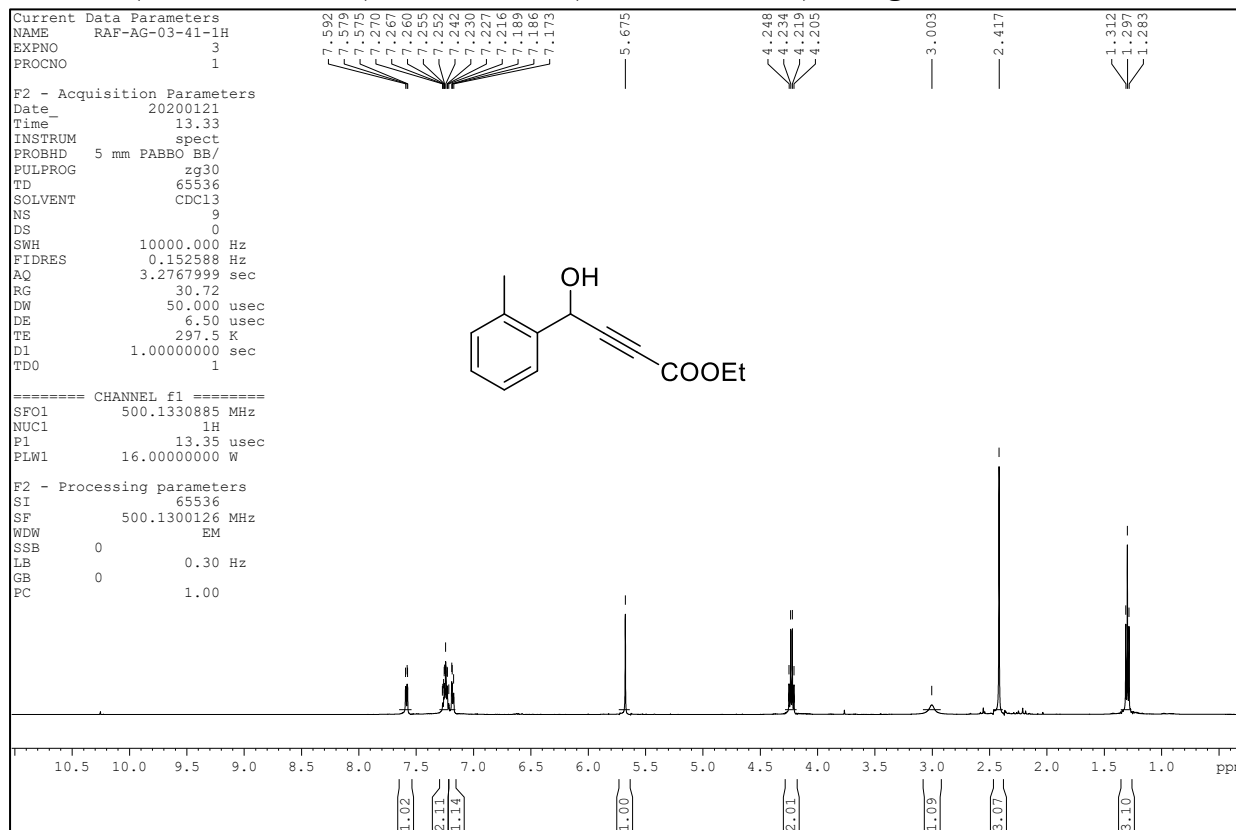
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **12f**



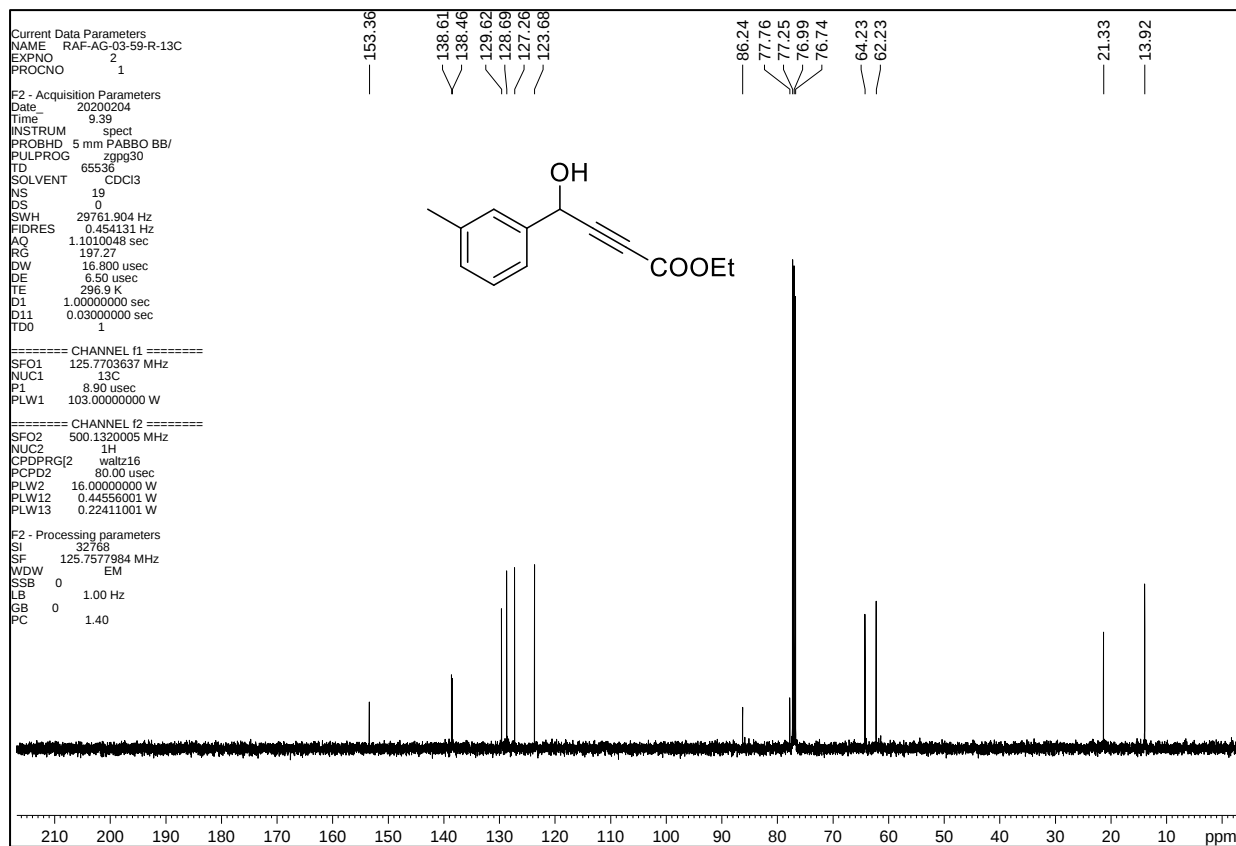
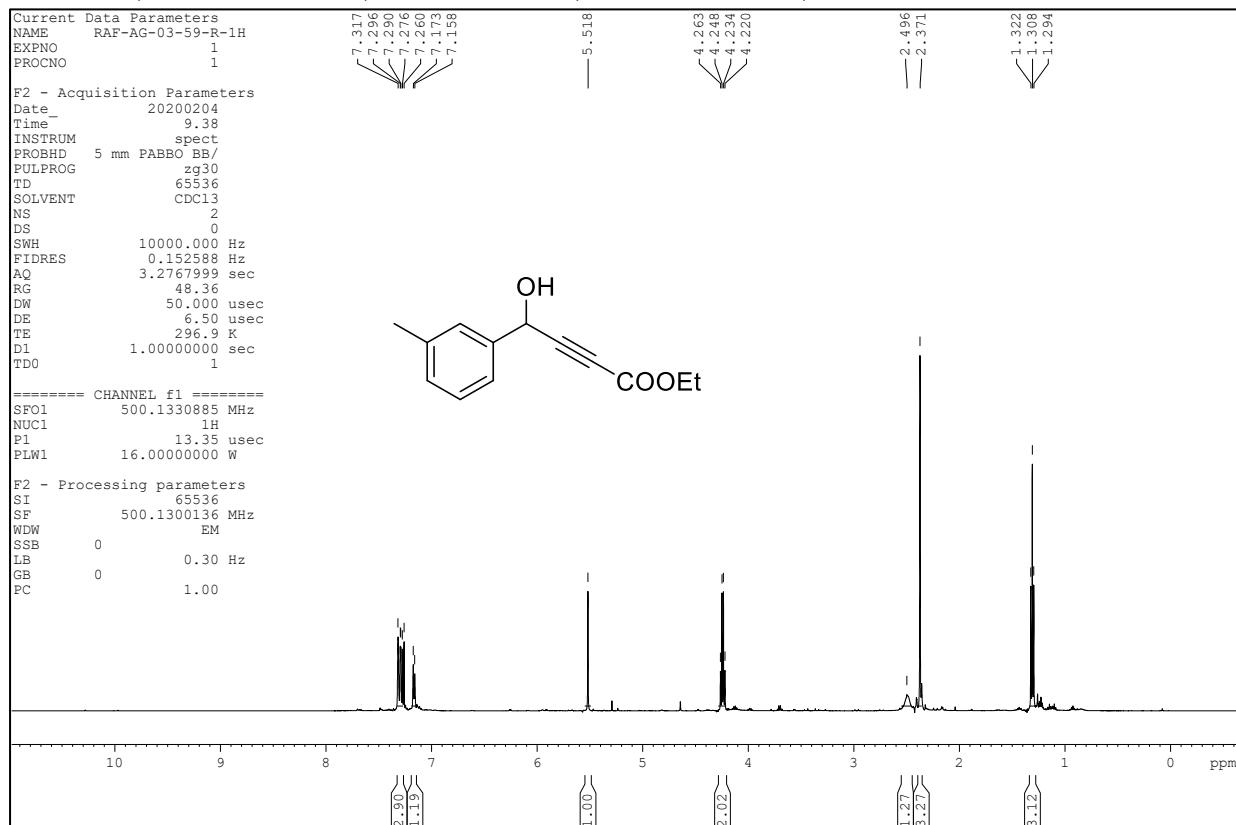
<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) of **12f**



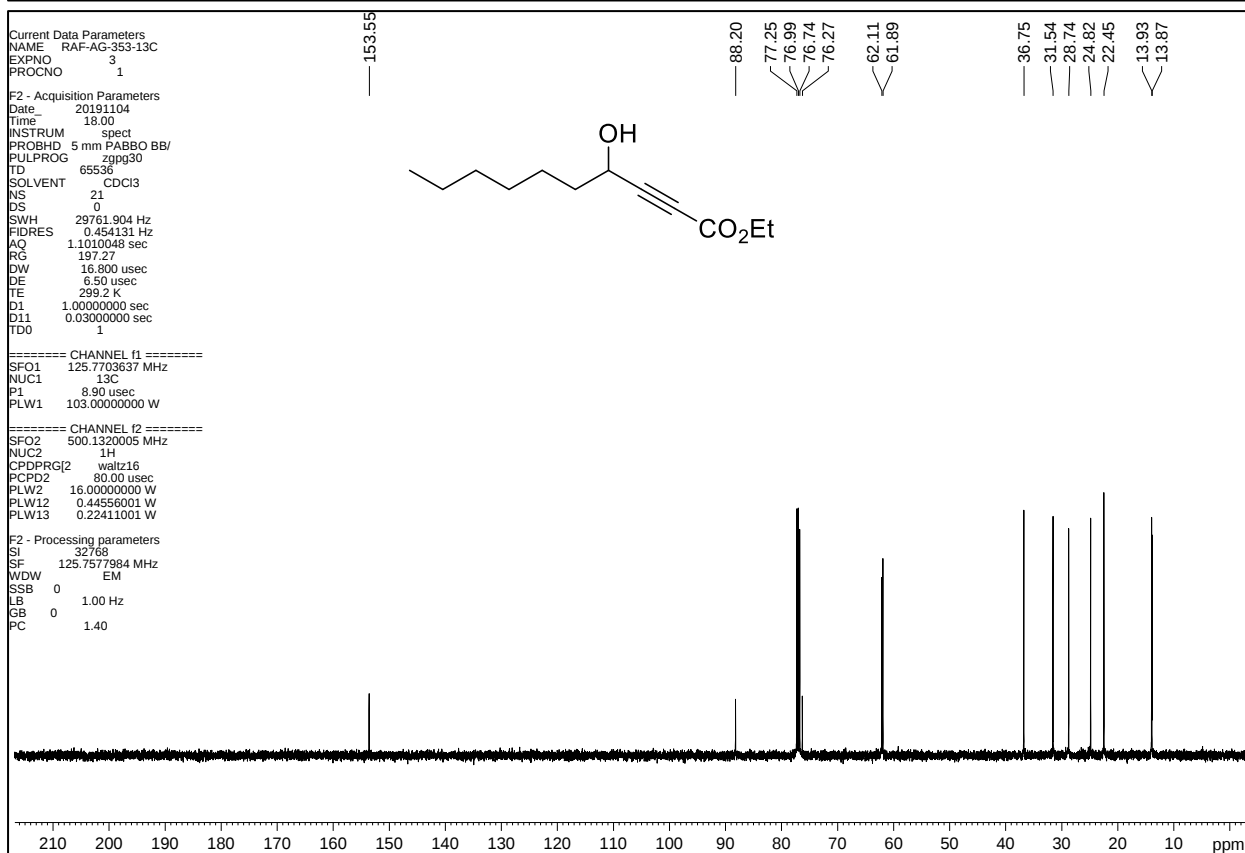
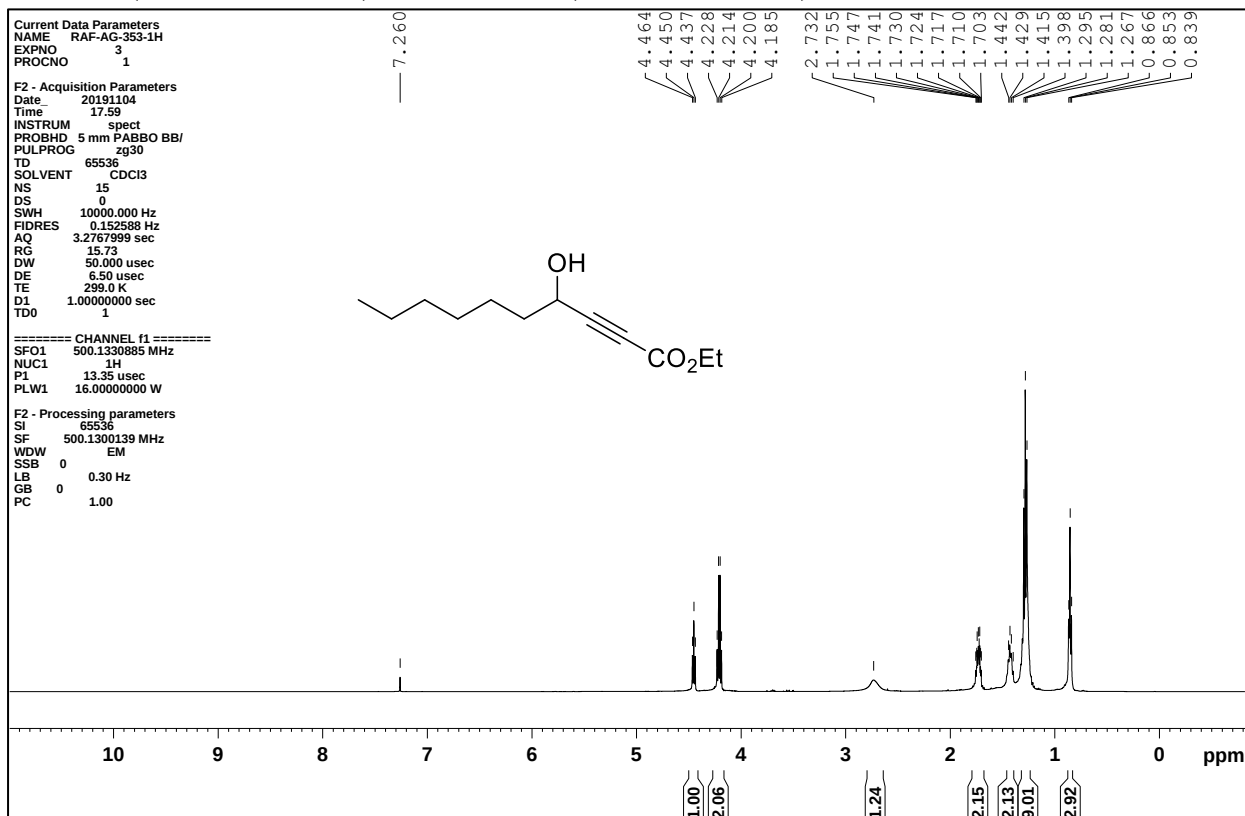
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **12g**



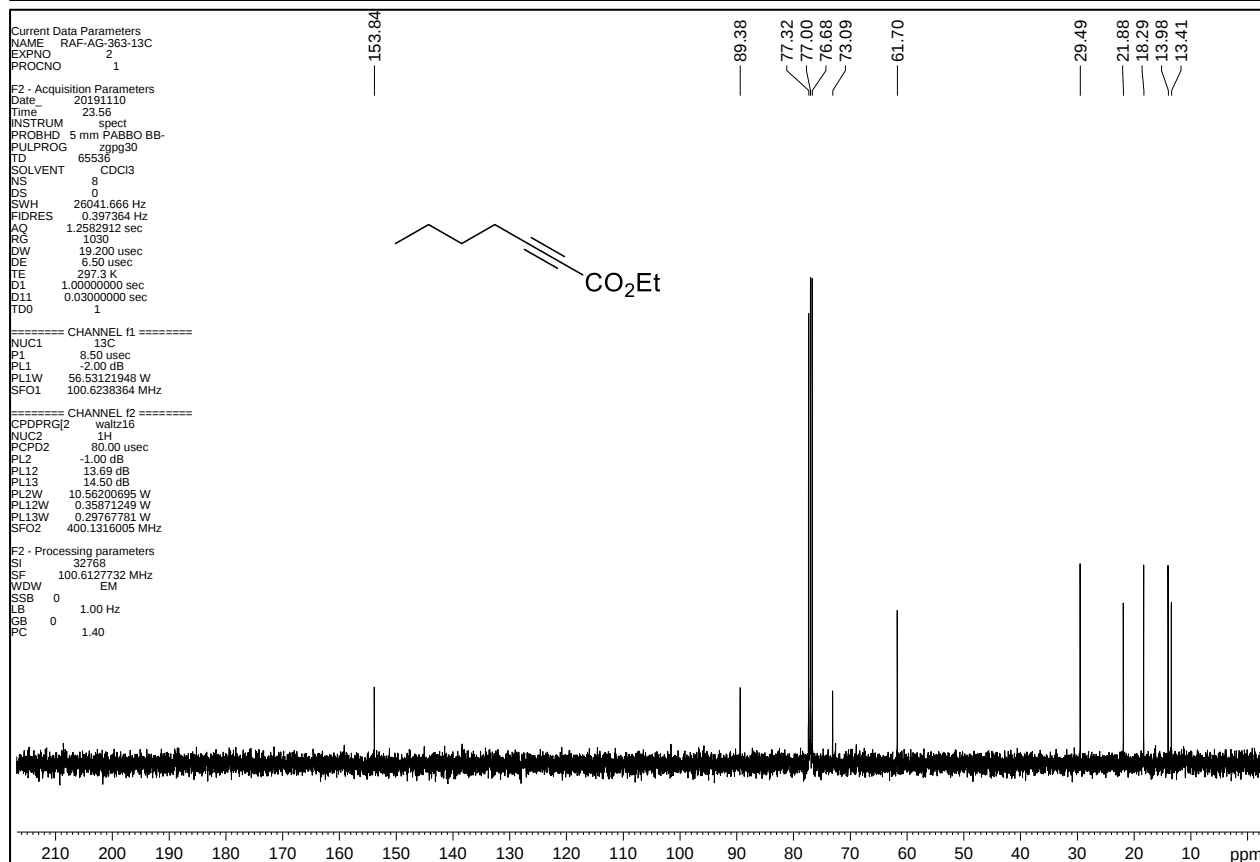
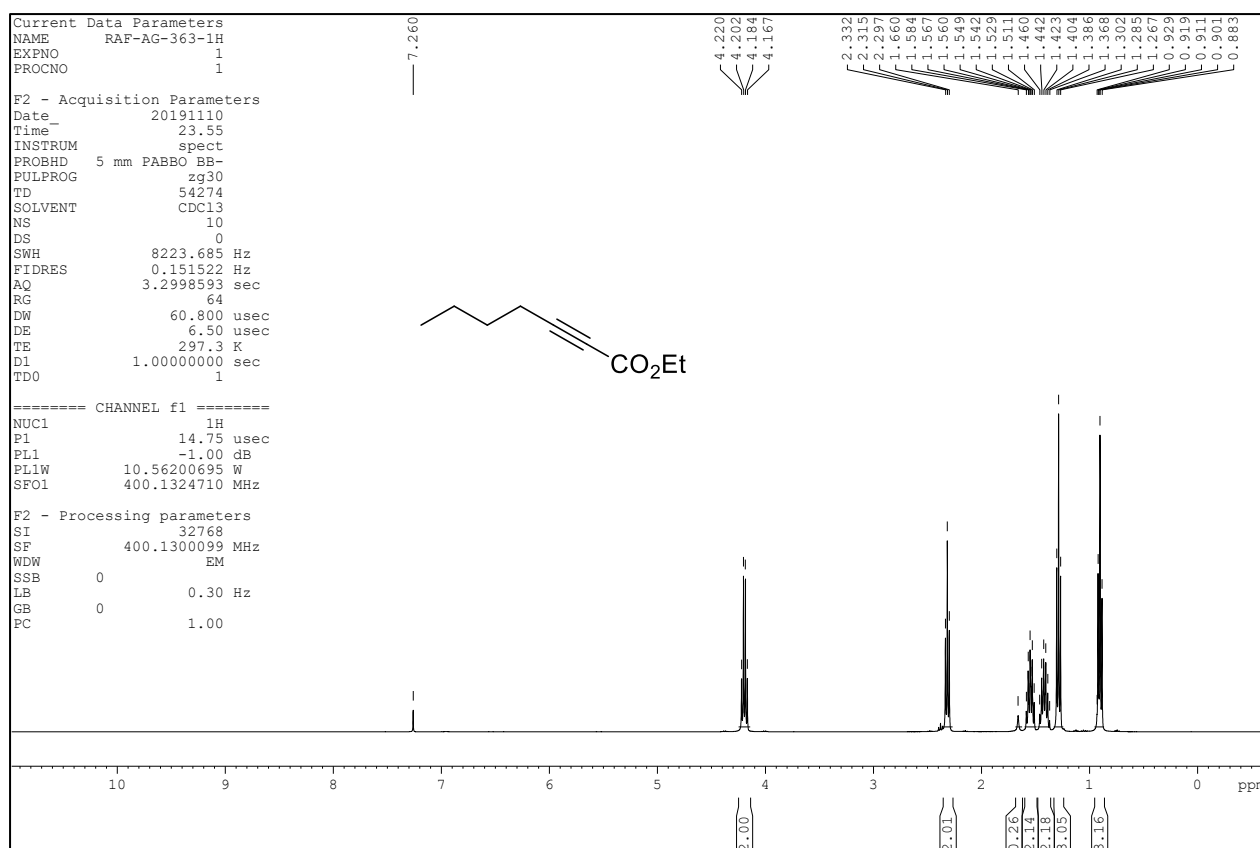
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **12h**



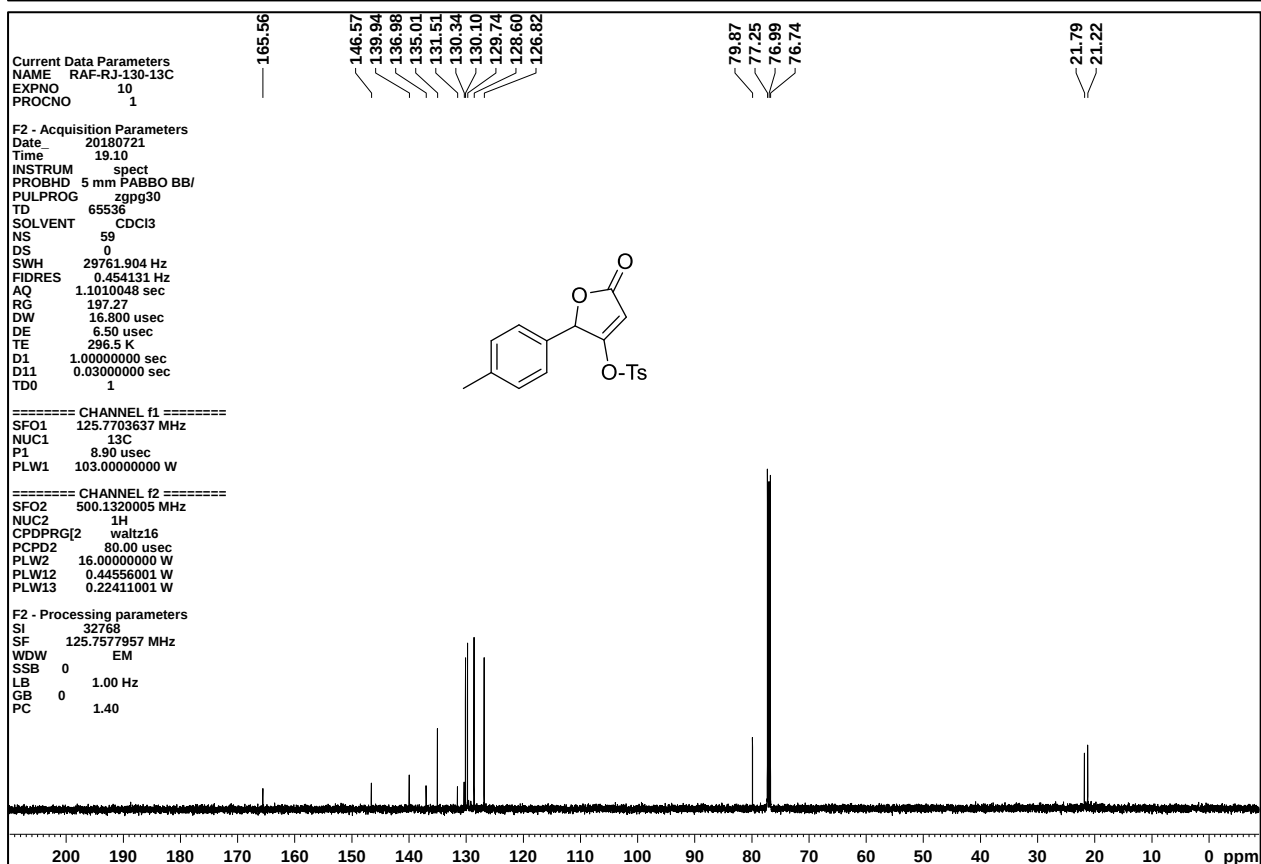
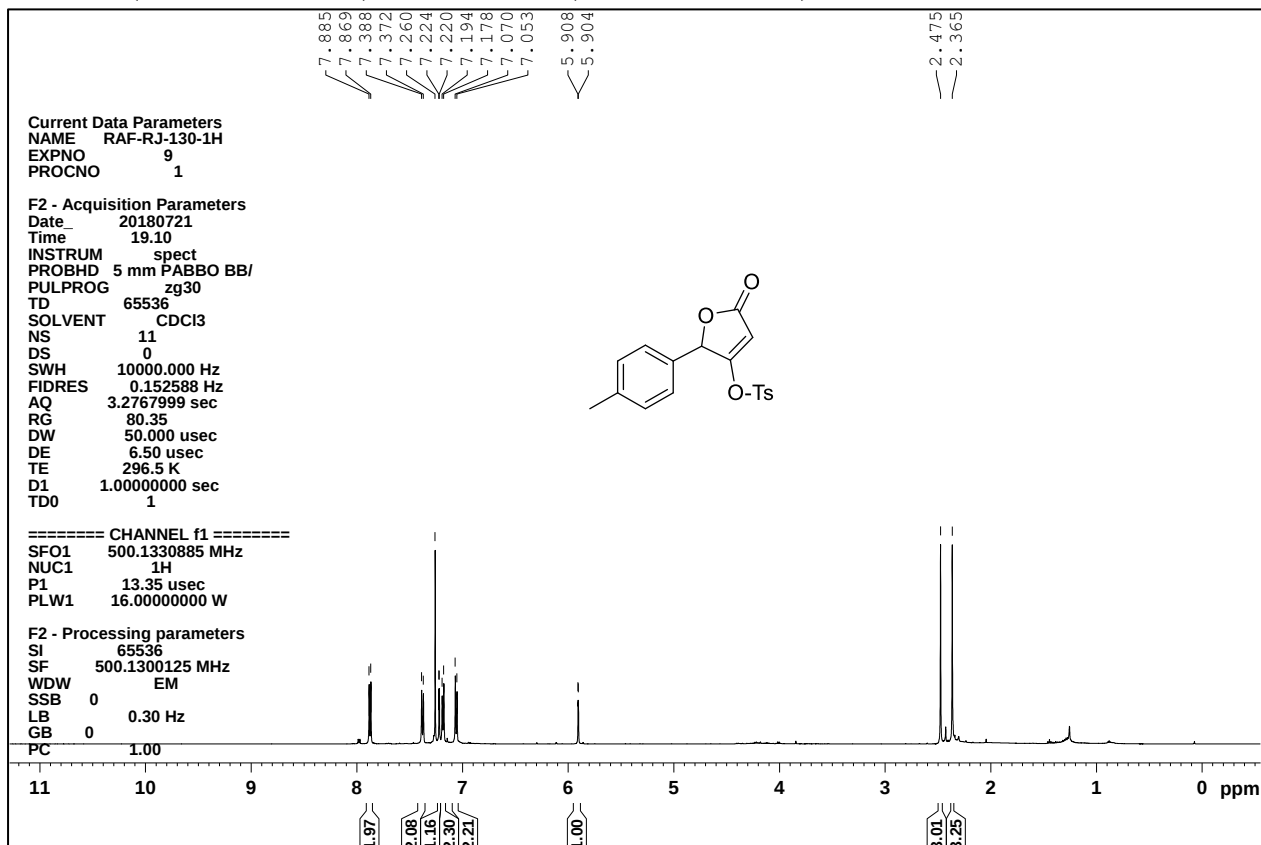
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **12i**



