Synthesis of Guaianolide Analogues with a Tunable α-Methylene–γ-lactam Electrophile and Correlating Bioactivity with Thiol Reactivity

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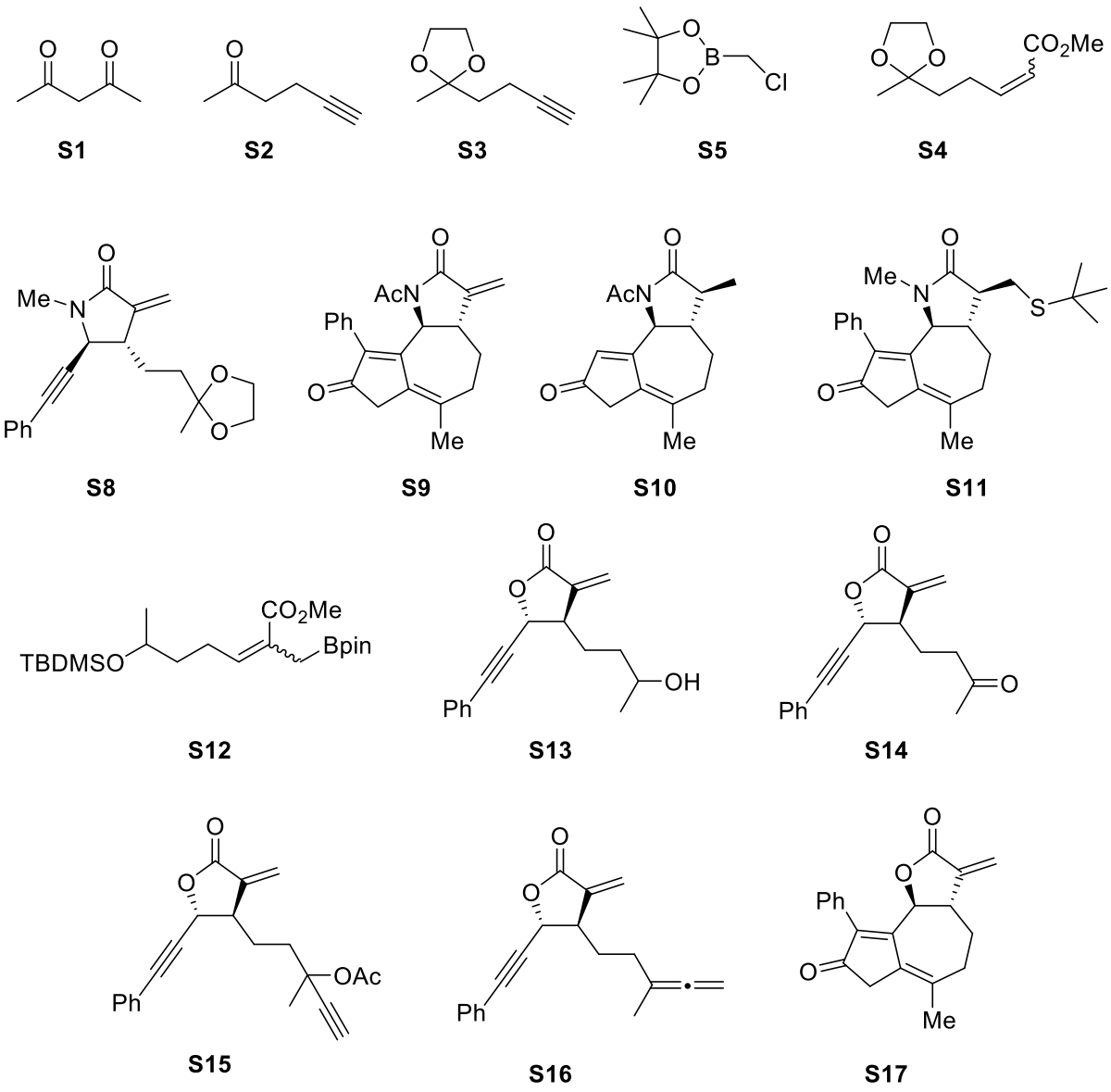



**Figure S1**. Structures of Supporting Information Compounds

**Supporting Information Table S1**

| | A549/NF-κB-Luciferase Activity (%) | | | | | |
|---|---|---|---|---|---|---|
| | A549/NF-κB Luciferase Reporter Assay | | | Alamar Blue Cell Viability Assay | | |
| **Compound** | 20μM | 10μM | 5μM | 20μM | 10μM | 5μM |
| **45** | 93.6 ± 8.2 | 97.3 ± 9.0 | 96.2 ± 12.1 | 102.0 ± 11.3 | 97.3 ± 22.0 | 91.1 ± 13.9 |
| **47** | 100.4 ± 7.7 | 106.2 ± 13.4 | 100.5 ± 15.2 | 87.9 ± 26.9 | 96.3 ± 13.8 | 96.8 ± 16.9 |
| **48** | 101.3 ± 4.2 | 95.7 ± 6.5 | 101.6 ± 7.4 | 111.6 ± 17.4 | 98.5 ± 10.8 | 99.9 ± 23.0 |
| **49** | 86.0 ± 17.6 | 95.3 ± 15.9 | 105.8 ± 16.4 | 108.4 ± 19.6 | 100.6 ± 11.9 | 97.2 ± 31.2 |
| **51** | 20.9 ± 2.7 | 61.9 ± 8.5 | 85.6 ± 7.2 | 88.0 ± 38.3 | 96.8 ± 21.8 | 103.7 ± 22.0 |
| **52** | 80.8 ± 12.8 | 95.7 ± 7.2 | 98.9 ± 11.5 | 99.2 ± 7.5 | 99.0 ± 11.1 | 96.6 ± 16.2 |
| **54** | 2.6 ± 1.3 | 8.8 ± 2.2 | 33.1 ± 5.2 | 64.1 ± 7.7 | 63.6 ± 12.1 | 80.7 ± 7.0 |
| **60** | 28.3 ± 13.7 | 59.1 ± 6.1 | 97.8 ± 19.5 | 82.3 ± 8.2 | 93.3 ± 10.9 | 89.0 ± 5.3 |

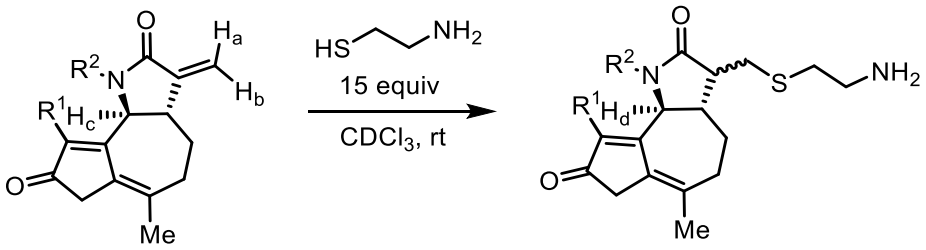
**Table S1.** Relative NF-κB activities (%; left) and relative cellular viabilities (%; right) for lactams dosed at various concentrations to A549/NF-κB-luc cells (Figure 4). Values are shown as mean ± S.D. for $n \geq 3$ biological replicates.

**Supporting Information Table S2**

| | HEK293-NF-κB/SEAP Activity (%) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | HEK293-NF-κB/SEAP Reporter Assay | | | | Alamar Blue Cell Viability Assay | | | |
| **Compound** | 7.5μM | 5μM | 2.5μM | 1μM | 7.5μM | 5μM | 2.5μM | 1μM |
| **PTL** | 19.7 ± 4.6 | 21.1 ± 5.0 | 49.7 ± 10.3 | 85.2 ± 15.8 | 74.4 ± 5.4 | 87.1 ± 9.6 | 93.3 ± 14.5 | 104.7±17.7 |
| **49** | 102.0±43.1 | 113.2 ± 37.3 | 97.4 ± 29.9 | 143.5 ± 29.0 | 99.1 ± 12.2 | 101.3 ± 7.11 | 97.9 ± 12.1 | 94.6 ± 7.1 |
| **51** | 44.1 ± 13.8 | 59.7 ± 18.3 | 93.2 ± 20.3 | 104.4 ± 39.3 | 101.2±12.3 | 110.7 ± 11.9 | 107.4 ± 6.6 | 113.5 ± 6.3 |
| **54** | 20.1 ± 4.1 | 27.6 ± 6.4 | 54.2 ± 13.7 | 85.1 ± 26.6 | 82.0 ± 5.2 | 87.8 ± 8.7 | 92.8 ± 8.0 | 101.7 ± 9.1 |
| **60** | 18.9 ± 3.5 | 20.3 ± 4.0 | 34.5 ± 13.3 | 63.4 ± 20.2 | 68.7 ± 9.1 | 76.3 ± 5.4 | 90.3 ± 12.7 | 102.6±17.1 |

**Table S2.** Relative NF-κB activities (%; left) and relative cellular viabilities (%; right) for lactams dosed at various concentrations to HEK293/NF-κB-SEAP cells (Figure 4). Values are shown as mean ± S.D. for $n \geq 3$ biological replicates.

**Reaction of α-Methylene-γ-Lactams and Lactone S17 with Cysteamine: Monitoring Reaction Progress by $^1$H NMR.**




(eq S1)

$$\text{Fraction Remaining}_x = \frac{\frac{H_a+H_b}{2}}{H_c+H_d} \quad \text{(eq S2)}$$

$$\ln(Fraction\ Remaining_x) = -k_{pseudo1st}t + \ln(Fraction\ Remaining_{x_o}) \quad \text{(eq S3)}$$

$$t_{1/2} = \frac{\ln 2}{k_{pseudo1st}} \quad \text{(eq S4)}$$

$$Fraction\ Remaining_x = \frac{\frac{H_a+H_b}{2}}{\frac{H_{hexamethylbenzene}}{32}} \quad \text{(eq S5)}$$

$$Fraction\ Remaining_x = \frac{\frac{H_a+H_b}{2}}{\frac{H_{hexamethylbenzene}}{18}} \quad \text{(eq S6)}$$

**Table S3. $^1$H NMR Integration and ln(Fraction Remaining) Values Used to Determine Pseudo-first Order Rate Constant for Reaction of Cysteamine with Lactam 45**

| | | Integration Values | | | | |
|---|---|---|---|---|---|---|
| Time | Time Point | $H_a$ | $H_b$ | $H_c$+$H_d$ | | |
| mm/dd/yy hh:mm | d:h:min | δ 5.95 | δ 5.25 | δ 4.8 | Fraction Remaining | ln(Fraction Remaining) |
| 6/3/15 12:55 | 1:0:00 | 1 | 1.03 | 1.57 | 0.646497 | -0.43619 |
| 6/4/15 10:10 | 1:21:15 | 1 | 1.02 | 2.07 | 0.487923 | -0.7176 |
| 6/5/15 10:30 | 2:21:35 | 1 | 1 | 2.54 | 0.393701 | -0.93216 |
| 6/8/15 11:41 | 5:22:46 | 1 | 1.22 | 8.56 | 0.129673 | -2.04274 |

**Figure S1. $^1$H NMR Spectra at Reaction Time Points for Cysteamine and Lactam 45**

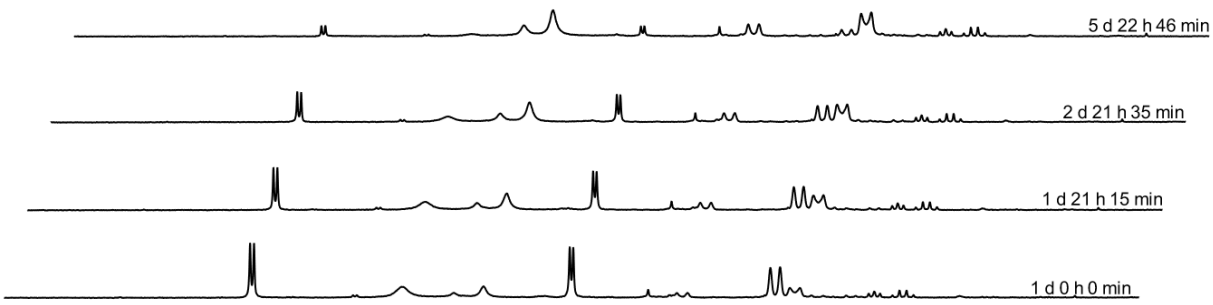


**Figure S2. Fraction Remaining Values Plotted Against Time Points for Lactam 45**

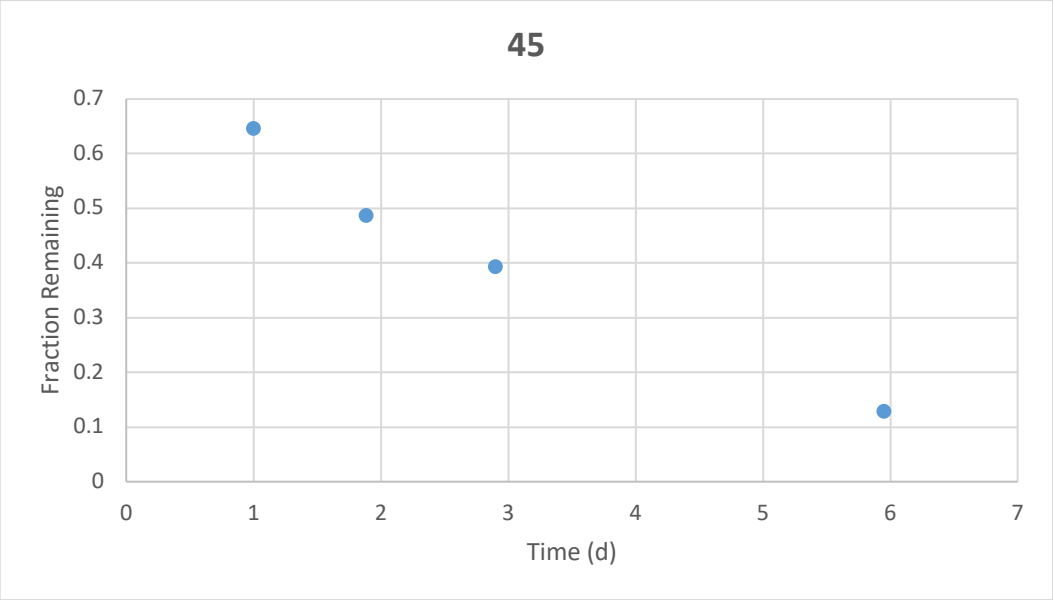
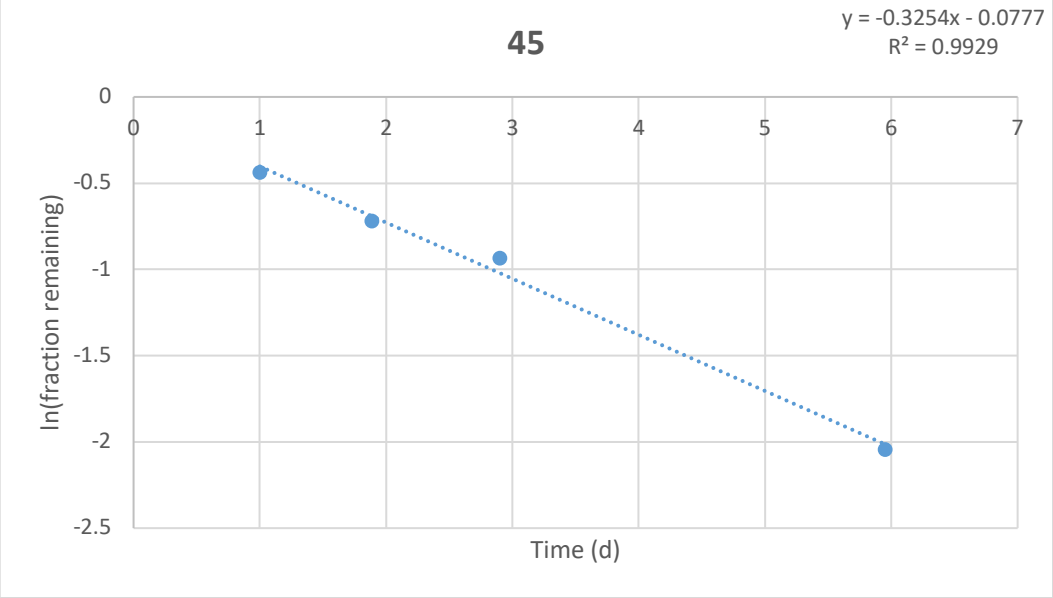
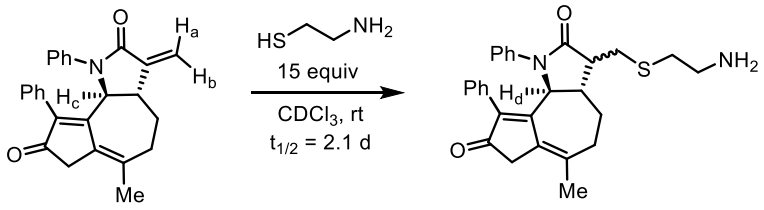



**Figure S3. ln(Fraction Remaining) Values Plotted Against Time Points for Lactam 45**






**49** **S19**

**Table S4. $^1$H NMR Integration and ln(Fraction Remaining) Values Used to Determine Pseudo-first Order Rate Constant for Reaction of Cysteamine with Lactam 49**

| | | Integration values | | | | |
|---|---|---|---|---|---|---|
| **Time** | **Time Point** | **$H_a$** | **$H_b$** | **$H_c$+$H_d$** | | |
| **mm/dd/yy hh:mm** | **min** | **δ 6.24** | **δ 5.46** | **δ 5.35** | **Fraction Remaining** | **ln(Fraction Remaining)** |
| 5/22/19 12:05 | 0:0:00 | 1 | 1.06 | 1.07 | 0.962617 | -0.0381 |
| 5/22/19 12:07 | 0:0:02 | 1 | 1.1 | 1.07 | 0.981308 | -0.01887 |
| 5/22/19 12:43 | 0:0:38 | 1 | 1.1 | 1.08 | 0.972222 | -0.02817 |
| 5/22/19 12:45 | 0:0:40 | 1 | 1.04 | 1.07 | 0.953271 | -0.04786 |
| 5/22/19 13:36 | 0:1:31 | 1 | 1.06 | 1.11 | 0.927928 | -0.0748 |
| 5/22/19 13:38 | 0:1:33 | 1 | 1.06 | 1.12 | 0.919643 | -0.08377 |
| 5/22/19 14:06 | 0:2:01 | 1 | 1.06 | 1.12 | 0.919643 | -0.08377 |
| 5/22/19 18:05 | 0:6:00 | 1 | 1.06 | 1.2 | 0.858333 | -0.15276 |
| 5/23/19 7:34 | 0:19:29 | 1 | 1.12 | 1.53 | 0.69281 | -0.367 |
| 5/23/19 9:39 | 0:21:34 | 1 | 1.19 | 1.65 | 0.663636 | -0.41002 |
| 5/23/19 9:41 | 0:21:36 | 1 | 1.15 | 1.61 | 0.667702 | -0.40391 |
| 5/23/19 13:44 | 1:1:39 | 1 | 1.13 | 1.73 | 0.615607 | -0.48515 |
| 5/23/19 13:46 | 1:1:41 | 1 | 1.16 | 1.76 | 0.613636 | -0.48835 |
| 5/23/19 15:31 | 1:0:00 | 1 | 1.17 | 1.82 | 0.596154 | -0.51726 |
| 5/23/19 15:33 | 1:3:28 | 1 | 1.16 | 1.75 | 0.617143 | -0.48265 |
| 5/23/19 16:49 | 1:4:44 | 1 | 1.15 | 1.89 | 0.568783 | -0.56426 |
| 5/23/19 16:51 | 1:4:46 | 1 | 1.13 | 1.81 | 0.588398 | -0.53035 |
| 5/24/19 10:32 | 1:22:27 | 1 | 1.18 | 2.41 | 0.452282 | -0.79345 |
| 5/24/19 10:34 | 1:22:29 | 1 | 1.19 | 2.37 | 0.462025 | -0.77214 |
| 5/24/19 13:43 | 2:1:38 | 1 | 1.26 | 2.62 | 0.431298 | -0.84096 |
| 5/24/19 15:36 | 2:3:31 | 1 | 1.27 | 2.71 | 0.418819 | -0.87032 |
| 5/24/19 17:56 | 2:5:51 | 1 | 1.27 | 2.77 | 0.409747 | -0.89221 |
| 5/25/19 13:35 | 3:1:30 | 1 | 1.35 | 3.77 | 0.311671 | -1.16581 |
| 5/25/19 22:16 | 3:10:11 | 1 | 1.33 | 4.11 | 0.283455 | -1.2607 |
| 5/25/19 22:18 | 3:10:13 | 1 | 1.41 | 4.24 | 0.284198 | -1.25808 |
| 5/26/19 11:16 | 3:23:11 | 1 | 1.57 | 5.16 | 0.249031 | -1.39018 |
| 5/26/19 11:18 | 3:23:13 | 1 | 1.51 | 5.02 | 0.25 | -1.38629 |
| 5/27/19 10:19 | 4:22:14 | 1 | 1.74 | 6.75 | 0.202963 | -1.59473 |

**Figure S4. $^{1}$H NMR Spectra at Reaction Time Points for Cysteamine and Lactam 49**

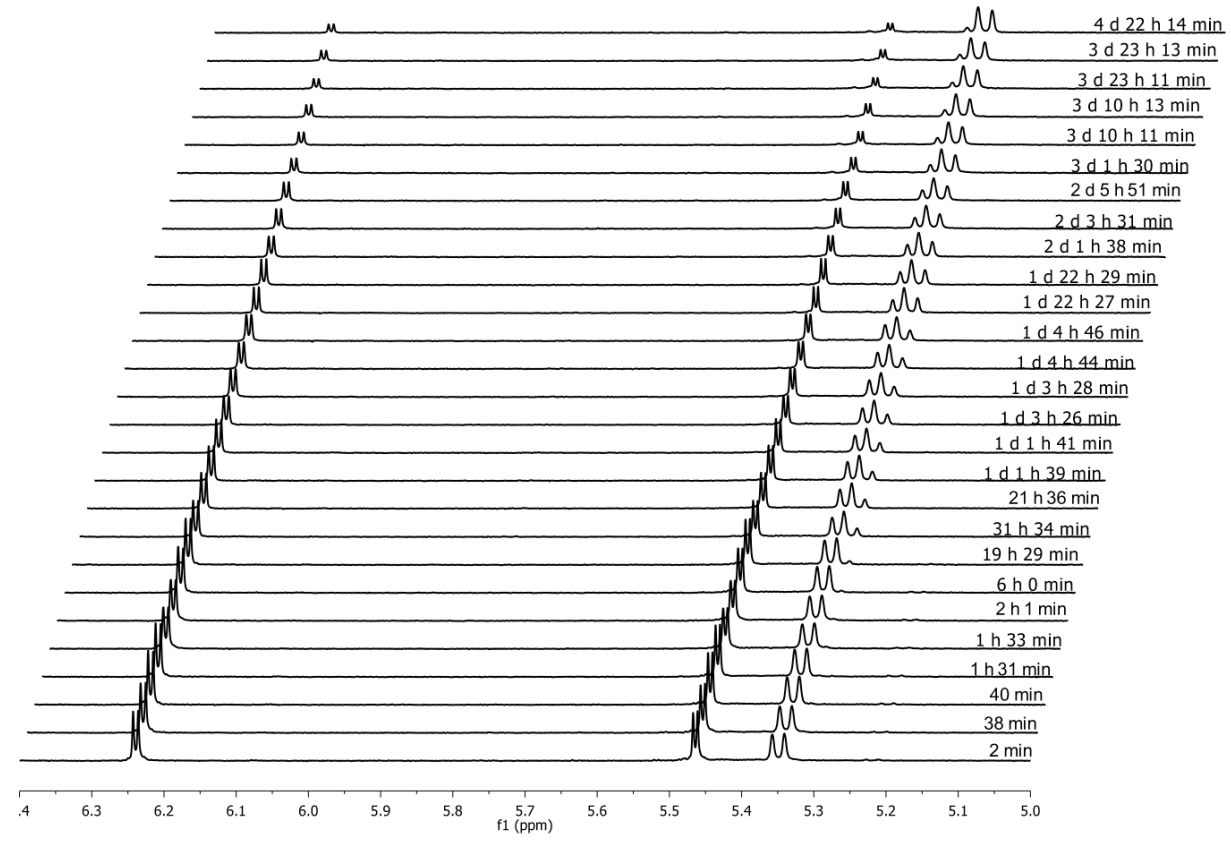


**Figure S5. Fraction Remaining Values Plotted Against Time Points for Lactam 49**

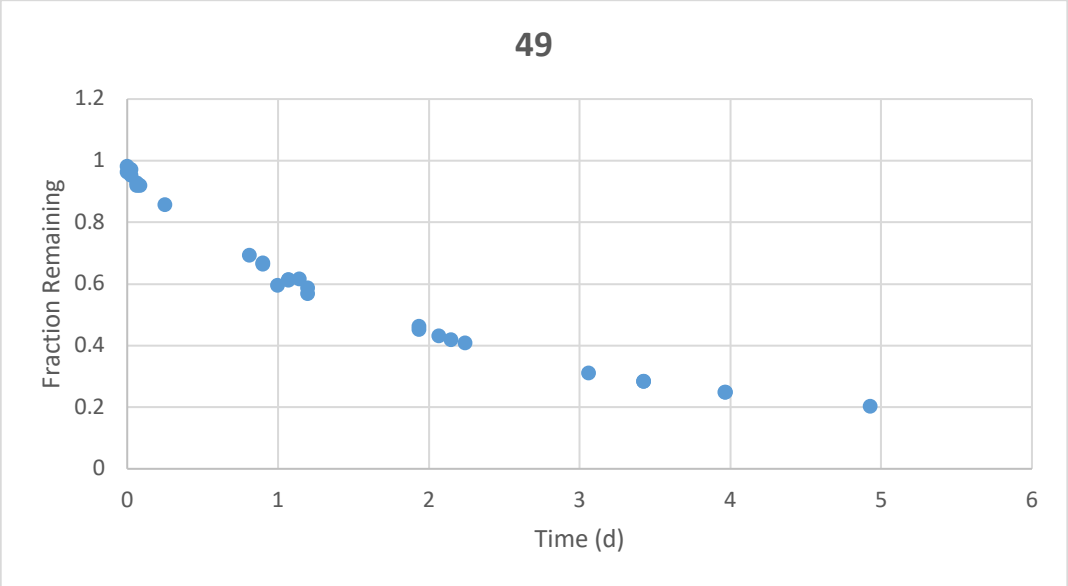



**Figure S6. ln(Fraction Remaining) Values Plotted Against Time Points for Lactam 49**

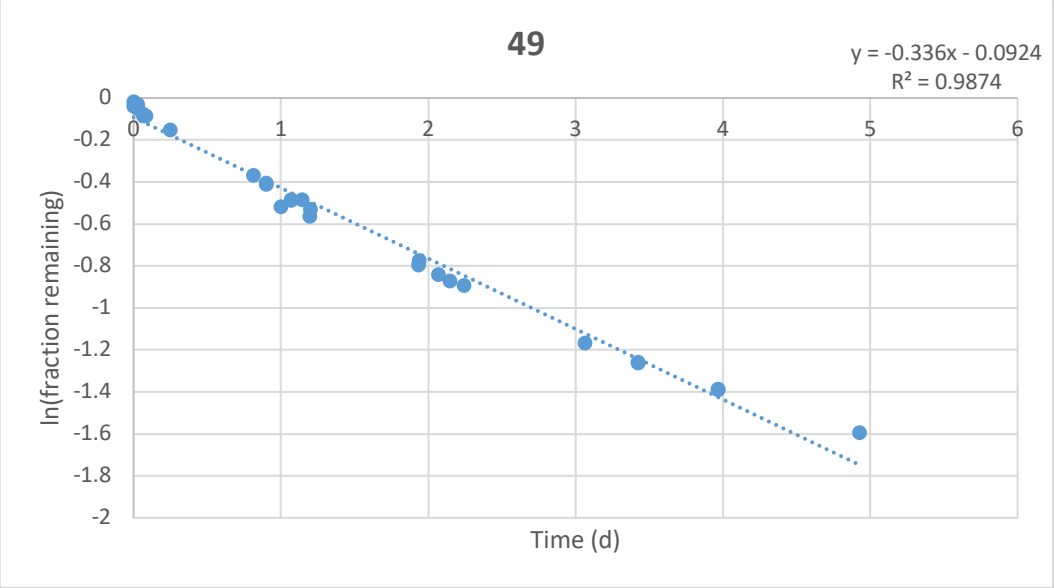


**Table S5. $^1$H NMR Integration and ln(Fraction Remaining) Values Used to Determine Pseudo-first Order Rate Constant for Reaction of Cysteamine with Lactam 49 and Internal Standard**