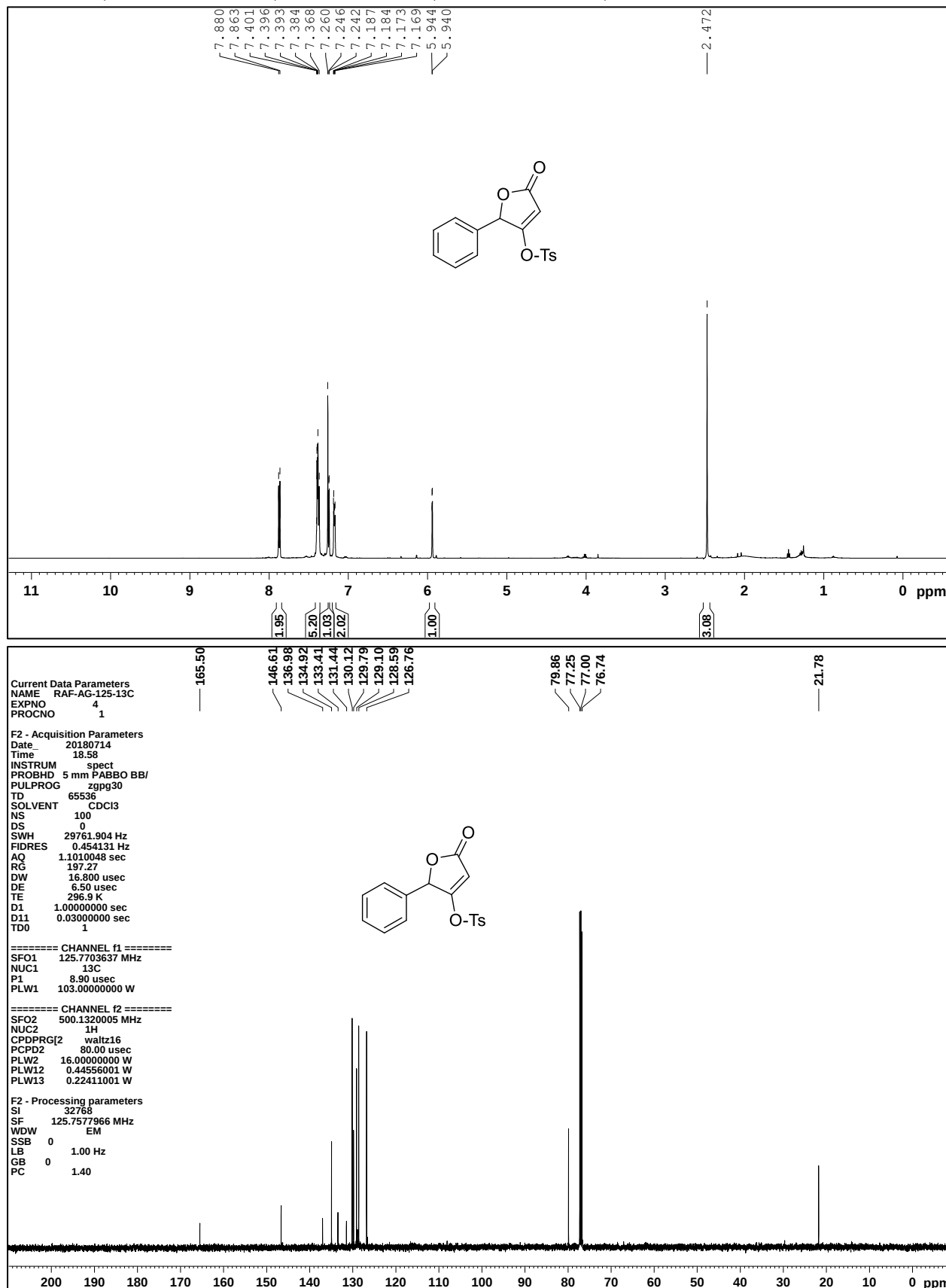
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **12j**



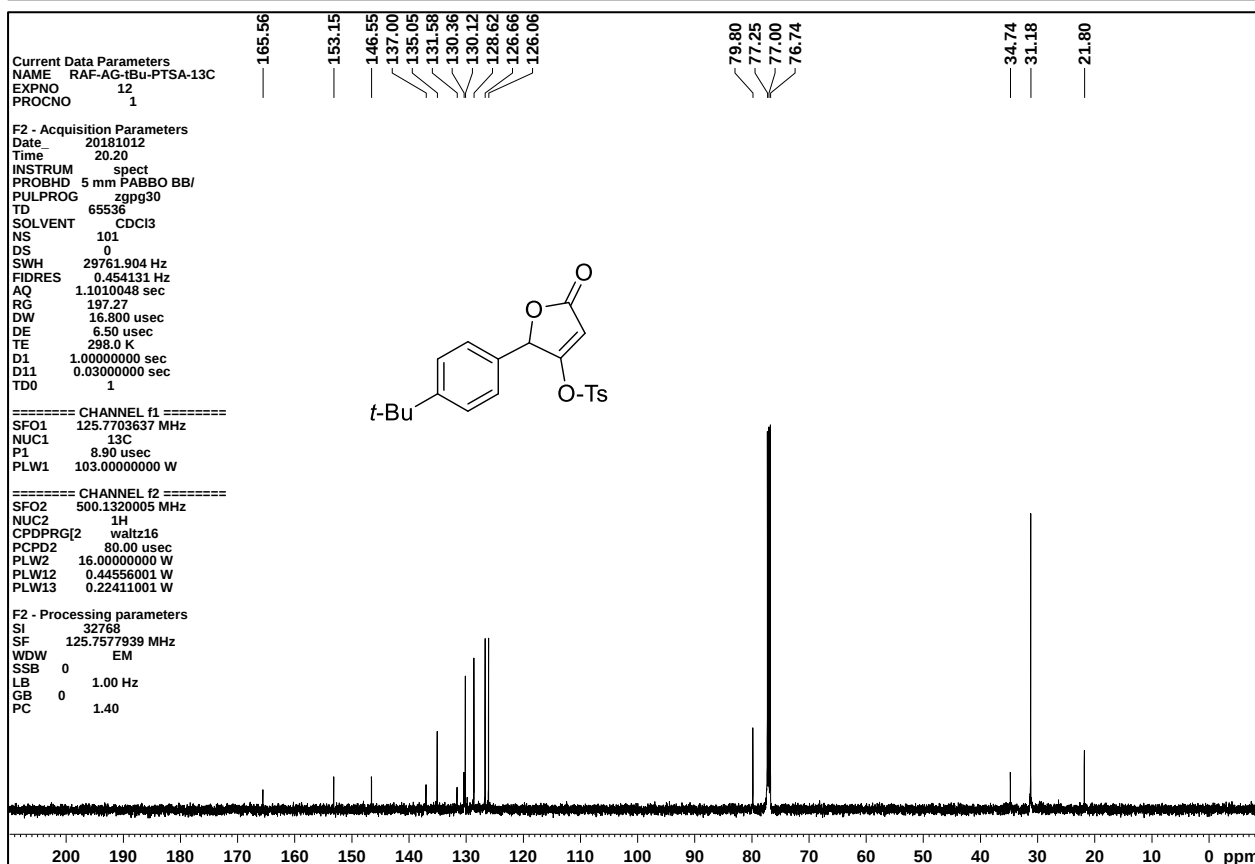
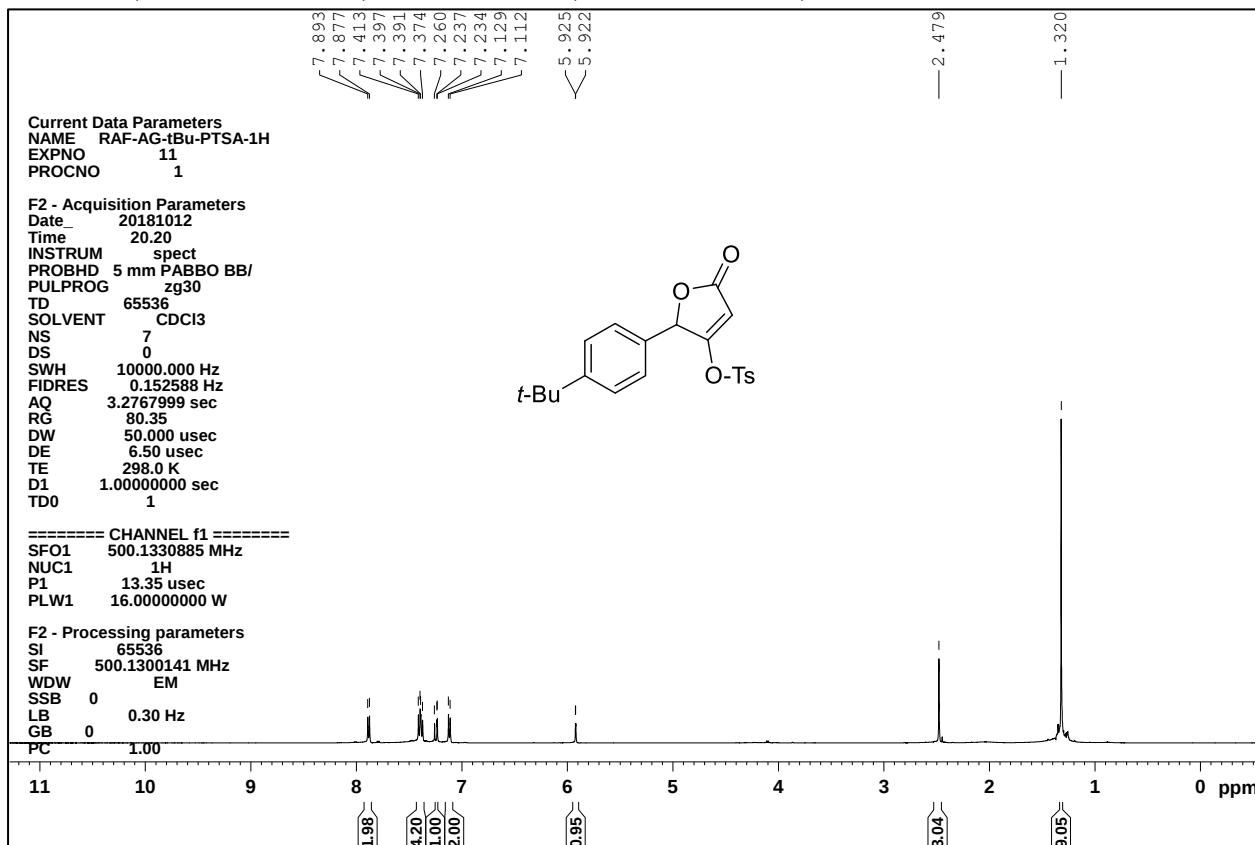
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of **13a**



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **13b**



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of **13c**



Current Data Parameters  
NAME RAF-AG-200-1H  
EXPNO 9  
PROCNO 1

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Time 0.47  
INSTRUM spect  
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PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 13  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
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RG 161  
DW 60.800 usec  
DE 6.50 usec  
TE 297.8 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
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PL1W 10.56200695 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
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SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

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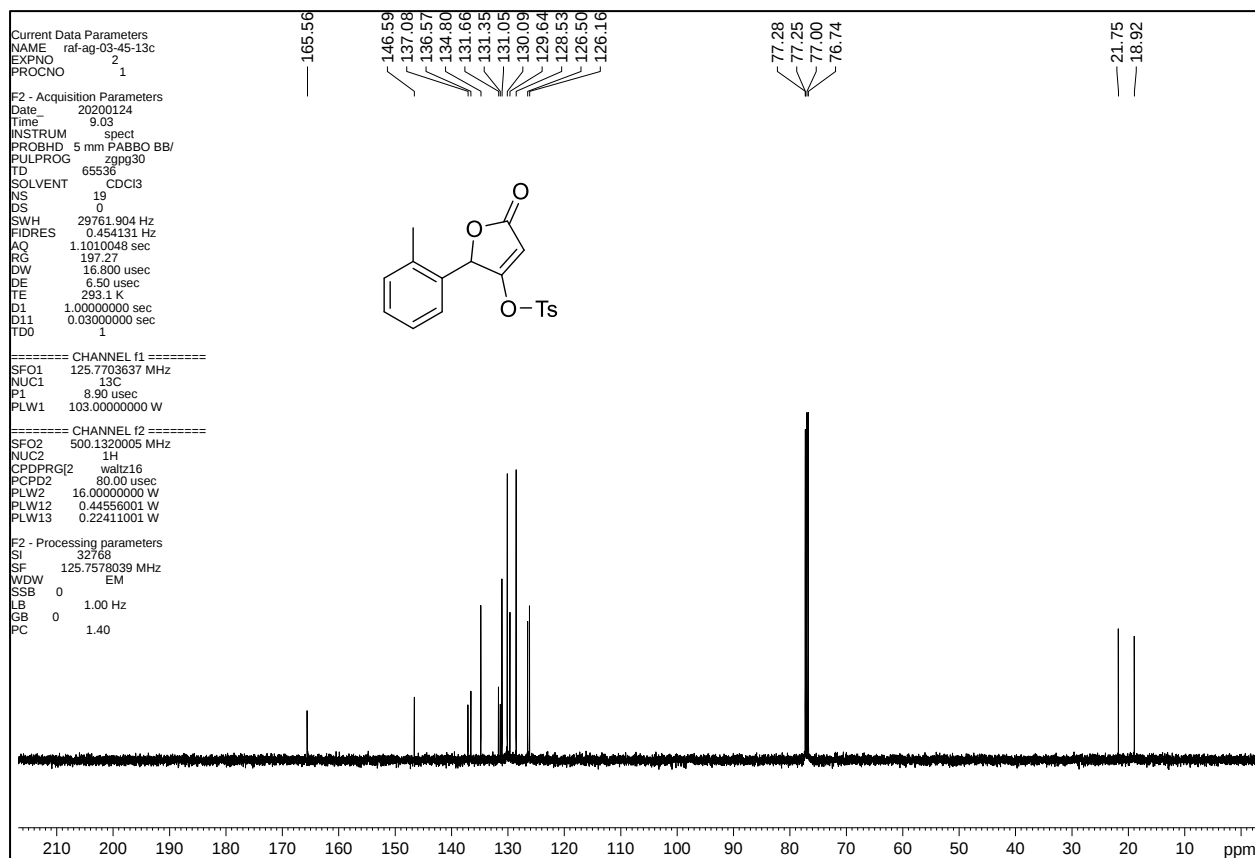
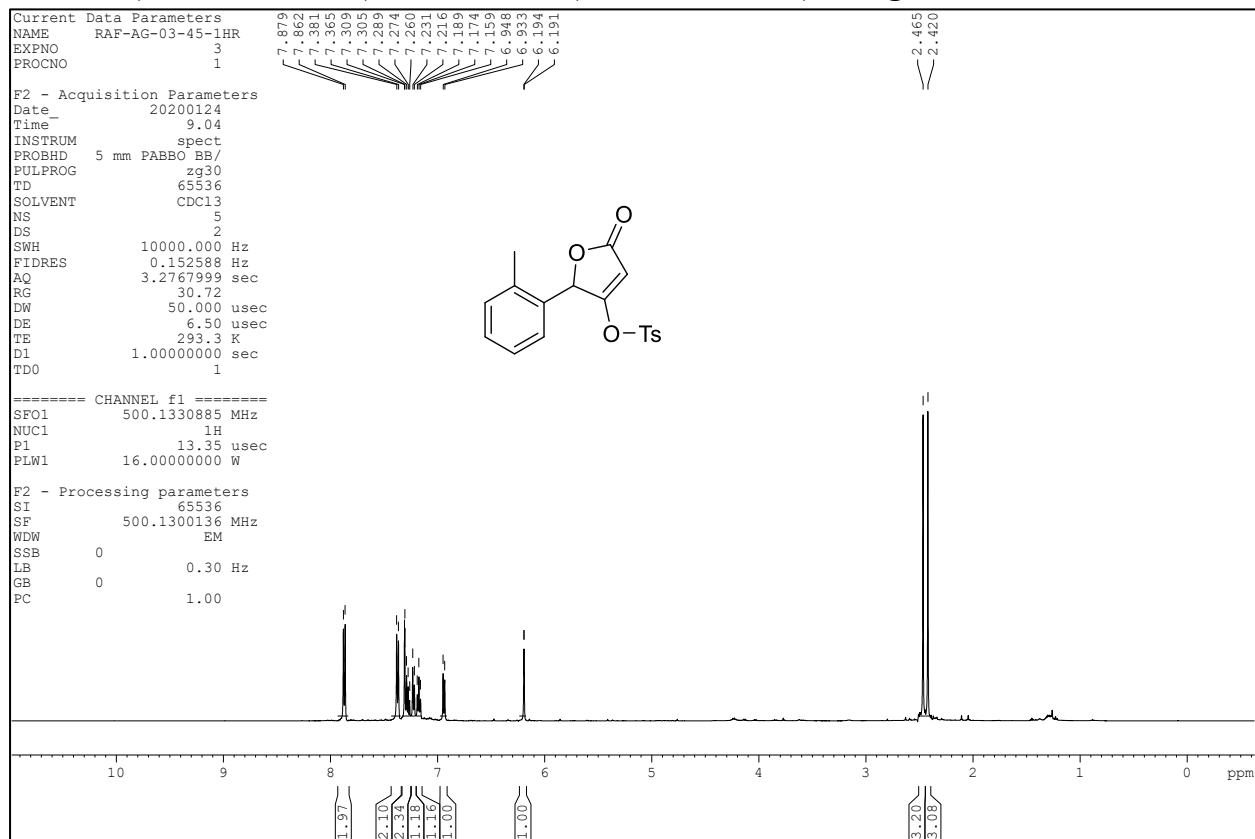
Ph

O-Ts

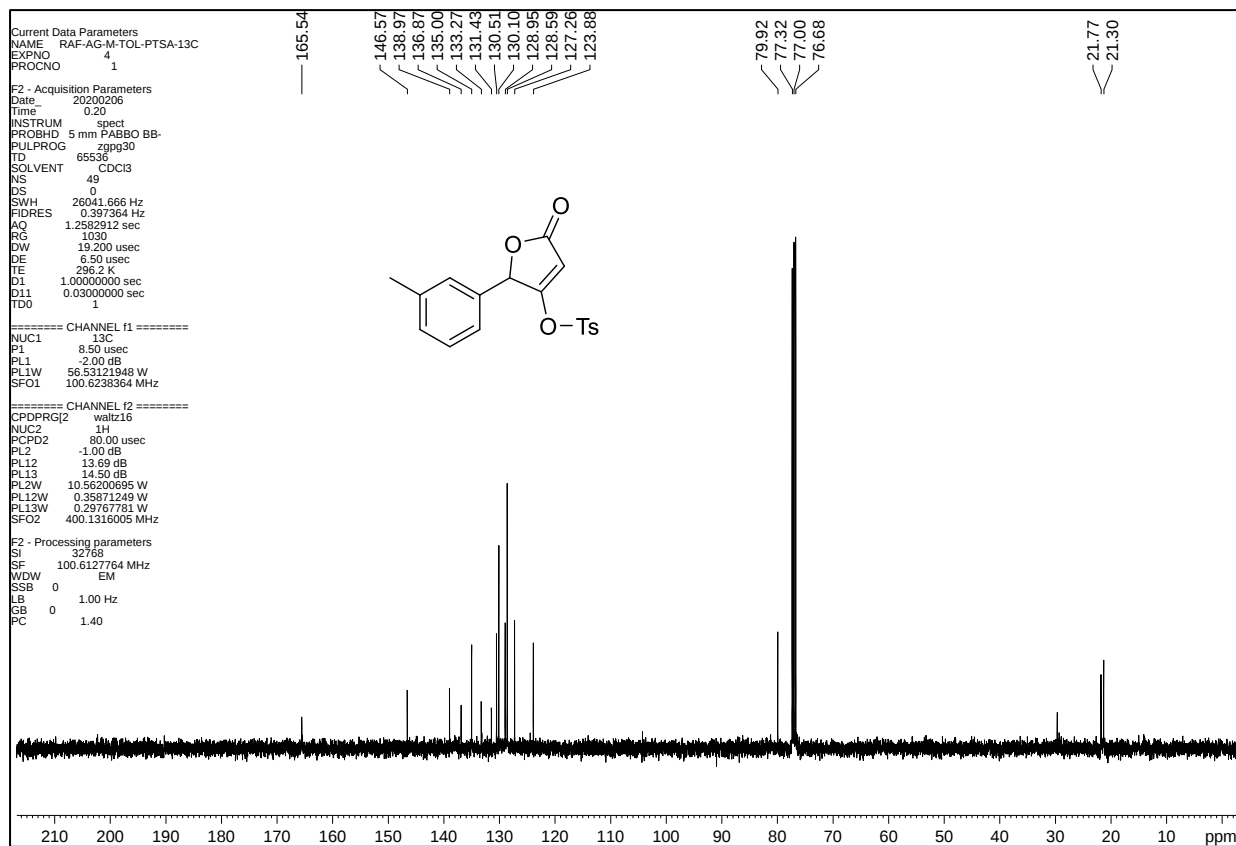
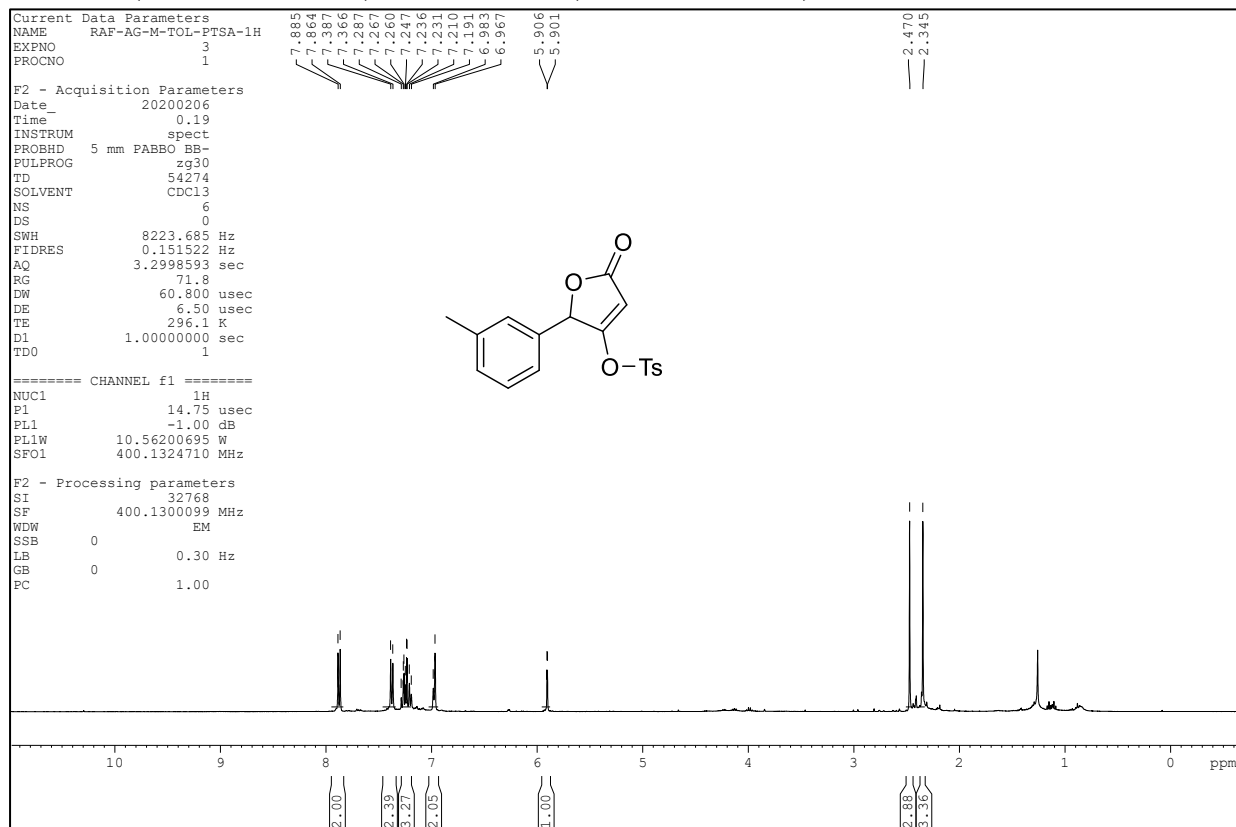
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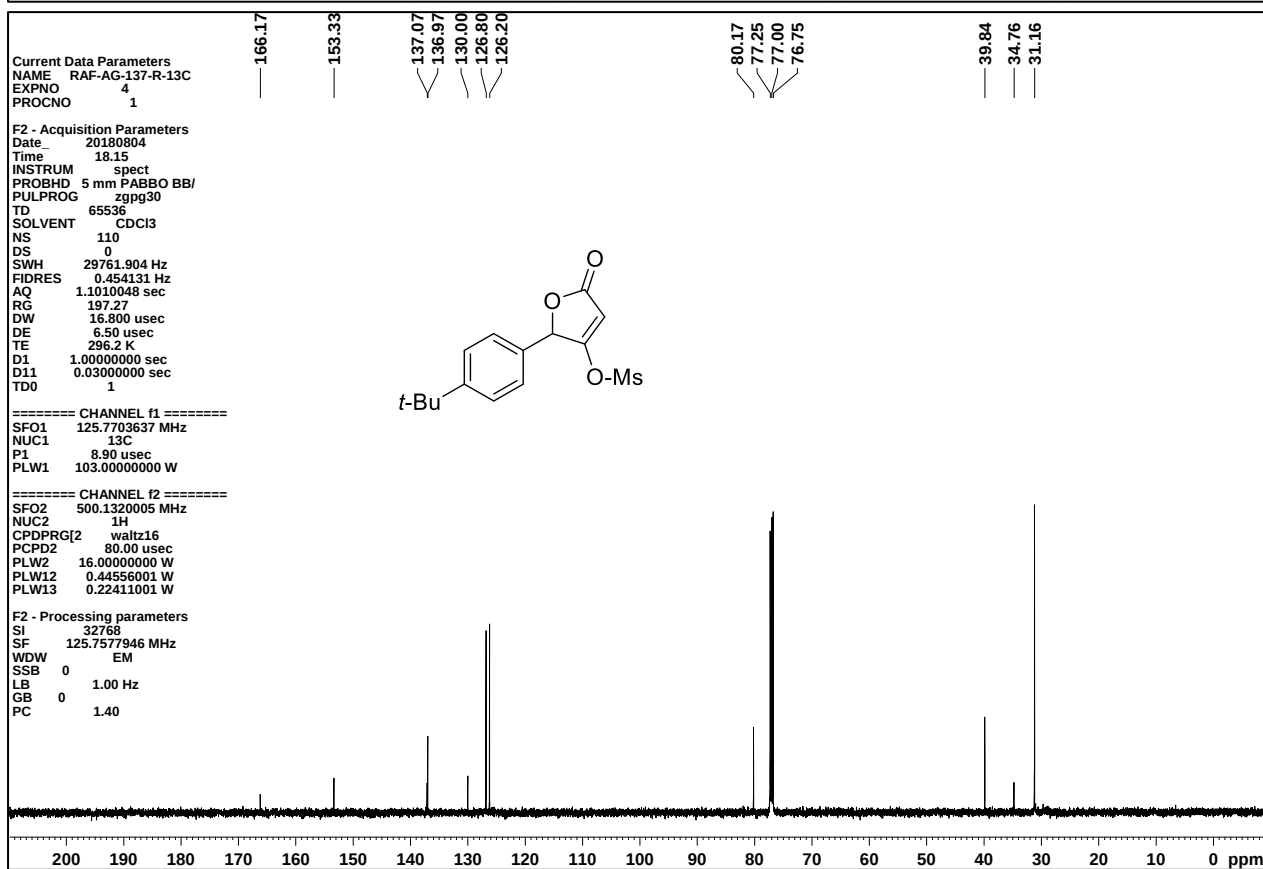
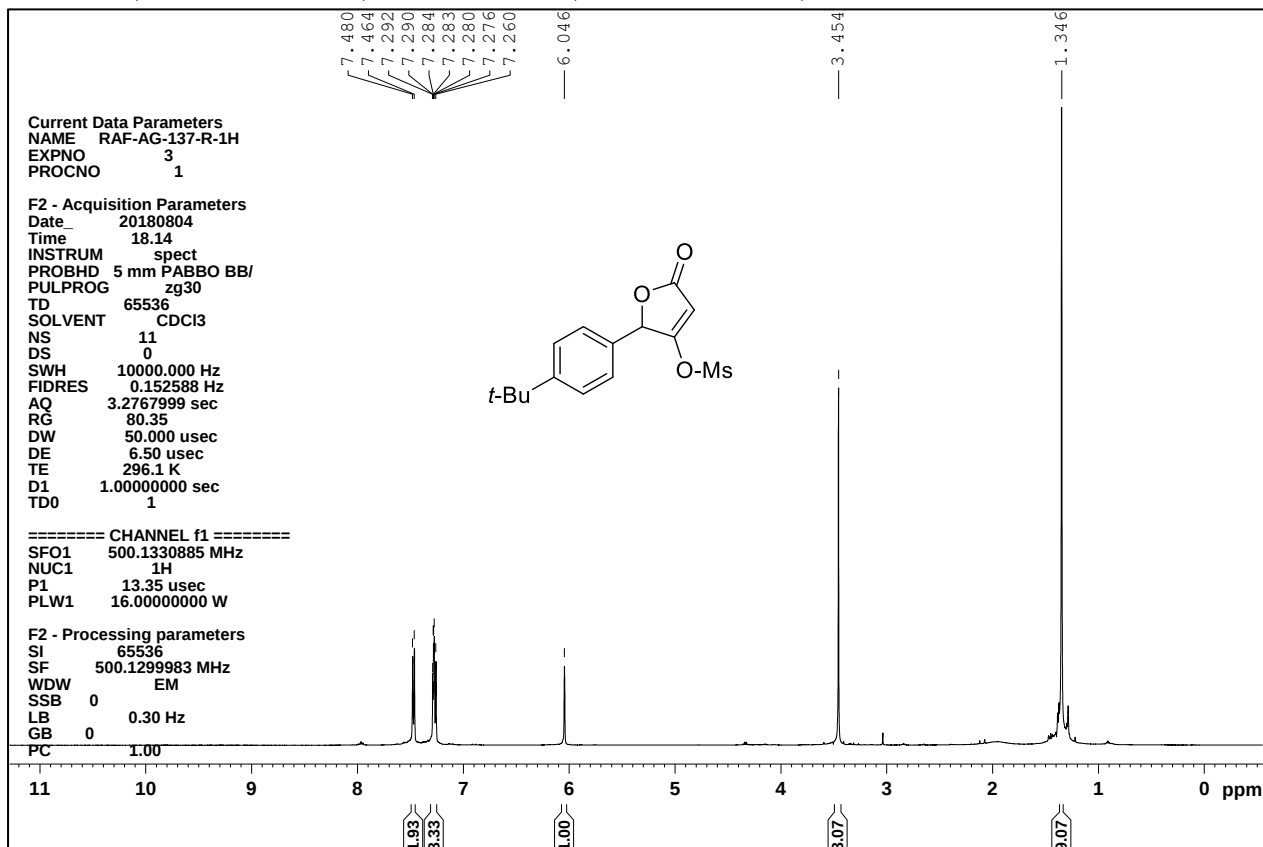
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **13g**