| | | Integration Values | | | | |
|---|---|---|---|---|---|---|
| **Time** | **Time Point** | **$H_a$** | **$H_b$** | **Internal standard** | **Fraction Remaining** | **ln(Fraction Remaining)** |
| **m/dd/yy hh:mm** | **d:h:min** | δ 6.18 | δ 5.4 | δ 2.2 | | |
| 5/22/2019 12:05 | 0:0:00 | 1 | 1.06 | 32.59 | 1.011353 | 0.011289 |
| 5/22/2019 12:07 | 0:0:02 | 1 | 1.1 | 33.12 | 1.014493 | 0.014389 |
| 5/22/2019 12:43 | 0:0:38 | 1 | 1.1 | 33.79 | 0.994377 | -0.00564 |
| 5/22/2019 12:45 | 0:0:40 | 1 | 1.04 | 33.72 | 0.967972 | -0.03255 |
| 5/22/2019 13:36 | 0:1:31 | 1 | 1.06 | 34.53 | 0.954532 | -0.04653 |
| 5/22/2019 13:38 | 0:1:33 | 1 | 1.06 | 35.12 | 0.938497 | -0.06348 |
| 5/22/2019 14:06 | 0:2:01 | 1 | 1.06 | 35.04 | 0.940639 | -0.0612 |
| 5/22/2019 18:05 | 0:6:00 | 1 | 1.06 | 38.49 | 0.856326 | -0.1551 |
| 5/23/2019 7:34 | 0:19:29 | 1 | 1.12 | 50.04 | 0.677858 | -0.38882 |
| 5/23/2019 9:39 | 0:21:34 | 1 | 1.19 | 53.7 | 0.652514 | -0.42692 |
| 5/23/2019 | 0:21:36 | 1 | 1.15 | 52.37 | 0.656865 | -0.42028 |
| 5/23/2019 13:44 | 1:1:39 | 1 | 1.13 | 56.2 | 0.606406 | -0.50021 |
| 5/23/2019 13:46 | 1:1:41 | 1 | 1.16 | 56.51 | 0.611573 | -0.49172 |
| 5/23/2019 15:31 | 1:0:00 | 1 | 1.17 | 57.77 | 0.601004 | -0.50915 |
| 5/23/2019 15:33 | 1:3:28 | 1 | 1.16 | 56.65 | 0.610062 | -0.4942 |
| 5/23/2019 16:49 | 1:4:44 | 1 | 1.15 | 59.86 | 0.574674 | -0.55395 |
| 5/23/2019 16:51 | 1:4:46 | 1 | 1.13 | 59.34 | 0.574317 | -0.55457 |
| 5/24/2019 10:32 | 1:22:27 | 1 | 1.18 | 78.15 | 0.446321 | -0.80672 |
| 5/24/2019 10:34 | 1:22:29 | 1 | 1.19 | 78.49 | 0.446426 | -0.80648 |
| 5/24/2019 13:43 | 2:1:38 | 1 | 1.26 | 83.98 | 0.430579 | -0.84263 |
| 5/24/2019 15:36 | 2:3:31 | 1 | 1.27 | 88.54 | 0.41021 | -0.89109 |
| 5/24/2019 17:56 | 2:5:51 | 1 | 1.27 | 95.41 | 0.380673 | -0.96581 |
| 5/25/2019 13:35 | 3:1:30 | 1 | 1.35 | 124.4 | 0.302251 | -1.1965 |
| 5/25/2019 22:16 | 3:10:11 | 1 | 1.33 | 144.36 | 0.258243 | -1.35385 |
| 5/25/2019 | 3:10:13 | 1 | 1.41 | 146.97 | 0.262366 | -1.33801 |
| 5/26/2019 11:16 | 3:23:11 | 1 | 1.57 | 184.74 | 0.222583 | -1.50245 |
| 5/26/2019 11:18 | 3:23:13 | 1 | 1.51 | 178.01 | 0.225605 | -1.48897 |
| 5/27/2019 10:19 | 4:22:14 | 1 | 1.74 | 248.15 | 0.176667 | -1.73349 |

**Figure S7. $^{1}$H NMR Spectra at Reaction Time Points for Cysteamine and Lactam 49 with Internal Standard**

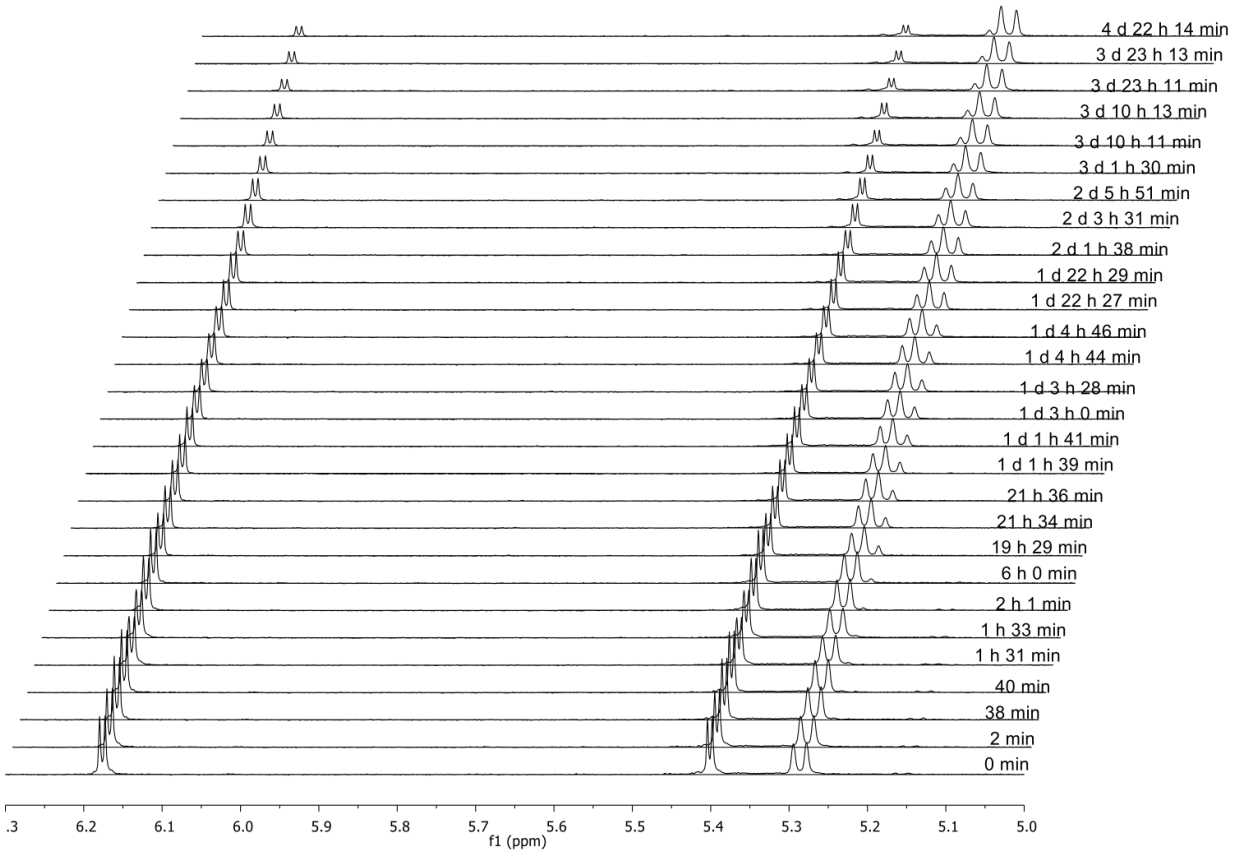


**Figure S8. Fraction Remaining Values Plotted Against Time Points for Lactam 49**

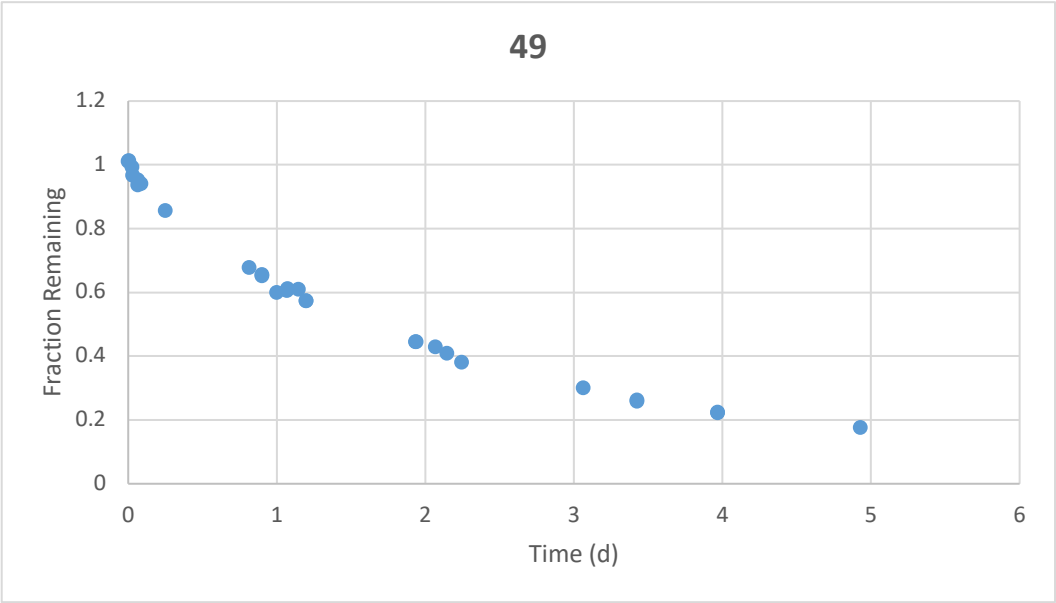
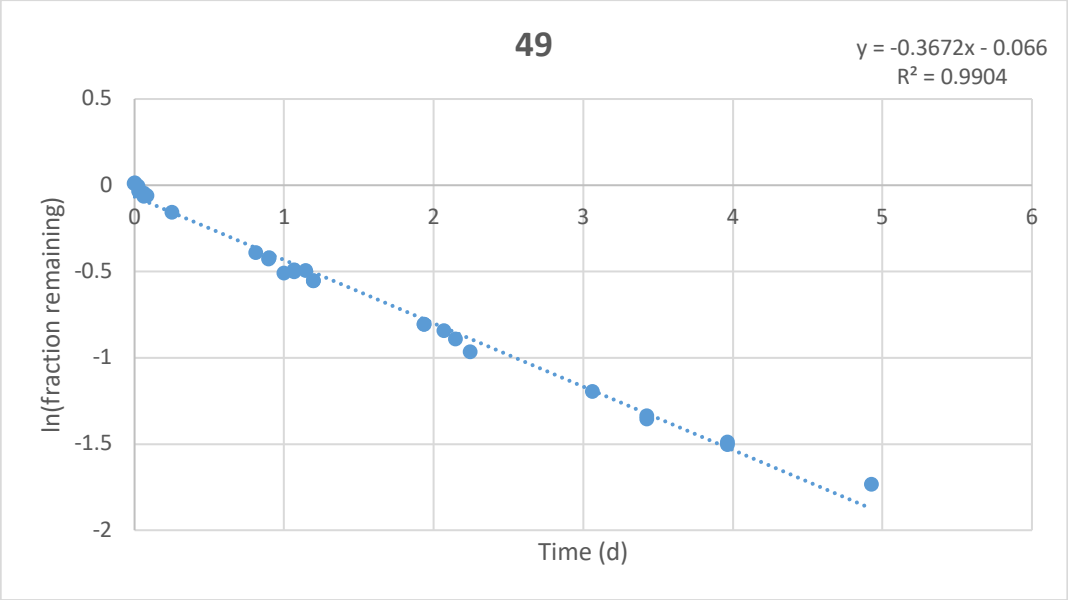
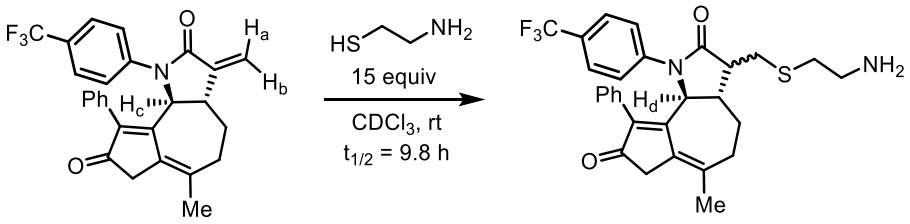



**Figure S9. ln(Fraction Remaining) Values Plotted Against Time Points for Lactam 49**






51 S20

**Table S6. $^1$H NMR Integration and ln(Fraction Remaining) Values Used to Determine Pseudo-first Order Rate Constant for Reaction of Cysteamine with Lactam 51**

| | | Integration | | | | |
|---|---|---|---|---|---|---|
| Time | Timepoint | Proton Ha | Proton $H_b$ | Proton Hc+HD | | |
| m/dd/yy hh:mm | d:h:min | 6.28 ppm | 5.52 ppm | 5.34 ppm | %rem | ln%rem |
| 5/14/18 13:38 | 0:0:02 | 1 | 1 | 1.15 | 0.869565 | -0.13976 |
| 5/14/18 14:55 | 0:1:19 | 1 | 0.98 | 1.31 | 0.755725 | -0.28008 |
| 5/14/18 15:43 | 0:2:07 | 1 | 1.09 | 1.49 | 0.701342 | -0.35476 |
| 5/14/18 16:42 | 0:3:06 | 1 | 1.07 | 1.56 | 0.663462 | -0.41028 |
| 5/14/18 16:51 | 0:3:15 | 1 | 1.06 | 1.62 | 0.635802 | -0.45287 |
| 5/14/18 16:53 | 0:3:17 | 1 | 1 | 1.61 | 0.621118 | -0.47623 |
| 5/15/18 2:25 | 0:12:49 | 1 | 1.09 | 3.2 | 0.326563 | -1.11913 |
| 5/15/18 8:44 | 0:19:08 | 1 | 1.04 | 5.32 | 0.191729 | -1.65167 |
| 5/15/18 9:40 | 0:20:04 | 1 | 1.11 | 5.94 | 0.177609 | -1.72817 |
| 5/15/18 10:43 | 0:21:07 | 1 | 0.86 | 4.82 | 0.192946 | -1.64534 |
| 5/15/18 11:32 | 0:21:56 | 1 | 1.18 | 6.19 | 0.17609 | -1.73676 |
| 5/15/18 12:34 | 0:22:58 | 1 | 1.02 | 6.27 | 0.161085 | -1.82583 |
| 5/15/18 14:37 | 1:0:59 | 1 | 0.94 | 6.39 | 0.1518 | -1.88519 |

**Figure S10. $^{1}$H NMR Spectra at Reaction Time Points for Cysteamine and Lactam 51**

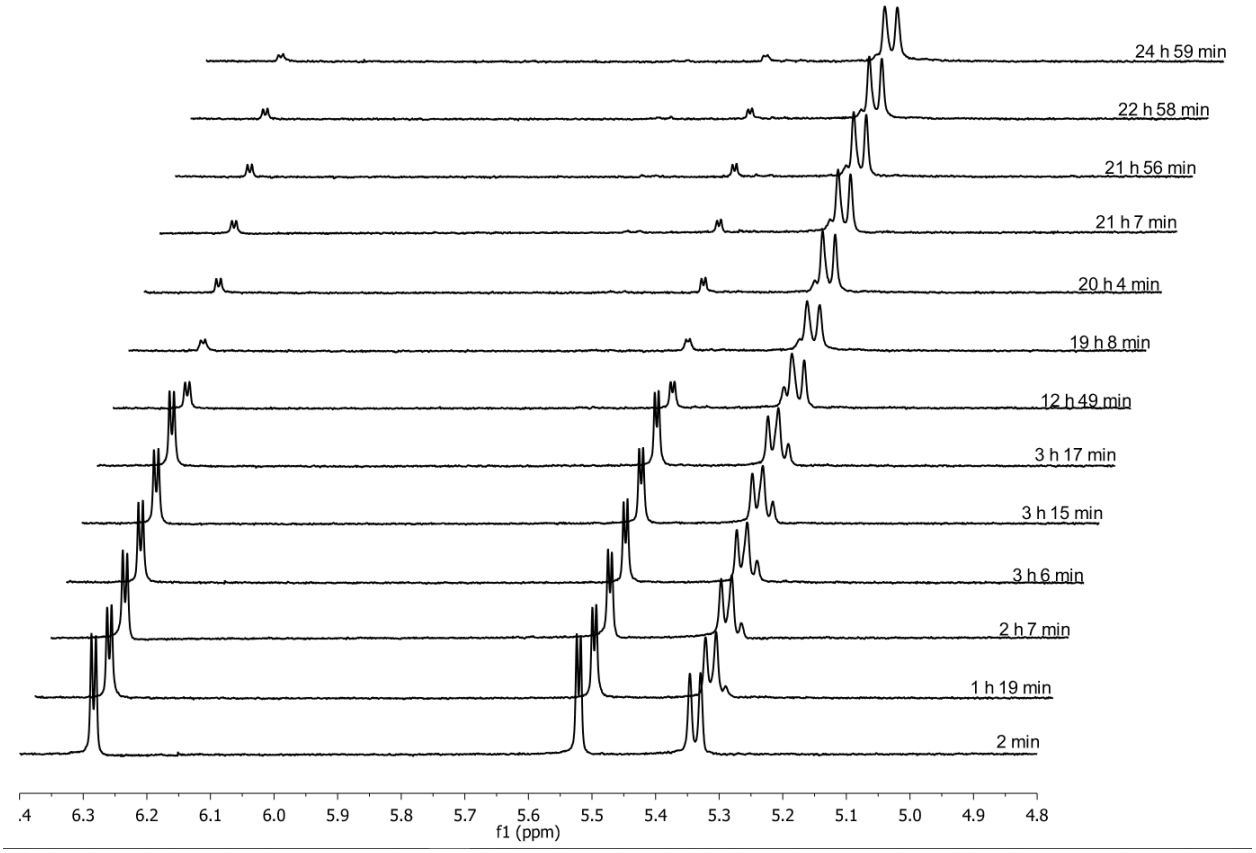


**Figure S11. Fraction Remaining Values Plotted Against Time Points for Lactam 51**

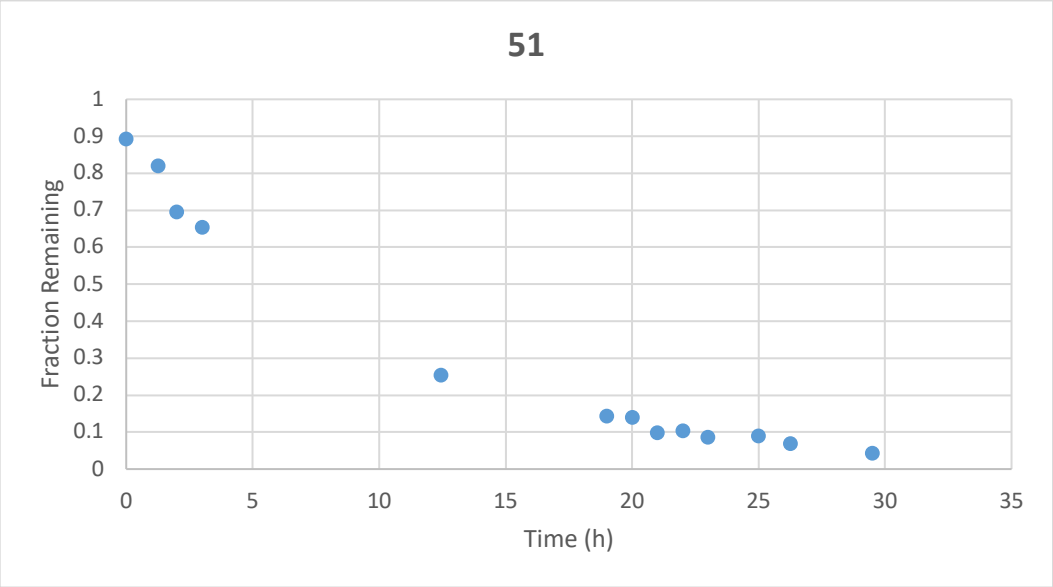
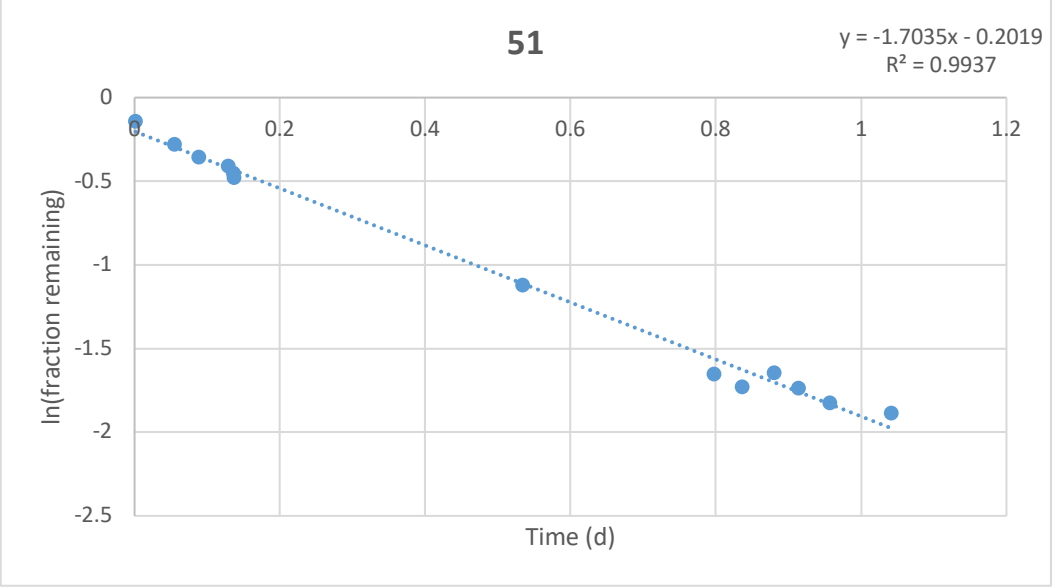
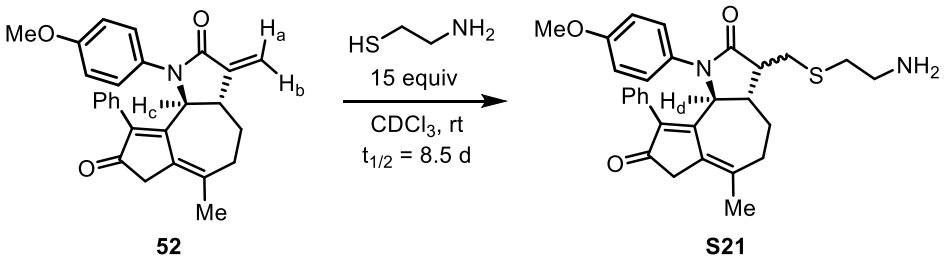



**Figure S12. ln(Fraction Remaining) Values Plotted Against Time Points for Lactam 51**






52 S21

**Table S7. $^1$H NMR Integration and ln(Fraction Remaining) Values Used to Determine Pseudo-first Order Rate Constant for Reaction of Cysteamine with Lactam 52**

| | | Integration Values | | | | |
|---|---|---|---|---|---|---|
| **Time** | **Time Point** | **$H_a$** | **$H_b$** | **$H_c$+$H_d$** | | |
| **mm/dd/yy hh:mm** | **d:h:min** | **δ 6.22** | **δ 5.45** | **δ 5.31** | **Fraction Remaining** | **ln(Fraction Remaining)** |
| 4/11/19 14:25 | 0:0:02 | 1 | 1.03 | 1.06 | 0.957547 | -0.04338 |
| 4/11/19 14:28 | 0:0:05 | 1 | 1.04 | 1.09 | 0.93578 | -0.06638 |
| 4/11/19 14:30 | 0:0:07 | 1 | 1.03 | 1.07 | 0.948598 | -0.05277 |
| 4/11/19 14:45 | 0:0:22 | 1 | 1.03 | 1.09 | 0.931193 | -0.07129 |
| 4/11/19 14:47 | 0:0:24 | 1 | 1.03 | 1.09 | 0.931193 | -0.07129 |
| 4/11/19 15:08 | 0:0:45 | 1 | 1.03 | 1.09 | 0.931193 | -0.07129 |
| 4/11/19 15:10 | 0:0:47 | 1 | 1.03 | 1.07 | 0.948598 | -0.05277 |
| 4/11/19 16:39 | 0:2:16 | 1 | 1.06 | 1.1 | 0.936364 | -0.06575 |
| 4/11/19 16:41 | 0:2:18 | 1 | 1.05 | 1.12 | 0.915179 | -0.08864 |
| 4/12/19 8:59 | 0:18:36 | 1 | 1.09 | 1.23 | 0.849593 | -0.163 |
| 4/12/19 10:13 | 0:19:50 | 1 | 1.09 | 1.26 | 0.829365 | -0.18709 |
| 4/12/19 10:15 | 0:19:52 | 1 | 1.03 | 1.23 | 0.825203 | -0.19213 |
| 4/12/19 10:17 | 0:19:54 | 1 | 1.05 | 1.2 | 0.854167 | -0.15763 |
| 4/12/19 12:52 | 0:22:29 | 1 | 1.04 | 1.21 | 0.842975 | -0.17082 |
| 4/12/19 12:54 | 0:22:31 | 1 | 1.05 | 1.26 | 0.813492 | -0.20642 |
| 4/12/19 13:47 | 0:23:24 | 1 | 1.05 | 1.25 | 0.82 | -0.19845 |
| 4/12/19 16:04 | 1:1:41 | 1 | 1.05 | 1.25 | 0.82 | -0.19845 |
| 4/12/19 16:52 | 1:2:29 | 1 | 1.07 | 1.26 | 0.821429 | -0.19671 |
| 4/13/19 12:01 | 1:21:38 | 1 | 1.07 | 1.32 | 0.784091 | -0.24323 |
| 4/13/19 14:13 | 1:23:50 | 1 | 1.09 | 1.34 | 0.779851 | -0.24865 |
| 4/13/19 17:46 | 2:3:23 | 1 | 1.04 | 1.26 | 0.809524 | -0.21131 |
| 4/13/19 17:48 | 2:3:25 | 1 | 1.05 | 1.27 | 0.807087 | -0.21432 |
| 4/14/19 12:29 | 2:22:06 | 1 | 1.06 | 1.38 | 0.746377 | -0.29252 |
| 4/14/19 12:32 | 2:22:09 | 1 | 1.09 | 1.4 | 0.746429 | -0.29246 |
| 4/15/19 10:10 | 3:19:47 | 1 | 1.09 | 1.56 | 0.669872 | -0.40067 |
| 4/15/19 10:13 | 3:19:50 | 1 | 1.07 | 1.54 | 0.672078 | -0.39738 |
| 4/15/19 13:54 | 3:23:31 | 1 | 1.08 | 1.6 | 0.65 | -0.43078 |
| 4/16/19 9:37 | 4:19:14 | 1 | 1.08 | 1.64 | 0.634146 | -0.45548 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 4/16/19 11:51 | 4:21:28 | 1 | 1.09 | 1.67 | 0.625749 | -0.46881 |
| 4/16/19 17:27 | 5:3:04 | 1 | 1.08 | 1.7 | 0.611765 | -0.49141 |
| 4/16/19 21:07 | 5:6:44 | 1 | 1.11 | 1.76 | 0.599432 | -0.51177 |
| 4/17/19 10:14 | 5:19:51 | 1 | 1.11 | 1.84 | 0.57337 | -0.55622 |

**Figure S13. $^{1}$H NMR Spectra at Reaction Time Points for Cysteamine and Lactam 52**

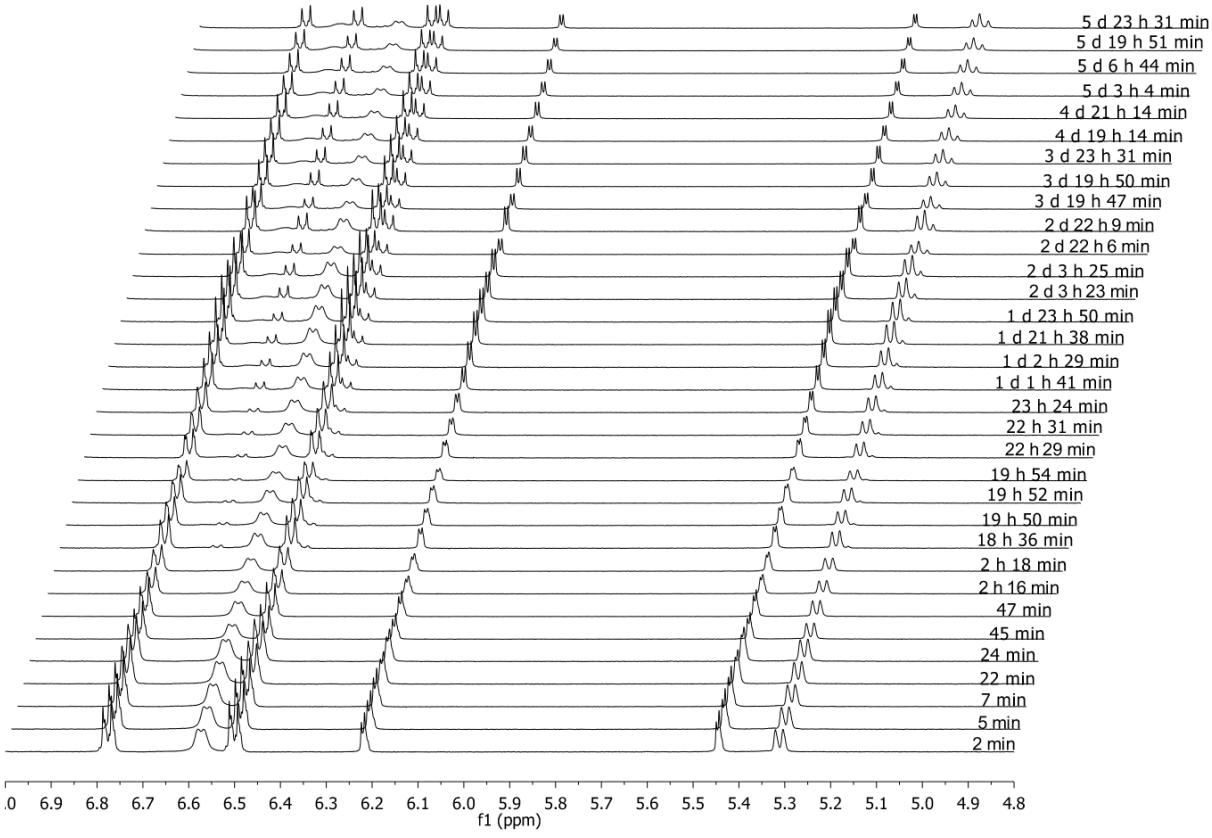


**Figure S14. Fraction Remaining Values Plotted Against Time Points for Lactam 52**

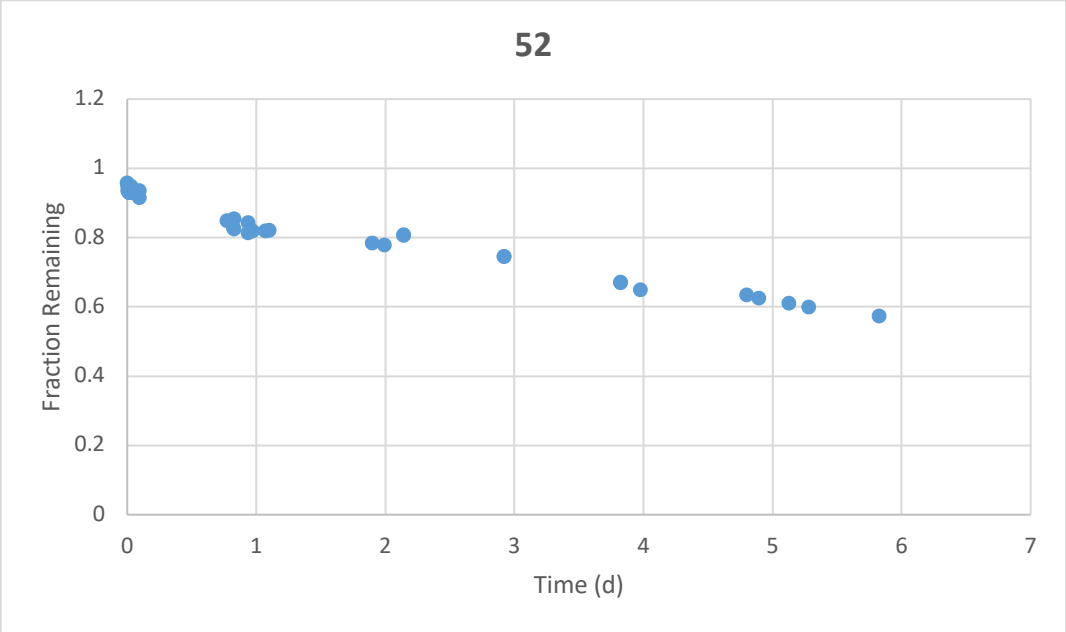



**Figure S15. ln(Fraction Remaining) Values Plotted Against Time Points for Lactam 52**

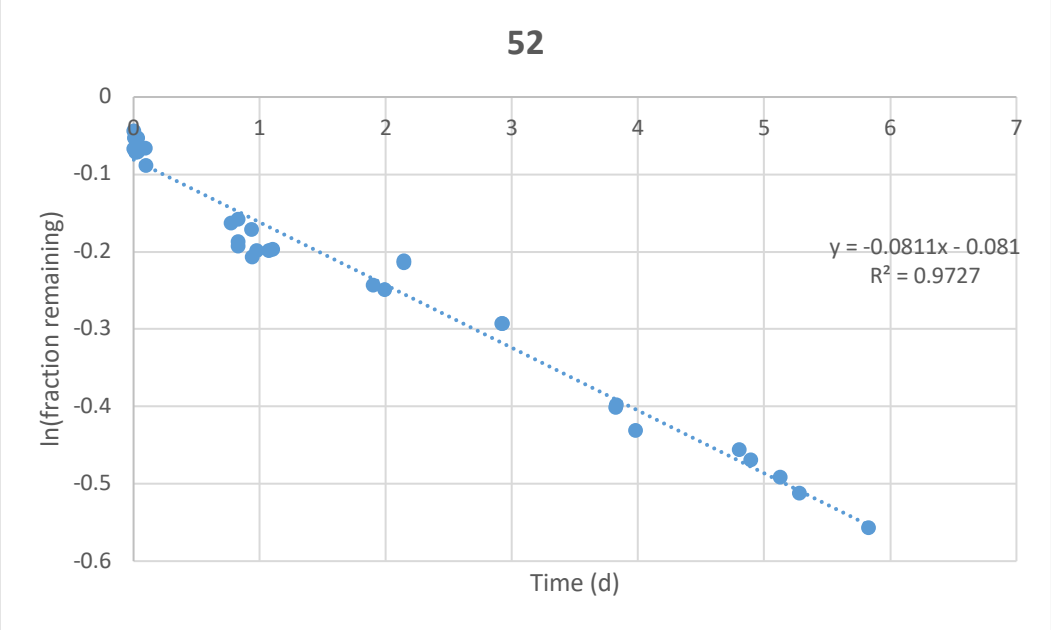


**Table S8. $^1$H NMR Integration and ln(Fraction Remaining) Values Used to Determine Pseudo-first Order Rate Constant for Reaction of Cysteamine with Lactam 52 with Internal Standard**

| | | Integration Values | | | | |
|---|---|---|---|---|---|---|
| **Time** | **Time Point** | **$H_a$** | **$H_b$** | **Internal standard** | **Fraction Remaining** | **ln(Fraction Remaining)** |
| **m/dd/yy hh:mm** | **d:h:min** | δ 6.22 | δ 5.45 | δ 2.20 | | |
| 5/3/2019 16:19 | 0:0:00 | 1 | 1.02 | 17.83 | 1.01963 | 0.01944 |
| 5/4/2019 12:07 | 0:19:48 | 1 | 1.07 | 20.23 | 0.92091 | -0.08239 |
| 5/4/2019 15:05 | 0:22:46 | 1 | 1.13 | 20.6 | 0.930583 | -0.07194 |
| 5/4/2019 15:07 | 0:22:48 | 1 | 1.05 | 20.63 | 0.894329 | -0.11168 |
| 5/6/2019 9:51 | 2:17:32 | 1 | 1.12 | 25.26 | 0.755344 | -0.28058 |
| 5/7/2019 13:30 | 3:21:11 | 1 | 1.19 | 27.59 | 0.714389 | -0.33633 |
| 5/8/2019 13:42 | 4:21:23 | 1 | 1.03 | 28.02 | 0.652034 | -0.42766 |
| 5/9/2019 8:14 | 5:15:55 | 1 | 1.12 | 32.13 | 0.593838 | -0.52115 |
| 5/9/2019 14:34 | 5:22:15 | 1 | 1.22 | 34.19 | 0.584381 | -0.5372 |
| 5/10/2019 4:08 | 6:11:49 | 1 | 1.31 | 36.35 | 0.571939 | -0.55872 |

**Figure S16. $^{1}$H NMR Spectra at Reaction Time Points for Cysteamine and Lactam 52 with Internal Standard**

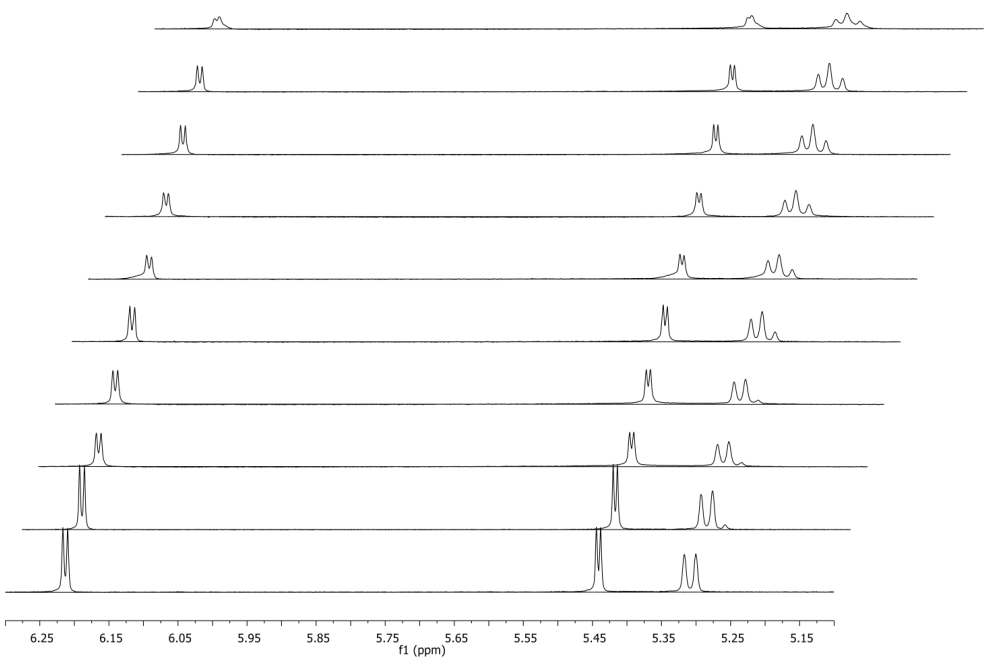


**Figure S17. Fraction Remaining Values Plotted Against Time Points for Lactam 52 with Internal Standard**

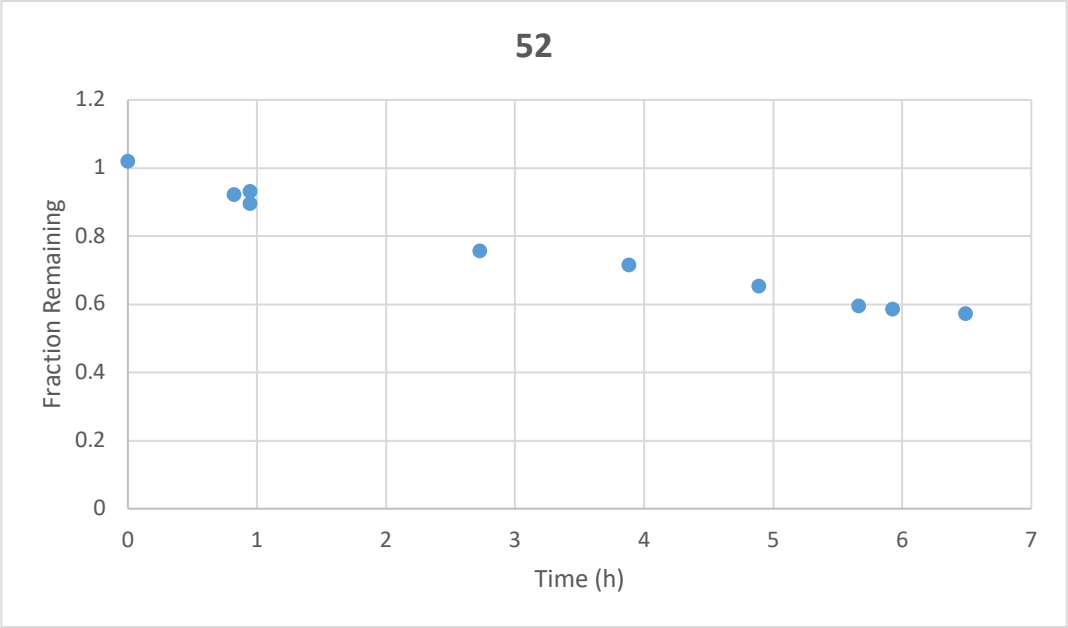
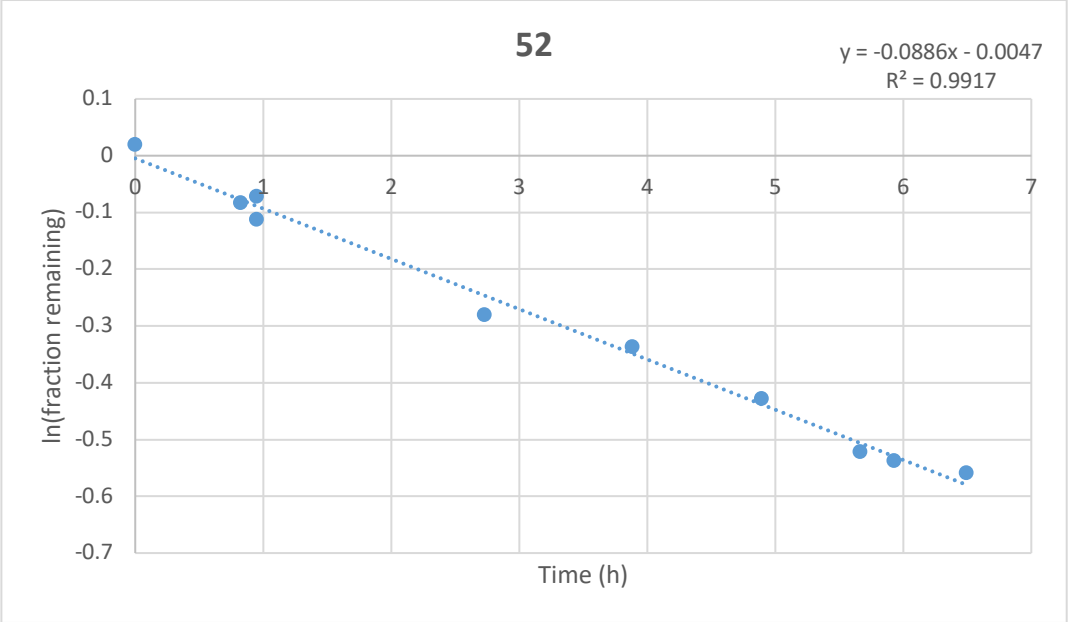
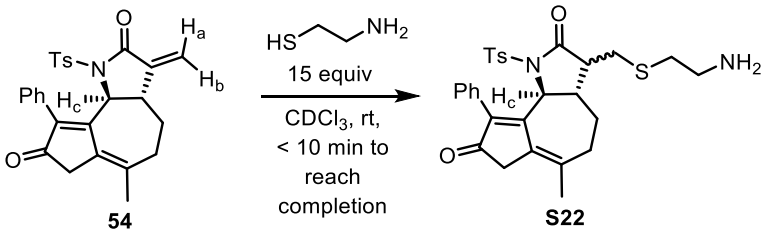



**Figure S18. ln(Fraction Remaining) Values Plotted Against Time Points for Lactam 52 with Internal Standard**





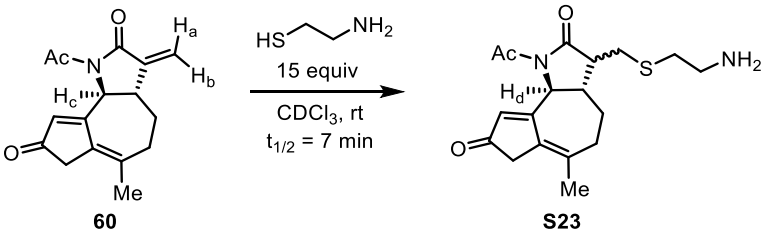




**Table S9. $^1$H NMR Integration and ln(Fraction Remaining) Values Used to Determine Pseudo-first Order Rate Constant for Reaction of Cysteamine with Lactam 60**

| | | Integration Values | | | | |
|---|---|---|---|---|---|---|
| Time | Time Point | $H_a$ | $H_b$ | $H_c$+$H_d$ | Fraction Remaining | ln(Fraction Remaining) |
| m/dd/yy hh:mm | min | δ 6.28 | δ 5.50 | δ 5.06 | | |
| 5/17/18 15:12 | 4 | 1 | 0.82 | 2.17 | 0.419354839 | -0.869037847 |
| 5/17/18 15:14 | 6 | 1 | 1.07 | 3.5 | 0.295714286 | -1.218361542 |
| 5/17/18 15:16 | 8 | 1 | 0.83 | 3.81 | 0.24015748 | -1.426460403 |

**Figure S19. $^1$H NMR Spectra at Reaction Time Points for Cysteamine and Lactam 60**

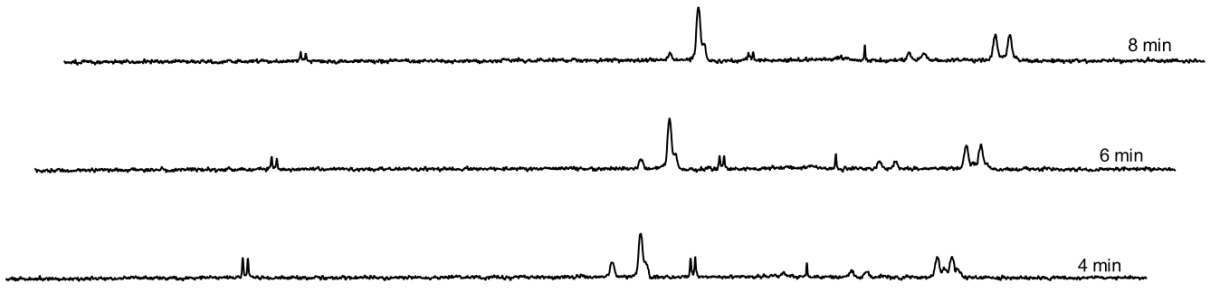


**Figure S20. Fraction Remaining Values Plotted Against Time Points for Lactam 60**

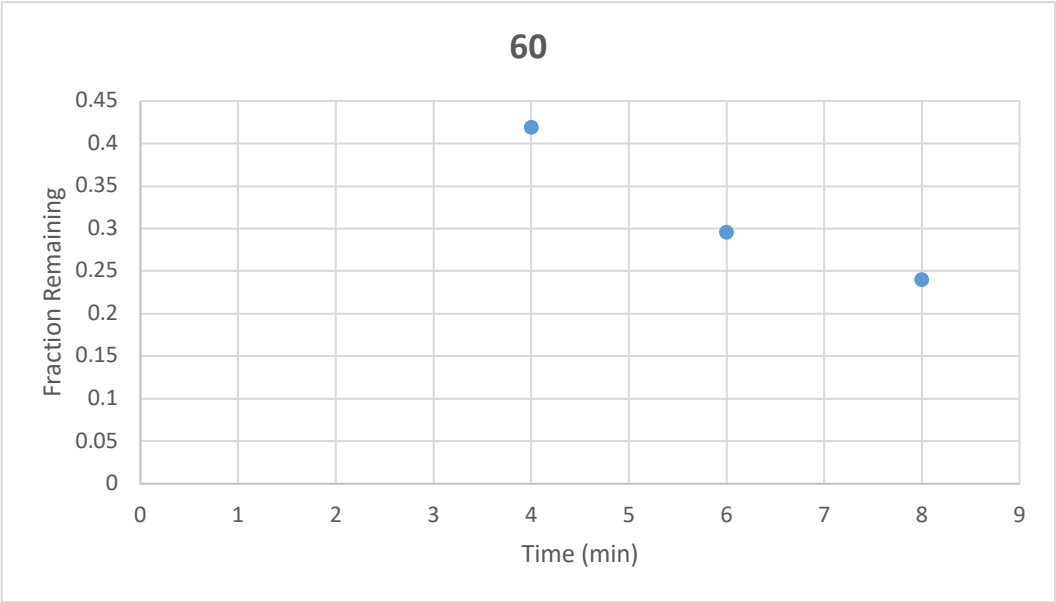
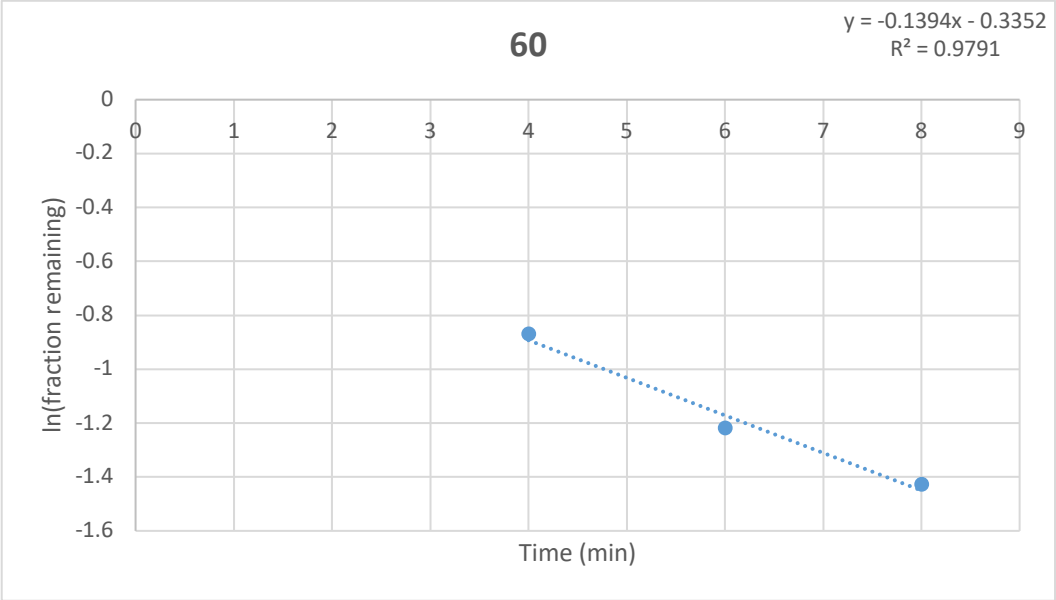
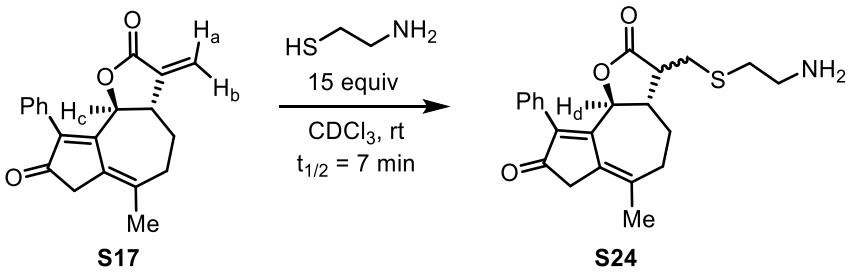



**Figure S21. ln(Fraction Remaining) Values Plotted Against Time Points for Lactam 60**






**S17** **S24**

**Table S10. $^1$H NMR Integration and ln(Fraction Remaining) Values Used to Determine Pseudo-first Order Rate Constant for Reaction of Cysteamine with Lactone S17**

| | Integration Values | | | | |
|---|---|---|---|---|---|
| **Time Point** | **$H_a$** | **$H_b$** | **$H_c$+$H_d$** | **Fraction Remaining** | **ln(Fraction Remaining)** |
| **min** | δ 6.22 | δ 5.53 | δ 5.45 | | |
| 3 | 1 | 1 | 1.15 | 0.869565 | -0.13976 |
| 6 | 1 | 0.98 | 1.31 | 0.755725 | -0.28008 |
| 8 | 1 | 1.09 | 1.49 | 0.701342 | -0.35476 |
| 10 | 1 | 1.07 | 1.56 | 0.663462 | -0.41028 |
| 12 | 1 | 1.06 | 1.62 | 0.635802 | -0.45287 |
| 14 | 1 | 1 | 1.61 | 0.621118 | -0.47623 |
| 17 | 1 | 1.09 | 3.2 | 0.326563 | -1.11913 |
| 19 | 1 | 1.04 | 5.32 | 0.191729 | -1.65167 |
| 21 | 1 | 1.11 | 5.94 | 0.177609 | -1.72817 |
| 24 | 1 | 0.86 | 4.82 | 0.192946 | -1.64534 |
| 26 | 1 | 1.18 | 6.19 | 0.17609 | -1.73676 |
| 28 | 1 | 1.02 | 6.27 | 0.161085 | -1.82583 |
| 30 | 1 | 0.94 | 6.39 | 0.1518 | -1.88519 |