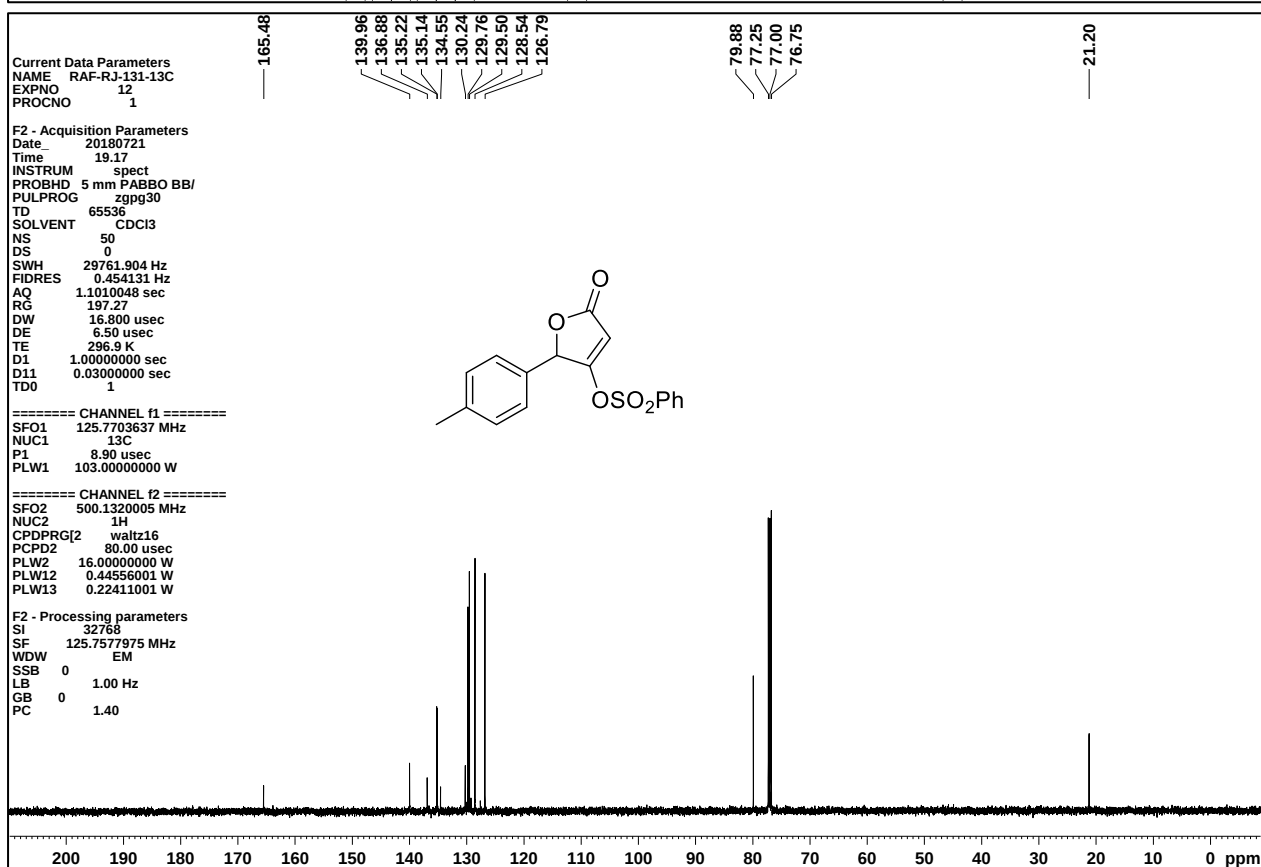
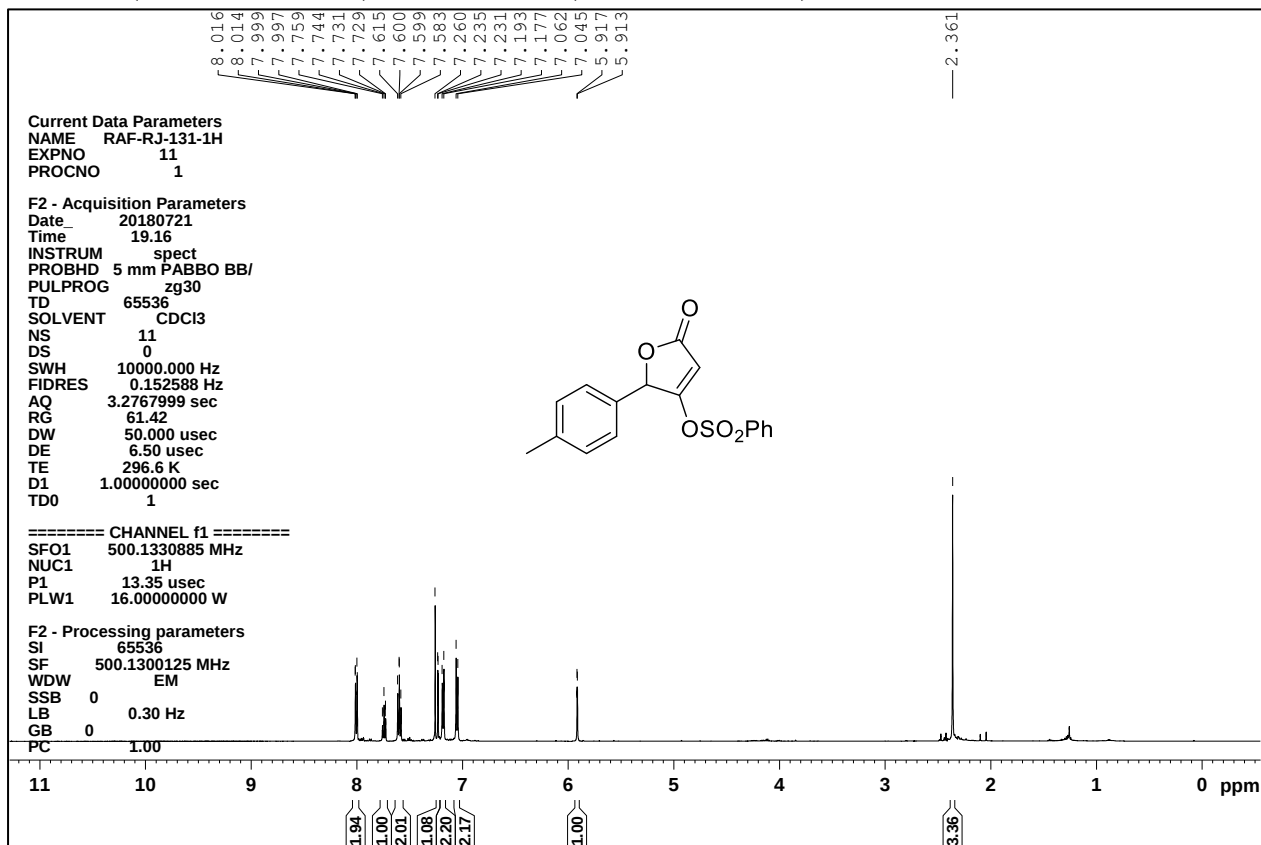
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **13h**



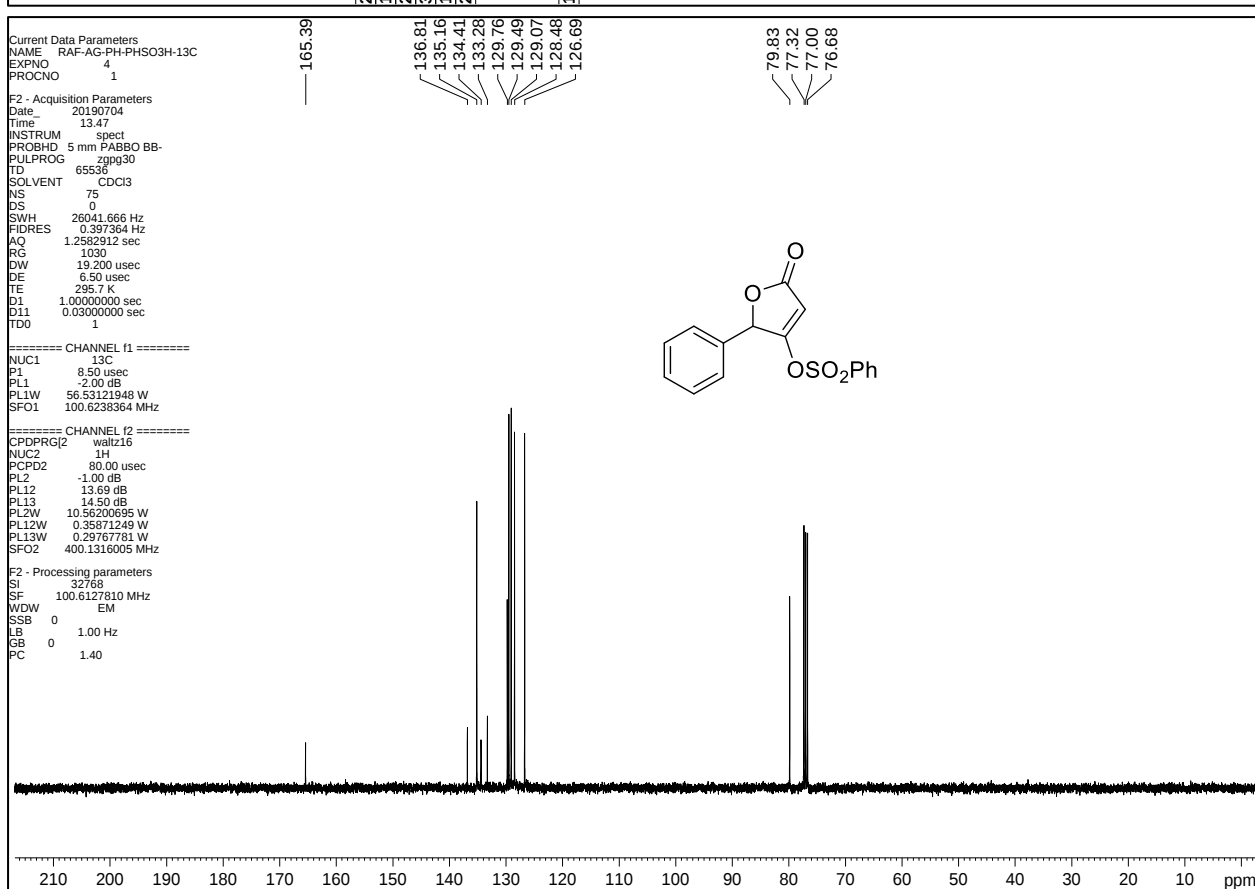
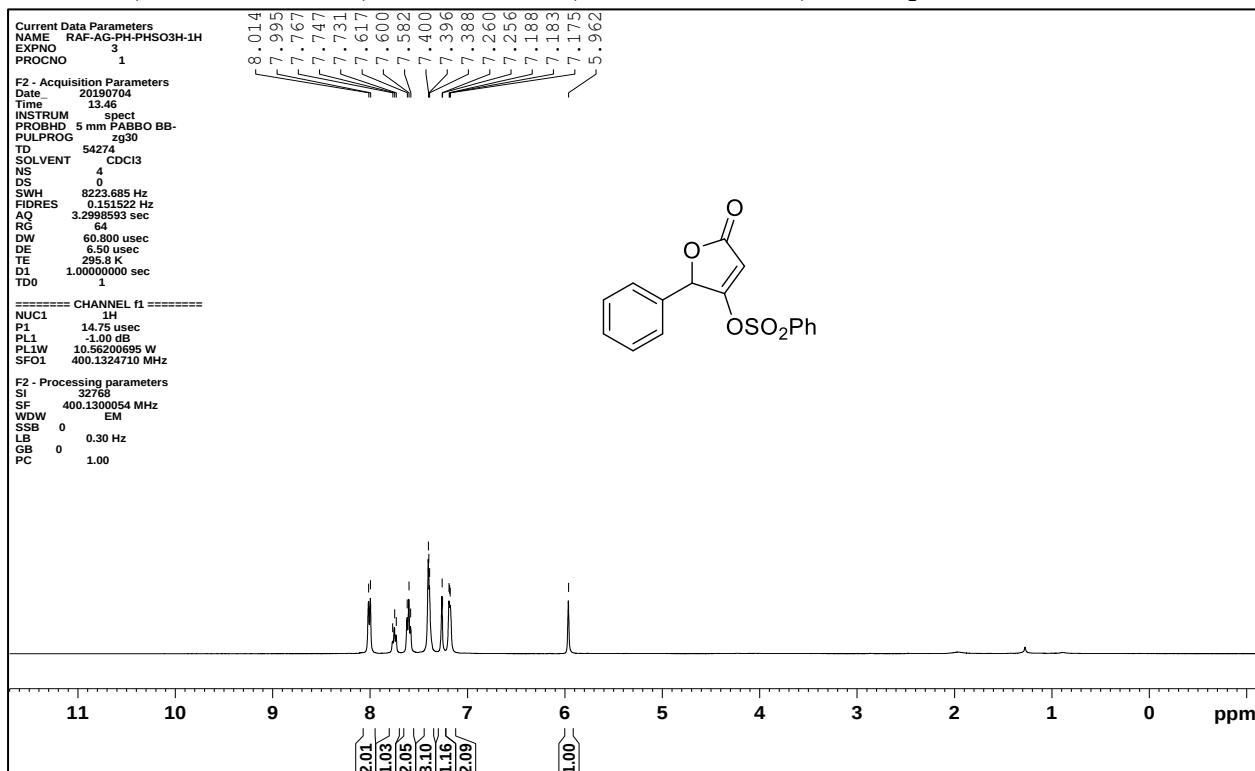
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **14**



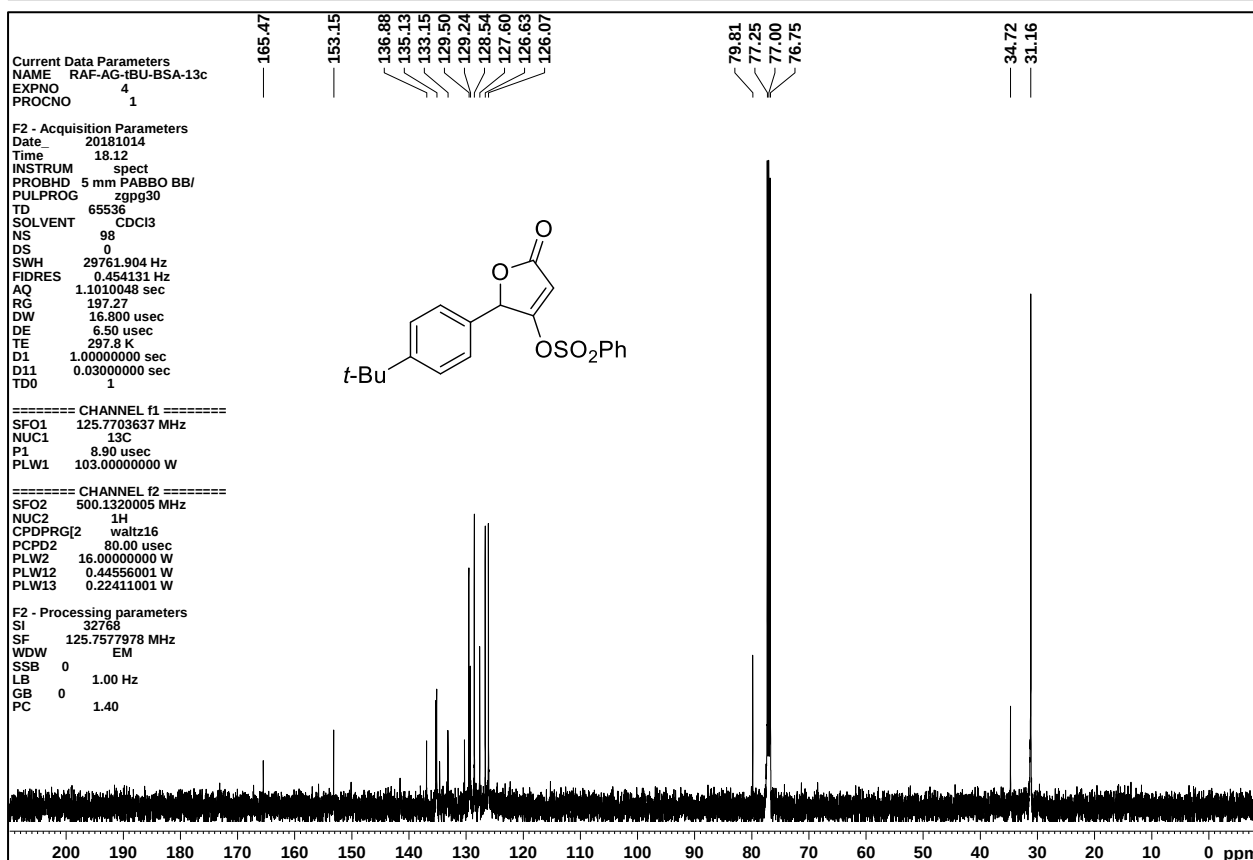
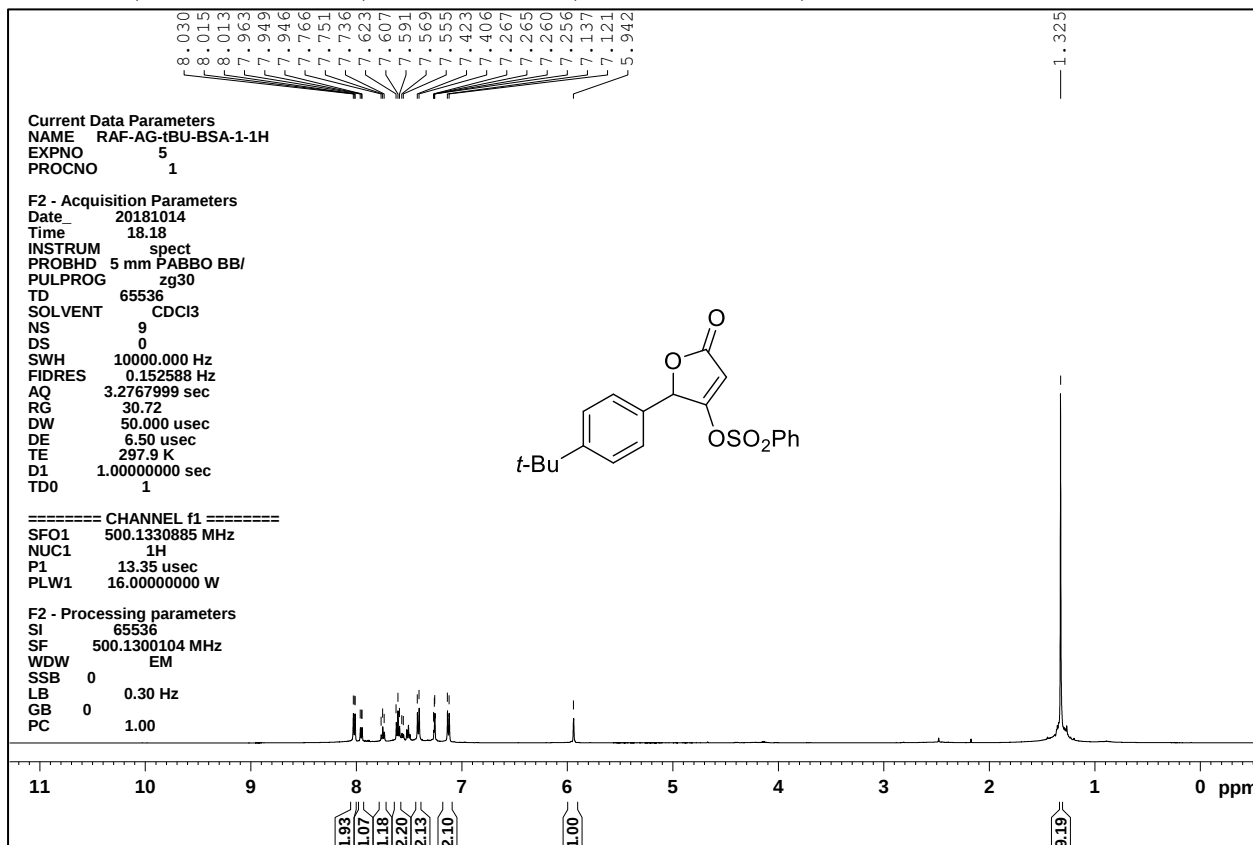
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **15a**



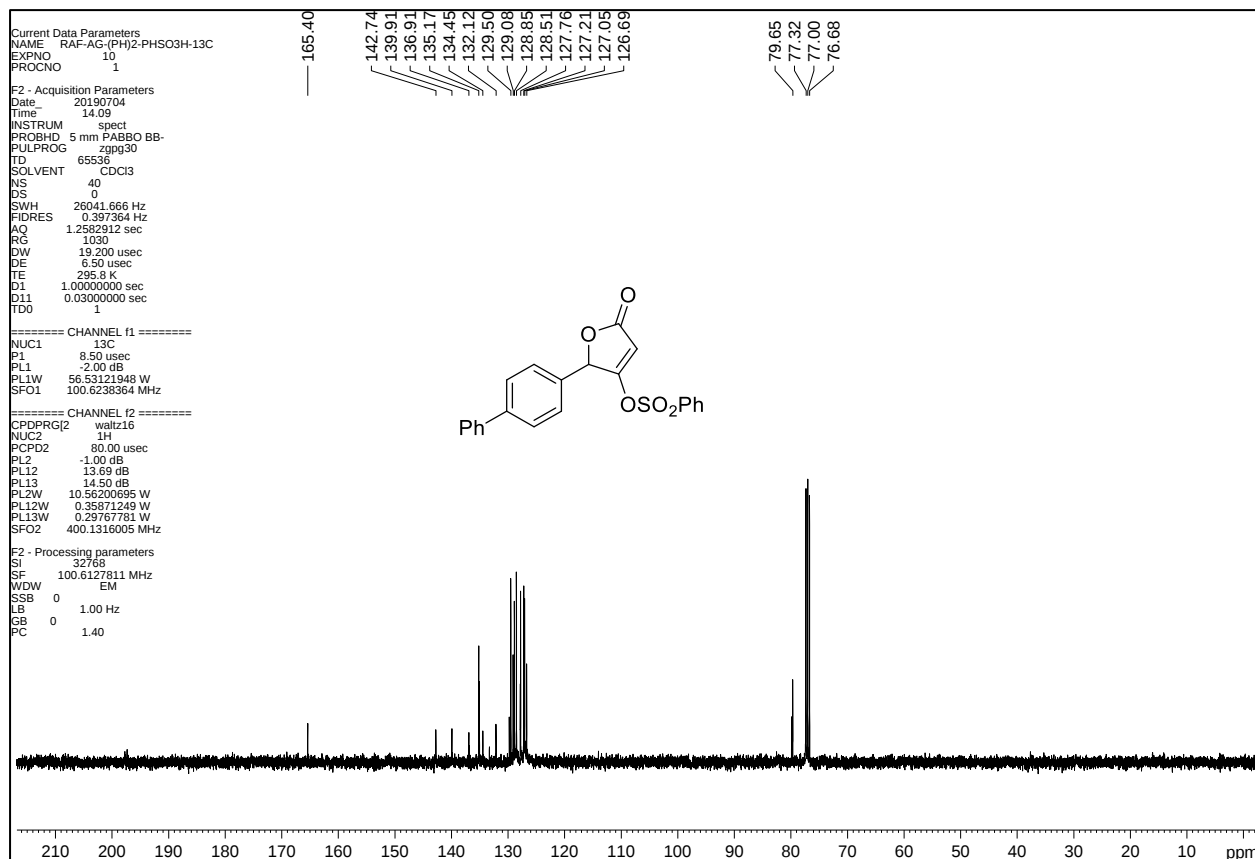
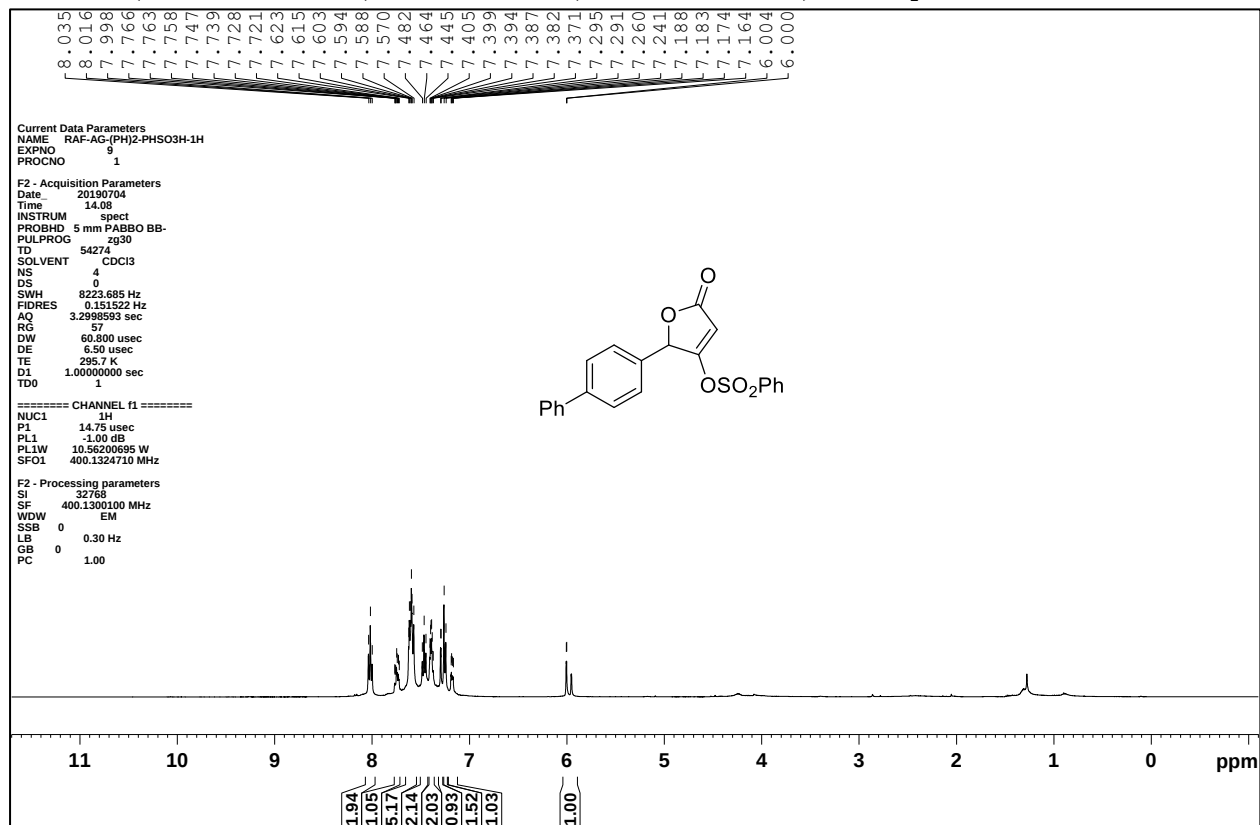
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of compound **15b**