**Figure 22. $^{1}$H NMR Spectra at Reaction Time Points for Cysteamine and Lactone S17**

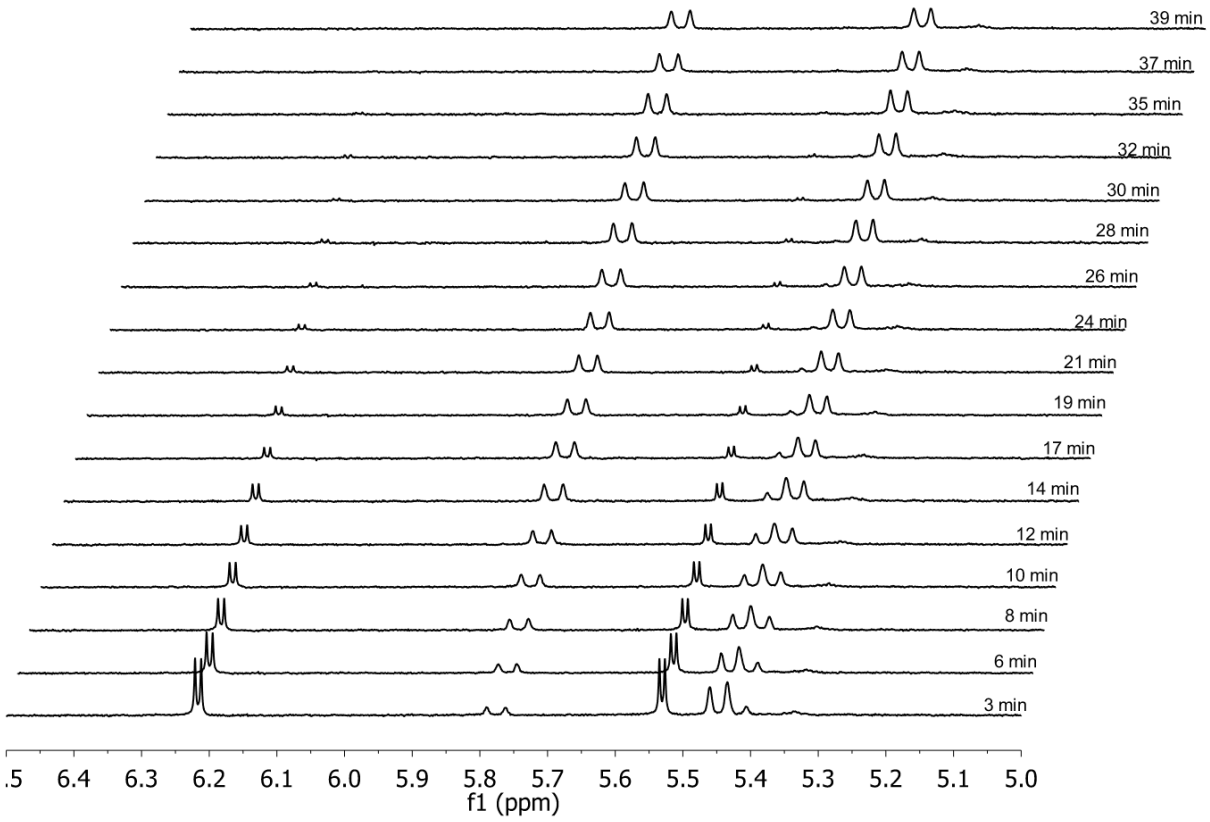


**Figure S23. Fraction Remaining Values Plotted Against Time Points for Lactone S17**

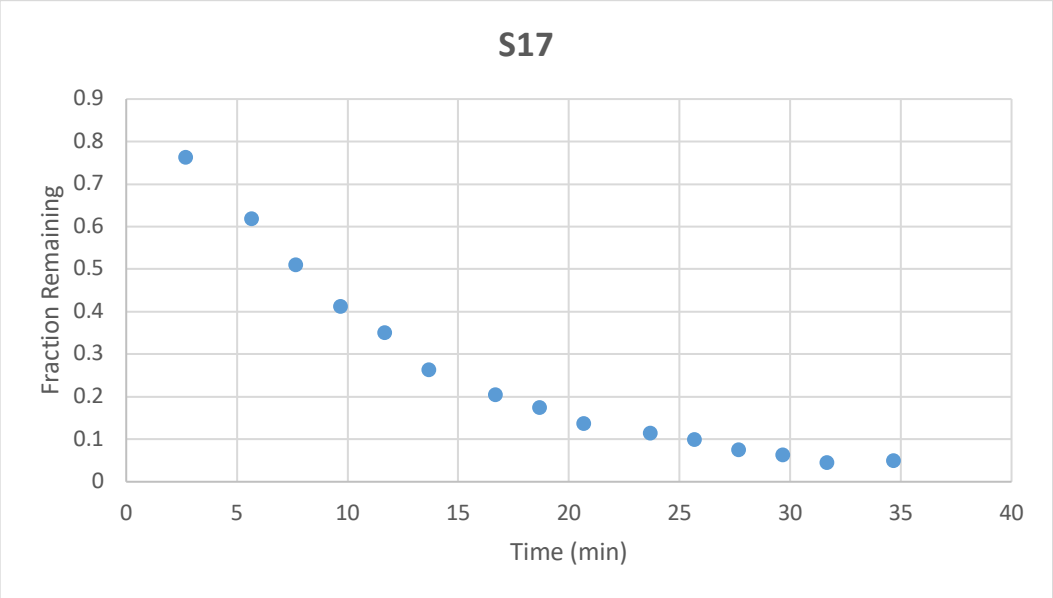
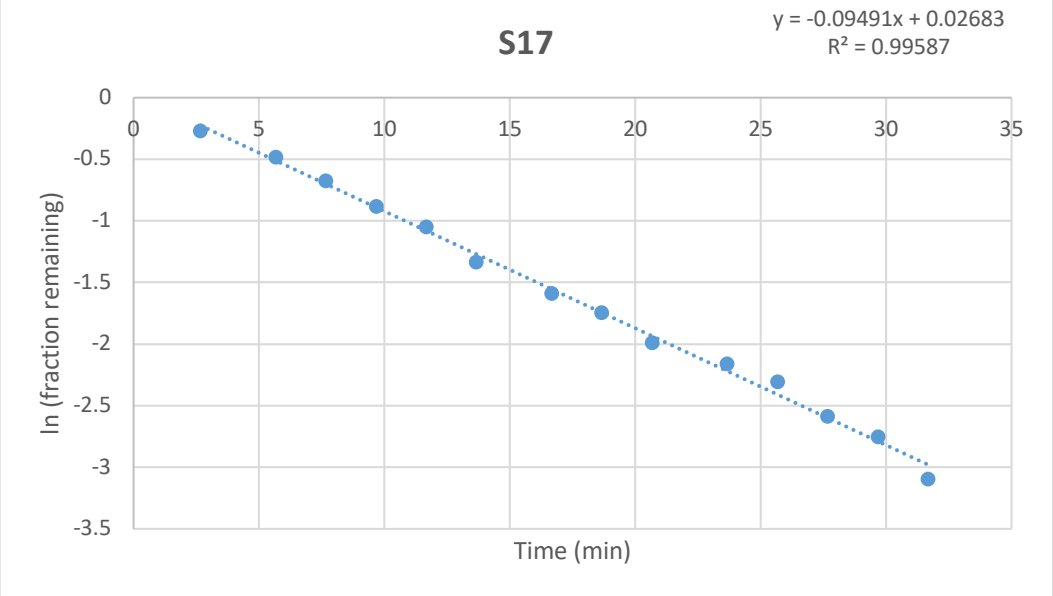



**Figure S24. ln(Fraction Remaining) Values Plotted Against Time Points for Lactone S17**



**Biological Materials and Methods**

**Table S11.** HPLC purities of compounds.

| Compound | Retention Time (min) | Purity (%) |
|---|---|---|
| **45** | 14.8 | >99 |
| **47** | 11.0 | 98.4 |
| **48** | 15.2 | 95.8 |
| **49** | 20.1 | 95.6 |
| **51** | 22.5 | 93.6 |
| **52** | 19.1 | 92.1 |
| **54** | 20.2 | 91.0 |
| **60** | 13.3 | 98.6 |

**Figure S25.** HPLC trace of **45**, > 99% pure. RT = 14.8 min. Peak at 1.5-3 min is DMSO.

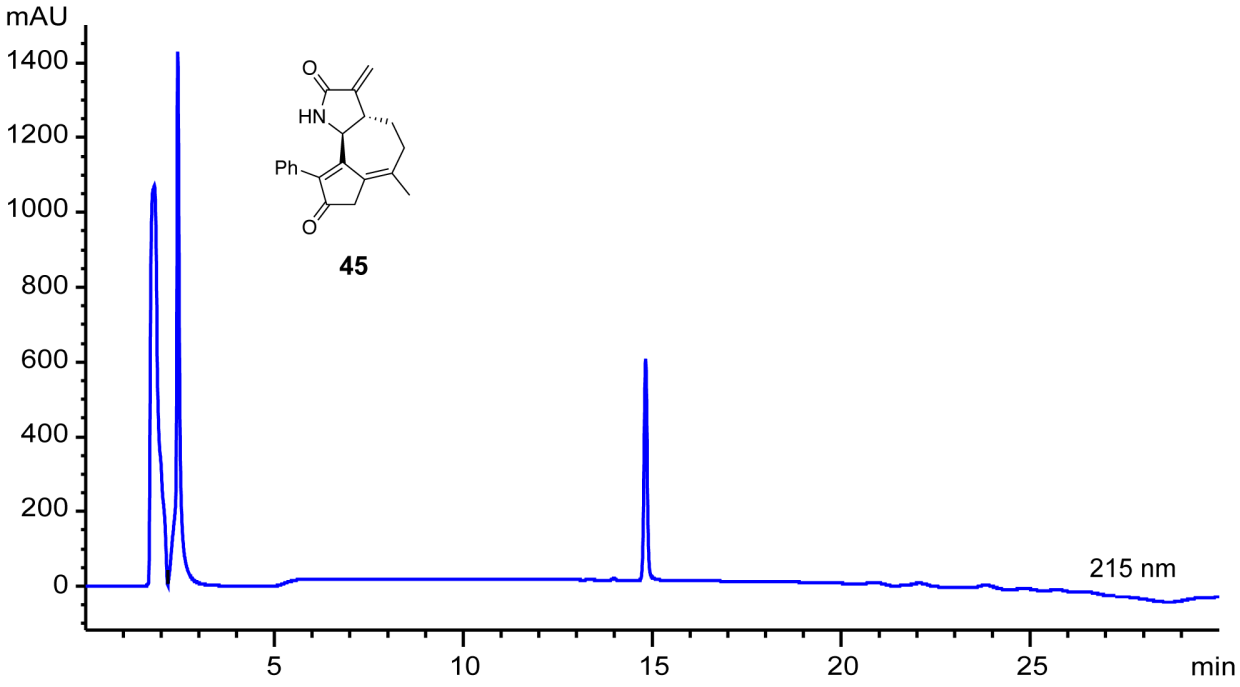


**Figure S26.** HPLC trace of **47**, 98.4% pure. RT = 11.0 min. Peak at 1.5-3 min is DMSO. The peak at 18.0 min appears in the blank run, and was excluded from purity analysis.

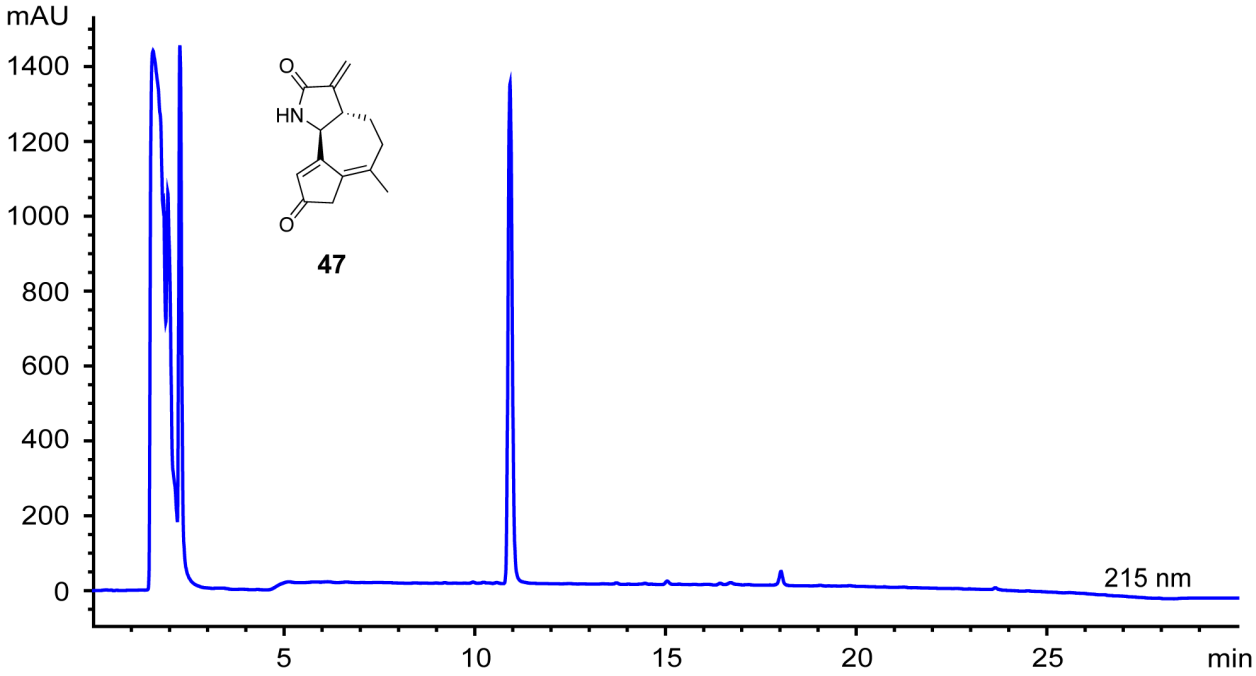



**Figure S27.** HPLC trace of **48**, 95.8% pure. RT = 15.2 min. Peak at 1.5-3 min is DMSO. The peak at 18.0 min appears in the blank run, and was excluded from purity analysis.

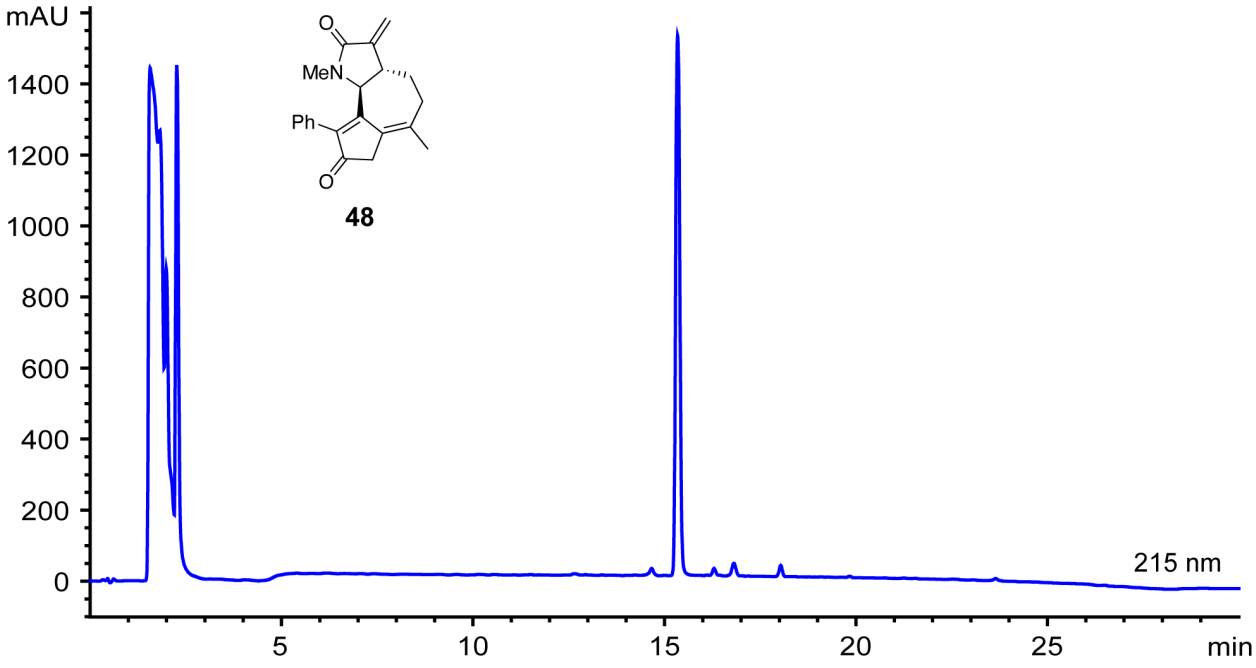


**Figure S28.** HPLC trace of **49**, 95.6% pure. RT = 20.1 min. The peak at 18.0 min appears in the blank run, and was excluded from purity analysis.

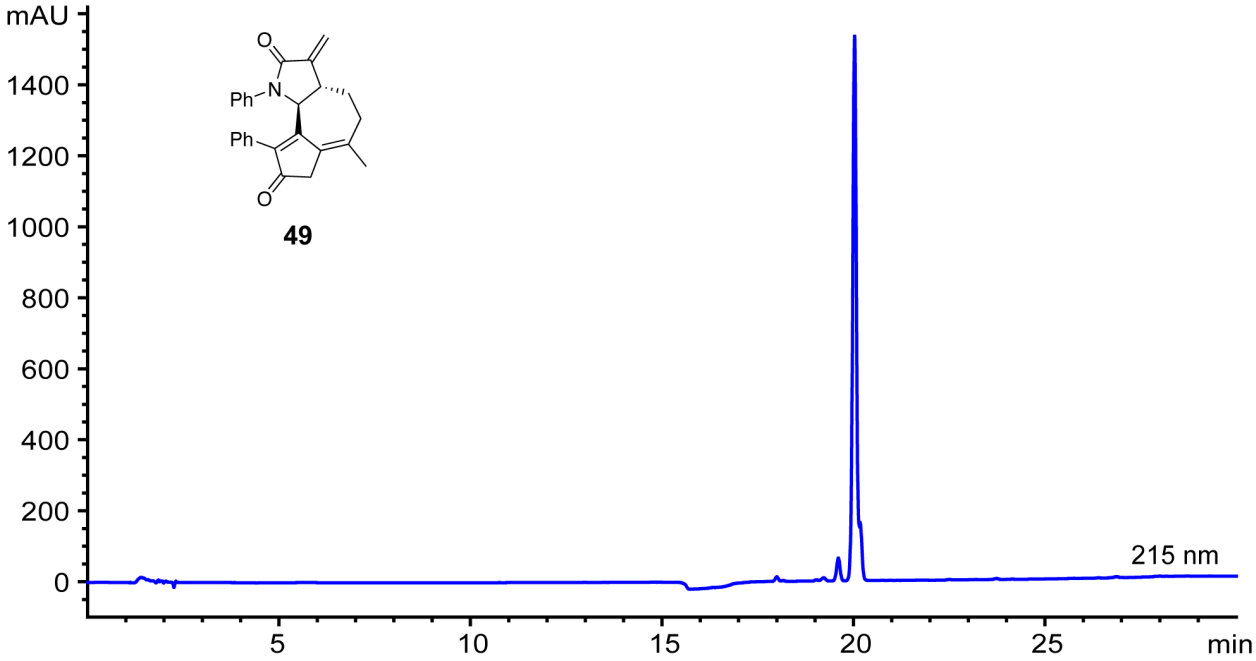



**Figure S29.** HPLC trace of **51**, 93.6% pure. RT = 22.5 min.

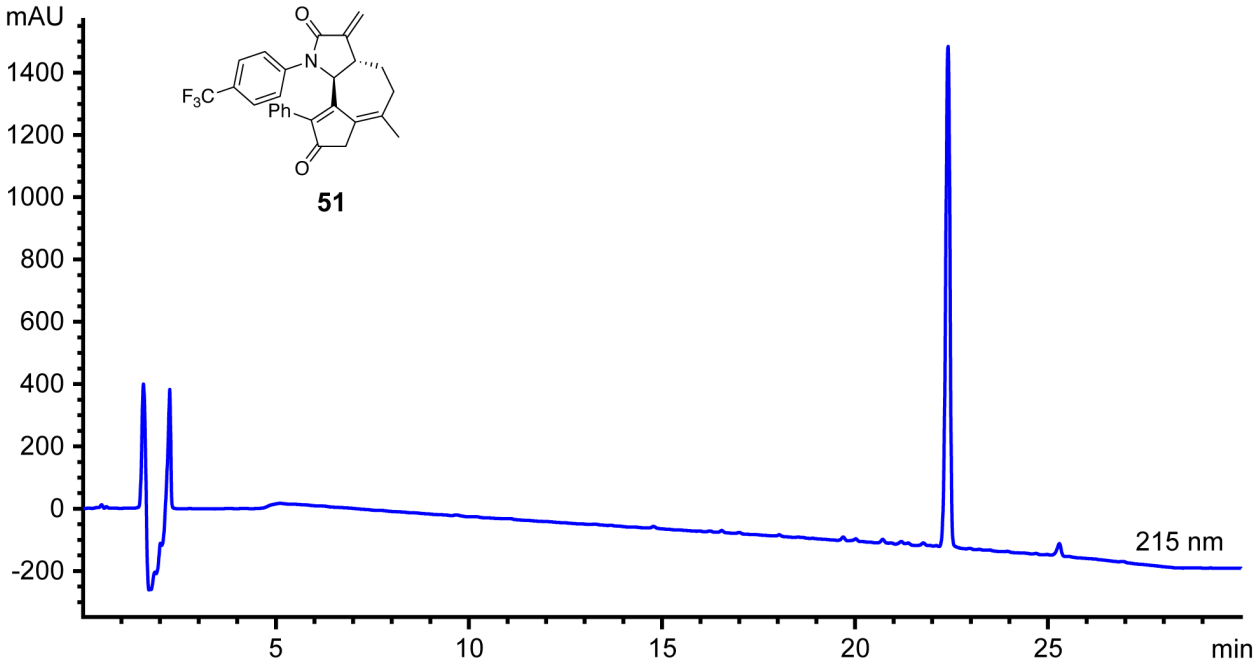


**Figure S30.** HPLC trace of **52**, 92.1% pure, RT = 19.1 min. Peak at 1.5-3 min is DMSO.

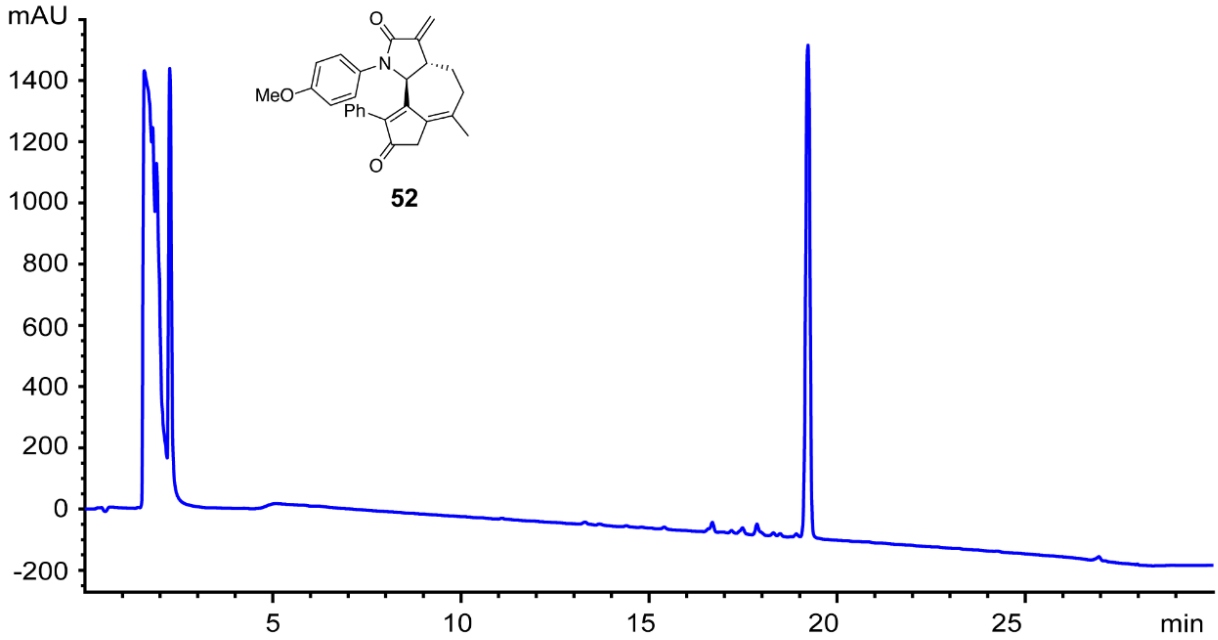



**Figure S31.** HPLC trace of **54**, 91.0% pure. RT = 20.2 min.

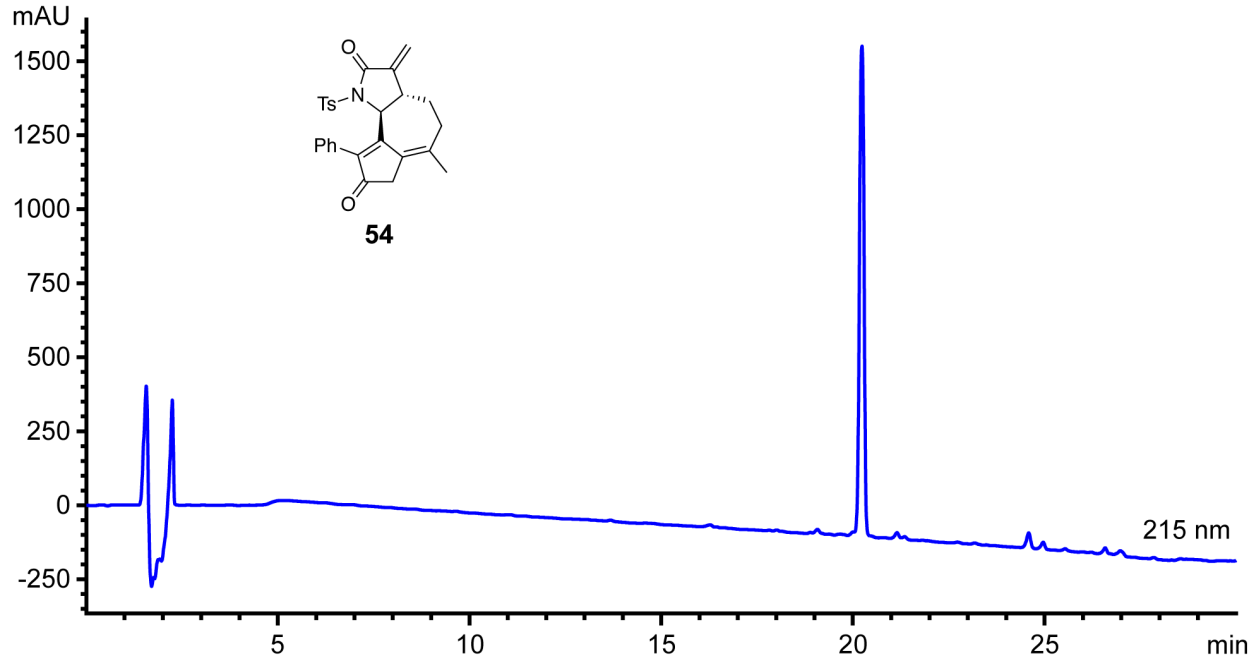


**Figure S32.** HPLC trace of **60**, 98.6% pure. RT = 13.3 min.

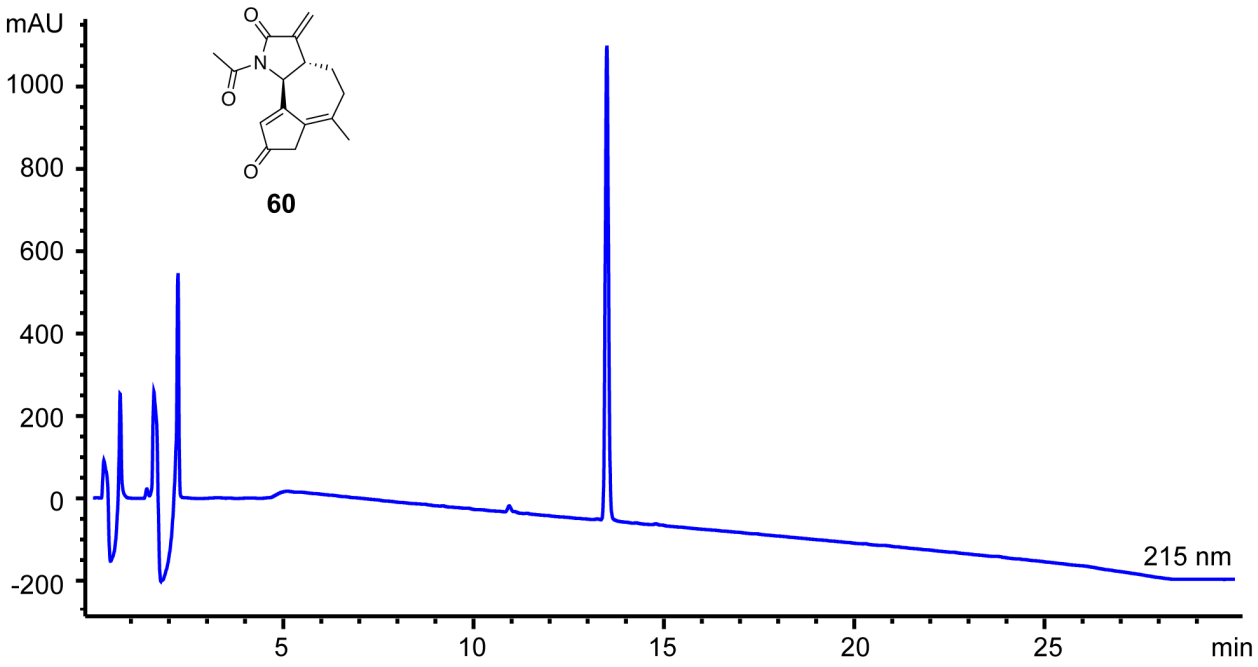


**Figure S33.** 4-15% SDS-PAGE analysis of La antigen protein expression, stained with Coomassie blue.

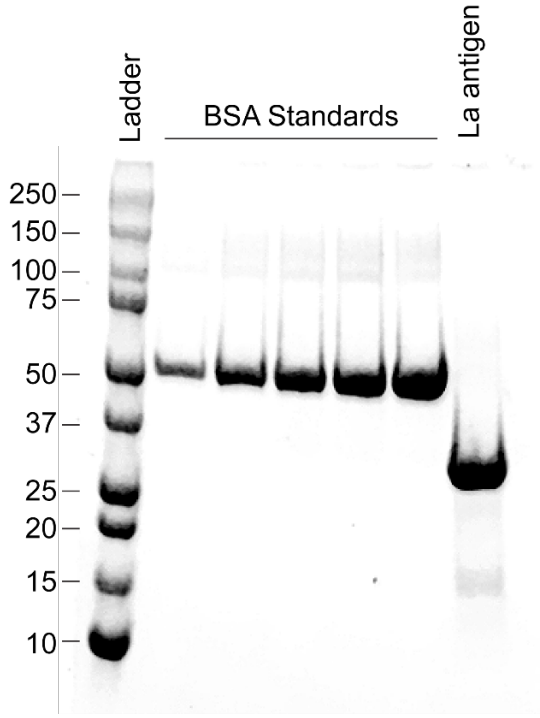



**Table S12.** Observed adducts from La Antigen mass spectrometry MS/MS analysis. Listed are the cysteine adducts identified for the three peptides searched. IAD = carbamidomethylation, D2-IAD = $d_2$-carbamidomethylation. Fluconazole and CPM are the same negative and positive controls, respectively, used in the ALARM NMR assay. N/A indicated the peptide was not identified. Samples included three biological replicates analyzed each in technical triplicate.

| Sample | Compound | SLEEKIG<u>C</u>$^{232}$LLK | IG<u>C</u>$^{232}$LLK | FSGDLDDQT<u>C</u>$^{245}$R |
|---|---|---|---|---|
| S1_1 | DMSO | IAD/D2-IAD | D2-IAD | N/A |
| S1_2 | | IAD/D2-IAD | D2-IAD | IAD |
| S1_3 | | IAD/D2-IAD | N/A | IAD |
| S2_1 | DMSO | IAD/D2-IAD | IAD/D2-IAD | IAD |
| S2_2 | | IAD/D2-IAD | IAD/D2-IAD | IAD |
| S2_3 | | IAD/D2-IAD | IAD/D2-IAD | IAD |
| S3_1 | DMSO | D2-IAD | IAD/D2-IAD | IAD |
| S3_2 | | IAD/D2-IAD | IAD/D2-IAD | IAD |
| S3_3 | | IAD/D2-IAD | IAD/D2-IAD | IAD |

| | | | | |
|---|---|---|---|---|
| S4_1 | Fluconazole | IAD/D2-IAD | D2-IAD | IAD |
| S4_2 | | IAD/D2-IAD | D2-IAD | IAD |
| S4_3 | | IAD/D2-IAD | D2-IAD | IAD |
| S5_1 | Fluconazole | IAD/D2-IAD | IAD/D2-IAD | IAD |
| S5_2 | | IAD/D2-IAD | D2-IAD | IAD |
| S5_3 | | IAD/D2-IAD | D2-IAD | IAD |
| S6_1 | Fluconazole | IAD/D2-IAD | D2-IAD | IAD |
| S6_2 | | IAD/D2-IAD | IAD/D2-IAD | IAD |
| S6_3 | | IAD/D2-IAD | D2-IAD | IAD |
| S7_1 | CPM | D2-IAD | D2-IAD/CPM | D2-IAD |
| S7_2 | | D2-IAD/CPM | D2-IAD/CPM | D2-IAD |
| S7_3 | | D2-IAD/CPM | D2-IAD/CPM | D2-IAD |
| S8_1 | CPM | D2-IAD/CPM | D2-IAD/CPM | D2-IAD |
| S8_2 | | D2-IAD/CPM | D2-IAD/CPM | D2-IAD |
| S8_3 | | D2-IAD/CPM | D2-IAD/CPM | D2-IAD |
| S9_1 | CPM | D2-IAD/CPM | D2-IAD/CPM | N/A |
| S9_2 | | D2-IAD/CPM | D2-IAD/CPM | N/A |
| S9_3 | | D2-IAD/CPM | D2-IAD/CPM | N/A |
| S10_1 | **S17** | D2-IAD | D2-IAD/**S17** | D2-IAD |
| S10_2 | | D2-IAD/**S17** | D2-IAD/**S17** | D2-IAD |
| S10_3 | | D2-IAD/**S17** | D2-IAD/**S17** | D2-IAD |
| S11_1 | **S17** | D2-IAD/**S17** | D2-IAD/**S17** | D2-IAD |
| S11_2 | | D2-IAD/**S17** | D2-IAD/**S17** | D2-IAD |
| S11_3 | | D2-IAD/**S17** | D2-IAD/**S17** | D2-IAD |
| S12_1 | **S17** | D2-IAD/**S17** | D2-IAD/**S17** | D2-IAD |
| S12_2 | | D2-IAD/**S17** | D2-IAD/**S17** | D2-IAD |
| S12_3 | | D2-IAD/**S17** | D2-IAD/**S17** | D2-IAD |
| S13_1 | **51** | IAD | IAD/D2-IAD | IAD |
| S13_2 | | IAD/D2-IAD | IAD/D2-IAD | IAD |
| S13_3 | | IAD | IAD/D2-IAD | IAD |
| S14_1 | **51** | IAD/D2-IAD | D2-IAD/**51** | IAD |
| S14_2 | | IAD/D2-IAD | D2-IAD | IAD |
| S14_3 | | IAD/D2-IAD | D2-IAD/**51** | IAD |
| S15_1 | **51** | IAD/D2-IAD | IAD/D2-IAD/**51** | IAD |
| S15_2 | | IAD/D2-IAD | IAD/D2-IAD/**51** | IAD |
| S15_3 | | IAD/D2-IAD | IAD/D2-IAD/**51** | IAD |

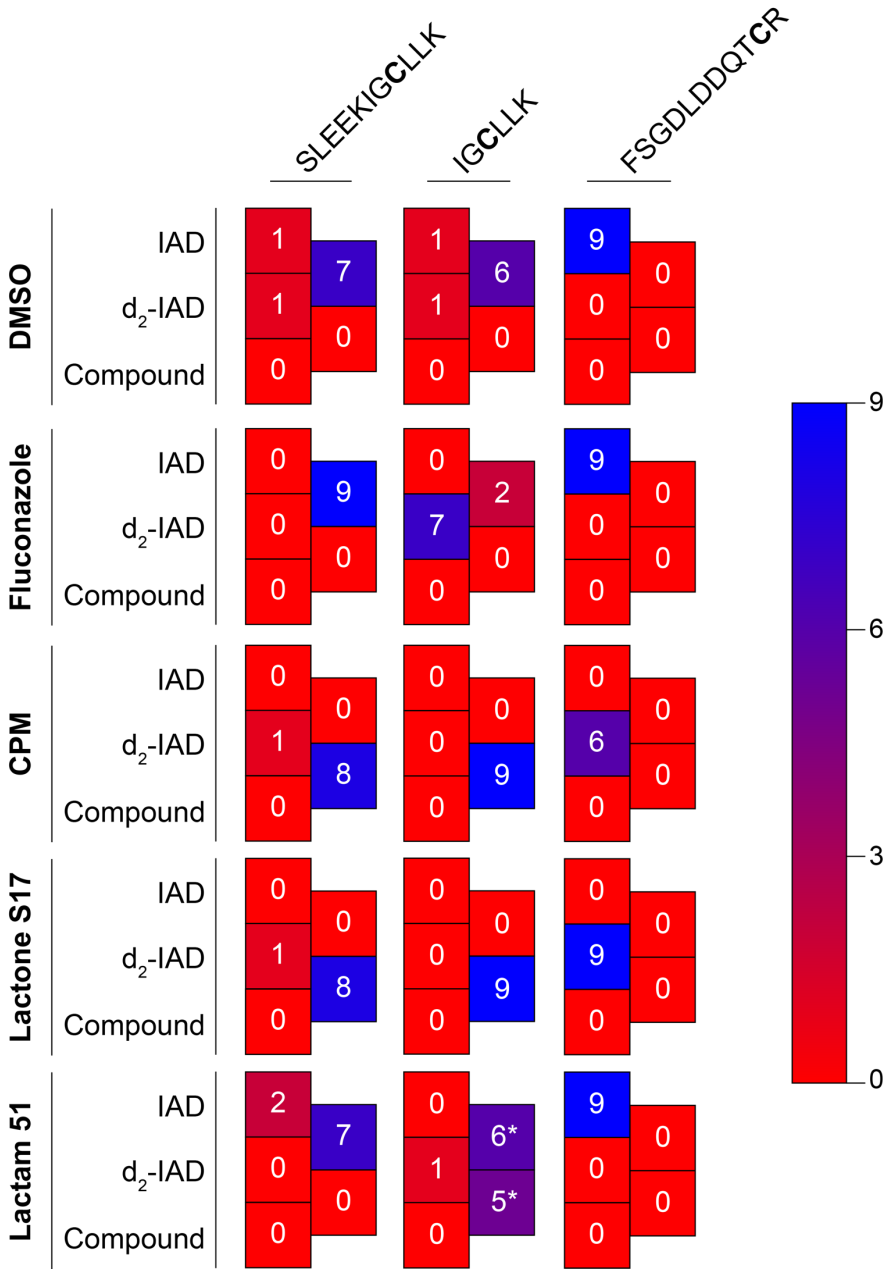
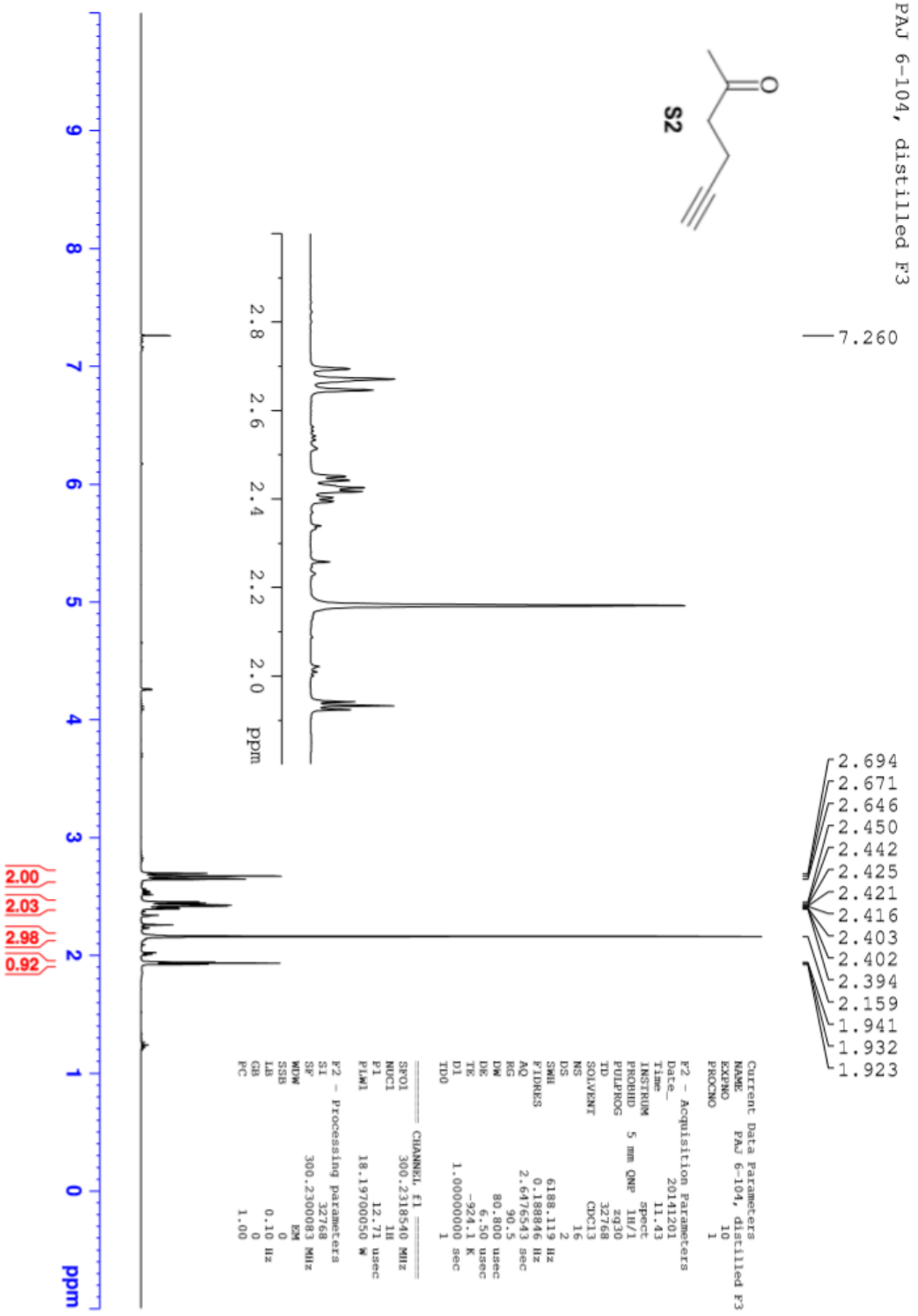



**Figure S34.** Heat map analysis of the observed adducts from La Antigen mass spectrometry MS/MS analysis (Table 12). Fluconazole is the negative control (non-thiol reactive) and CPM is the positive control (thiol-reactive). Asterisk indicates three samples overlap between these two categories (i.e., in three samples, IG**C**$^{232}$LLK is found adducted with IAD, $d_2$-IAD, and compound). Boxes in the middle of two possible adducts indicates the number of samples in which

the peptide is found with both adducts. Samples included three biological replicates analyzed each in technical triplicate.

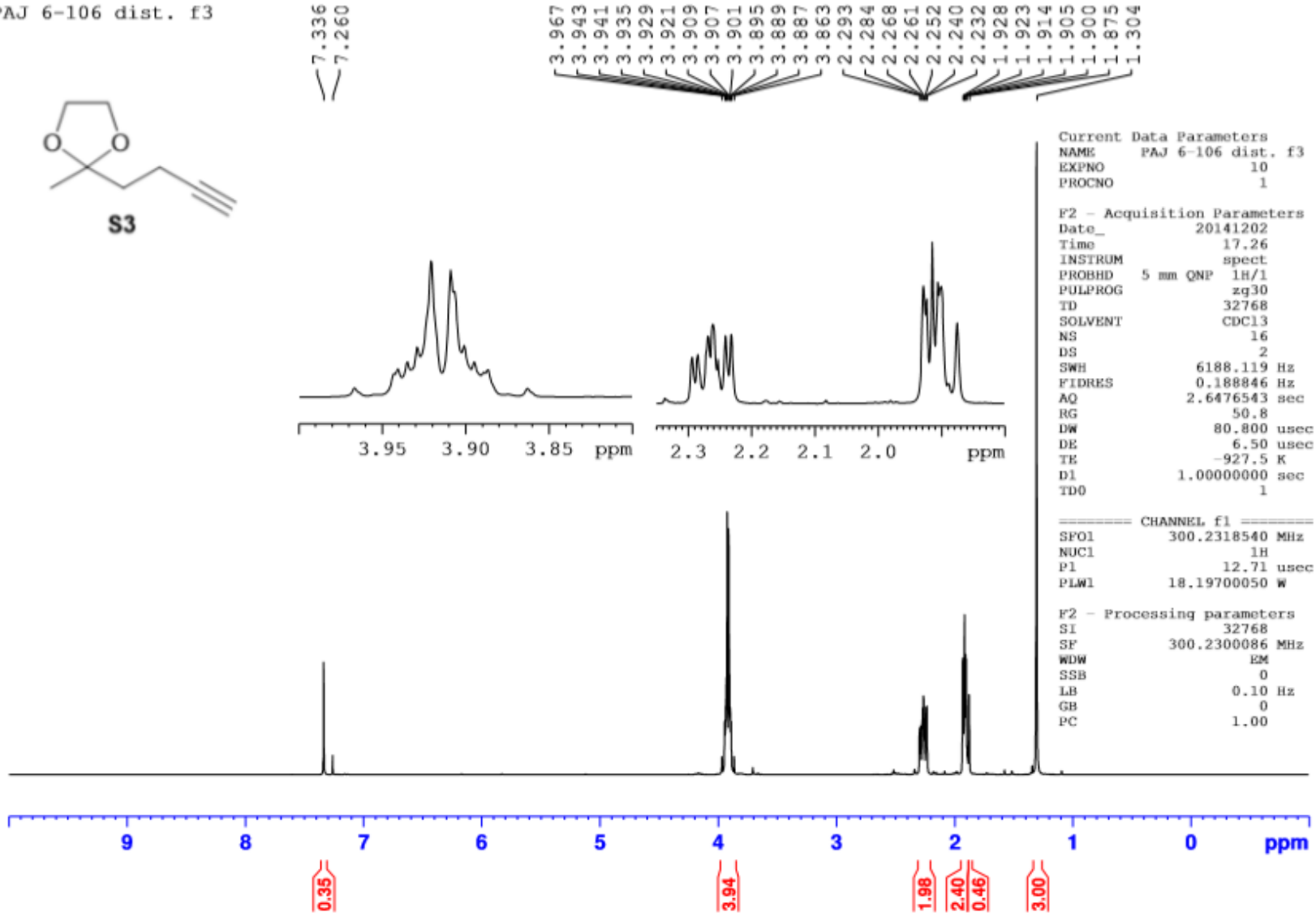
## $^{1}$H NMR, $^{13}$C NMR, and Peptide Mass Spectra