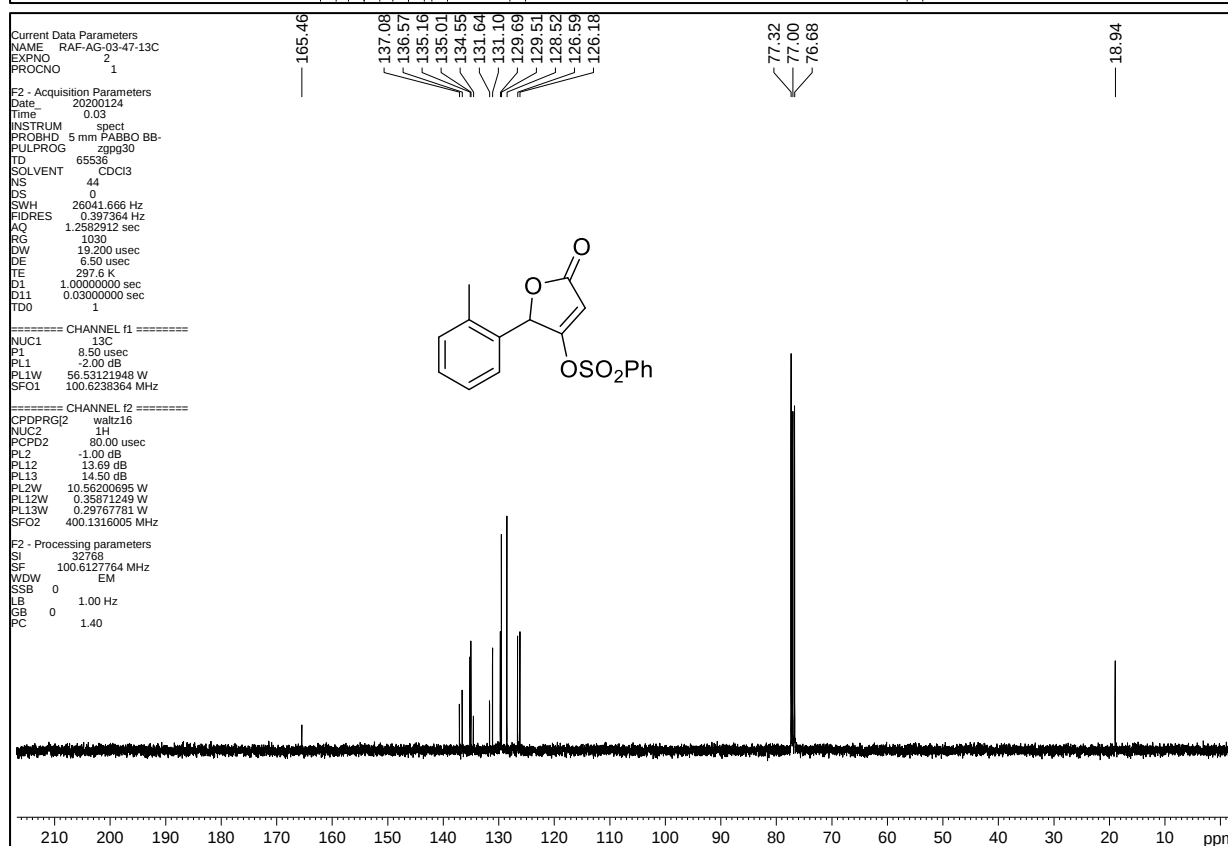
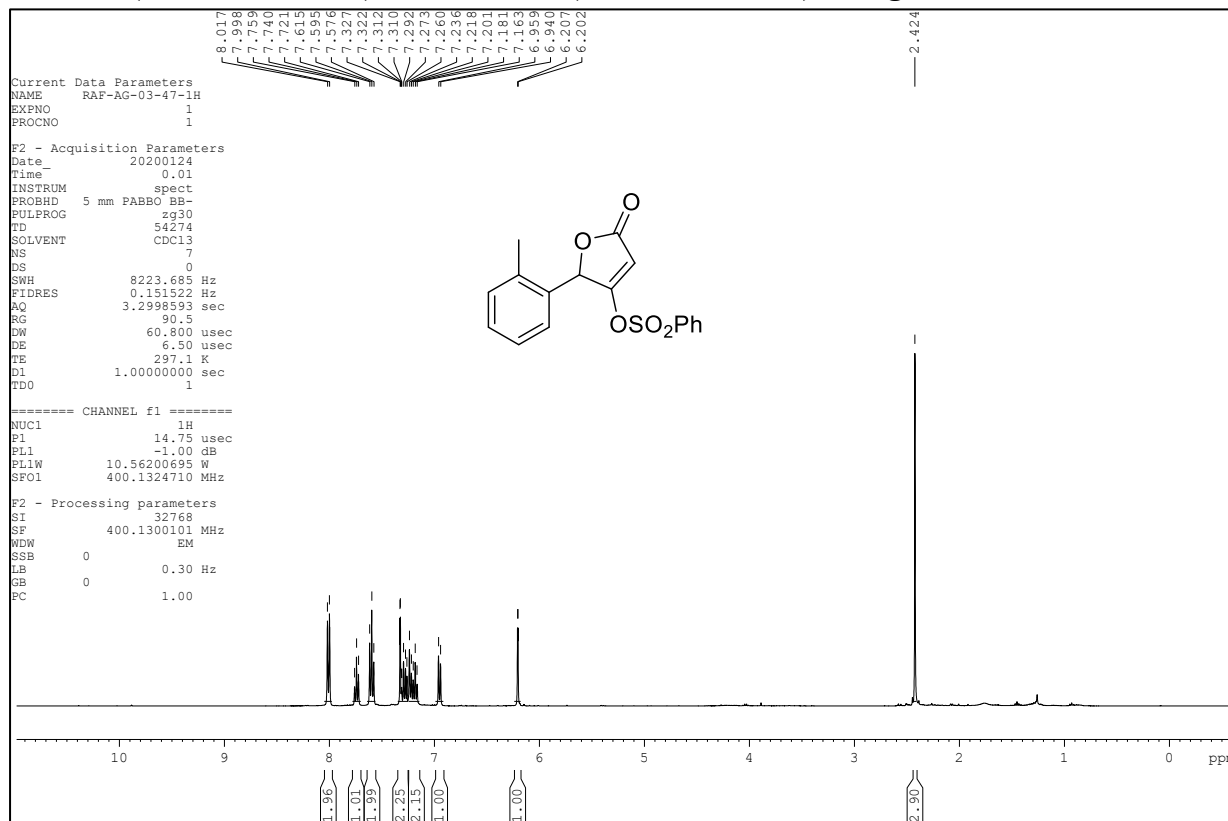
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **15c**



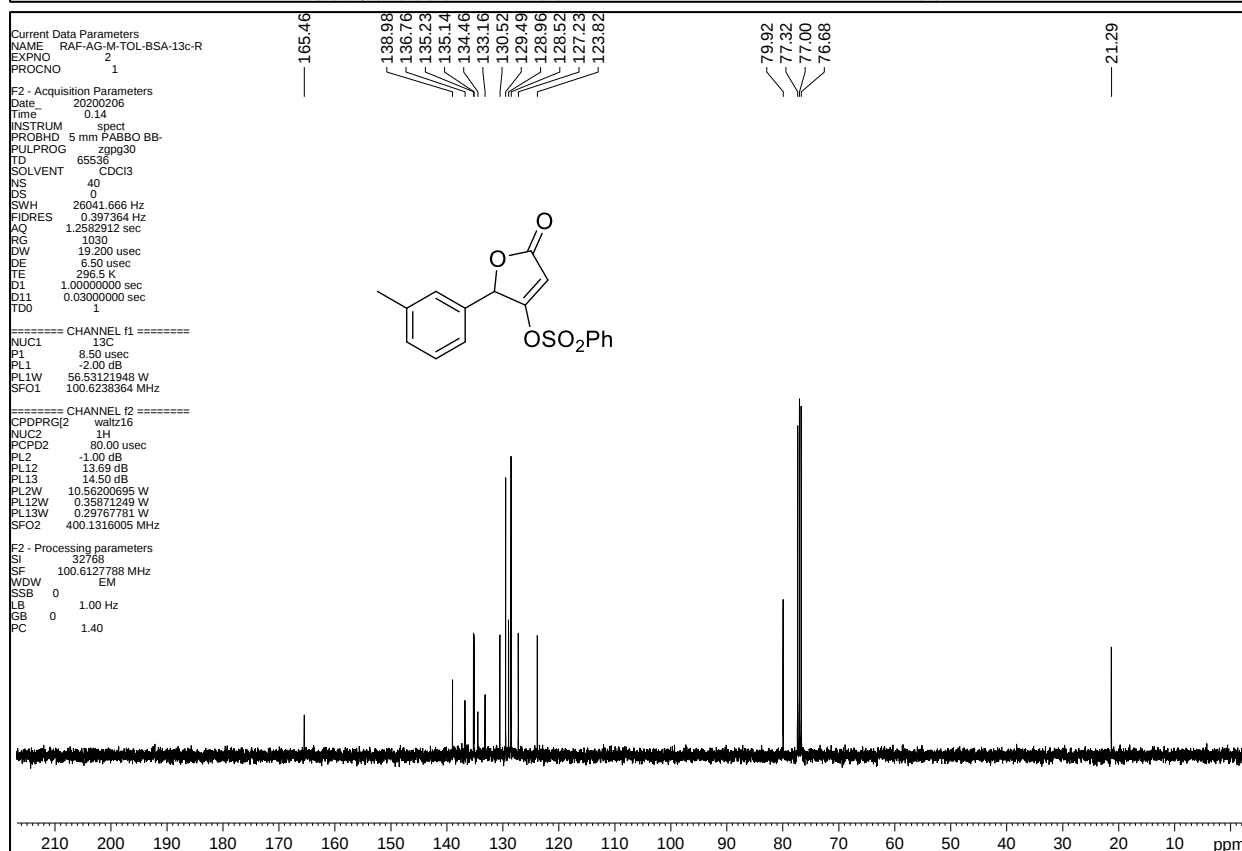
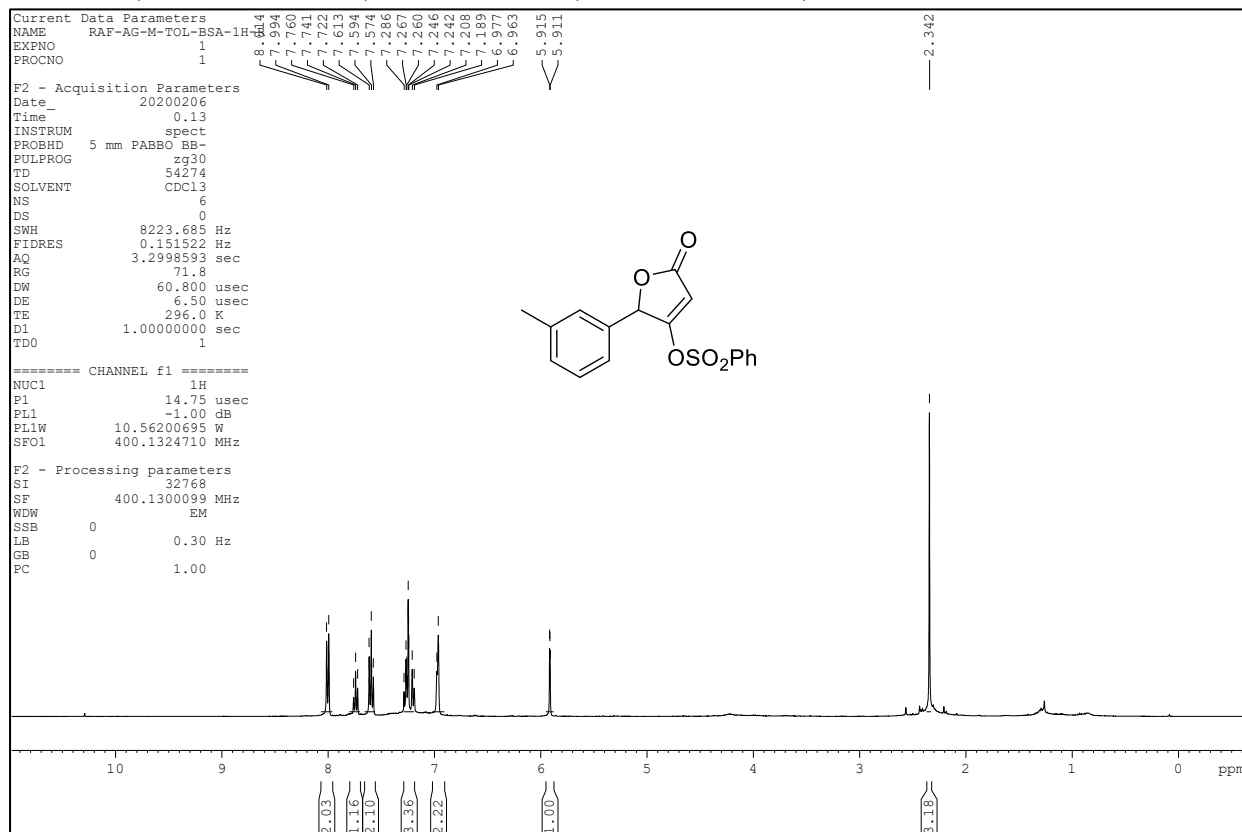
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound **15d**



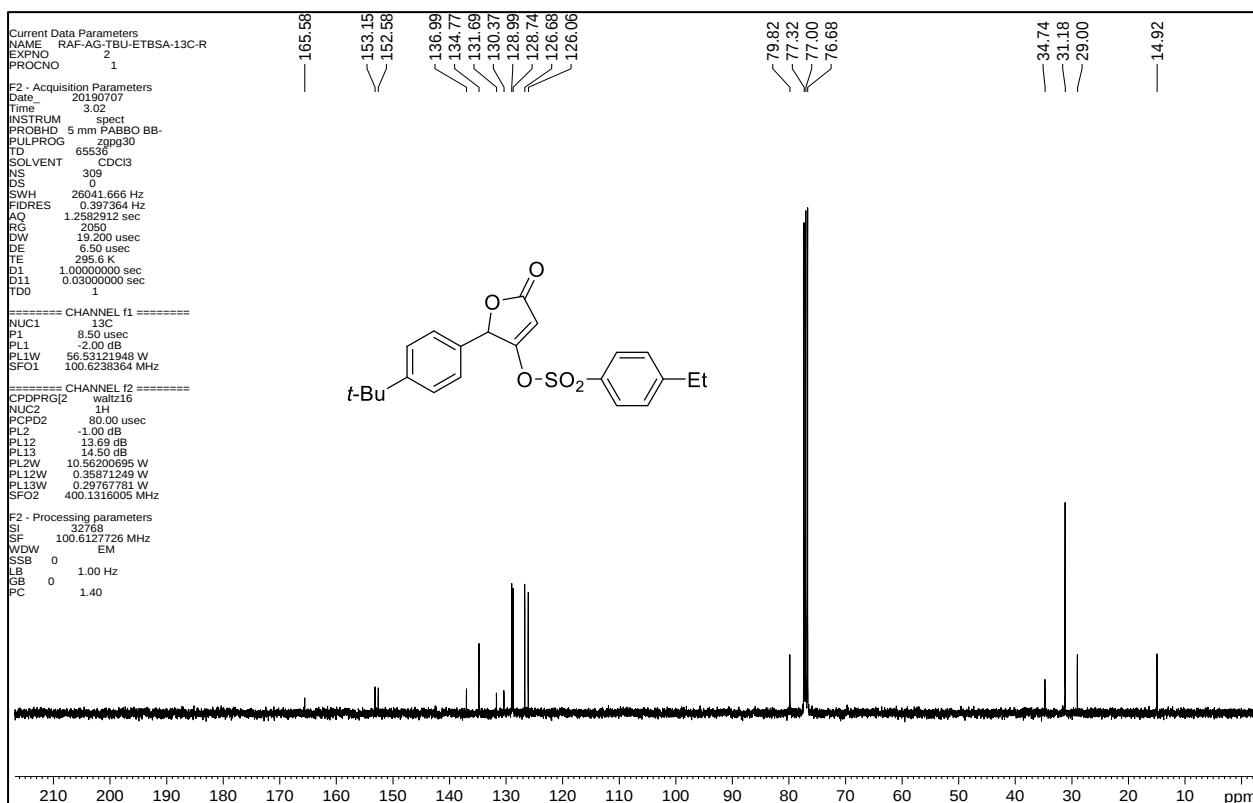
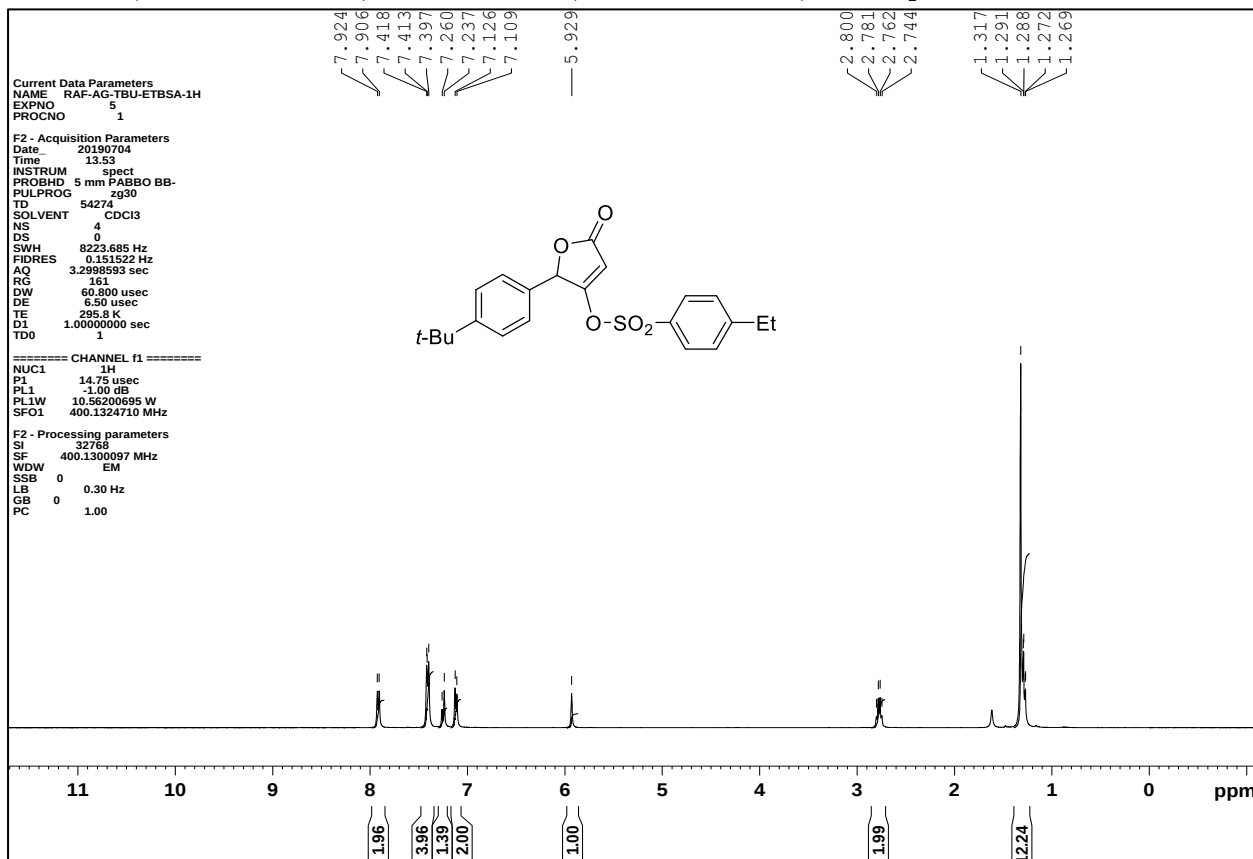
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **15g**



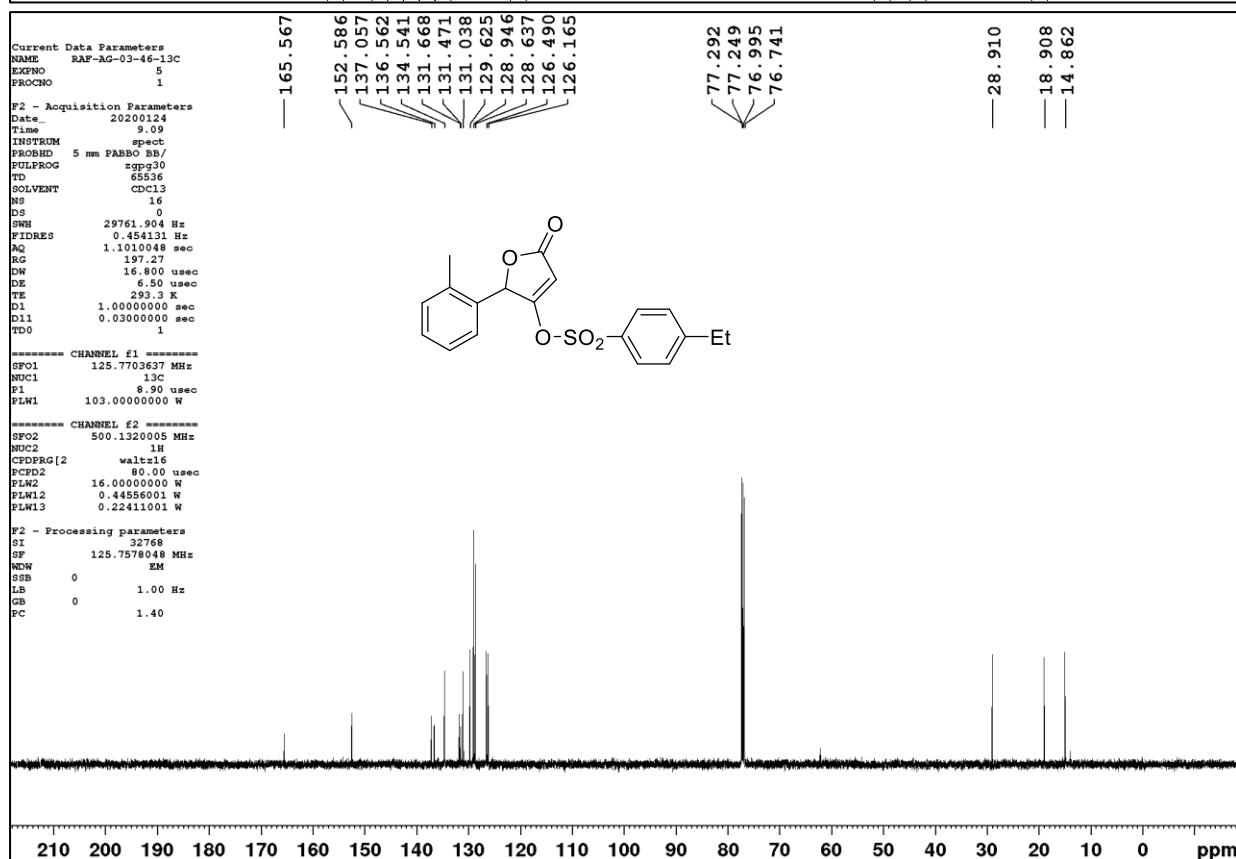
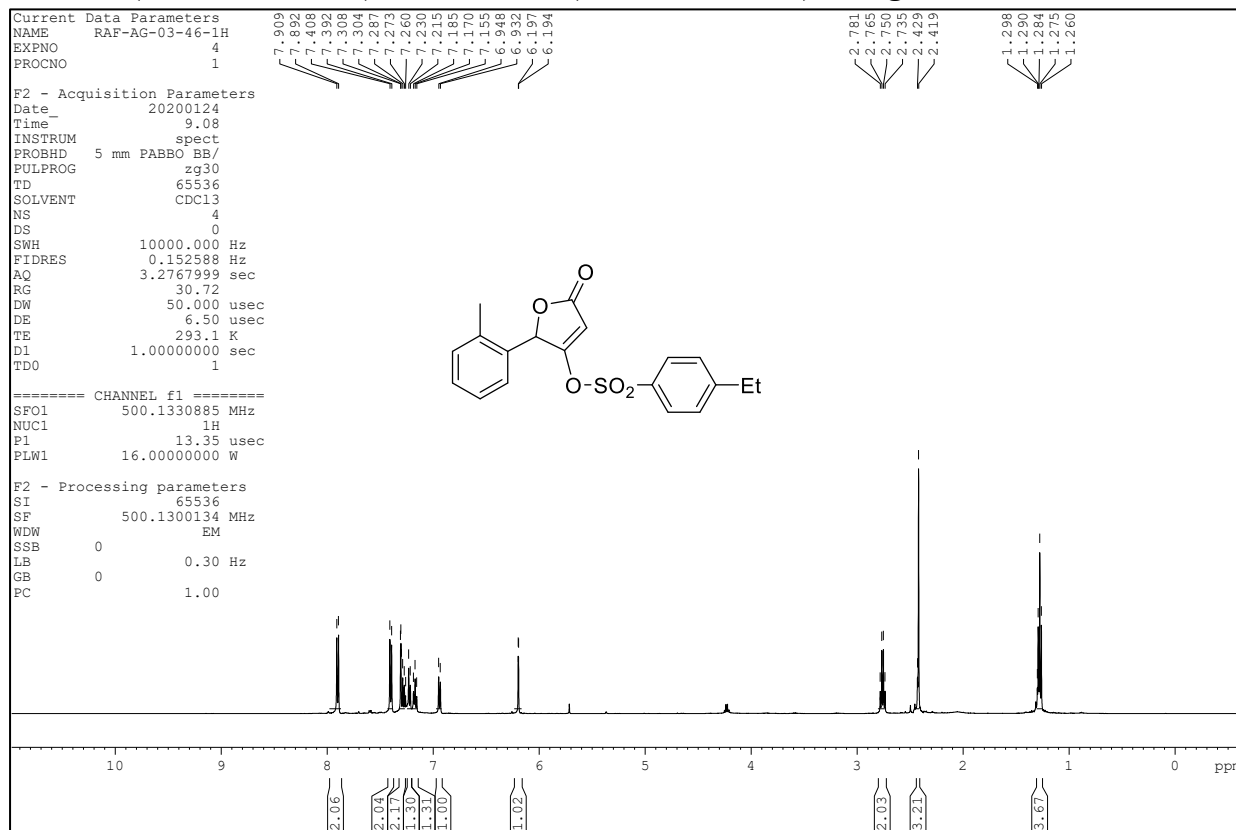
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **15h**



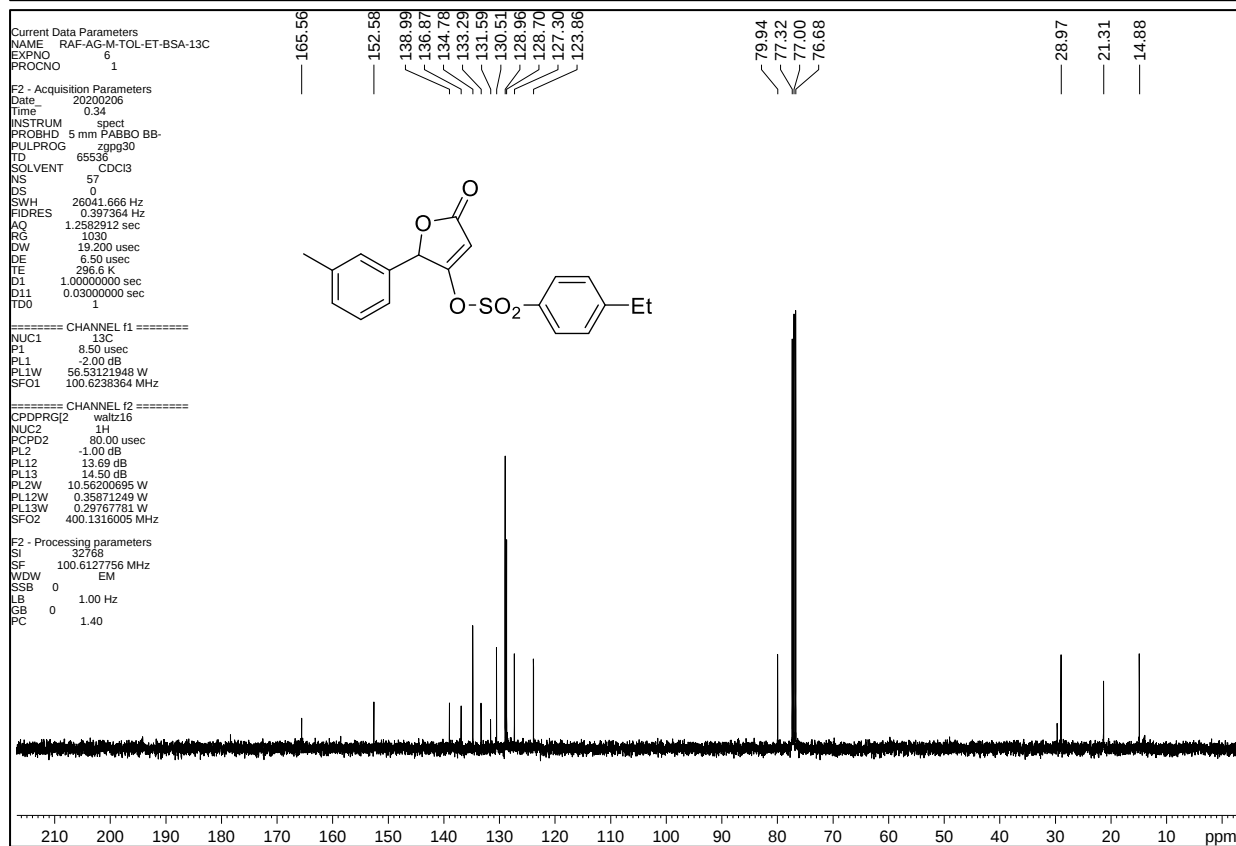
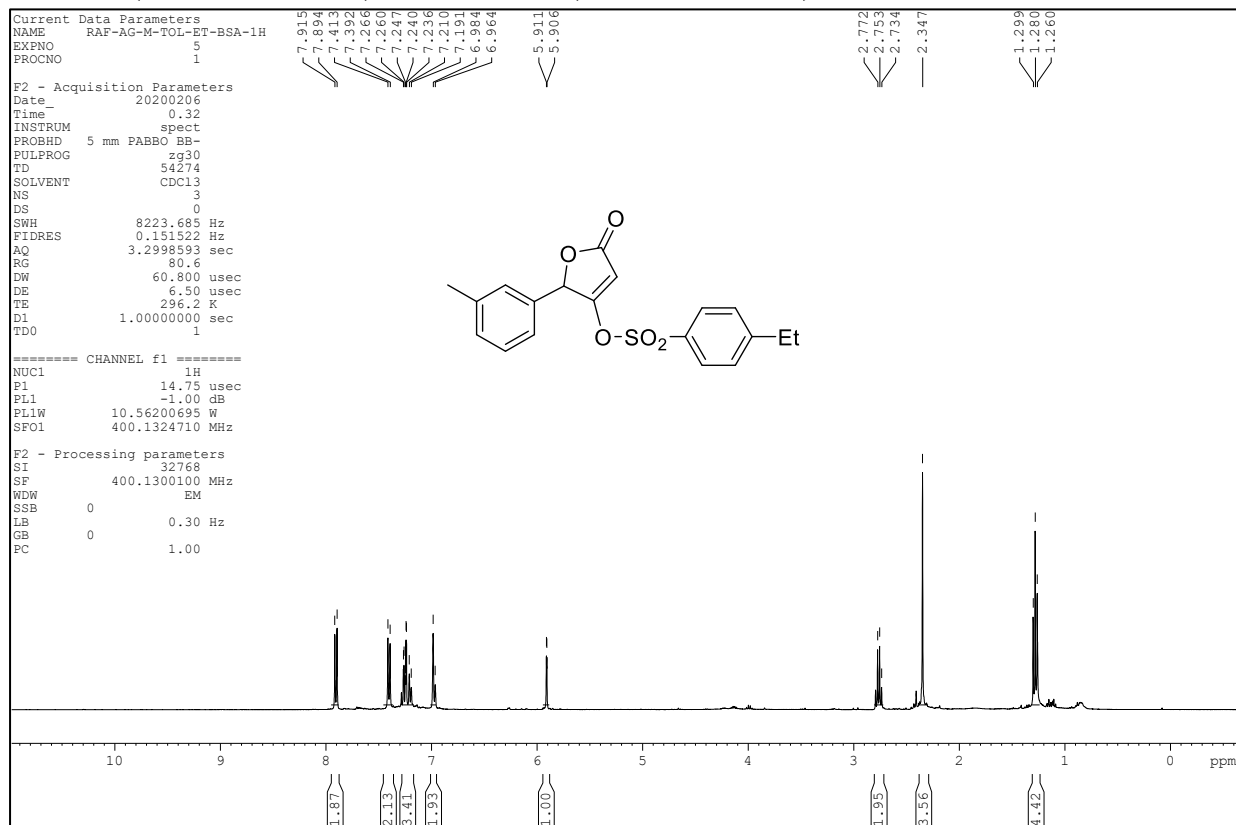
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound **16c**



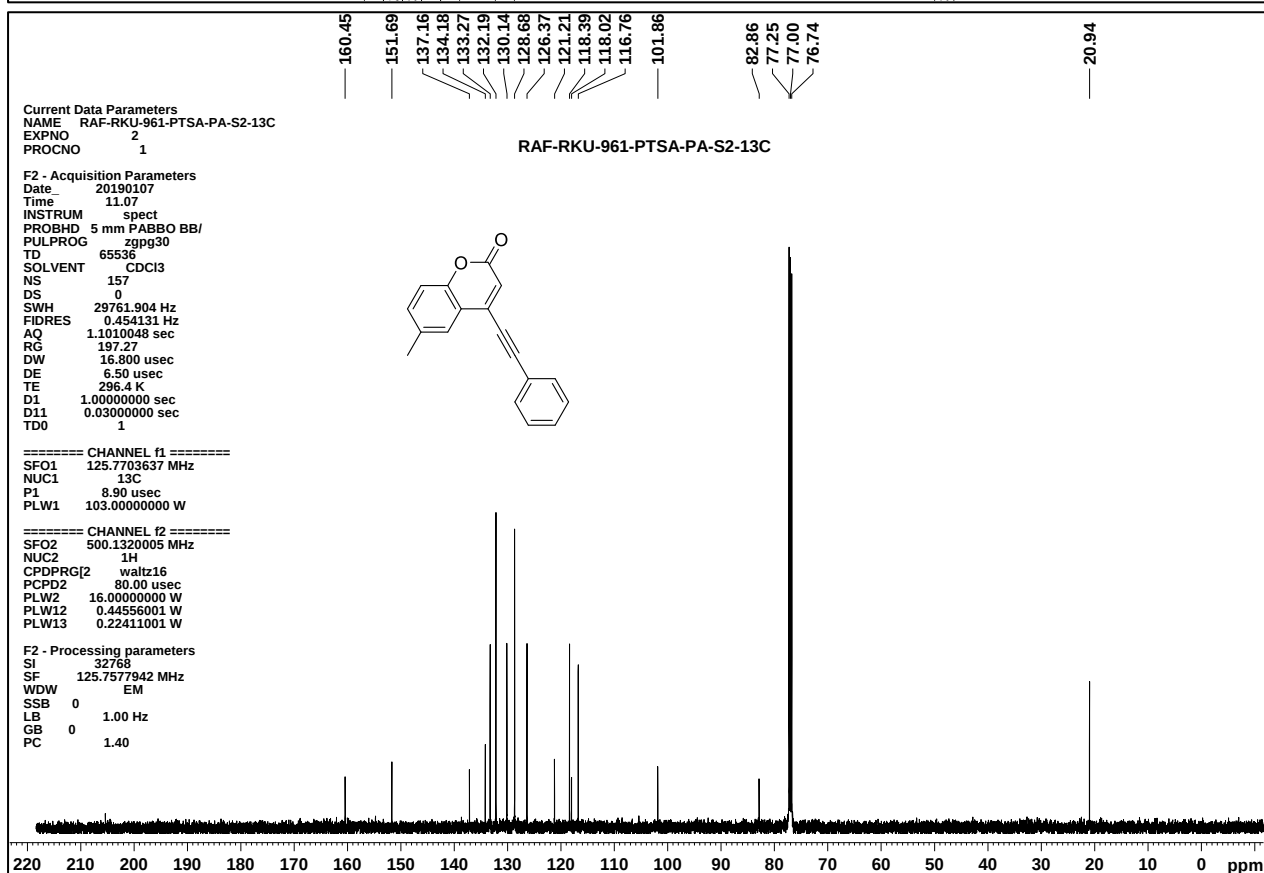
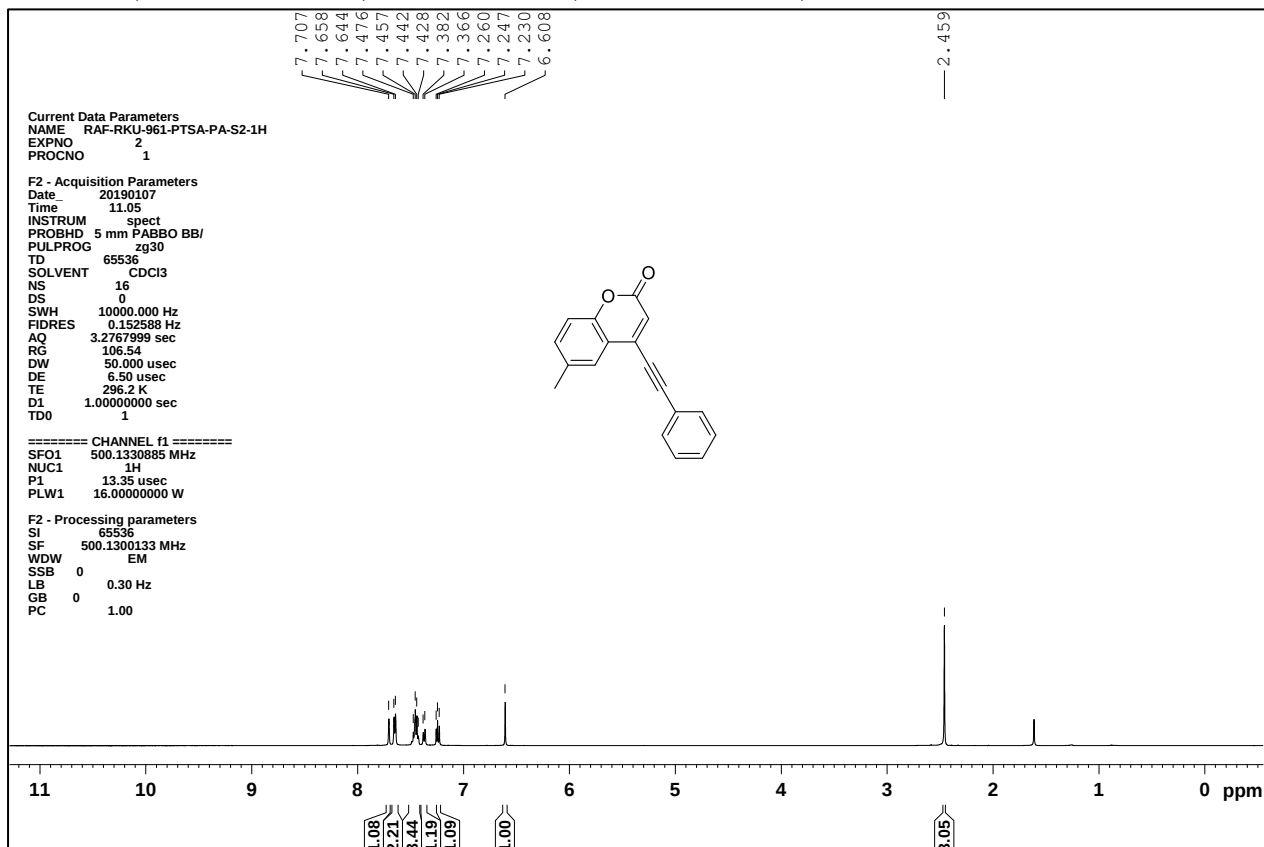
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **16g**



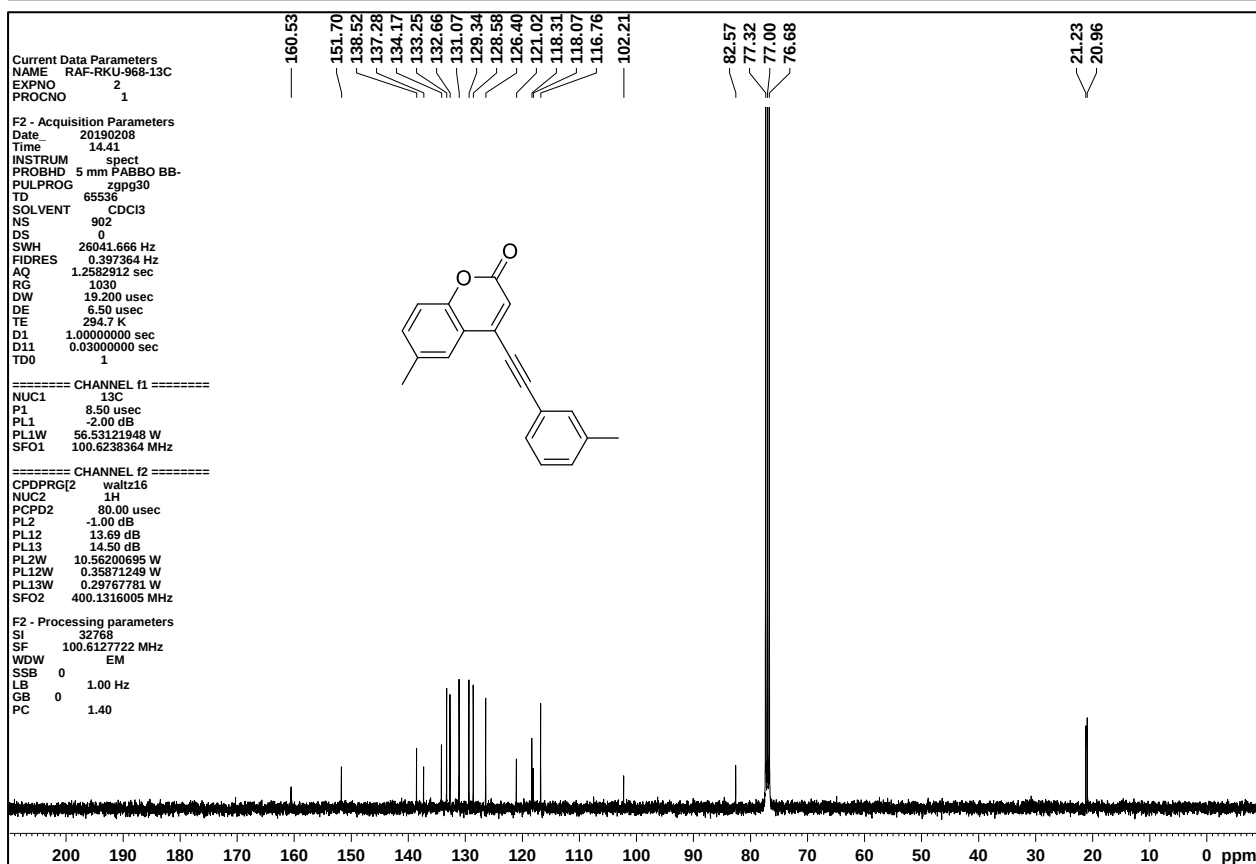
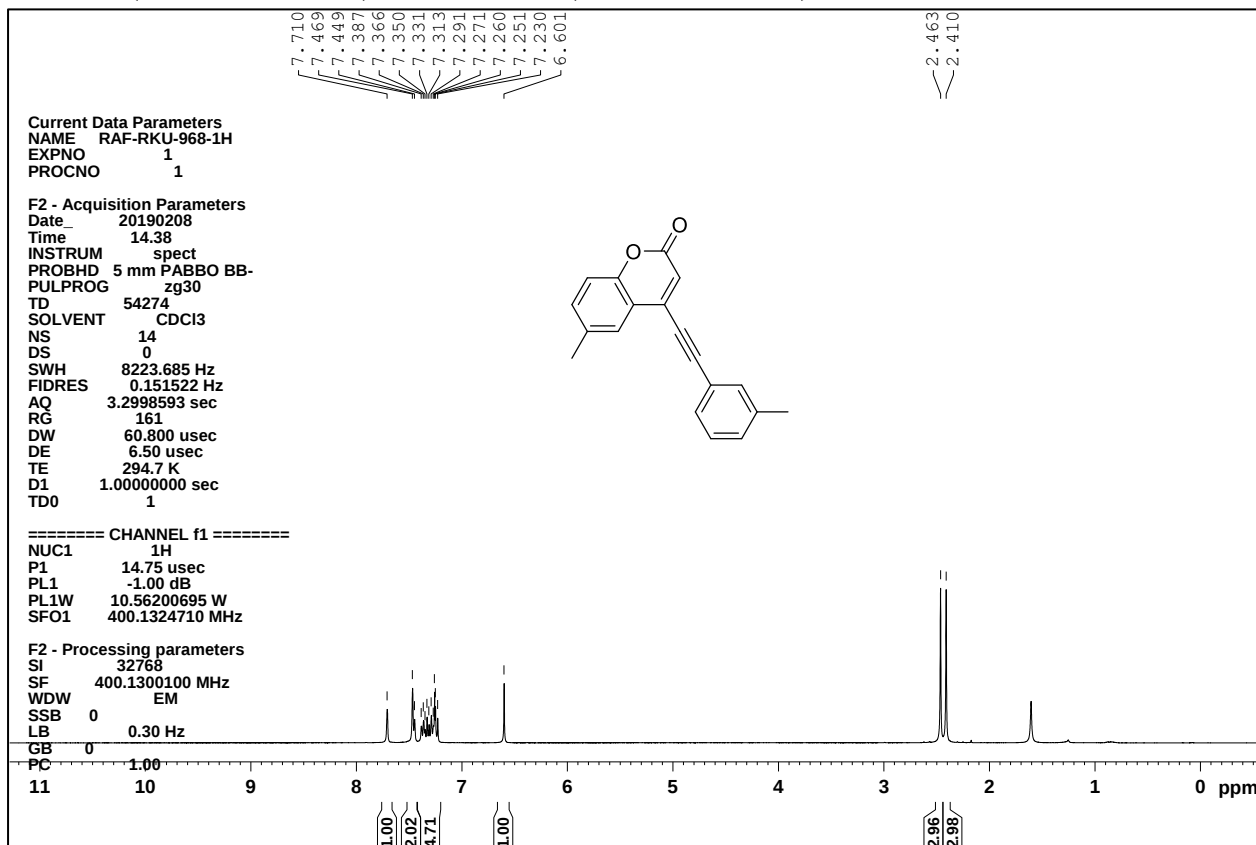
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **16h**



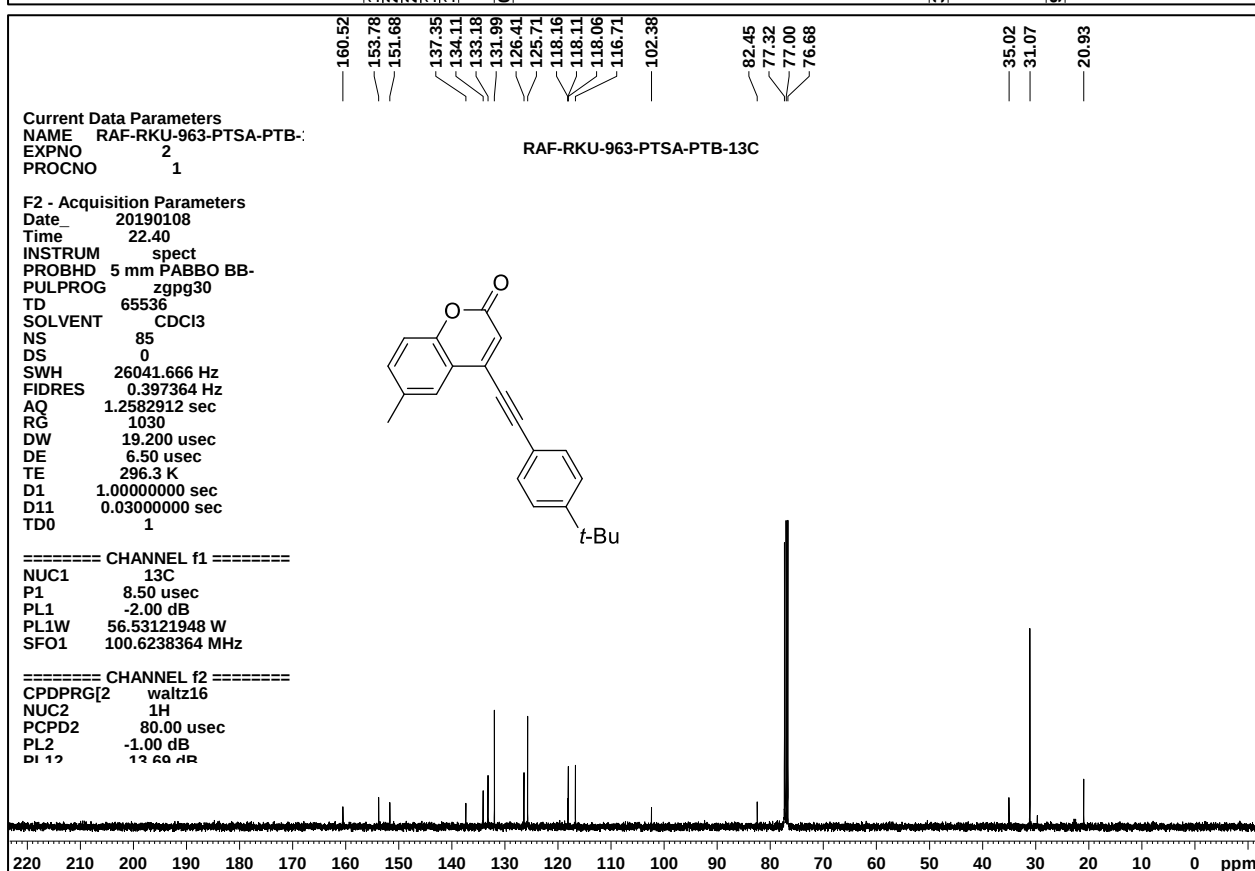
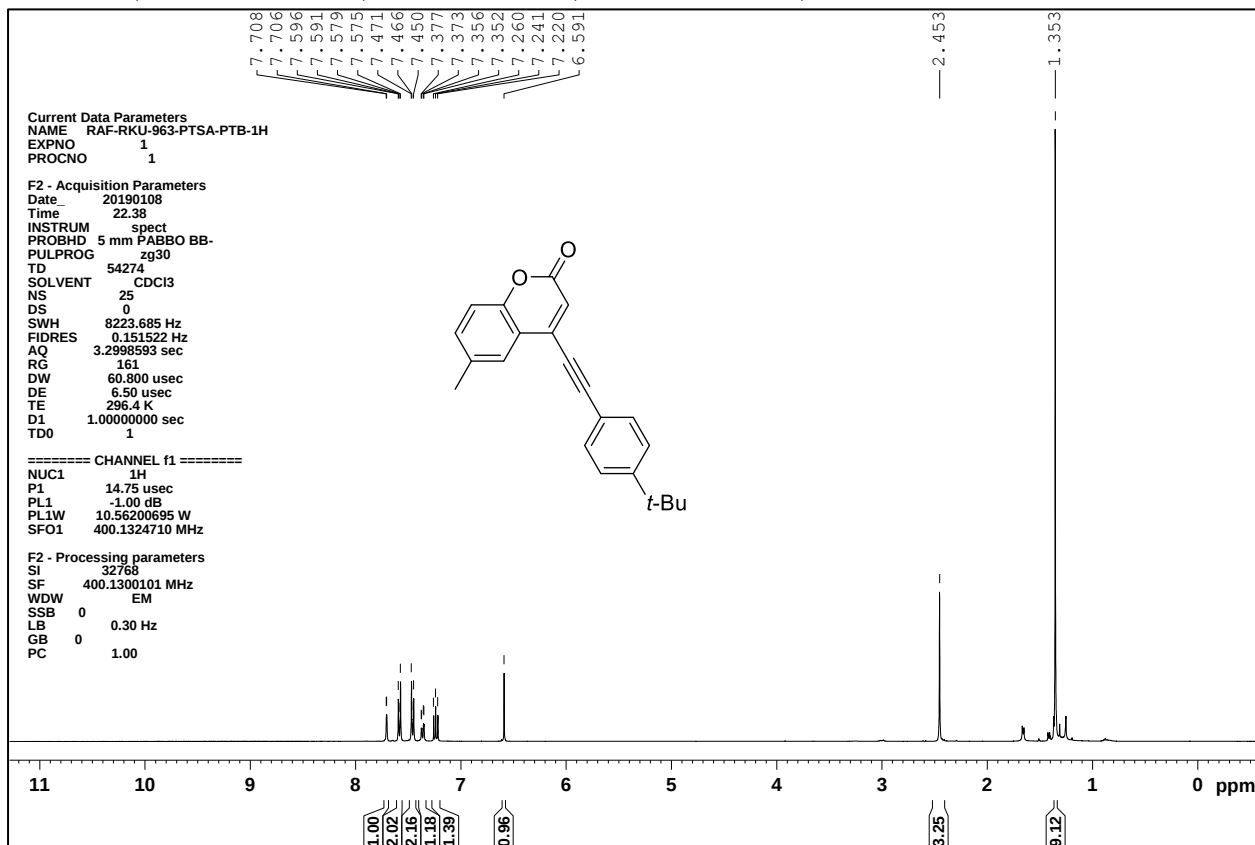
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **18a**



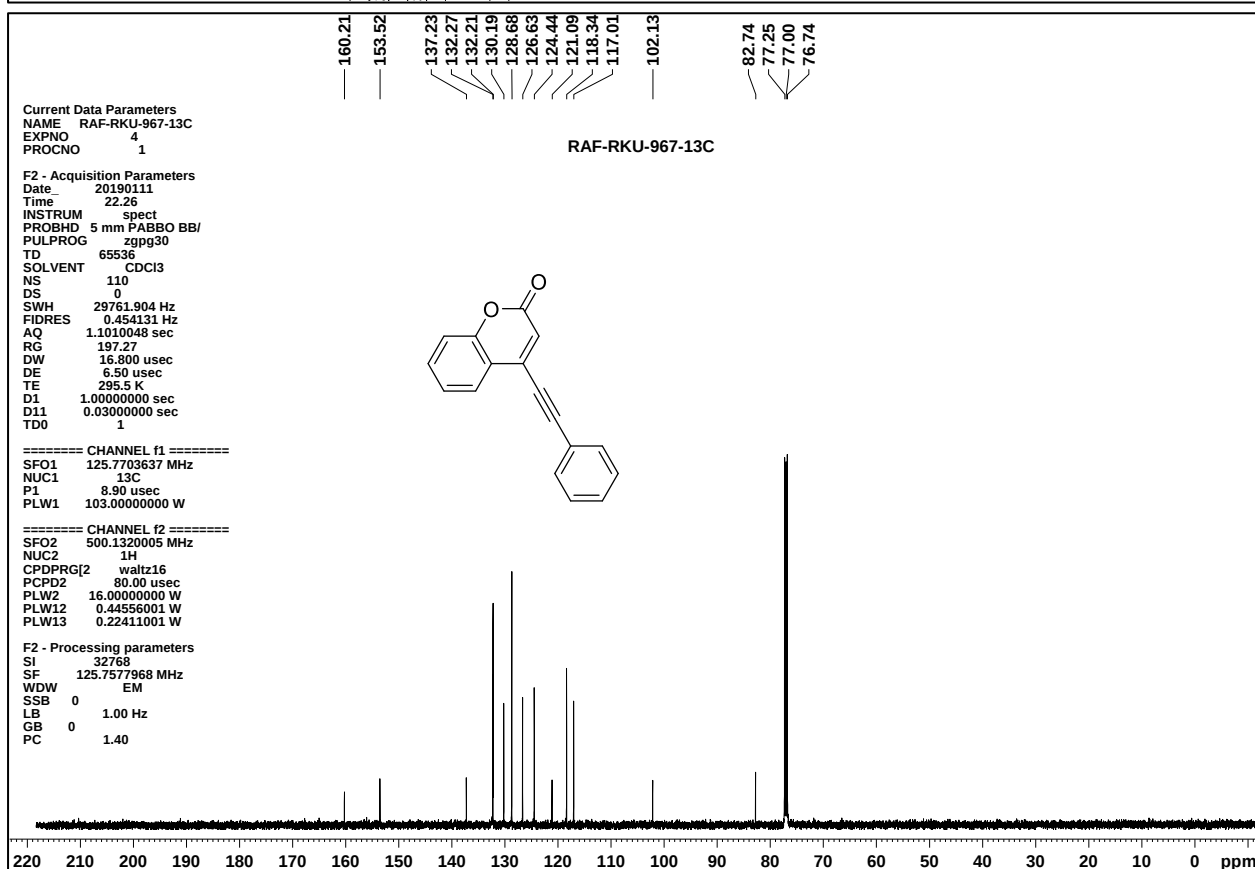
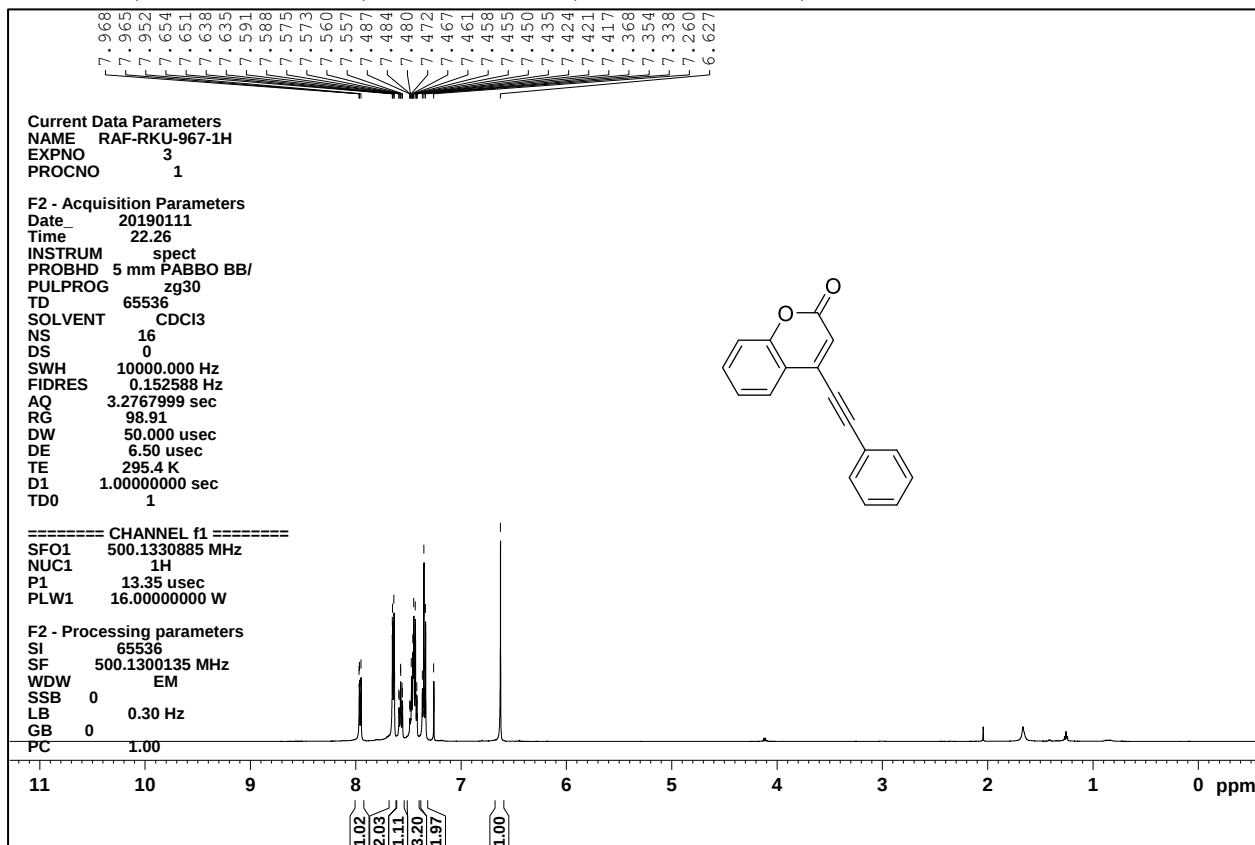
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **18b**