PAJ 6-106 dist. f3



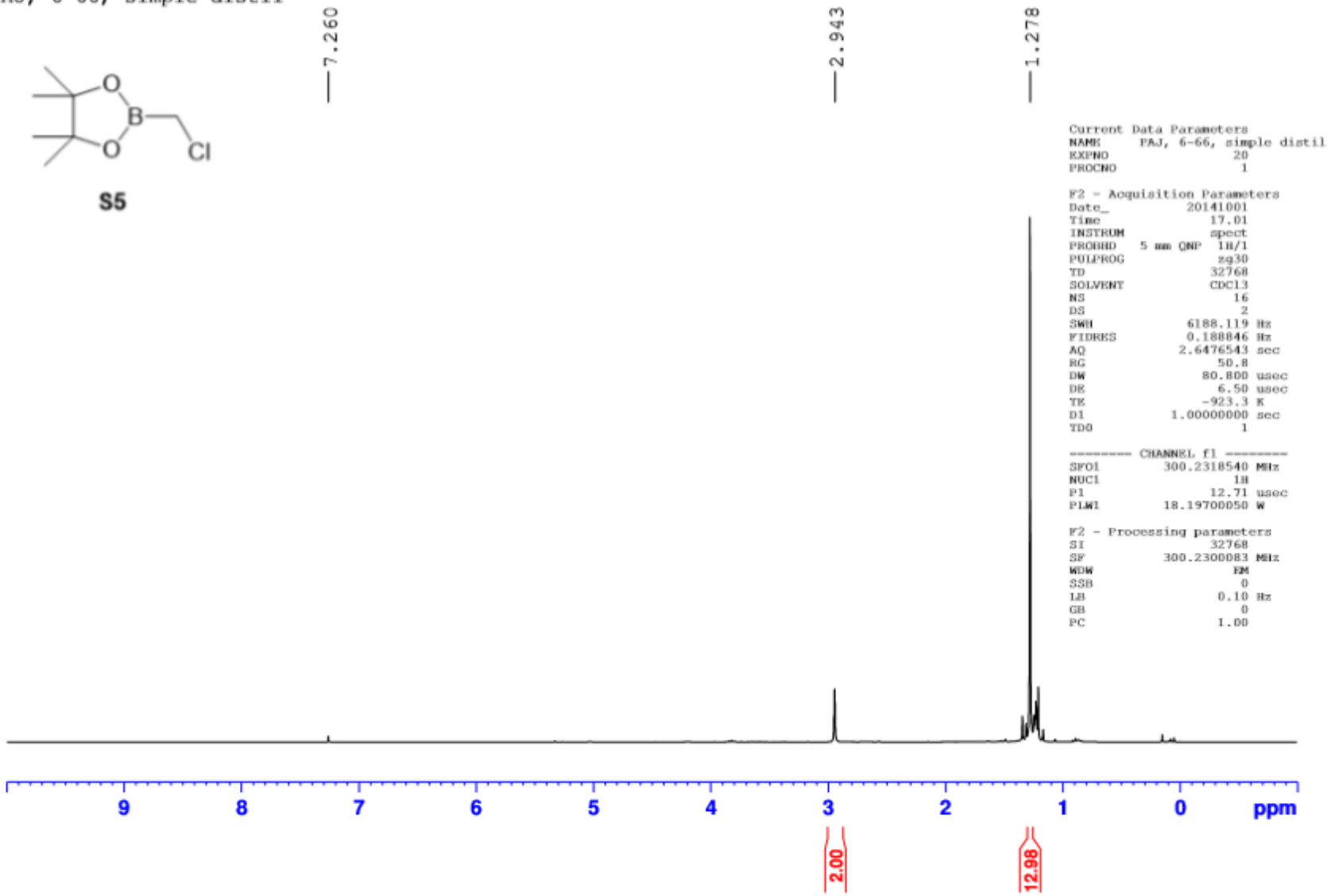
**S3**



PAJ, 6-66, simple distil



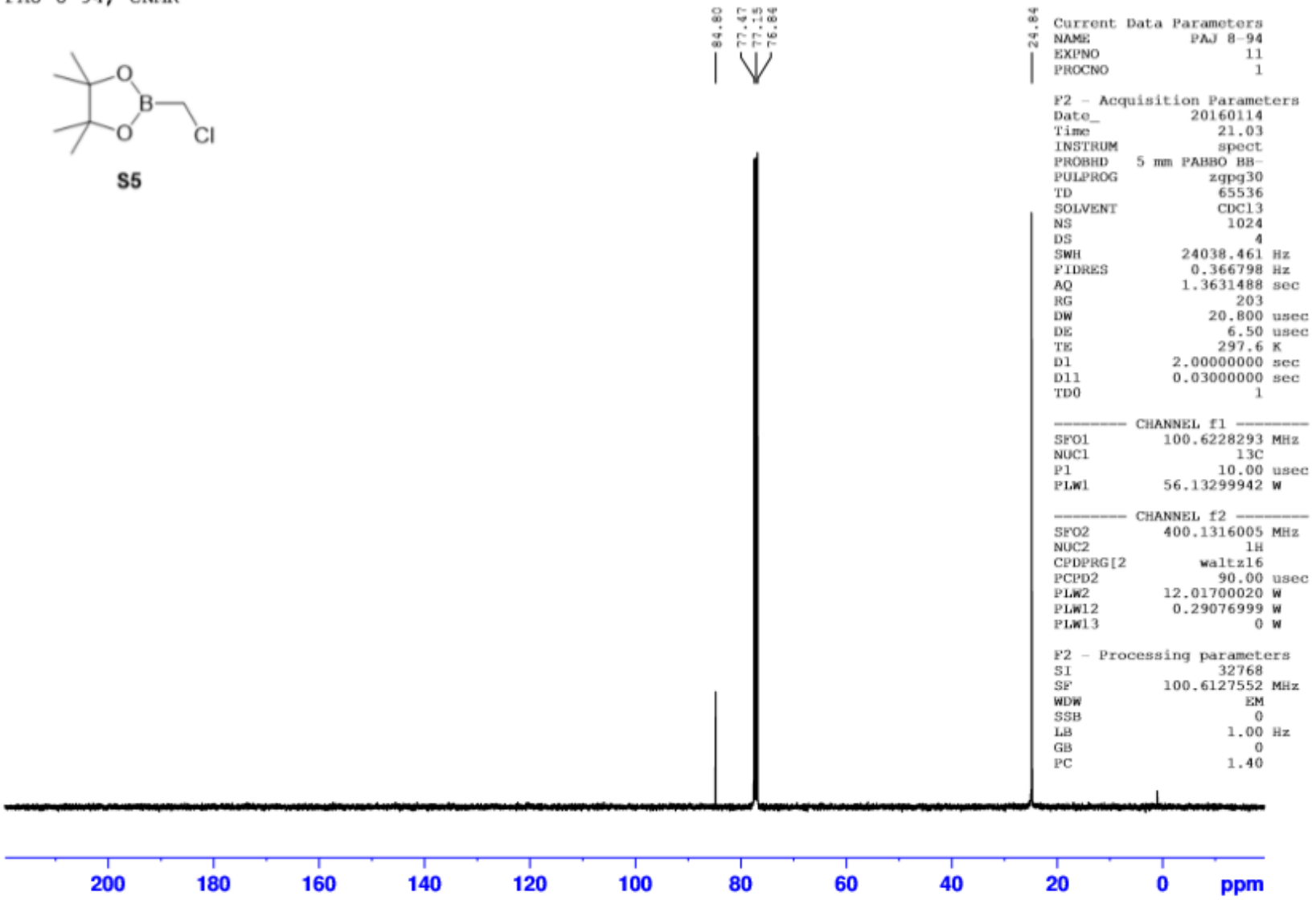

**S5**



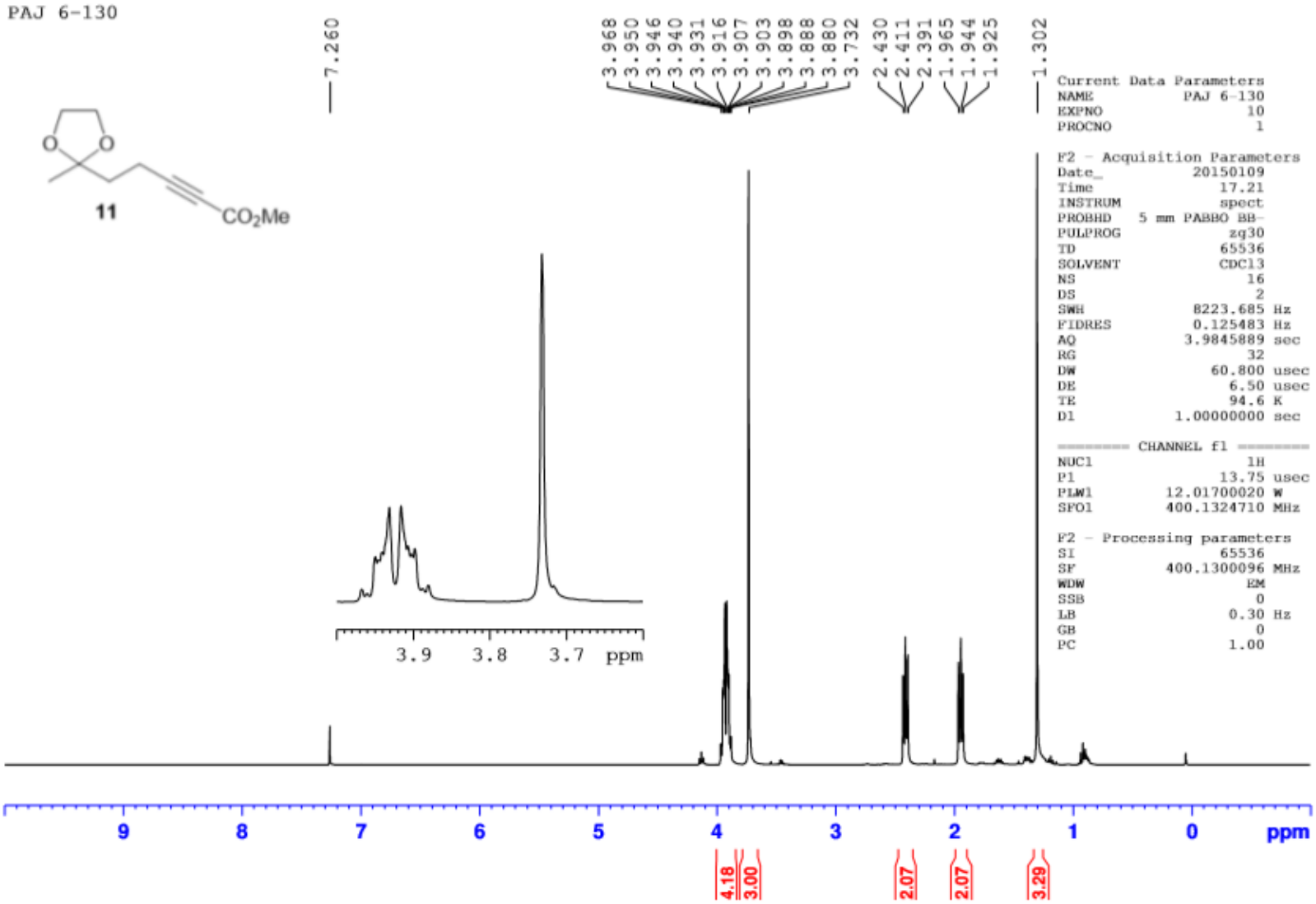
PAJ 8-94, CNMR

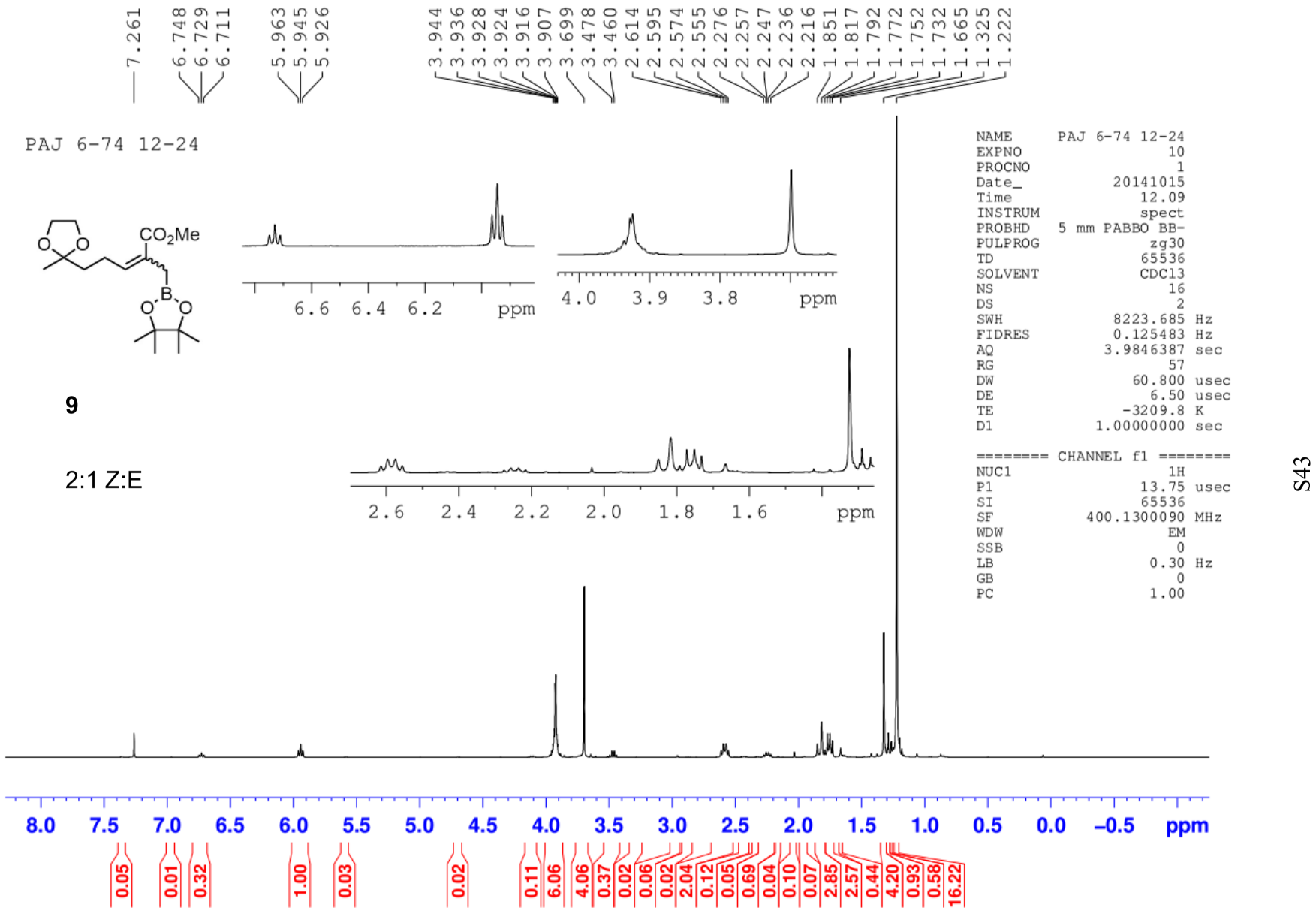


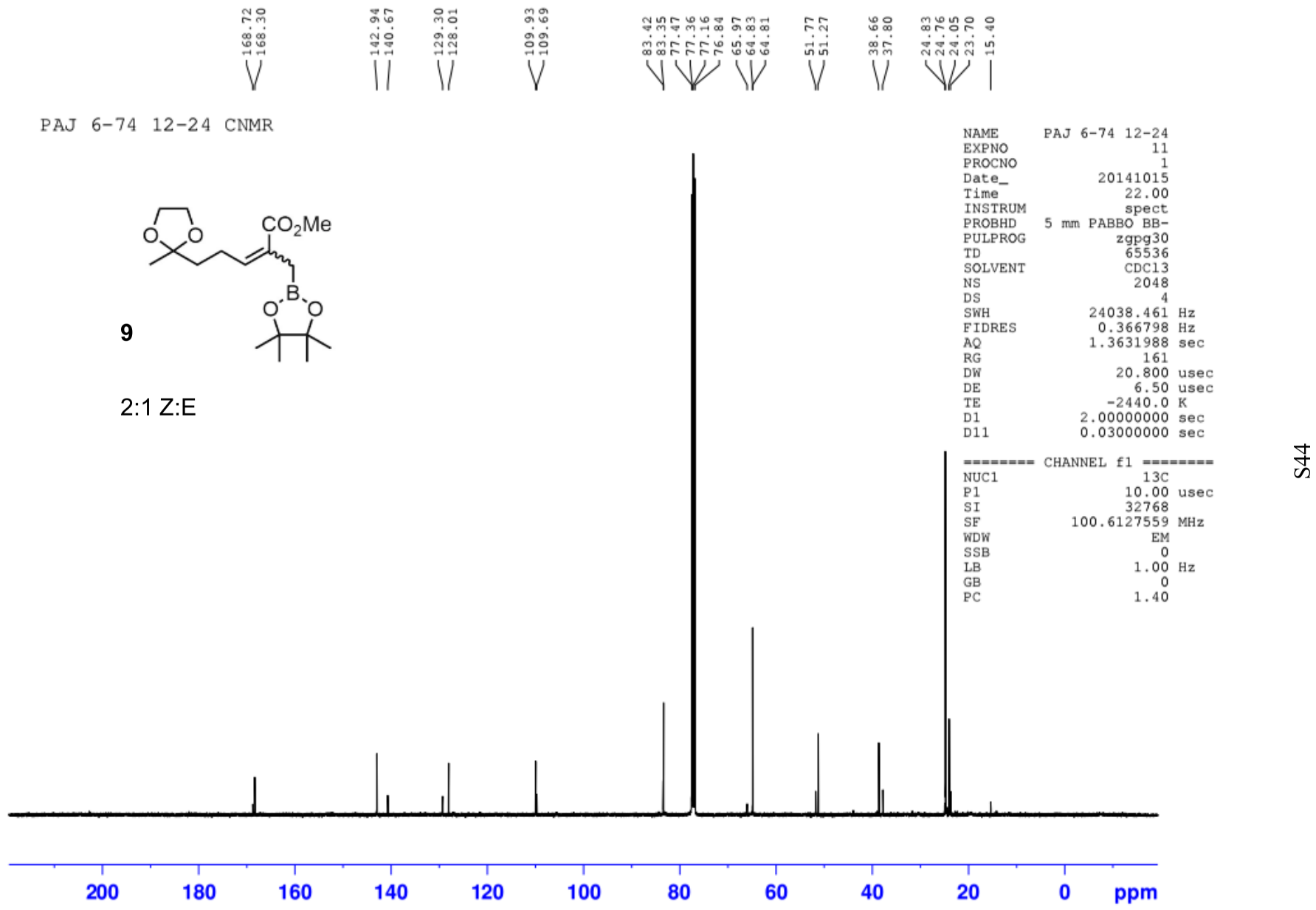

**S5**




```
Current Data Parameters
NAME                  PAJ 8-94
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_                  20160114
Time                      21.03
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1024
DS                           4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631488 sec
RG                         203
DW                      20.800 usec
DE                       6.50 usec
TE                      297.6 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                100.6228293 MHz
NUC1                       13C
P1                       10.00 usec
PLW1                56.13299942 W

======== CHANNEL f2 ========
SFO2                400.1316005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2                12.01700020 W
PLW12                0.29076999 W
PLW13                       0 W

F2 - Processing parameters
SI                         32768
SF                  100.6127552 MHz
WDW                          EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```


PAJ 6-130
7.260
3.968
3.950
3.946
3.940
3.931
3.916
3.907
3.903
3.898
3.888
3.880
3.732
2.430
2.411
2.391
1.965
1.944
1.925
1.302
11
CO2Me
3.9
3.8
3.7
ppm
Current Data Parameters
NAME PAJ 6-130
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150109
Time 17.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 32
DW 60.800 usec
DE 6.50 usec
TE 94.6 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 13.75 usec
PLW1 12.01700020 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
4.18
3.00
2.07
2.07
3.29


7.261
6.748
6.729
6.711
5.963
5.945
5.926
3.944
3.936
3.928
3.924
3.916
3.907
3.699
3.478
3.460
2.614
2.595
2.574
2.555
2.276
2.257
2.247
2.236
2.216
1.851
1.817
1.792
1.772
1.752
1.732
1.665
1.325
1.222
PAJ 6-74 12-24
CO2Me
B
O
6.6 6.4 6.2 ppm
4.0 3.9 3.8 ppm
9
2:1 Z:E
2.6 2.4 2.2 2.0 1.8 1.6 ppm
NAME PAJ 6-74 12-24
EXPNO 10
PROCNO 1
Date_ 20141015
Time 12.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 57
DW 60.800 usec
DE 6.50 usec
TE -3209.8 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
0.05 0.01 0.32 1.00 0.03 0.02 0.11 6.06 4.06 0.37 0.02 0.06 0.02 2.04 0.12 0.05 0.69 0.04 0.10 0.07 2.85 2.57 0.44 4.20 0.93 0.58 16.22

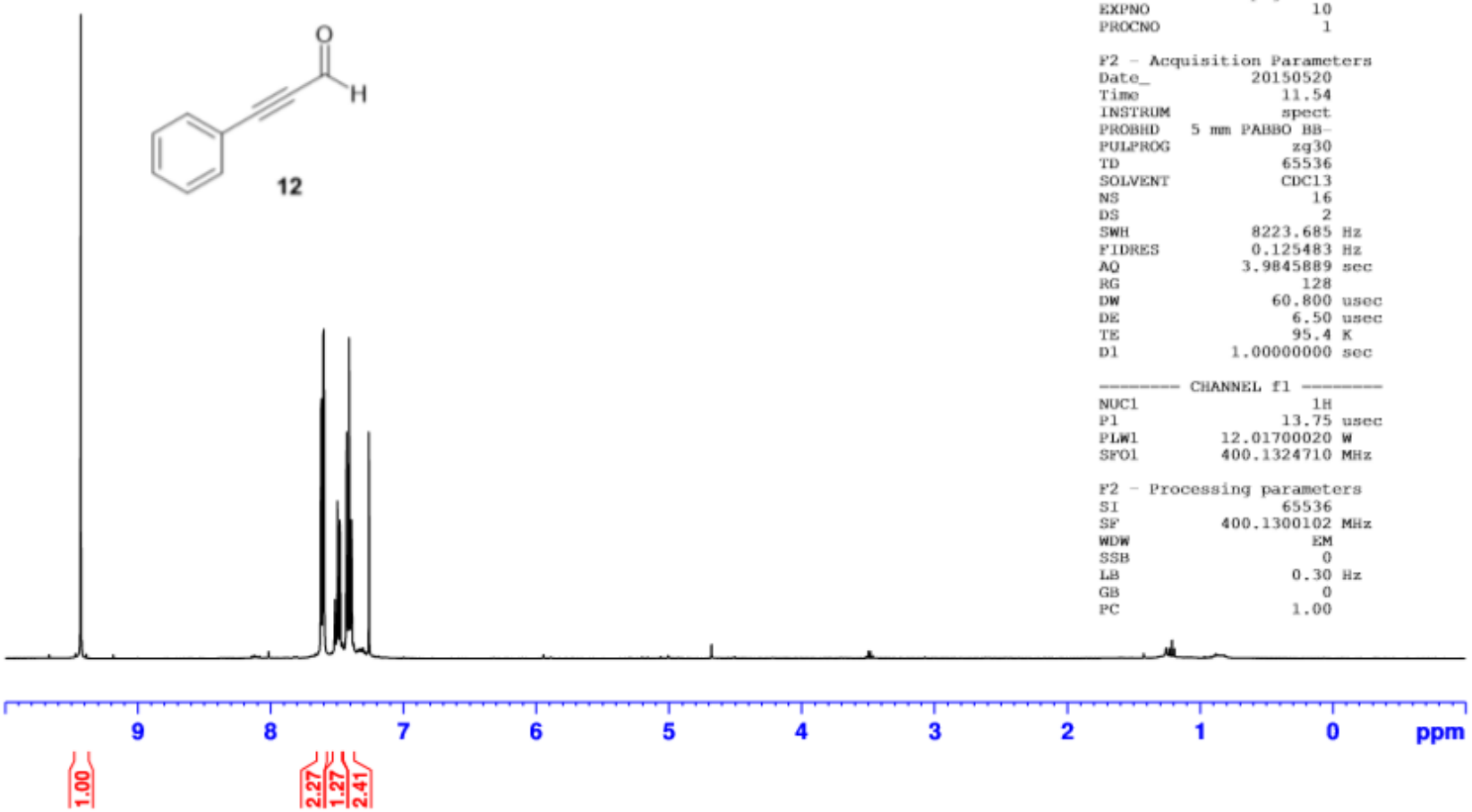

168.72
168.30
142.94
140.67
129.30
128.01
109.93
109.69
83.42
83.35
77.47
77.36
77.16
76.84
65.97
64.83
64.81
51.77
51.27
38.66
37.80
24.83
24.76
24.05
23.70
15.40
PAJ 6-74 12-24 CNMR
CO2Me
B
O
O
O
9
2:1 Z:E
NAME PAJ 6-74 12-24
EXPNO 11
PROCNO 1
Date_ 20141015
Time 22.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 161
DW 20.800 usec
DE 6.50 usec
TE -2440.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127559 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

PAJ 7-14

9.430
7.627
7.622
7.619
7.616
7.607
7.602
7.598
7.519
7.516
7.512
7.503
7.497
7.492
7.482
7.478
7.475
7.428
7.425
7.412
7.409
7.405
7.395
7.391
7.388
7.260

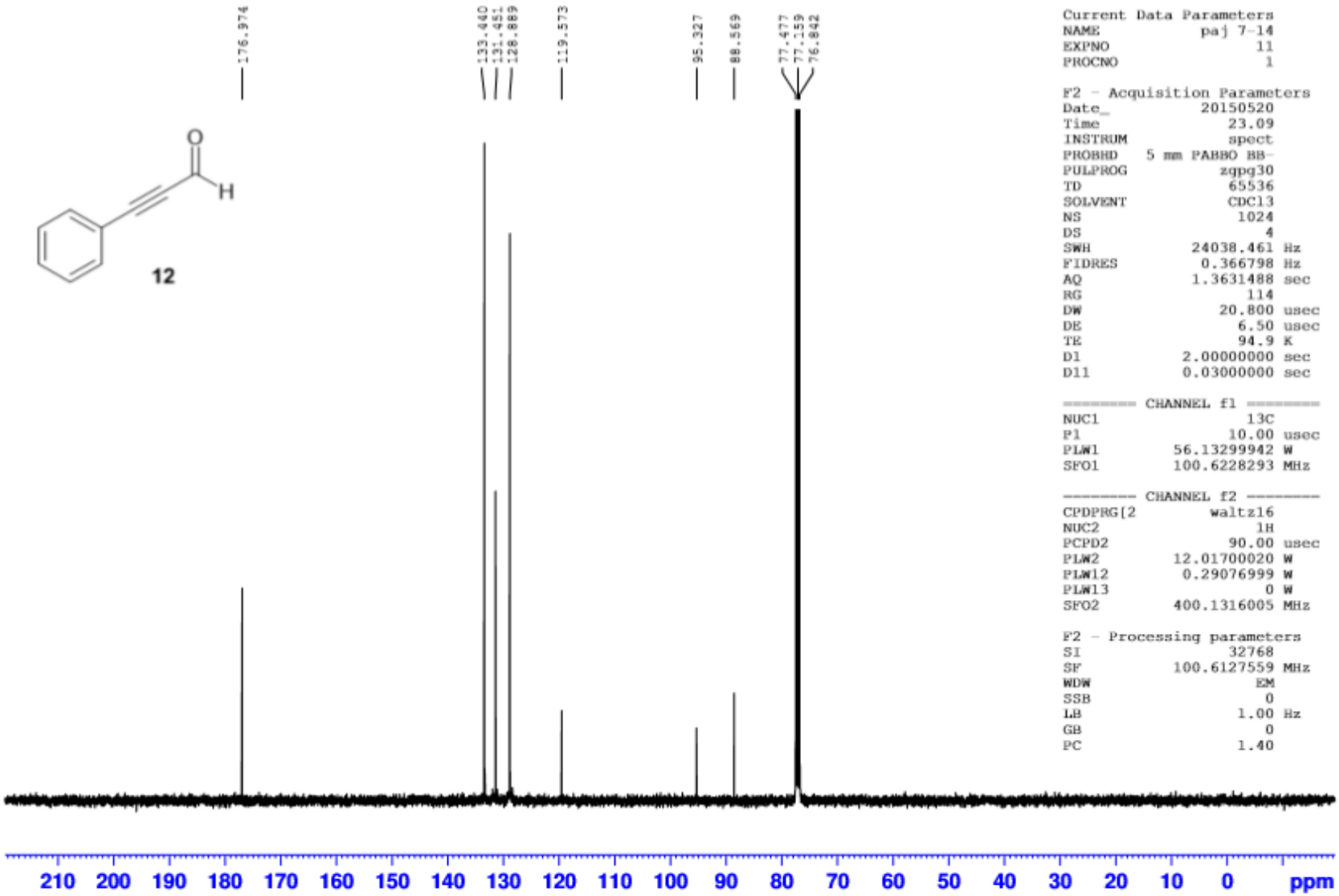



12

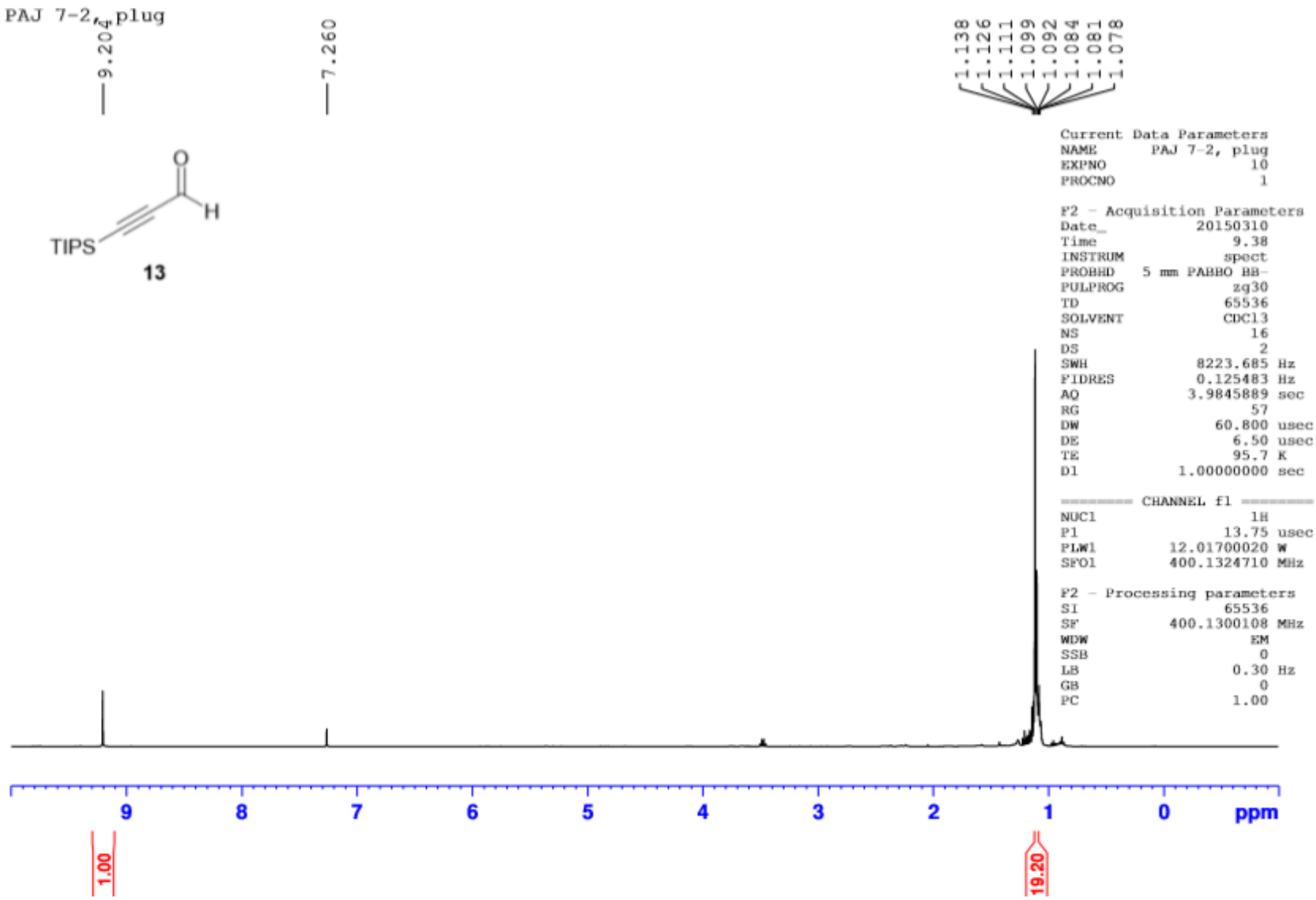



```
Current Data Parameters
NAME                  paj 7-14
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_               20150520
Time                    11.54
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         16
DS                          2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                   3.9845889 sec
RG                         128
DW                    60.800 usec
DE                     6.50 usec
TE                     95.4 K
D1                  1.00000000 sec

--------- CHANNEL f1 ---------
NUC1                       1H
P1                      13.75 usec
PLW1             12.01700020 W
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                     65536
SF                400.1300102 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                       1.00
```

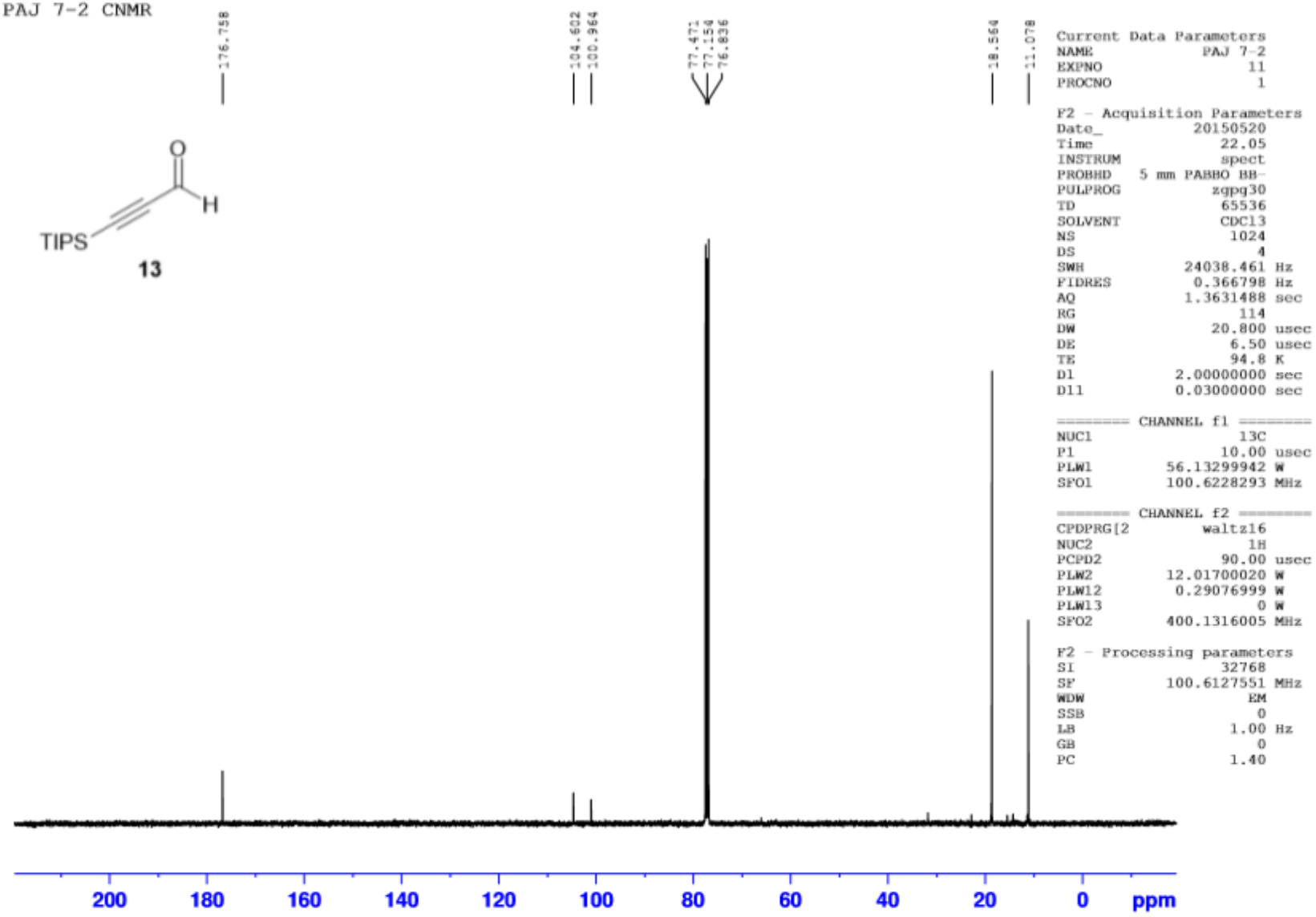
PAJ 7-14 CNMR




```
Current Data Parameters
NAME                  paj 7-14
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20150520
Time                     23.09
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1024
DS                           4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631488 sec
RG                         114
DW                      20.800 usec
DE                       6.50 usec
TE                       94.9 K
D1                    2.00000000 sec
D11                   0.03000000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                       10.00 usec
PLW1                56.13299942 W
SFO1               100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG[2                waltz16
NUC2                        1H
PCPD2                    90.00 usec
PLW2                12.01700020 W
PLW12                0.29076999 W
PLW13                       0 W
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                 100.6127559 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```


PAJ 7-2, plug
9.20
7.260
1.138
1.126
1.111
1.099
1.092
1.084
1.081
1.078
O
H
TIPS
13
Current Data Parameters
NAME PAJ 7-2, plug
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150310
Time 9.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 57
DW 60.800 usec
DE 6.50 usec
TE 95.7 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 13.75 usec
PLW1 12.01700020 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300108 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.00
19.20

PAJ 7-2 CNMR



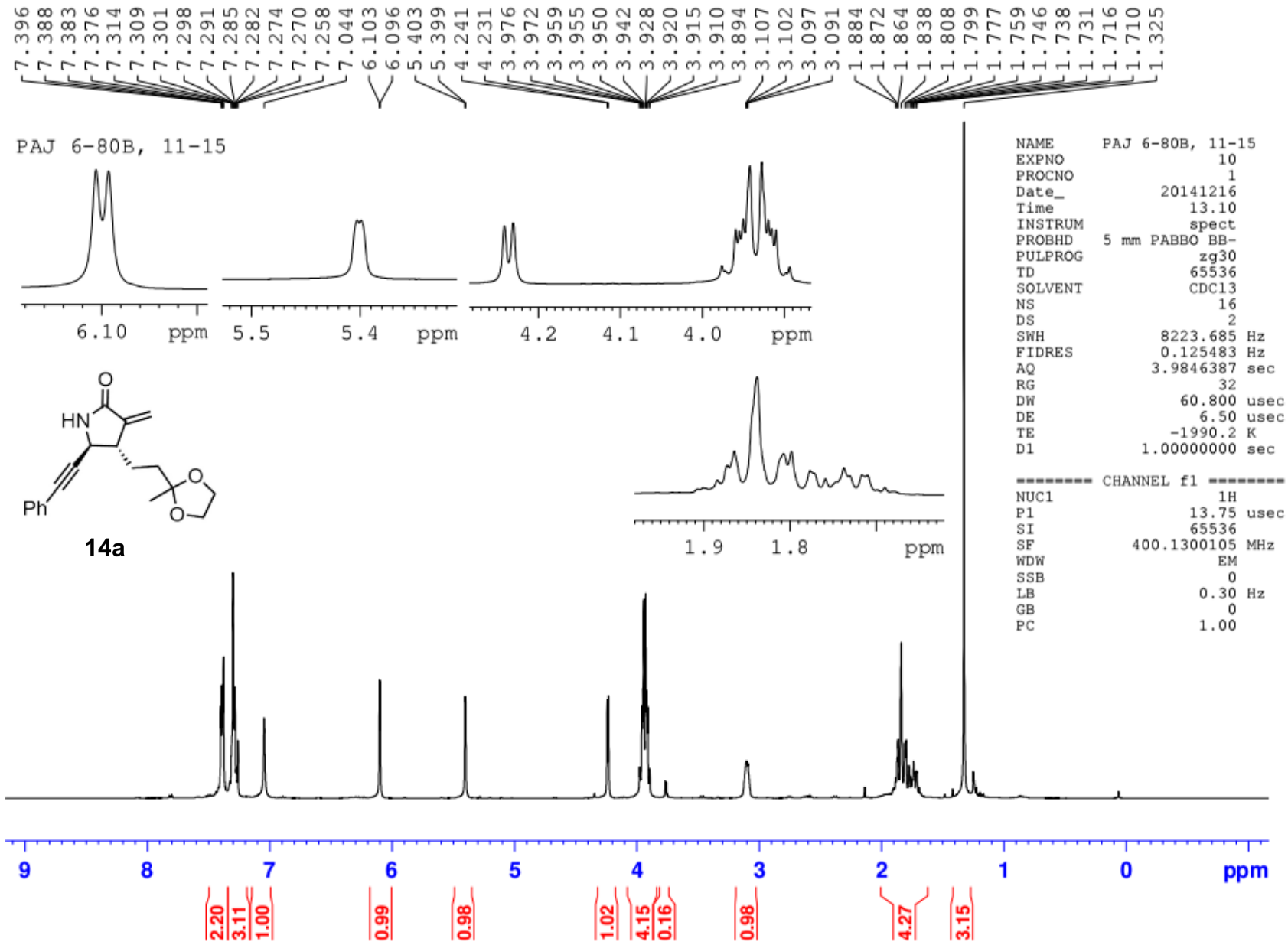

Current Data Parameters
NAME PAJ 7-2
EXPNO 11
PROCNO 1

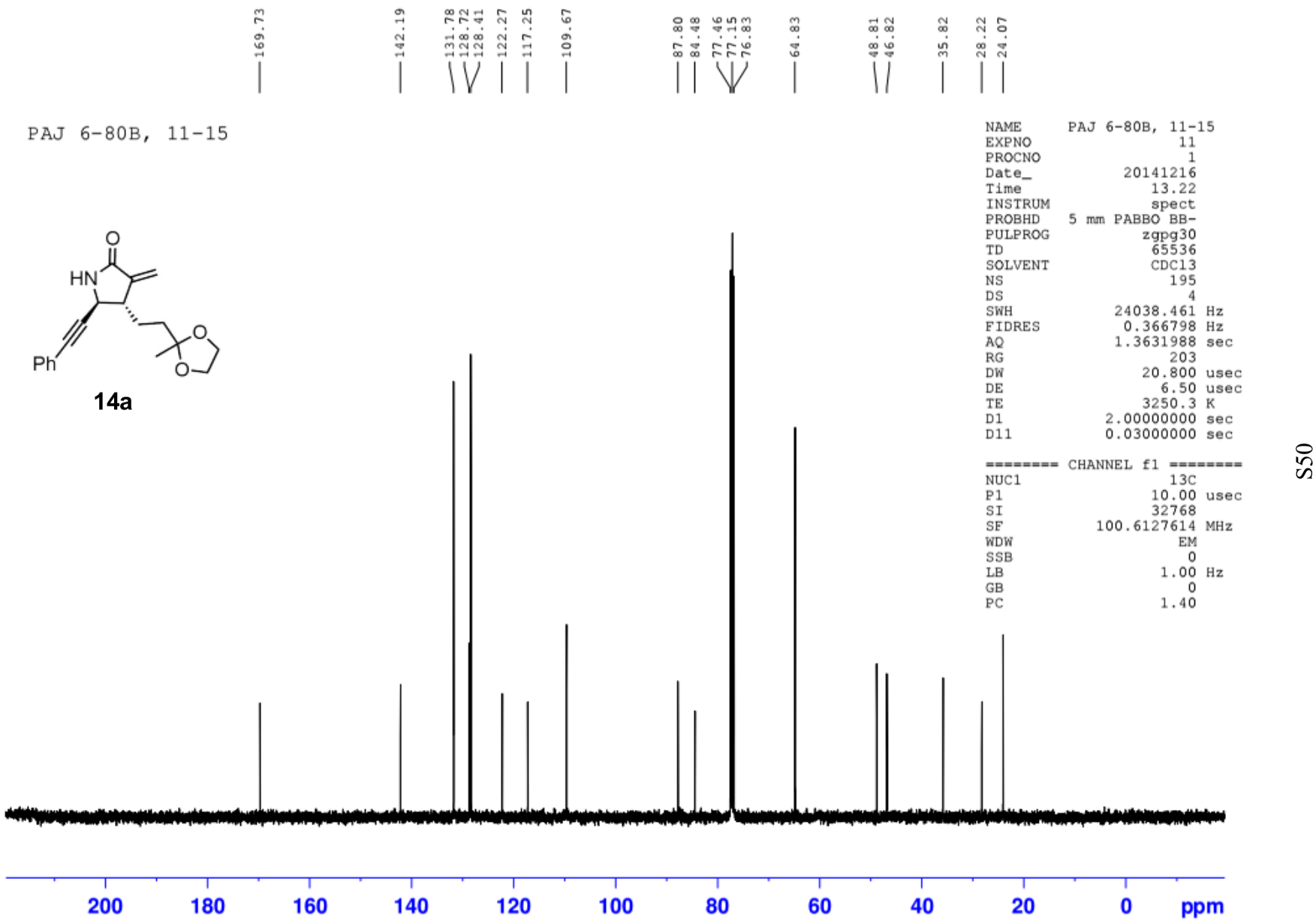
F2 - Acquisition Parameters
Date_ 20150520
Time 22.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 114
DW 20.800 usec
DE 6.50 usec
TE 94.8 K
D1 2.00000000 sec
D11 0.03000000 sec

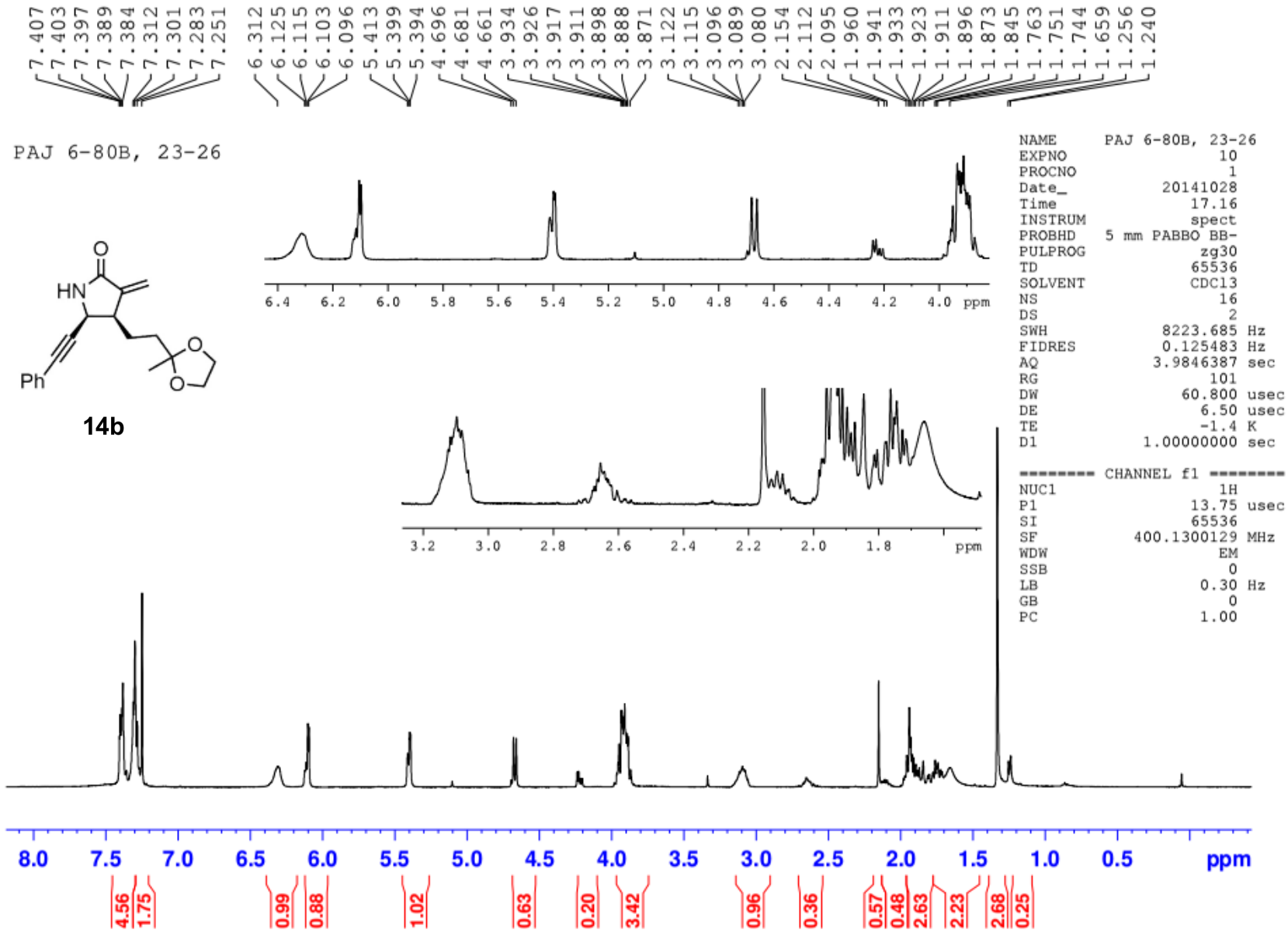
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 56.13299942 W
SFO1 100.6228293 MHz

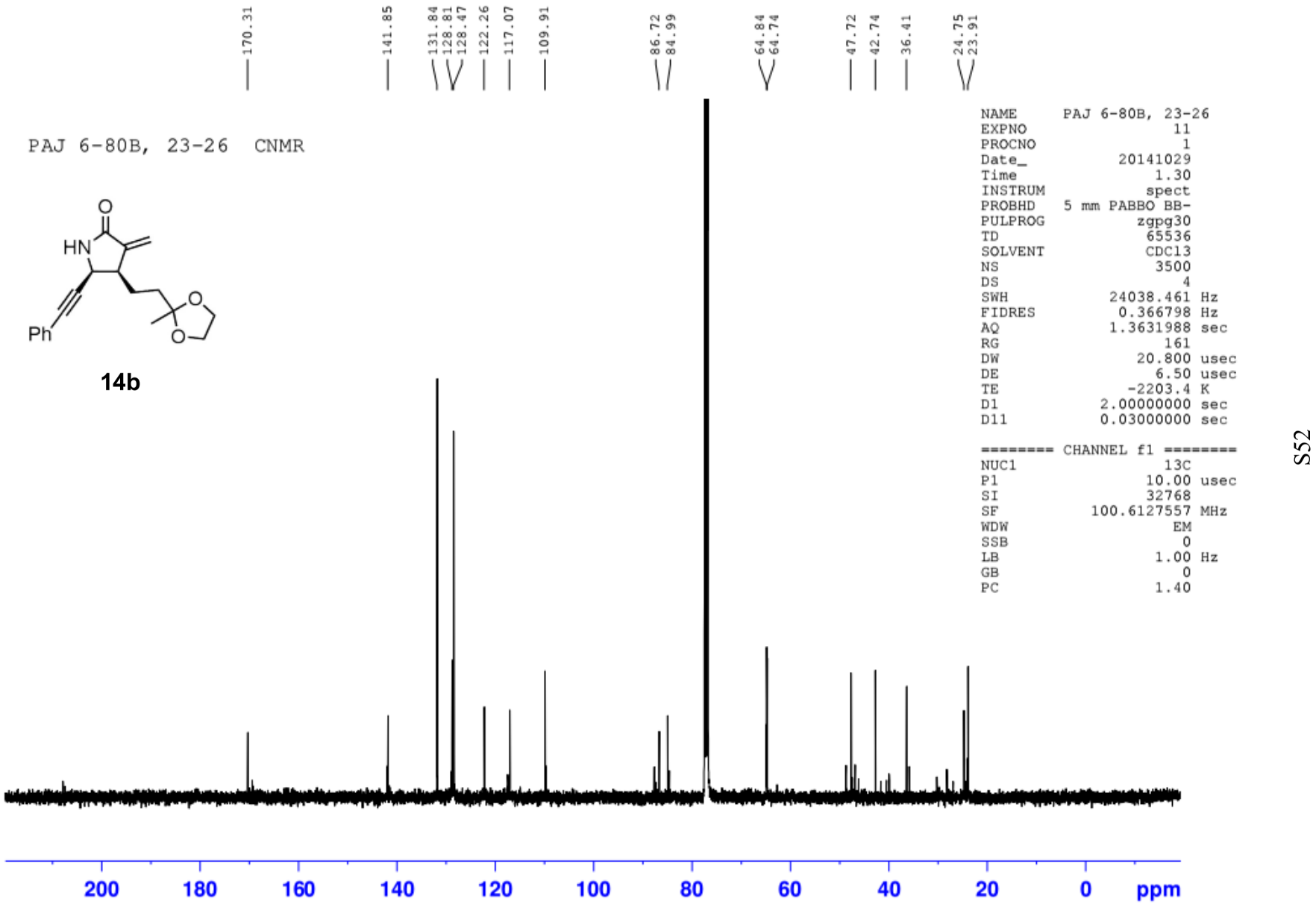
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.01700020 W
PLW12 0.29076999 W
PLW13 0 W
SFO2 400.1316005 MHz

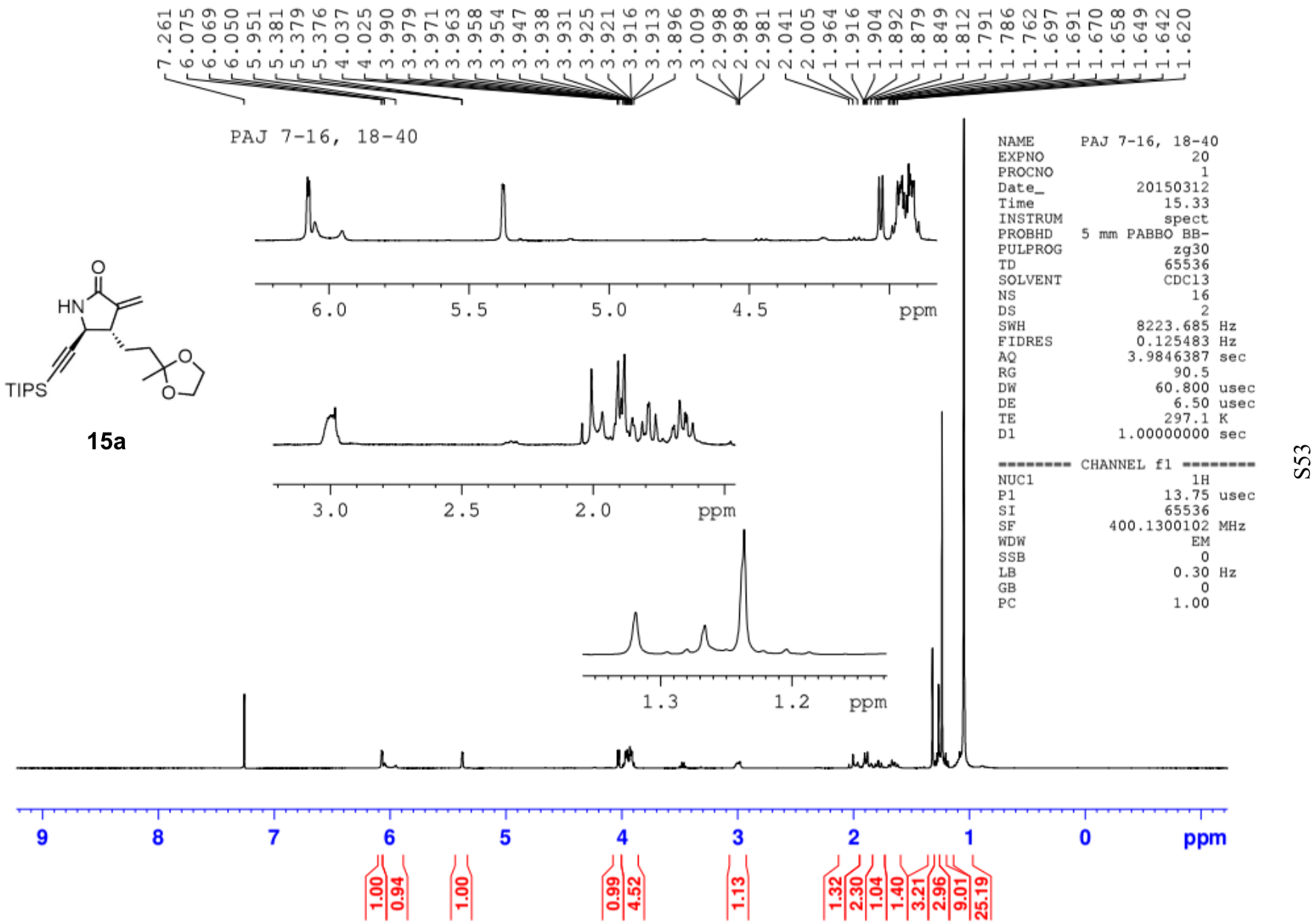
F2 - Processing parameters
SI 32768
SF 100.6127551 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

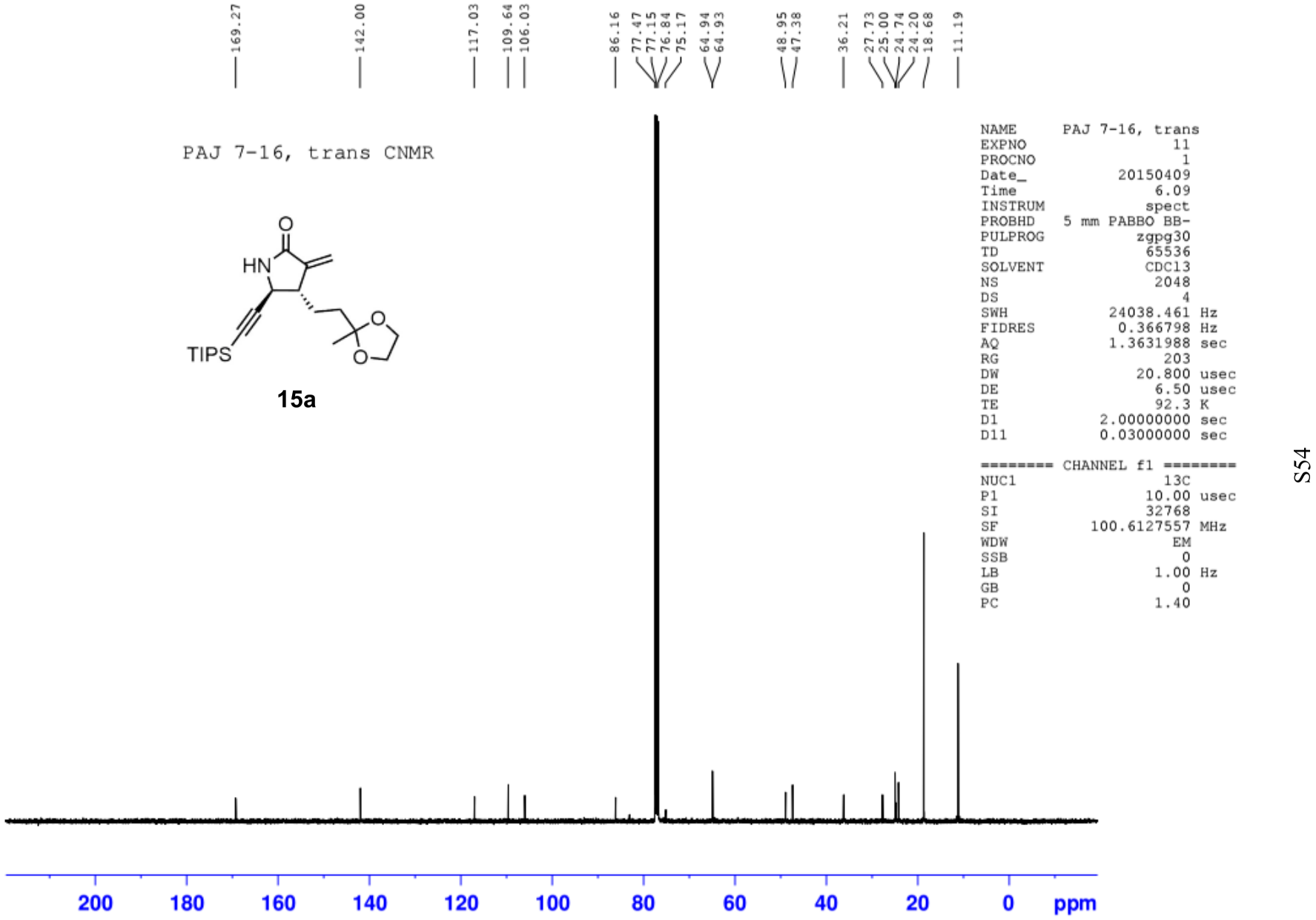

7.396 7.388 7.383 7.376 7.314 7.309 7.301 7.298 7.291 7.285 7.282 7.274 7.270 7.258 7.044 6.103 6.096 5.403 5.399 4.241 4.231 3.976 3.972 3.959 3.955 3.950 3.942 3.928 3.920 3.915 3.910 3.894 3.107 3.102 3.097 3.091 1.884 1.872 1.864 1.838 1.808 1.799 1.777 1.759 1.746 1.738 1.731 1.716 1.710 1.325
PAJ 6-80B, 11-15
6.10 ppm 5.5 5.4 ppm 4.2 4.1 4.0 ppm
1.9 1.8 ppm
HN
O
Ph
O
O
14a
NAME PAJ 6-80B, 11-15
EXPNO 10
PROCNO 1
Date_ 20141216
Time 13.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 32
DW 60.800 usec
DE 6.50 usec
TE -1990.2 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300105 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
2.20 3.11 1.00 0.99 0.98 1.02 4.15 0.16 0.98 4.27 3.15

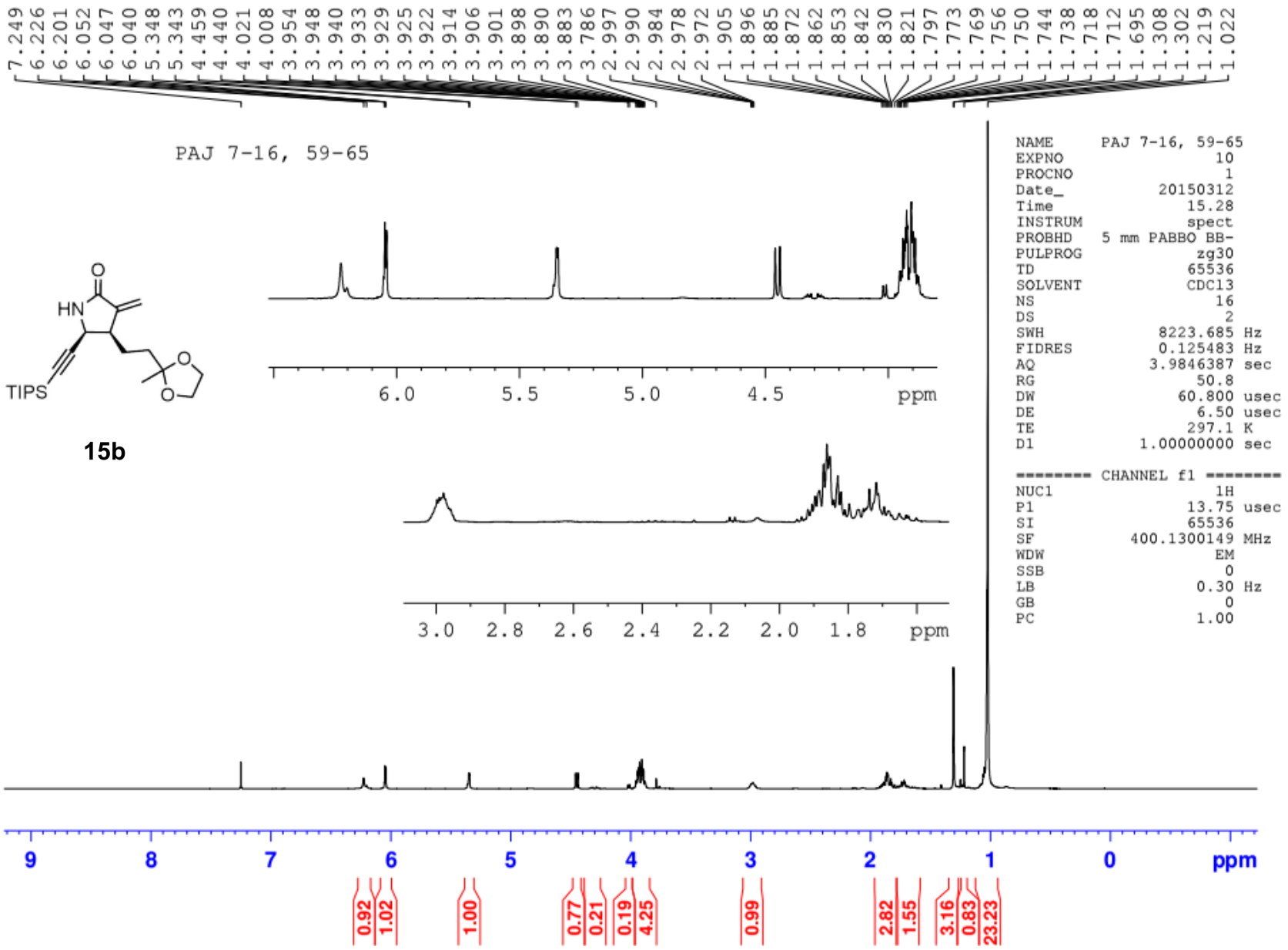

169.73
142.19
131.78
128.72
128.41
122.27
117.25
109.67
87.80
84.48
77.46
77.15
76.83
64.83
48.81
46.82
35.82
28.22
24.07
PAJ 6-80B, 11-15
NAME PAJ 6-80B, 11-15
EXPNO 11
PROCNO 1
Date_ 20141216
Time 13.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 195
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 3250.3 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127614 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HN
O
Ph
O
O
14a
200
180
160
140
120
100
80
60
40
20
0
ppm