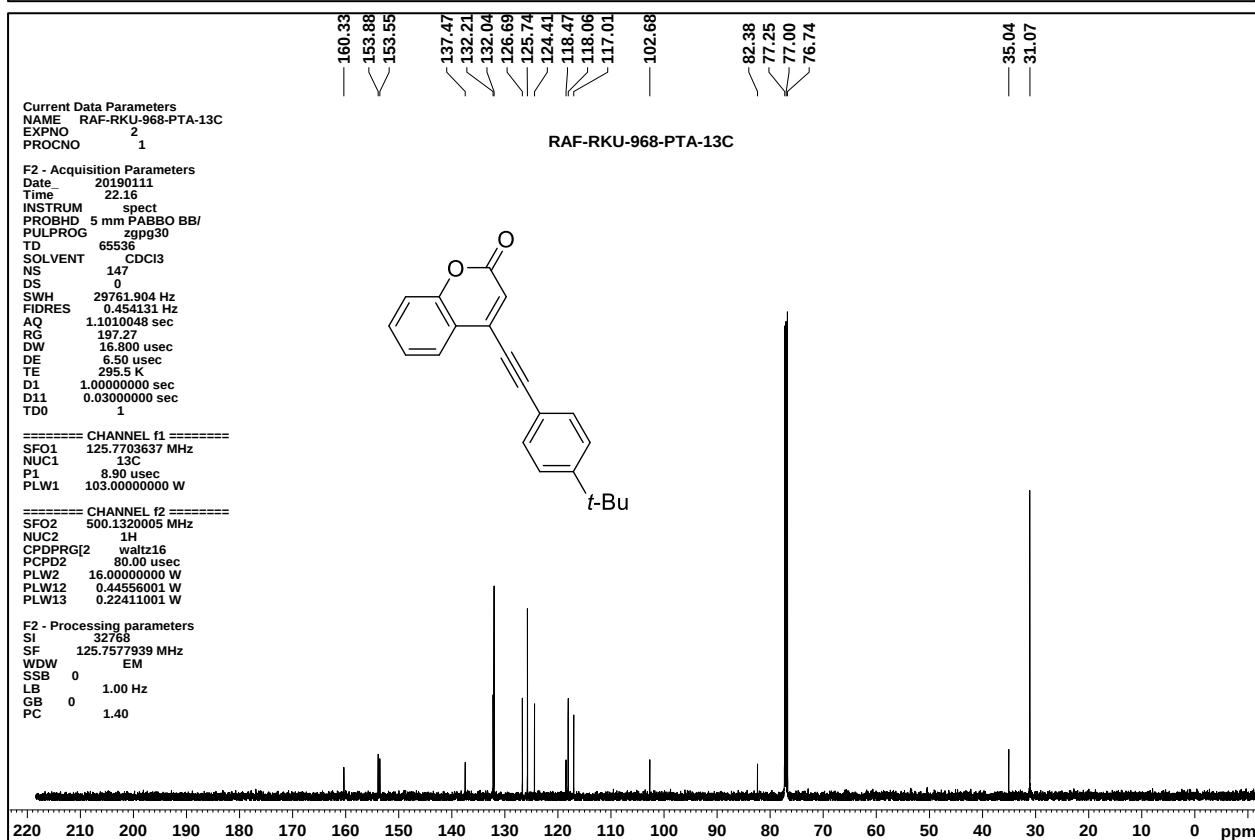
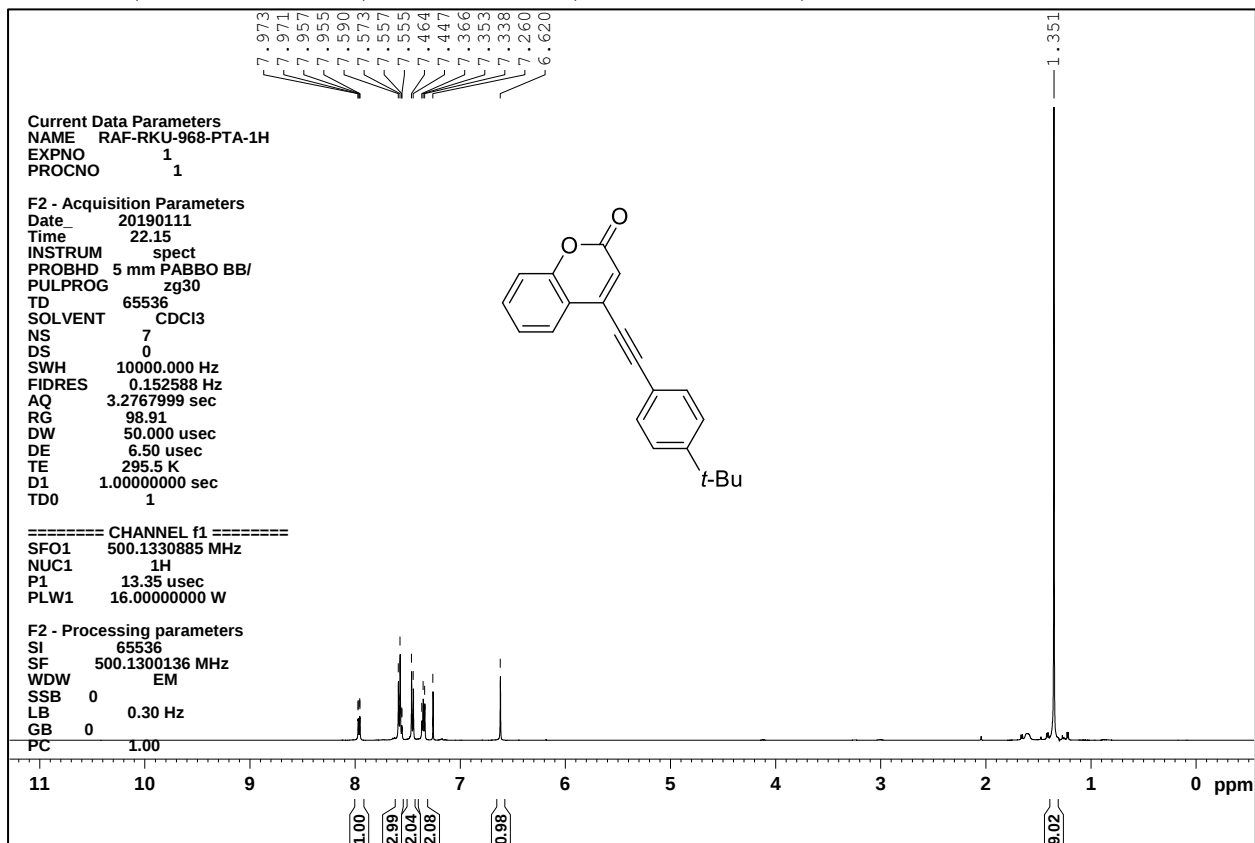
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **18c**



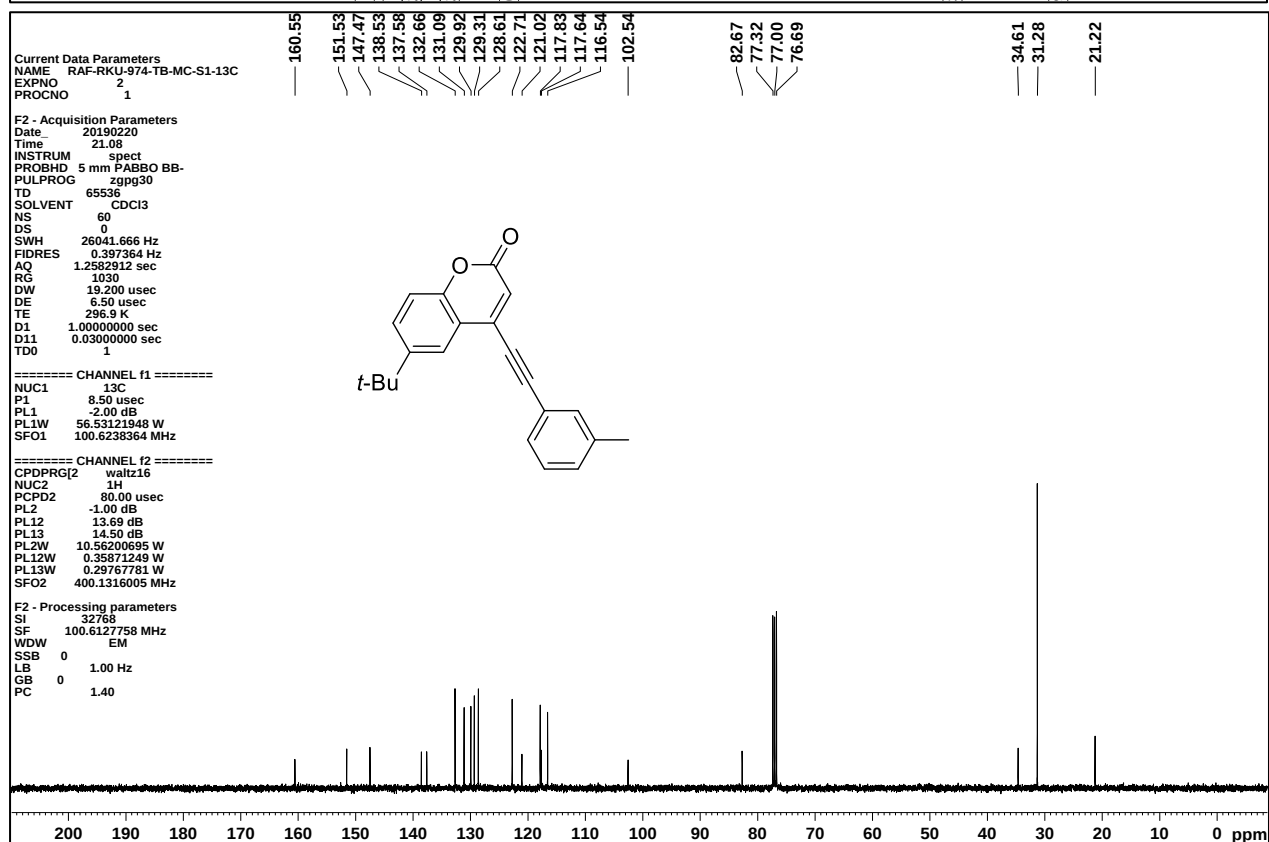
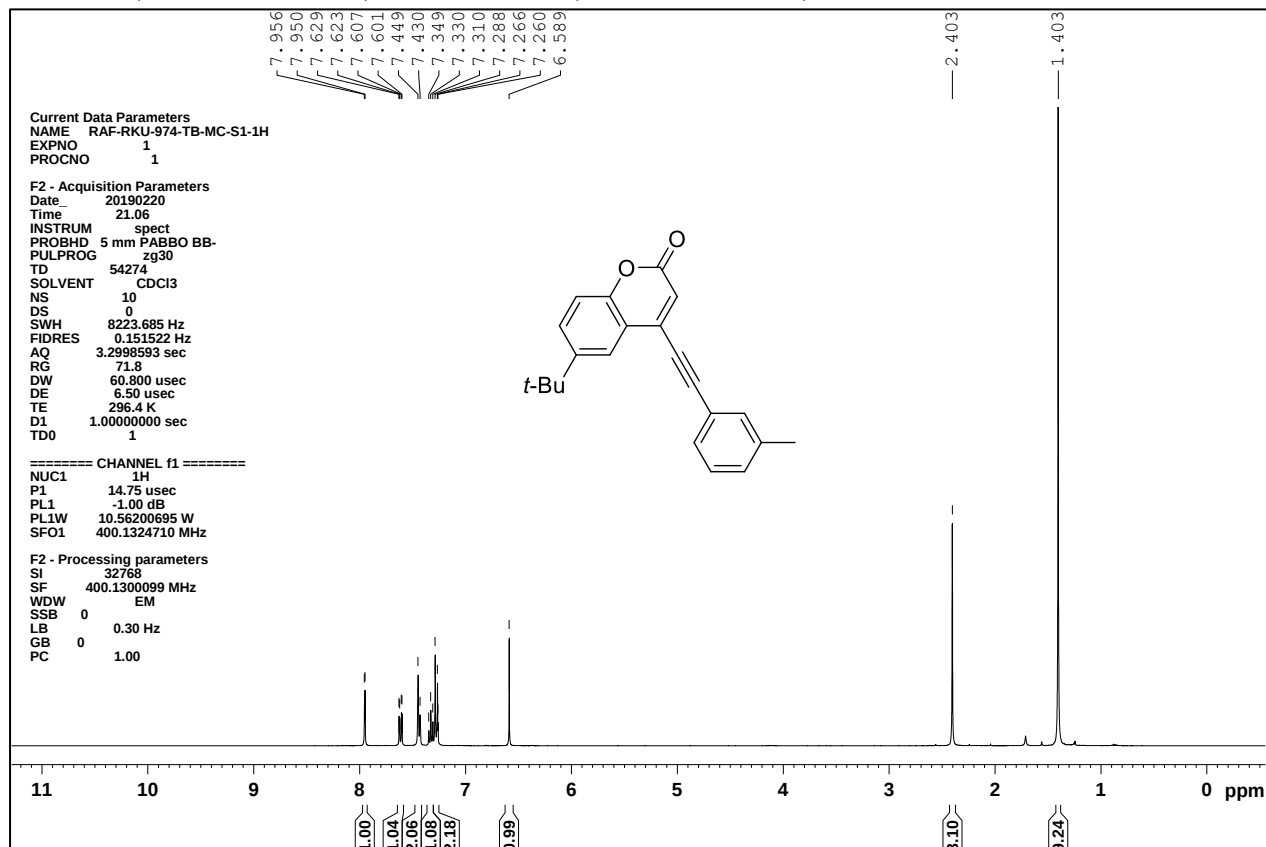
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **18d**



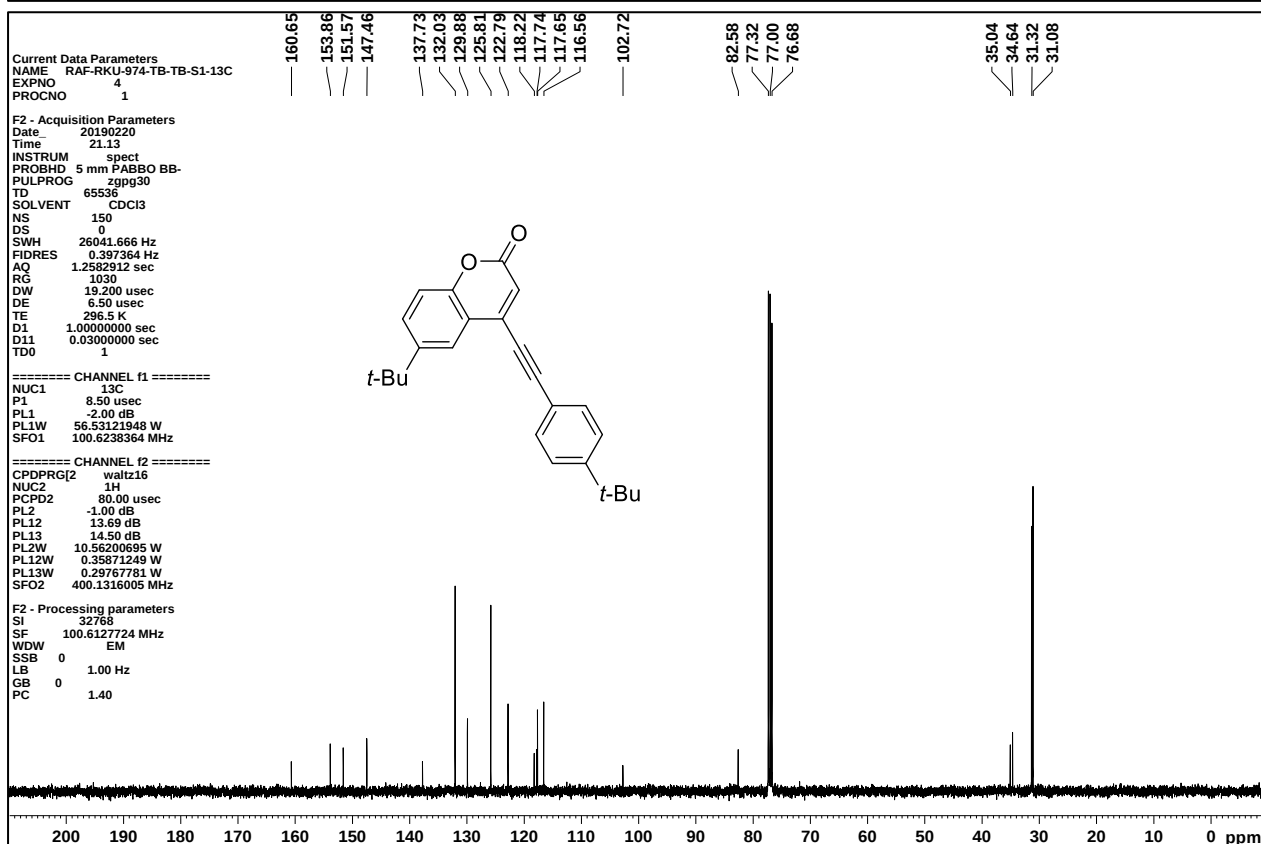
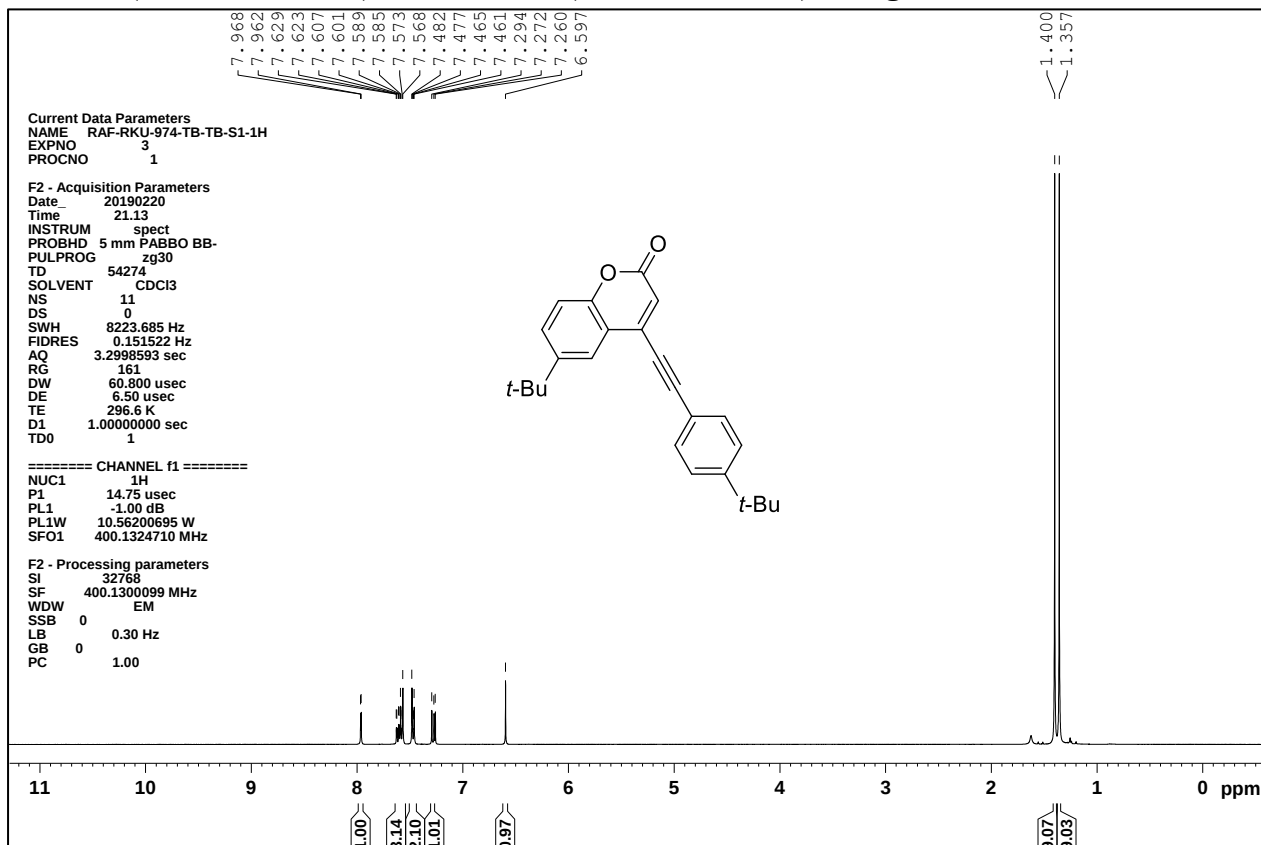
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **18e**



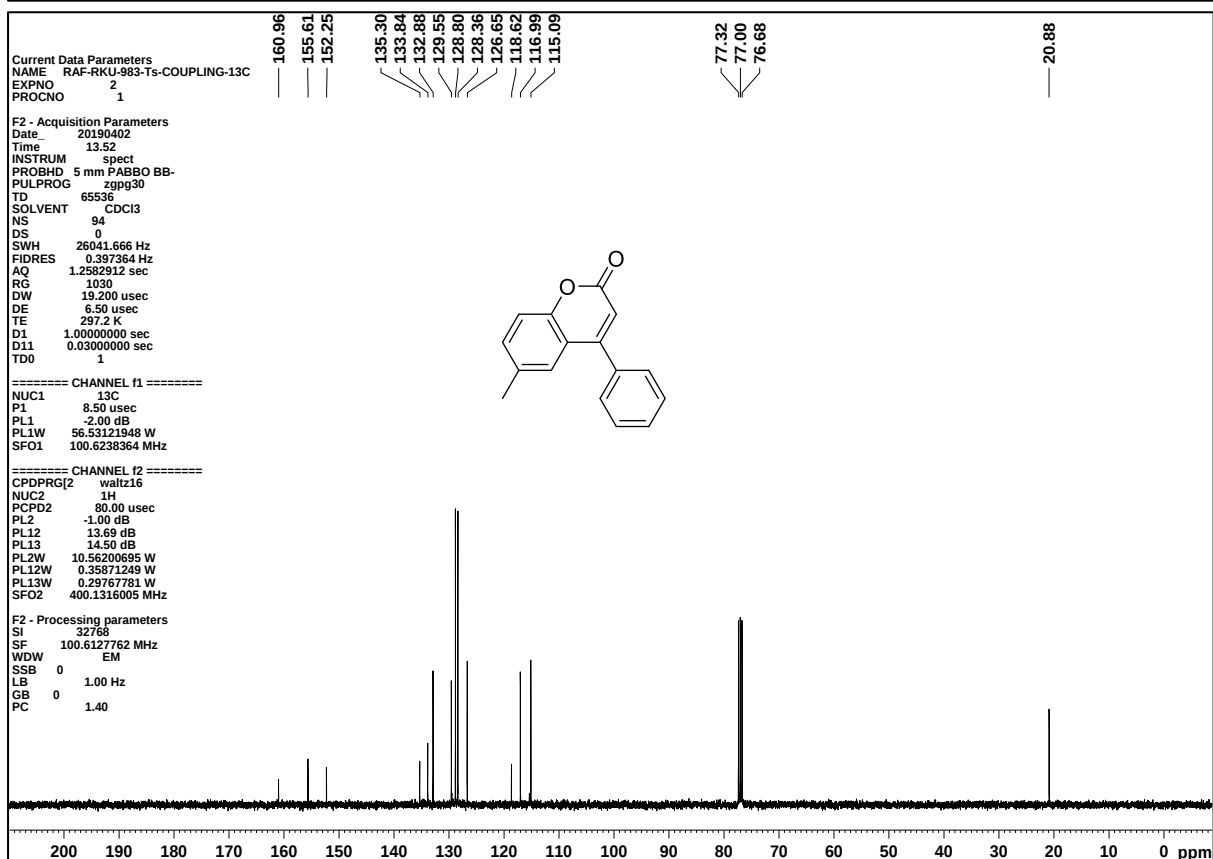
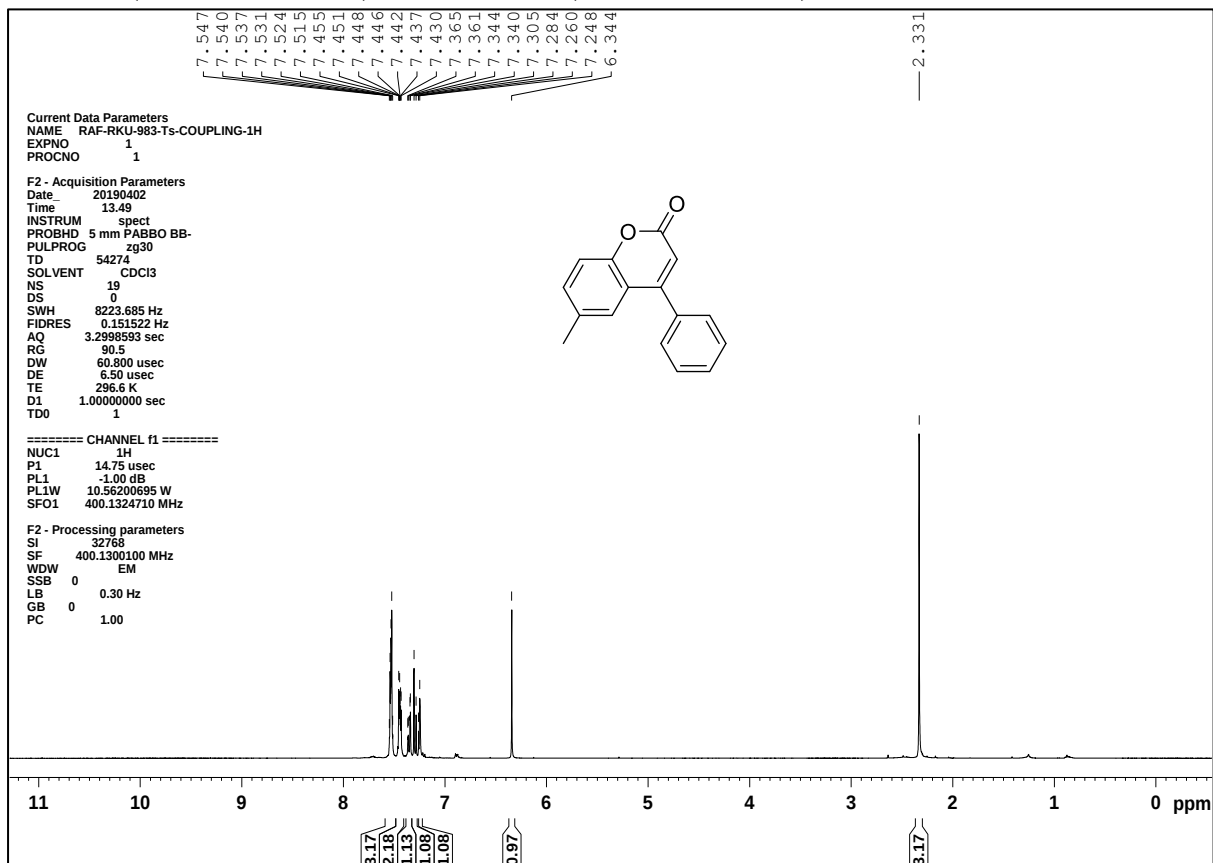
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of **18f**



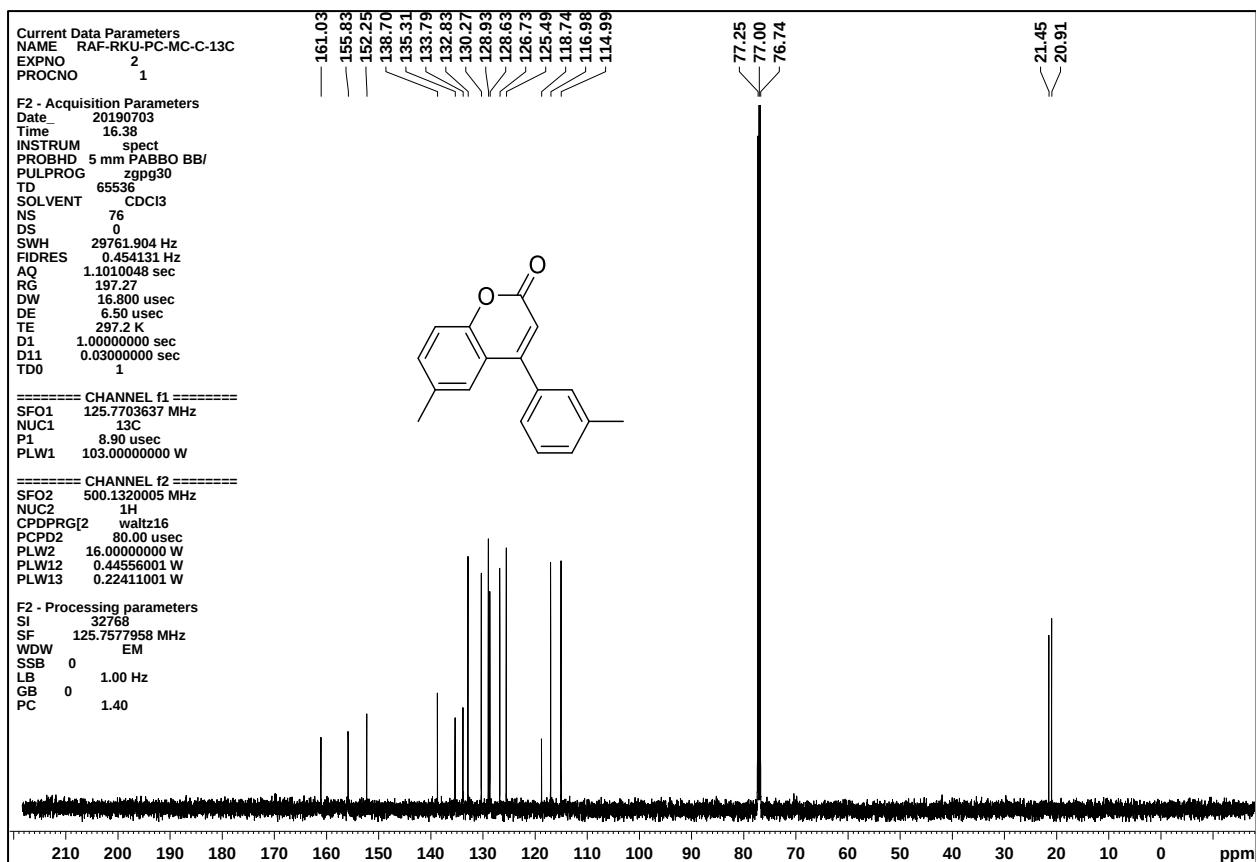
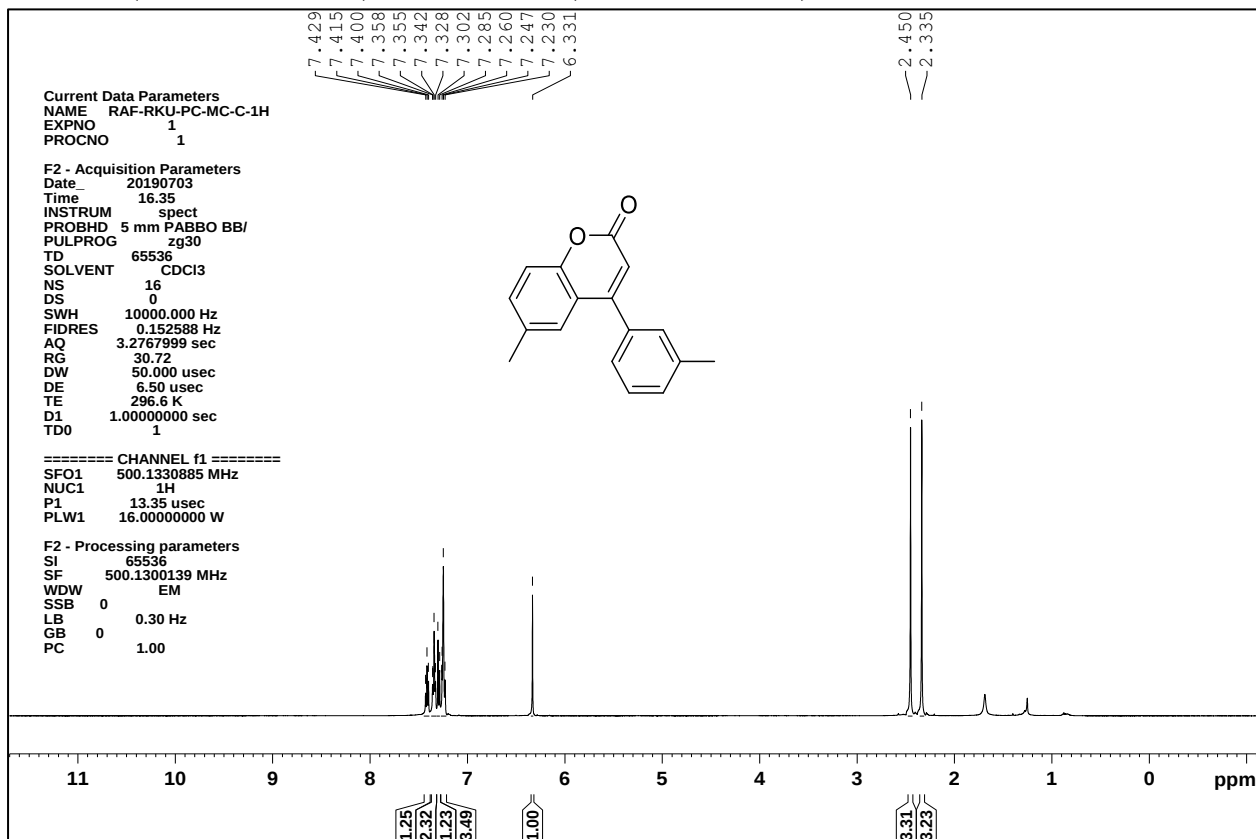
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of **18g**



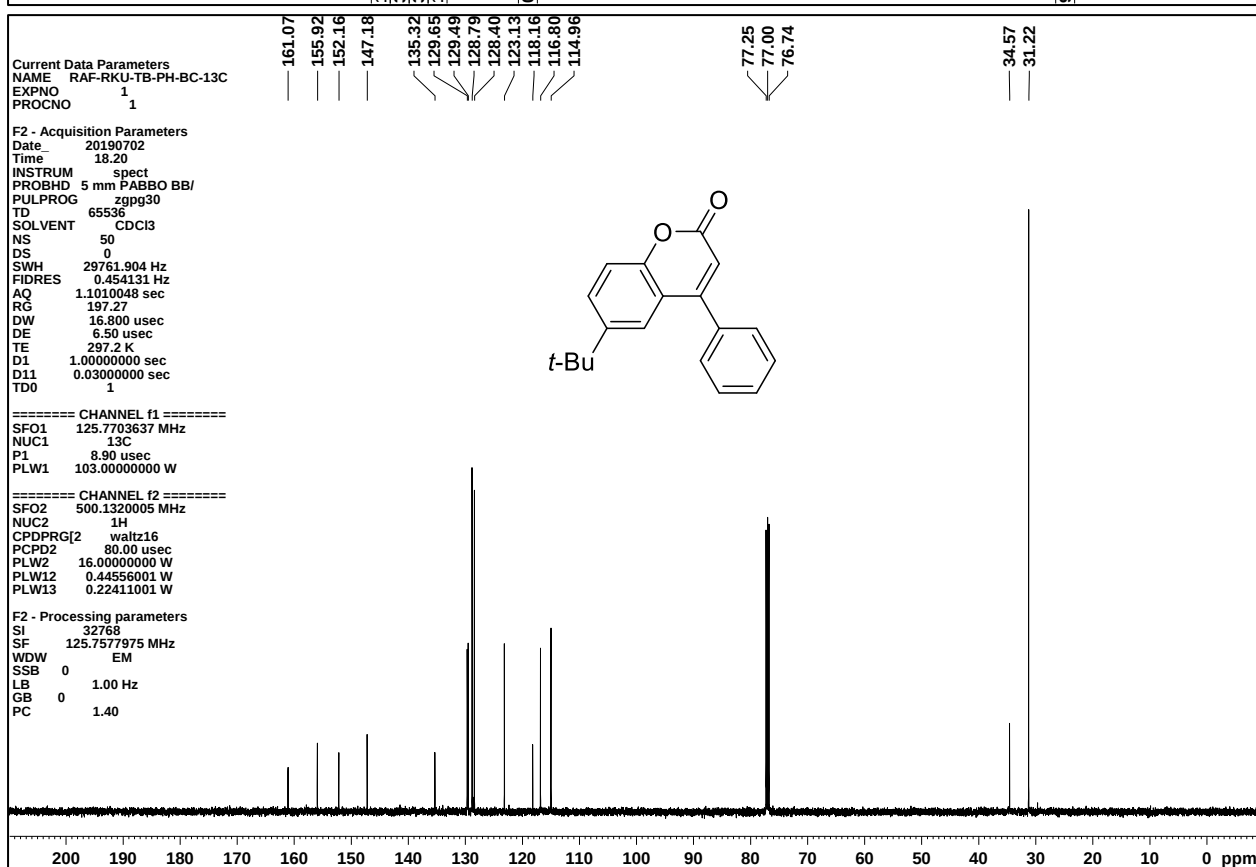
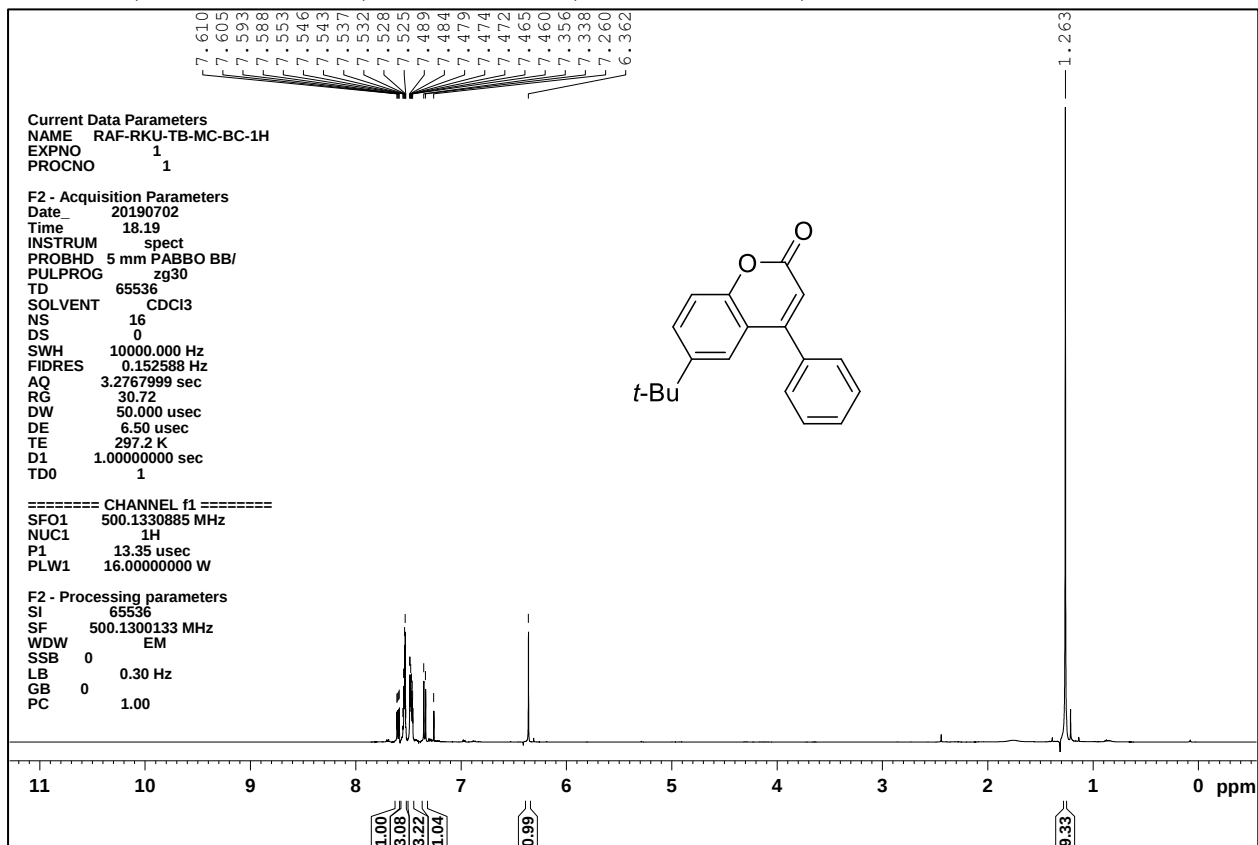
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of **20a**



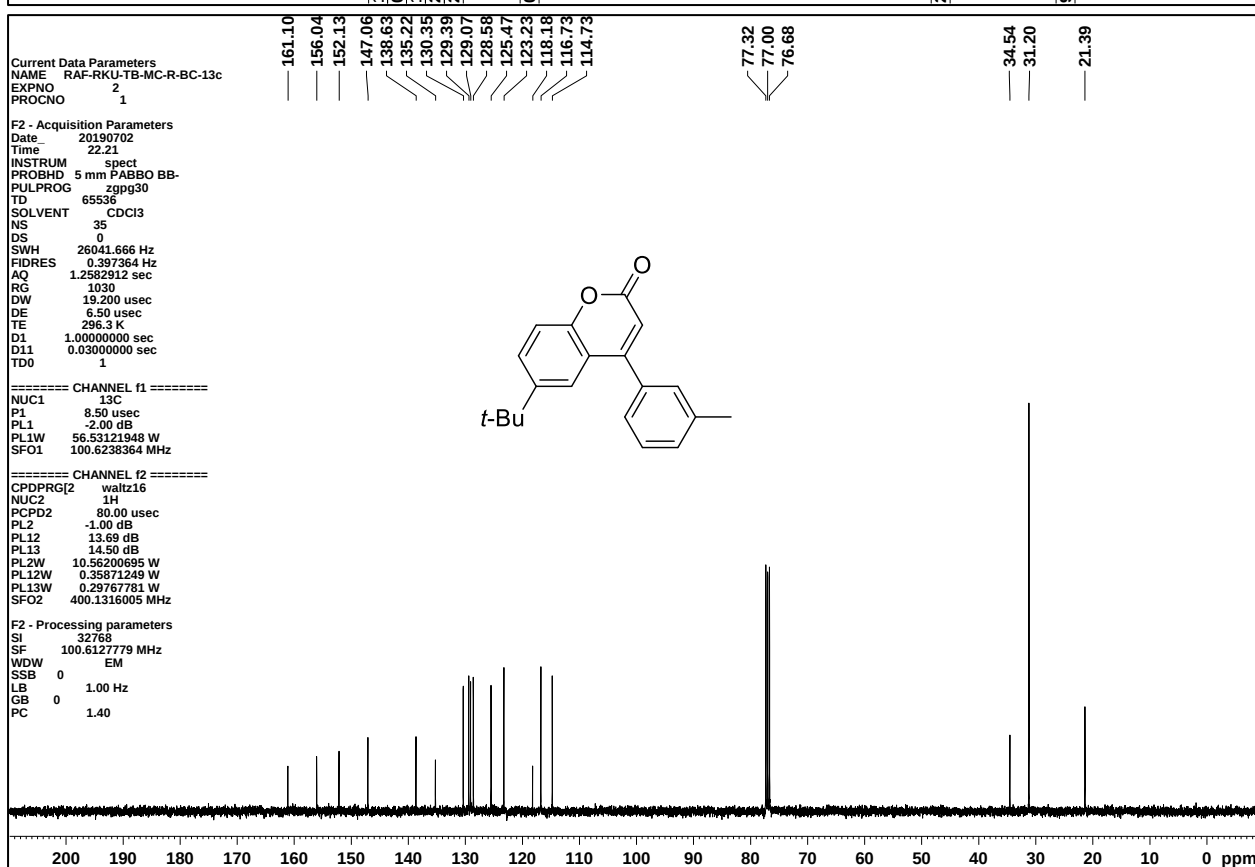
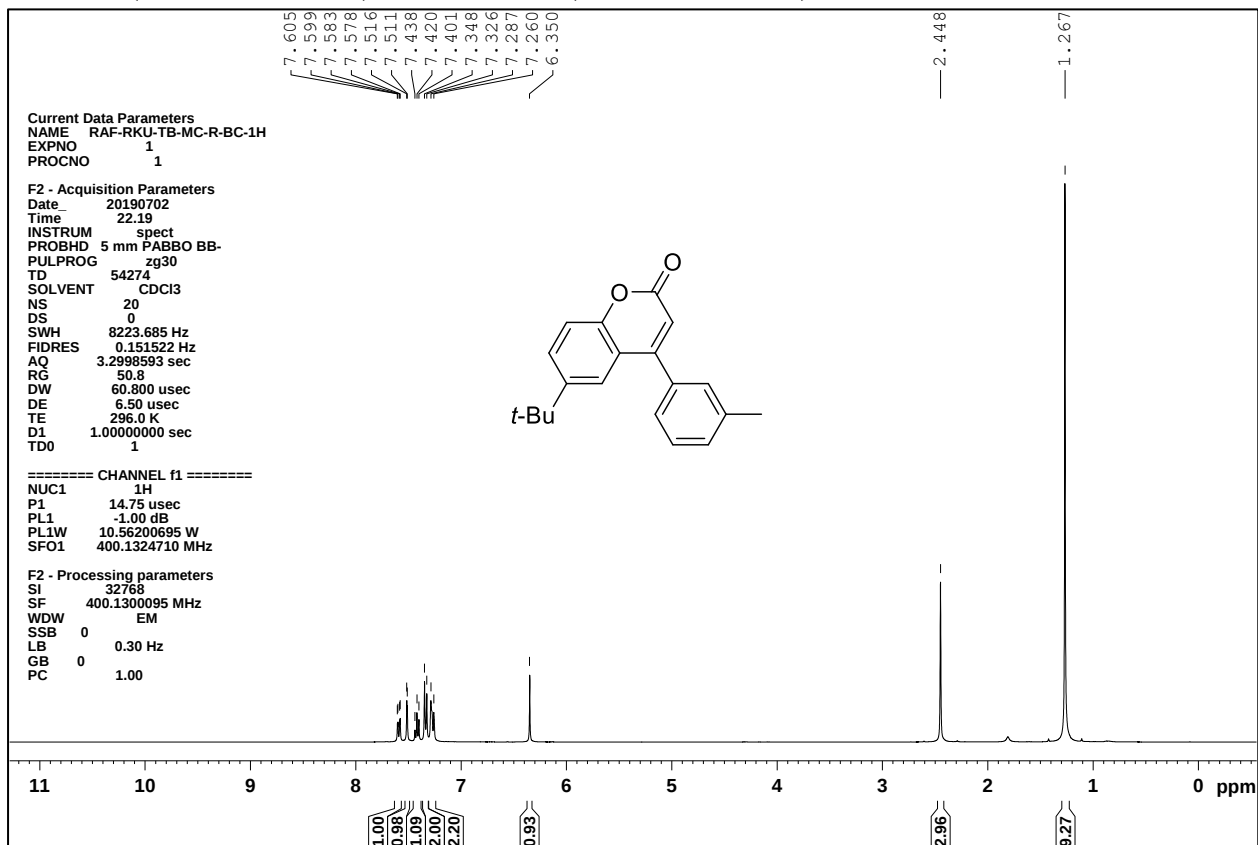
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **20b**



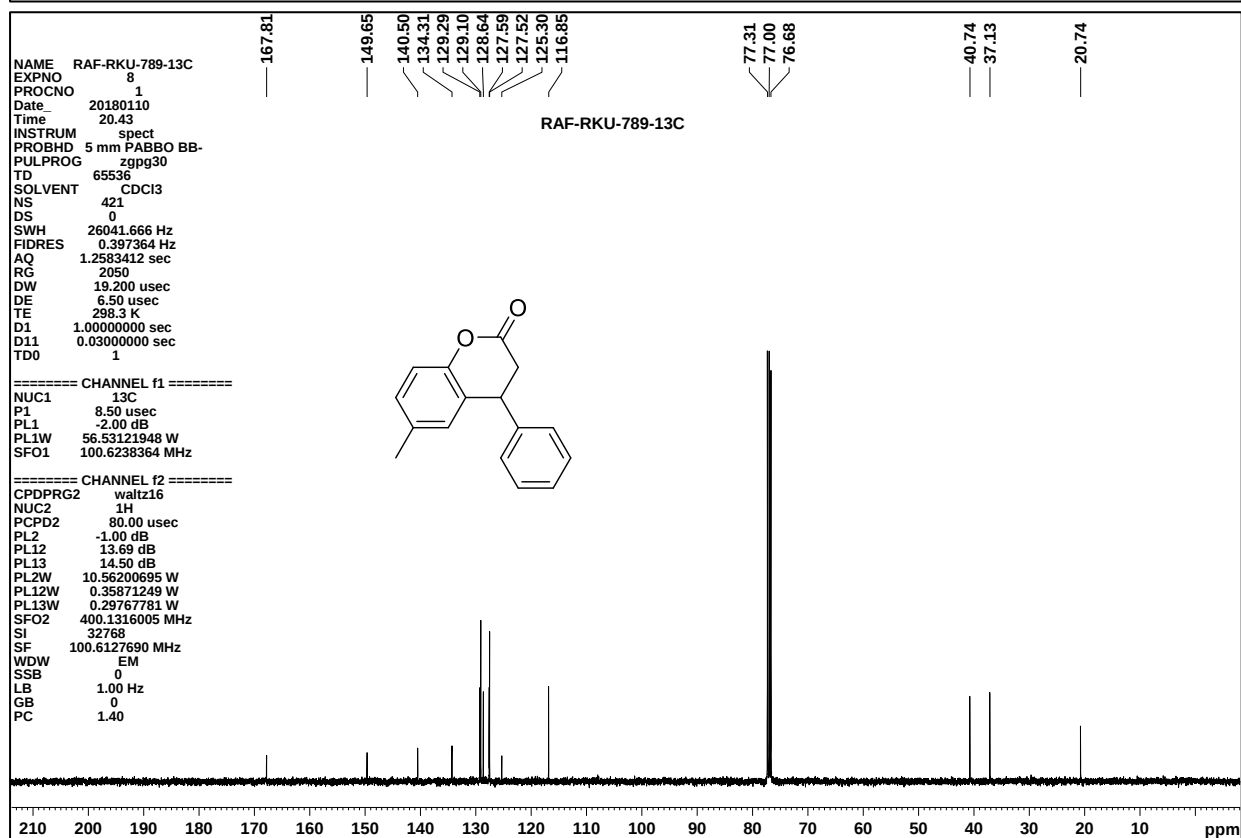
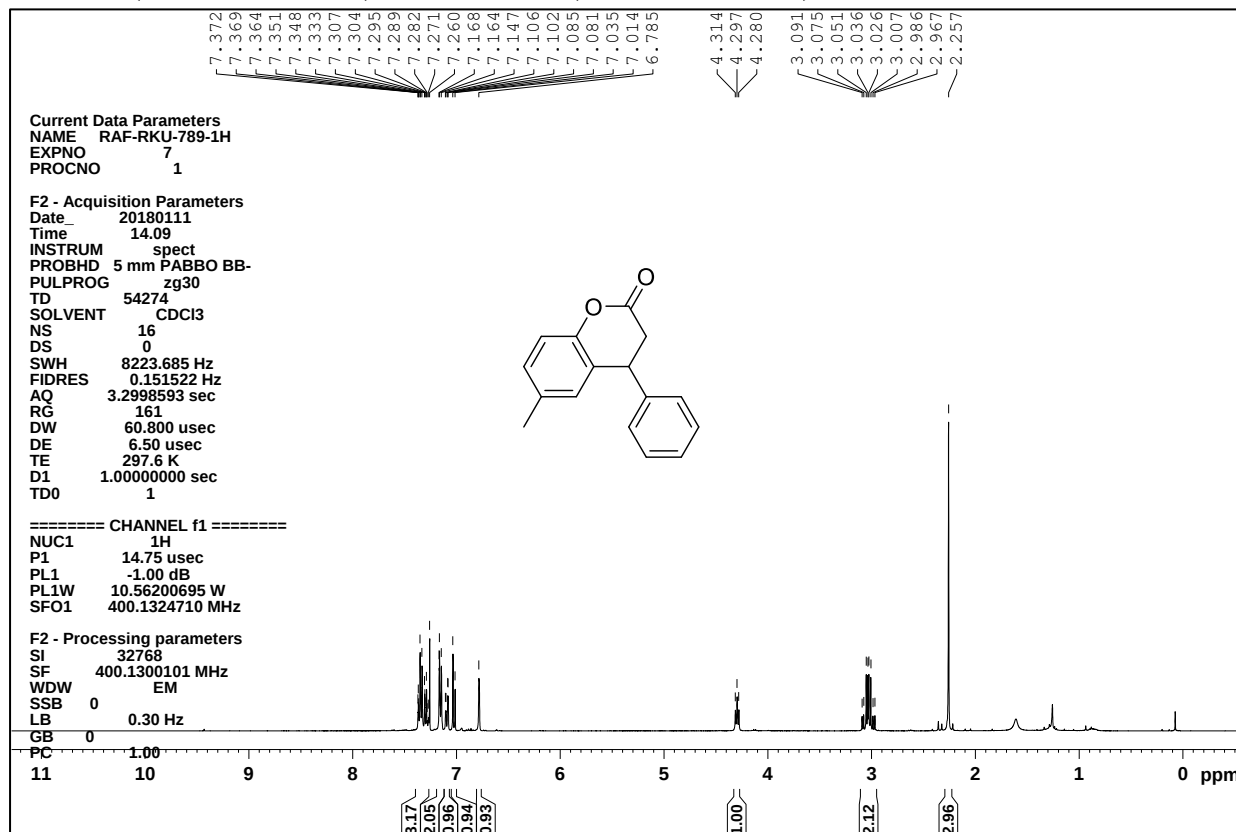
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of **20c**



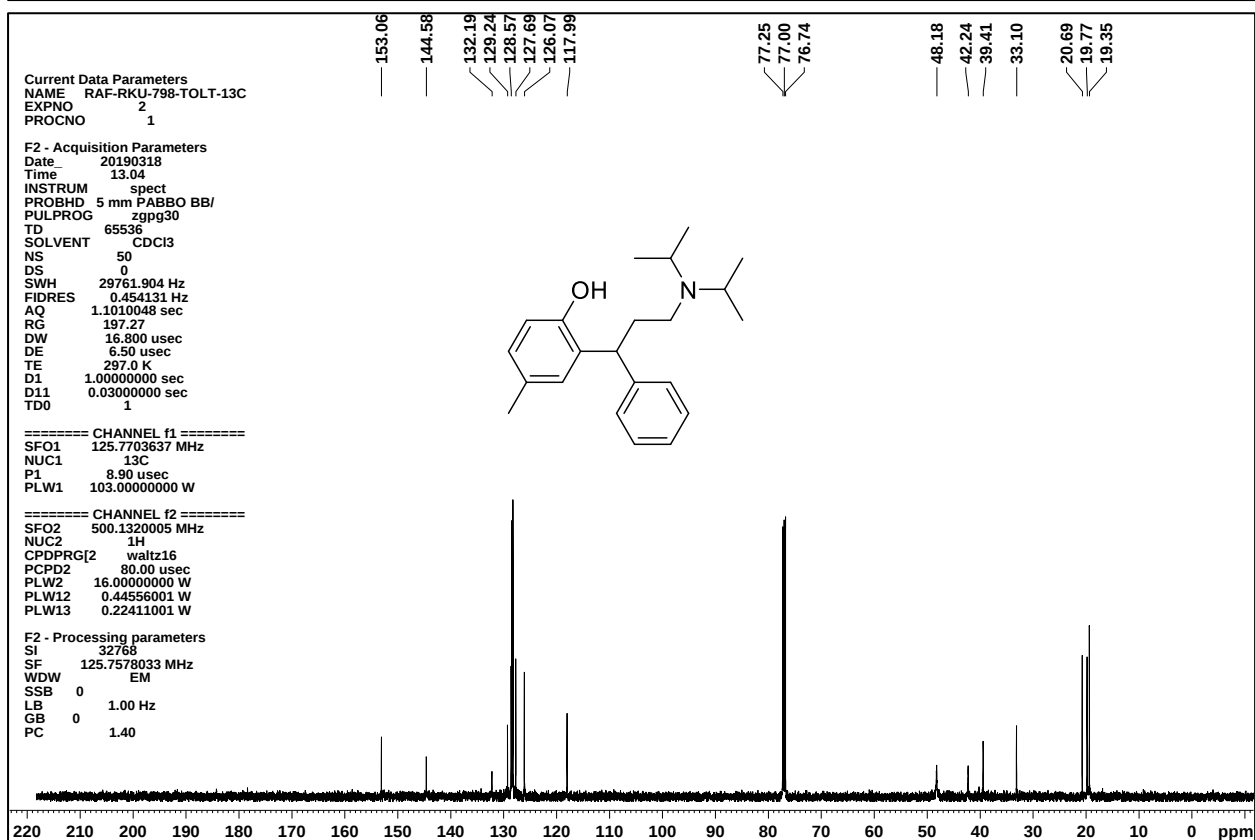
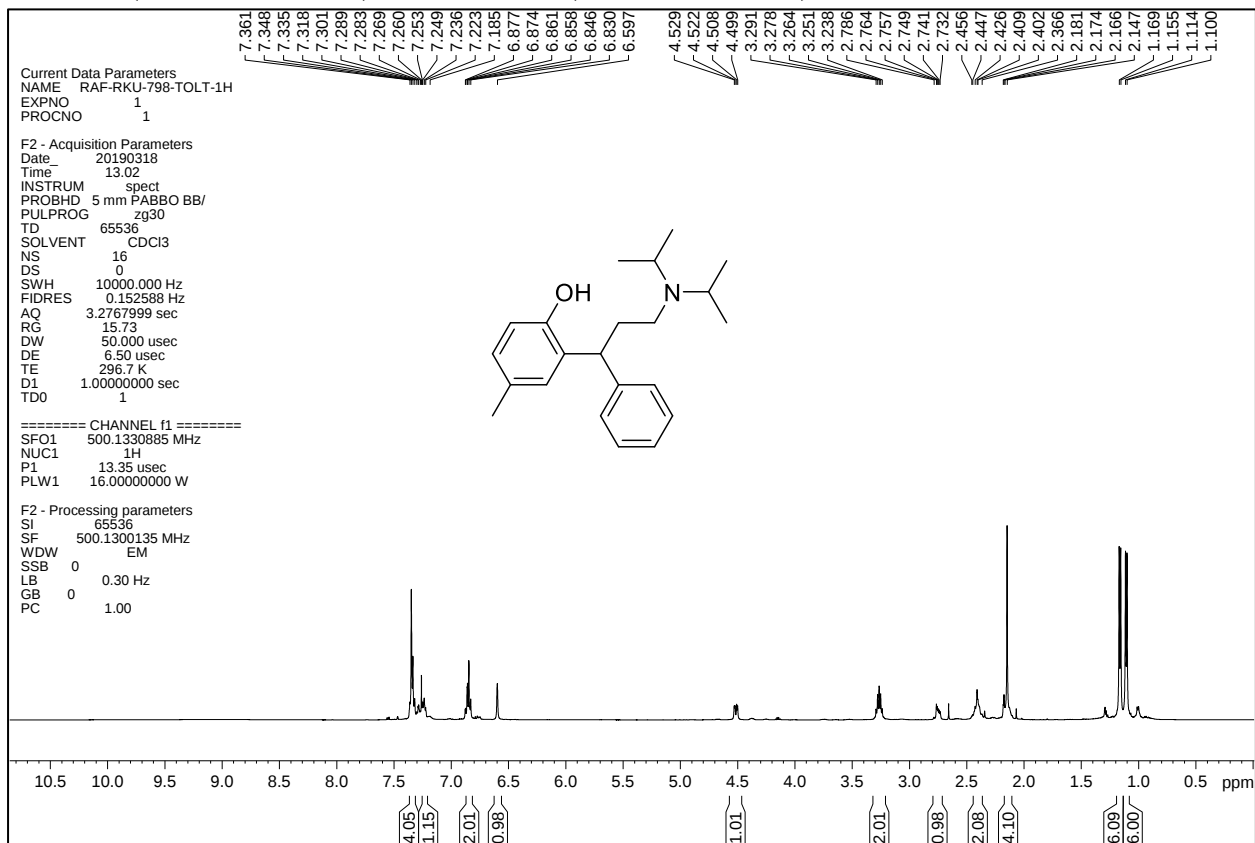
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of **20d**



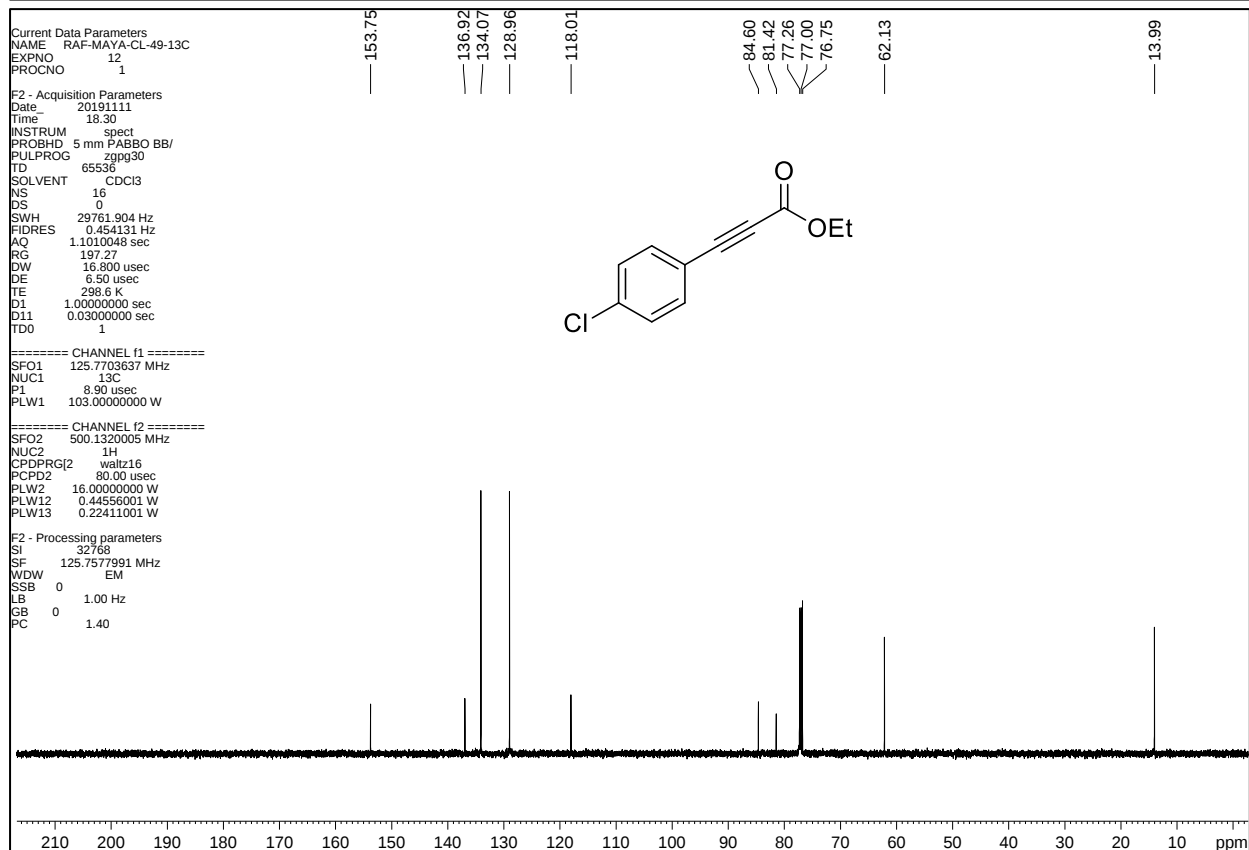
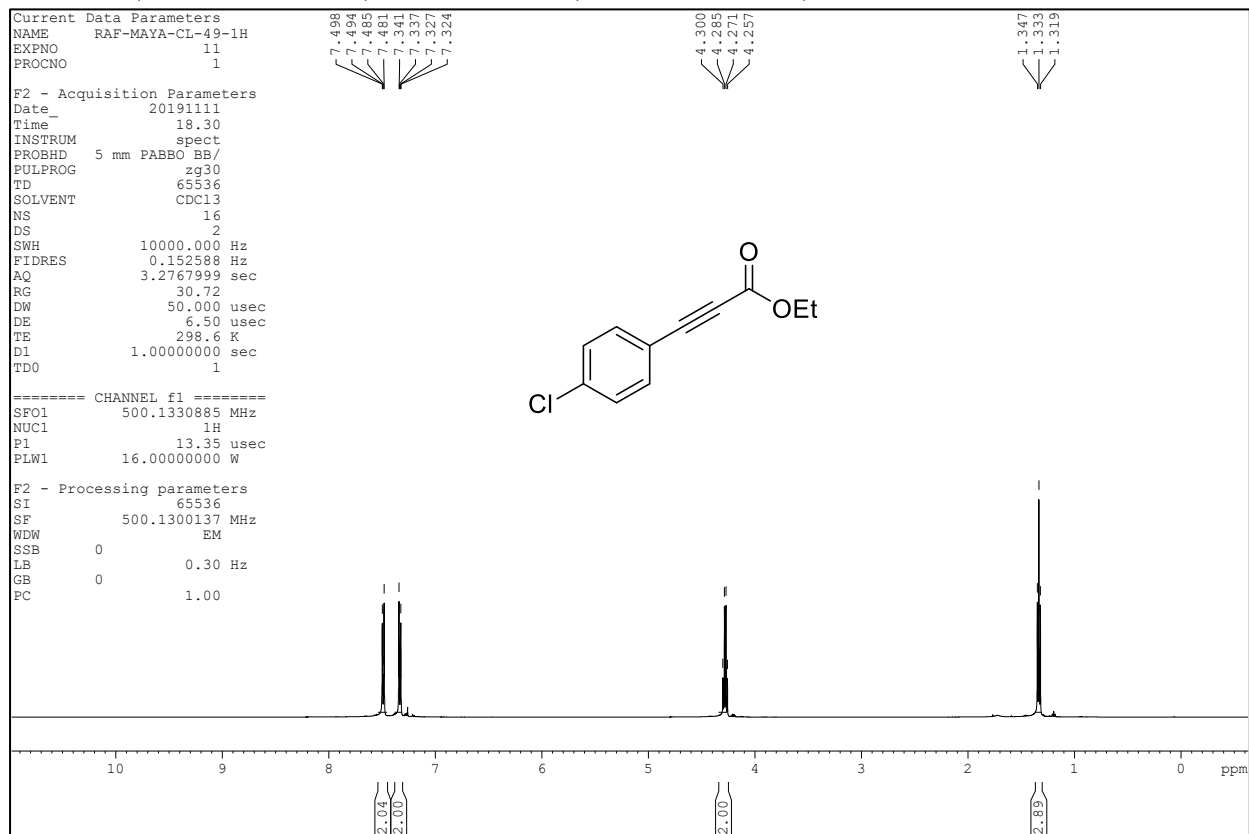
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **21**



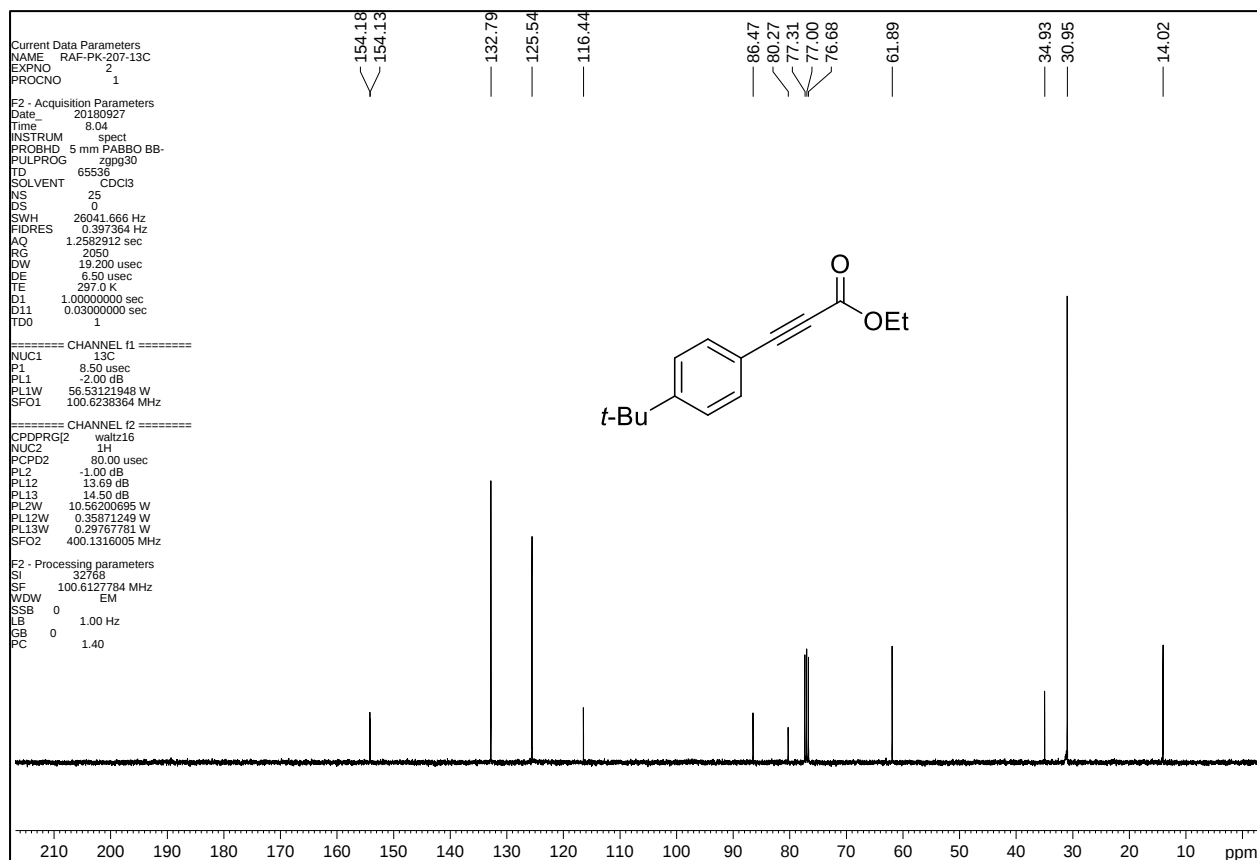
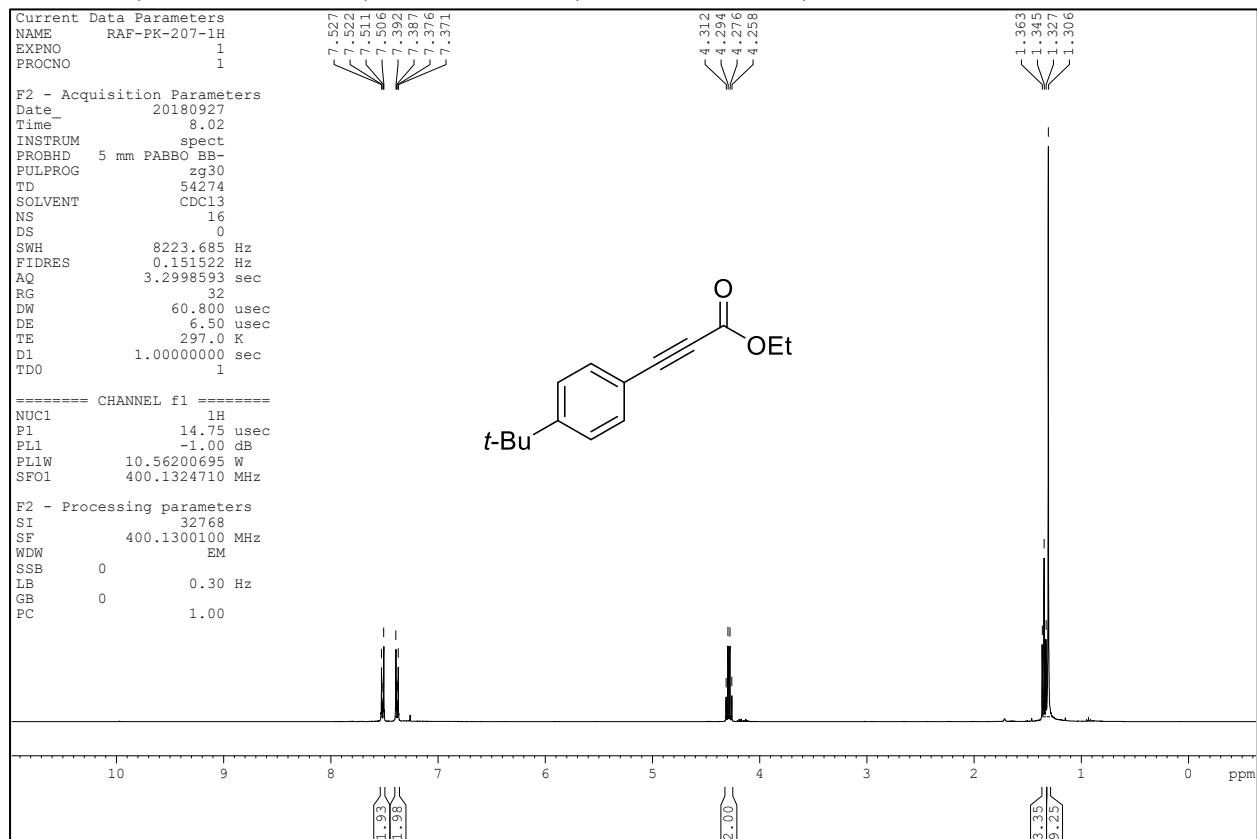
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of 22



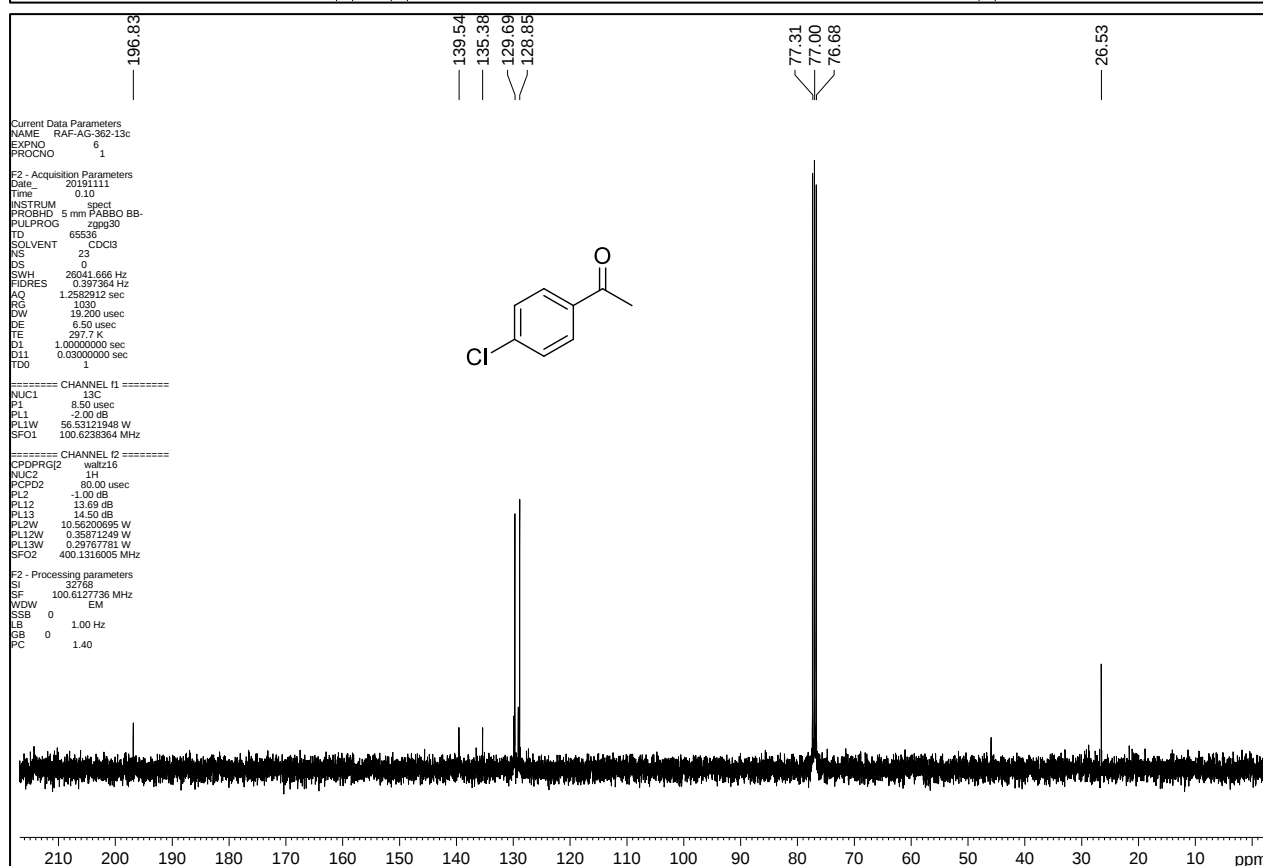
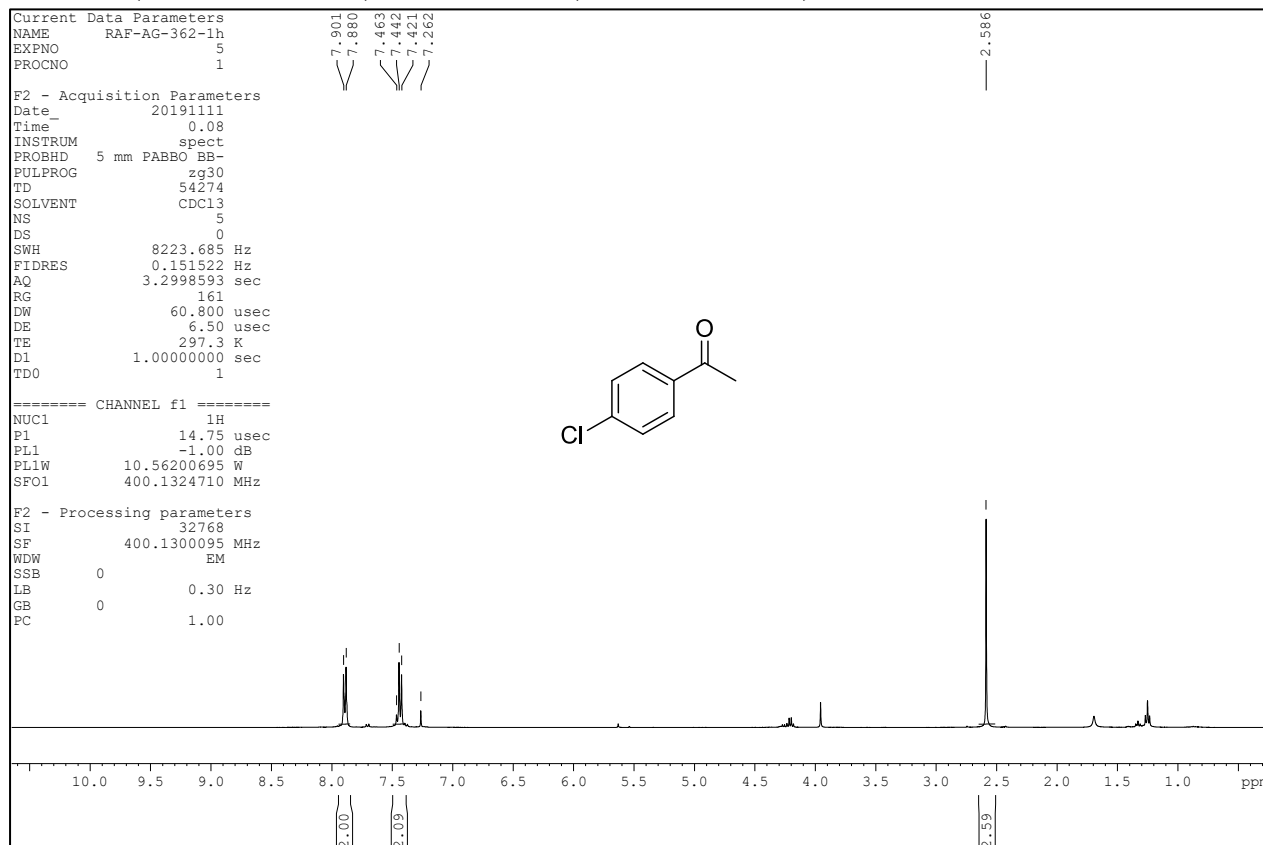
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of **23a**



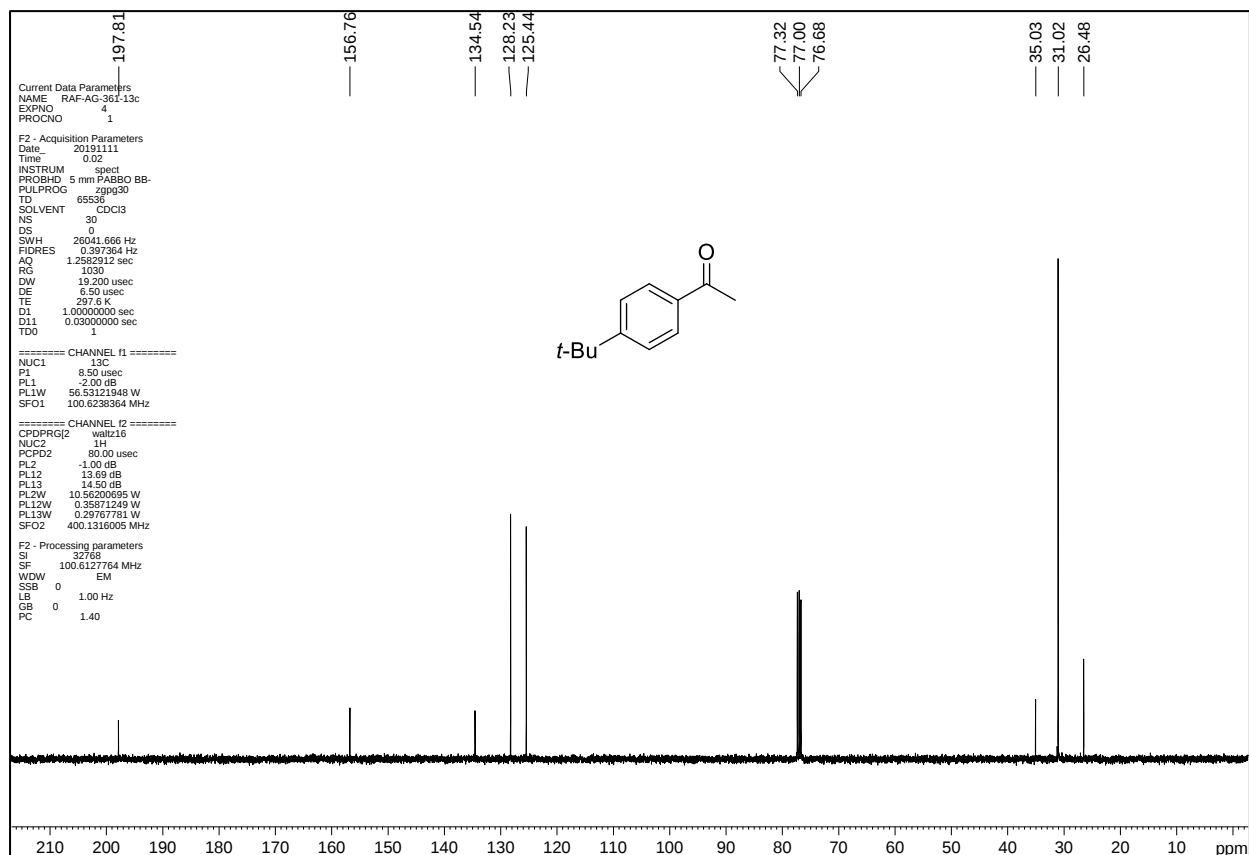
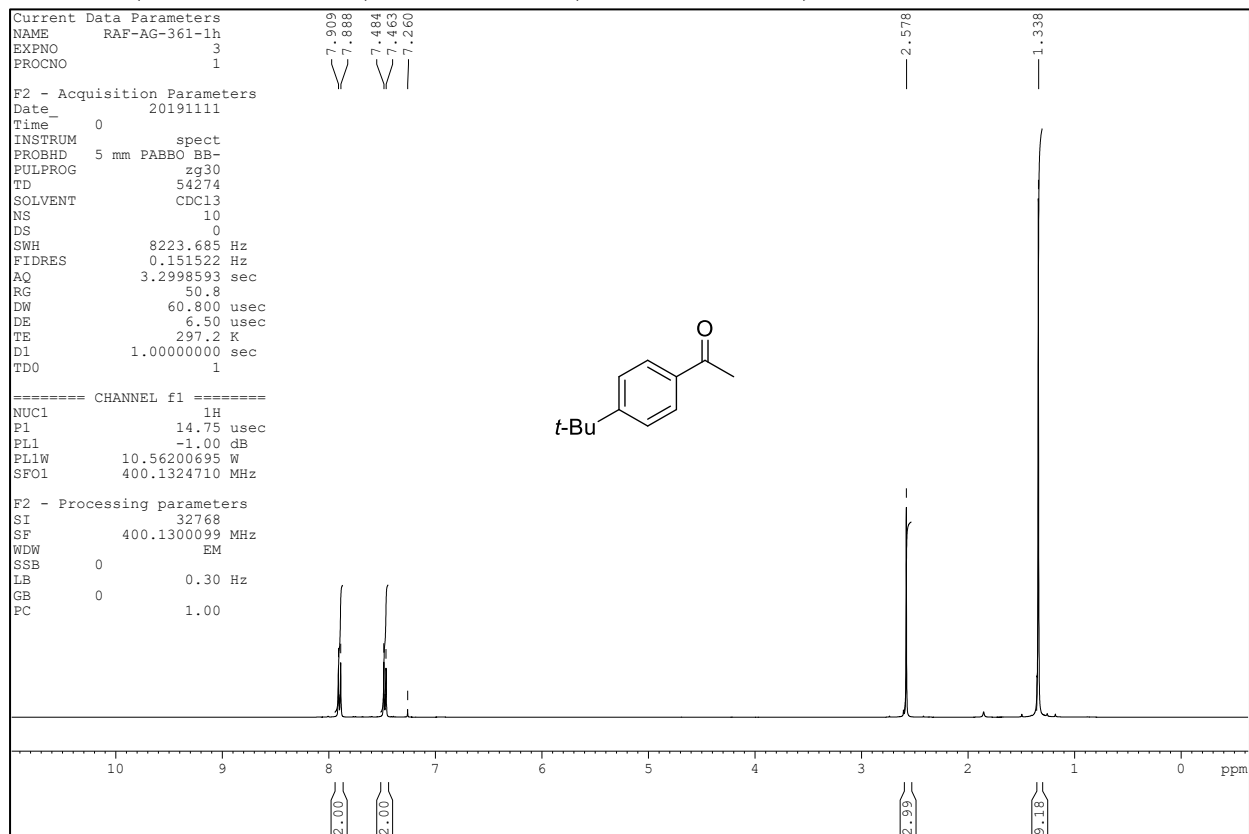
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **23b**



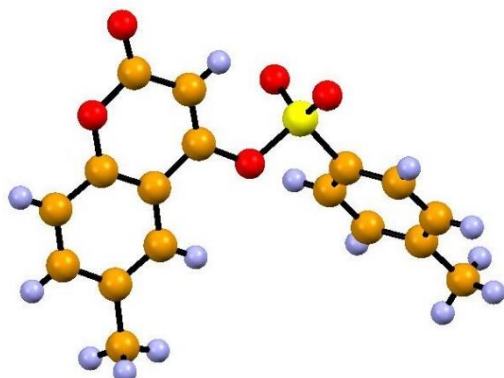
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **24a**



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) and <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of **24b**



# XRD parameters of 5a



|                                                |                                                                   |
|------------------------------------------------|-------------------------------------------------------------------|
| CCDC                                           | 1942552                                                           |
| Empirical formula                              | $C_{11.33}H_{9.33}O_{3.33}S_{0.67}$                               |
| Formula weight                                 | 220.23                                                            |
| Temperature/K                                  | 293(2)                                                            |
| Crystal system                                 | monoclinic                                                        |
| Space group                                    | $P2_1/n$                                                          |
| a/Å                                            | 9.5139(6)                                                         |
| b/Å                                            | 11.8830(8)                                                        |
| c/Å                                            | 13.6051(9)                                                        |
| $\alpha/^\circ$                                | 90                                                                |
| $\beta/^\circ$                                 | 103.300(6)                                                        |
| $\gamma/^\circ$                                | 90                                                                |
| Volume/Å <sup>3</sup>                          | 1496.86(18)                                                       |
| Z                                              | 6                                                                 |
| $\rho_{\text{calc}}/\text{g cm}^{-3}$          | 1.466                                                             |
| $\mu/\text{mm}^{-1}$                           | 0.240                                                             |
| F(000)                                         | 688.0                                                             |
| Crystal size/mm <sup>3</sup>                   | 0.20 × 0.12 × 0.05                                                |
| Radiation                                      | MoK $\alpha$ ( $\lambda$ = 0.71073)                               |
| 2 $\Theta$ range for data collection/ $^\circ$ | 4.606 to 49.992                                                   |
| Index ranges                                   | $-11 \leq h \leq 7$ , $-13 \leq k \leq 10$ , $-16 \leq l \leq 16$ |
| Reflections collected                          | 4668                                                              |
| Independent reflections                        | 2559 [ $R_{\text{int}}$ = 0.0496, $R_{\text{sigma}}$ = 0.0758]    |
| Data/restraints/parameters                     | 2559/0/210                                                        |
| Goodness-of-fit on $F^2$                       | 1.053                                                             |
| Final R indexes [ $I \geq 2\sigma(I)$ ]        | $R_1$ = 0.0510, $wR_2$ = 0.1134                                   |
| Final R indexes [all data]                     | $R_1$ = 0.0732, $wR_2$ = 0.1310                                   |
| Largest diff. peak/hole / e Å <sup>-3</sup>    | 0.29/-0.57                                                        |

**Table 1** Bond Lengths for **5a**.

| Atom | Atom | Length/Å  | Atom | Atom | Length/Å  |
|------|------|-----------|------|------|-----------|
| S01  | O002 | 1.624 (2) | C00A | C00G | 1.389 (4) |
| S01  | O003 | 1.420 (2) | C00A | C00N | 1.386 (4) |
| S01  | O004 | 1.427 (2) | C00B | C00D | 1.399 (4) |
| S01  | C00I | 1.744 (3) | C00C | C00N | 1.513 (4) |
| O002 | C007 | 1.383 (3) | C00D | C00J | 1.391 (4) |
| O005 | C00H | 1.372 (3) | C00E | C00M | 1.378 (4) |
| O005 | C00J | 1.385 (3) | C00F | C00H | 1.443 (4) |
| O006 | C00H | 1.213 (4) | C00G | C00I | 1.386 (4) |
| C007 | C00D | 1.444 (4) | C00I | C00K | 1.388 (4) |
| C007 | C00F | 1.343 (4) | C00J | C00M | 1.384 (4) |
| C008 | C009 | 1.513 (4) | C00K | C00L | 1.378 (4) |
| C008 | C00B | 1.381 (4) | C00L | C00N | 1.380 (4) |
| C008 | C00E | 1.396 (4) |      |      |           |

**Table 2** Bond Angles for **5a**.

| Atom | Atom | Atom | Angle/°     | Atom | Atom | Atom | Angle/°   |
|------|------|------|-------------|------|------|------|-----------|
| O002 | S01  | C00I | 98.40 (12)  | C00M | C00E | C008 | 122.0 (3) |
| O003 | S01  | O002 | 108.45 (12) | C007 | C00F | C00H | 121.4 (3) |
| O003 | S01  | O004 | 118.86 (14) | C00I | C00G | C00A | 118.5 (3) |
| O003 | S01  | C00I | 110.85 (14) | O005 | C00H | C00F | 117.3 (3) |
| O004 | S01  | O002 | 107.03 (12) | O006 | C00H | O005 | 117.3 (3) |
| O004 | S01  | C00I | 111.15 (13) | O006 | C00H | C00F | 125.4 (3) |
| C007 | O002 | S01  | 121.07 (18) | C00G | C00I | S01  | 119.1 (2) |
| C00H | O005 | C00J | 121.6 (2)   | C00G | C00I | C00K | 121.4 (3) |
| O002 | C007 | C00D | 113.5 (2)   | C00K | C00I | S01  | 119.4 (2) |
| C00F | C007 | O002 | 125.4 (3)   | O005 | C00J | C00D | 122.0 (3) |
| C00F | C007 | C00D | 121.2 (3)   | C00M | C00J | O005 | 116.9 (3) |
| C00B | C008 | C009 | 121.2 (3)   | C00M | C00J | C00D | 121.1 (3) |
| C00B | C008 | C00E | 118.2 (3)   | C00L | C00K | C00I | 118.5 (3) |
| C00E | C008 | C009 | 120.6 (3)   | C00K | C00L | C00N | 121.7 (3) |
| C00N | C00A | C00G | 121.1 (3)   | C00E | C00M | C00J | 118.8 (3) |
| C008 | C00B | C00D | 121.2 (3)   | C00A | C00N | C00C | 120.2 (3) |
| C00B | C00D | C007 | 124.8 (3)   | C00L | C00N | C00A | 118.7 (3) |
| C00J | C00D | C007 | 116.5 (3)   | C00L | C00N | C00C | 121.0 (3) |
| C00J | C00D | C00B | 118.8 (3)   |      |      |      |           |