7.407
7.403
7.397
7.389
7.384
7.312
7.301
7.283
7.251
6.312
6.125
6.115
6.103
6.096
5.413
5.399
5.394
4.696
4.681
4.661
3.934
3.926
3.917
3.911
3.898
3.888
3.871
3.122
3.115
3.096
3.089
3.080
2.154
2.112
2.095
1.960
1.941
1.933
1.923
1.911
1.896
1.873
1.845
1.763
1.751
1.744
1.659
1.256
1.240
PAJ 6-80B, 23-26
HN
O
Ph
O
O
14b
6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 ppm
3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 ppm
NAME PAJ 6-80B, 23-26
EXPNO 10
PROCNO 1
Date_ 20141028
Time 17.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 101
DW 60.800 usec
DE 6.50 usec
TE -1.4 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
4.56 1.75 0.99 0.88 1.02 0.63 0.20 3.42 0.96 0.36 0.57 0.48 2.63 2.23 2.68 0.25

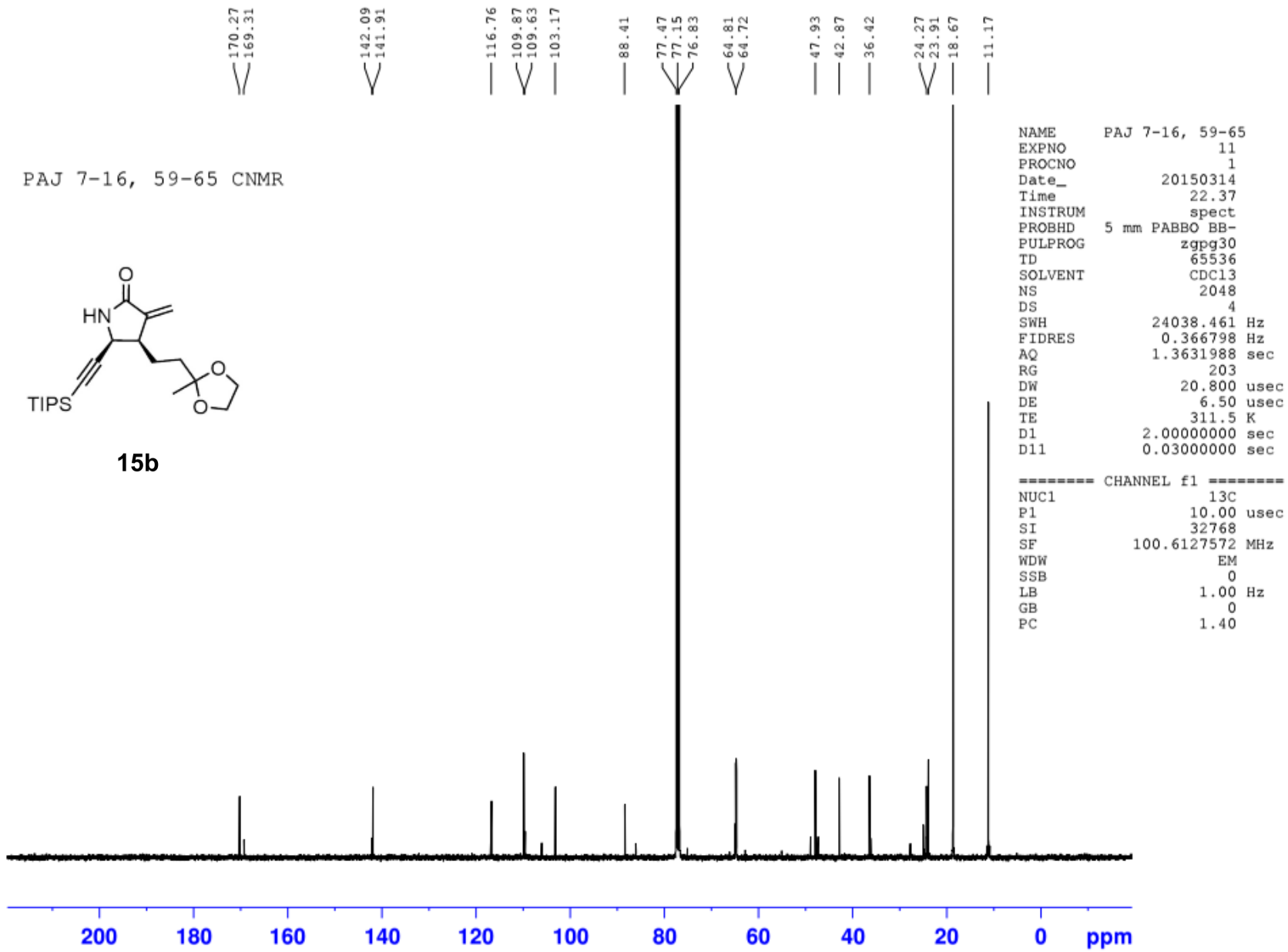

170.31
141.85
131.84
128.81
128.47
122.26
117.07
109.91
86.72
84.99
64.84
64.74
47.72
42.74
36.41
24.75
23.91
PAJ 6-80B, 23-26 CNMR
HN
O
Ph
O
O
14b
NAME PAJ 6-80B, 23-26
EXPNO 11
PROCNO 1
Date_ 20141029
Time 1.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 3500
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 161
DW 20.800 usec
DE 6.50 usec
TE -2203.4 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127557 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

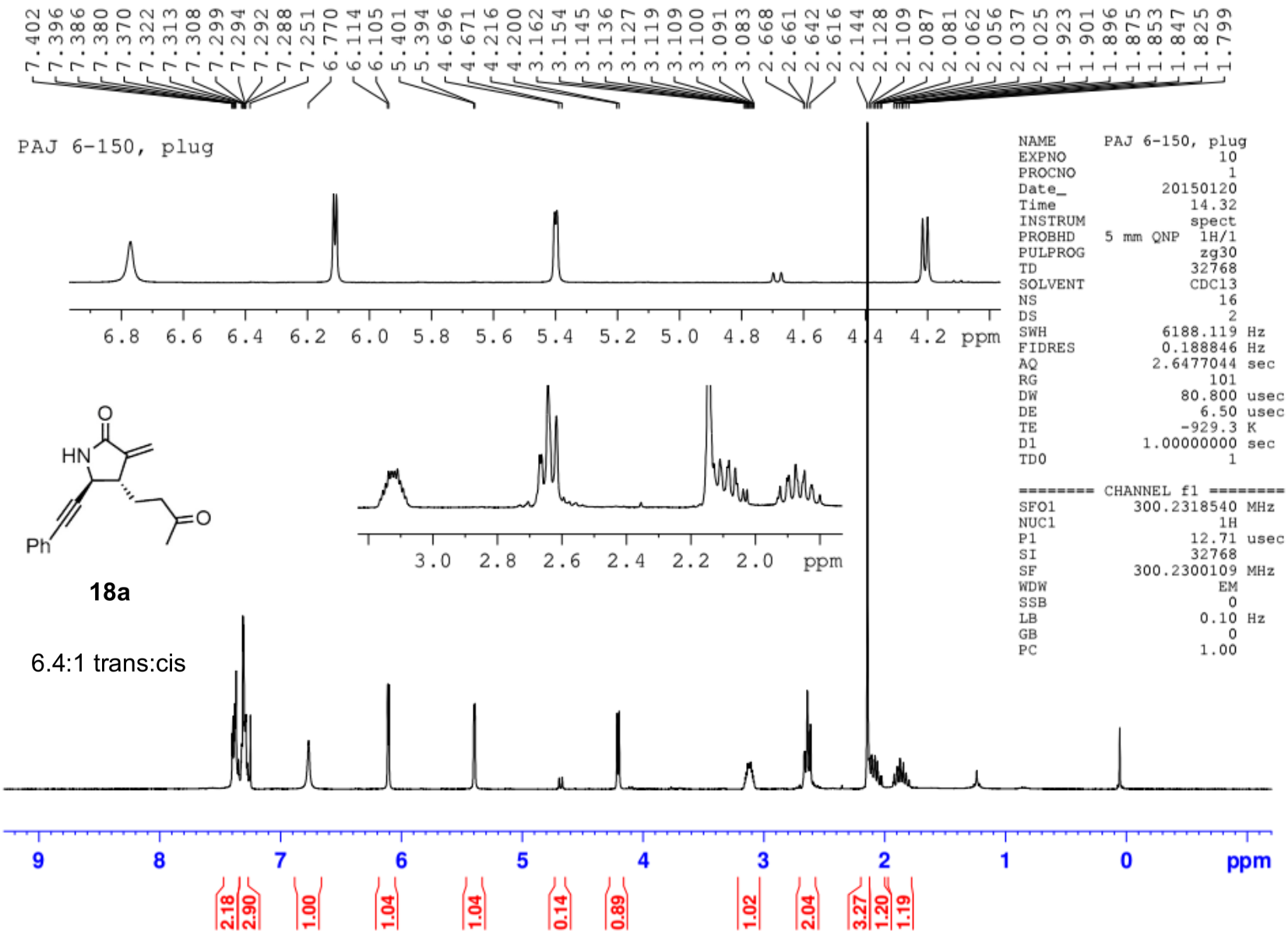

7.261
6.075
6.069
6.050
5.951
5.381
5.379
5.376
4.037
4.025
4.990
3.979
3.971
3.963
3.958
3.954
3.947
3.938
3.931
3.925
3.921
3.916
3.913
3.896
3.009
2.998
2.989
2.981
2.041
2.005
1.964
1.916
1.904
1.892
1.879
1.849
1.812
1.791
1.786
1.762
1.697
1.691
1.670
1.658
1.649
1.642
1.620
PAJ 7-16, 18-40
HN
O
TIPS
O
O
15a
6.0
5.5
5.0
4.5
ppm
3.0
2.5
2.0
ppm
1.3
1.2
ppm
NAME PAJ 7-16, 18-40
EXPNO 20
PROCNO 1
Date_ 20150312
Time 15.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.00
0.94
1.00
0.99
4.52
1.13
1.32
2.30
1.04
1.40
3.21
2.96
9.01
25.19

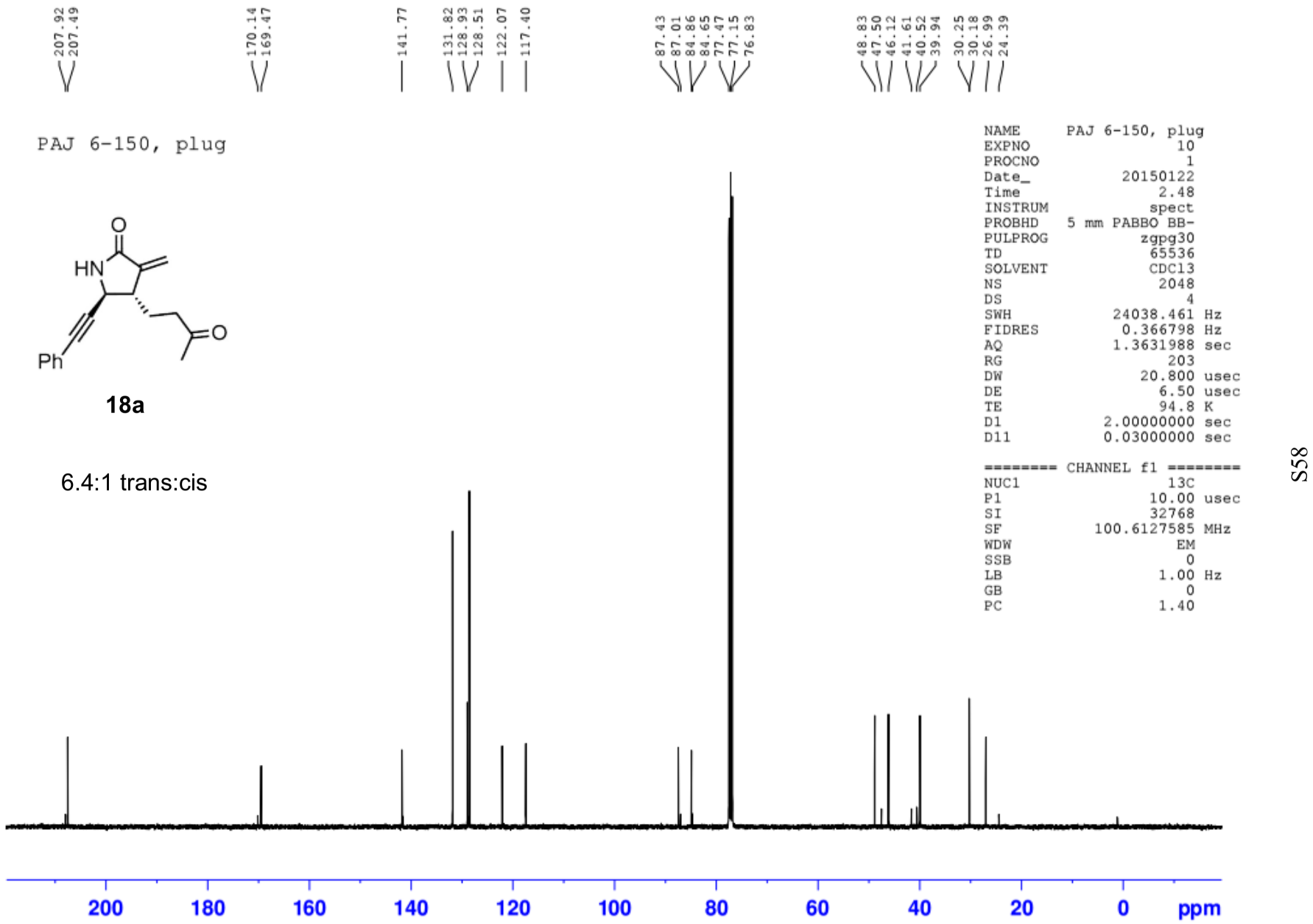

169.27
142.00
117.03
109.64
106.03
86.16
77.47
77.15
76.84
75.17
64.94
64.93
48.95
47.38
36.21
27.73
25.00
24.74
24.20
18.68
11.19
PAJ 7-16, trans CNMR
HN
O
TIPS
O
O
15a
NAME PAJ 7-16, trans
EXPNO 11
PROCNO 1
Date_ 20150409
Time 6.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 92.3 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127557 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

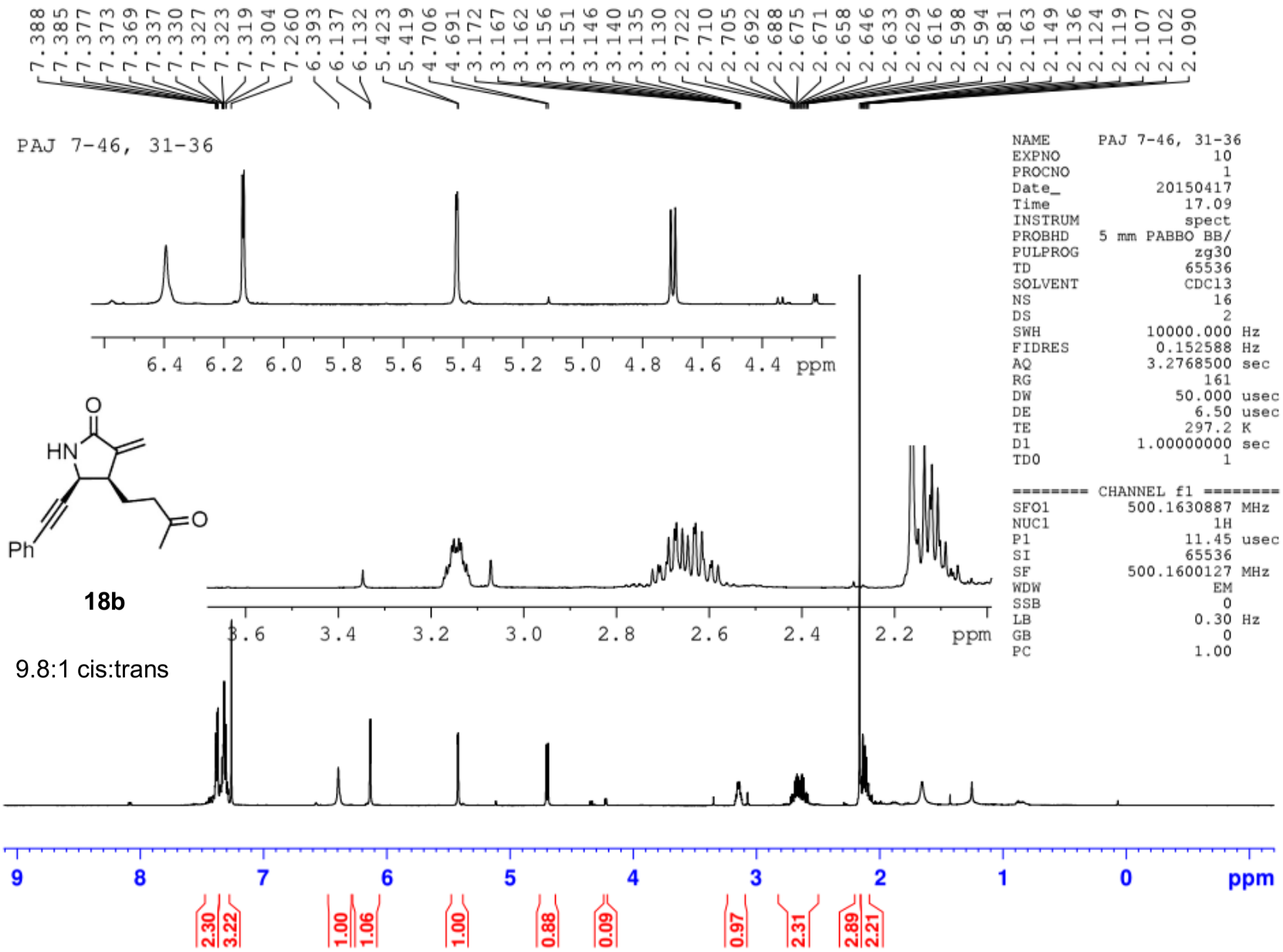

7.249 6.226 6.201 6.052 6.047 6.040 5.348 5.343 4.459 4.440 4.021 4.008 3.954 3.948 3.940 3.933 3.929 3.925 3.922 3.914 3.906 3.901 3.898 3.890 3.883 3.786 2.997 2.990 2.984 2.978 2.972 1.905 1.896 1.885 1.872 1.862 1.853 1.842 1.830 1.821 1.797 1.773 1.769 1.756 1.750 1.744 1.738 1.718 1.712 1.695 1.308 1.302 1.219 1.022
PAJ 7-16, 59-65
HN
O
TIPS
O
O
6.0 5.5 5.0 4.5 ppm
3.0 2.8 2.6 2.4 2.2 2.0 1.8 ppm
NAME PAJ 7-16, 59-65
EXPNO 10
PROCNO 1
Date_ 20150312
Time 15.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 50.8
DW 60.800 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300149 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
0.92 1.02 1.00 0.77 0.21 0.19 4.25 0.99 2.82 1.55 3.16 0.83 23.23

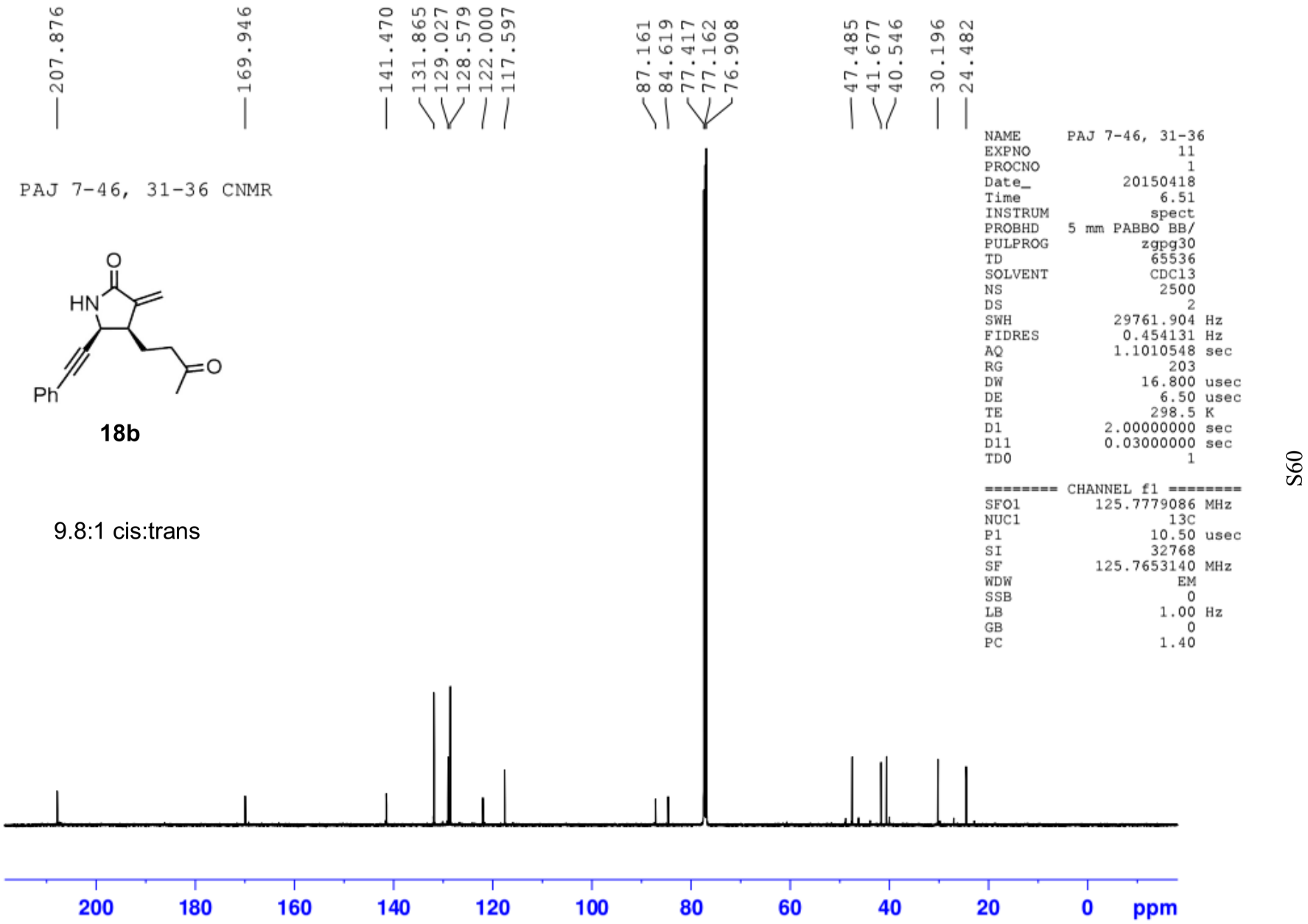
**15b**

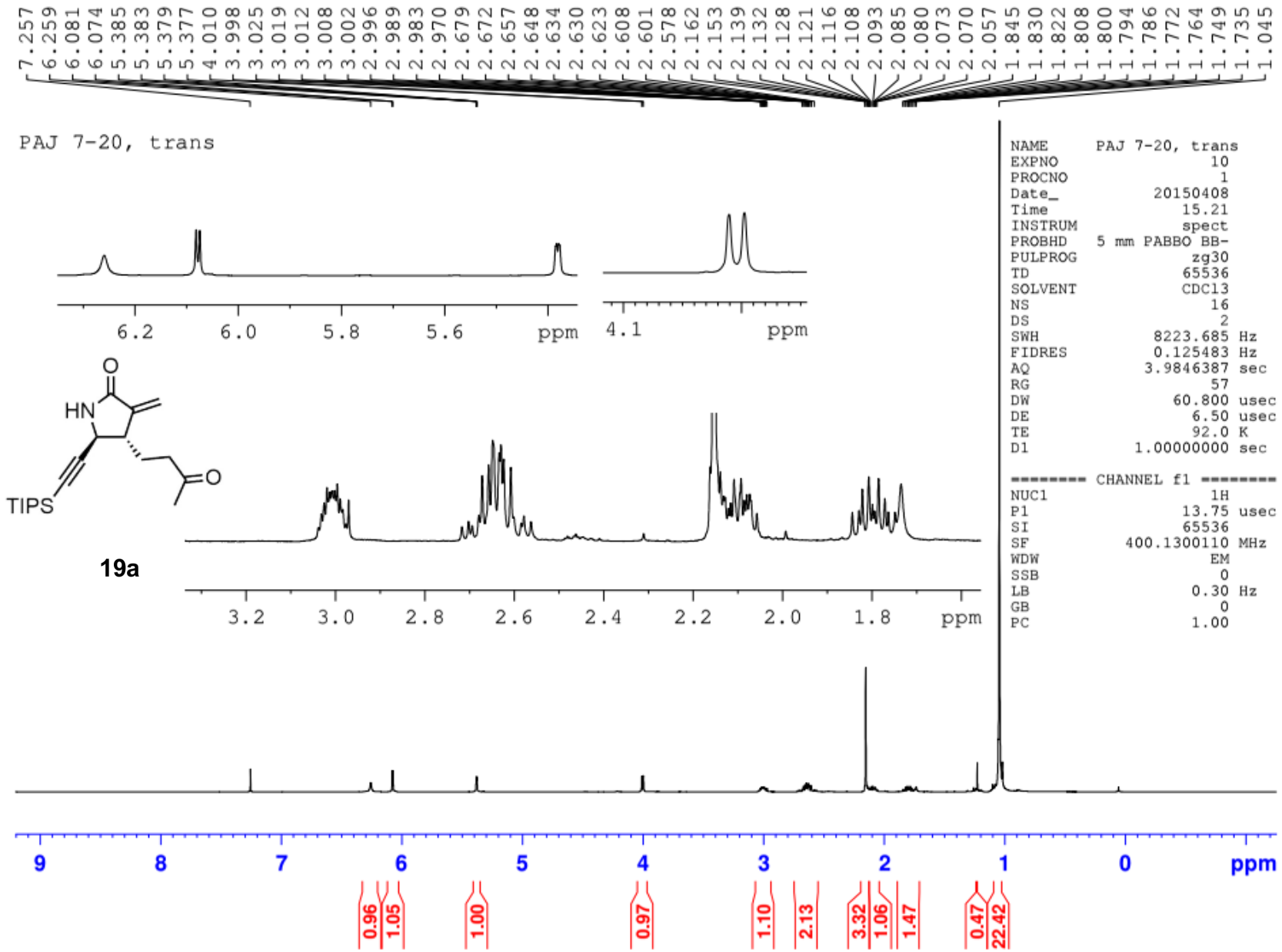

170.27
169.31
142.09
141.91
116.76
109.87
109.63
103.17
88.41
77.47
77.15
76.83
64.81
64.72
47.93
42.87
36.42
24.27
23.91
18.67
11.17
PAJ 7-16, 59-65 CNMR
HN
O
TIPS
O
O
15b
NAME PAJ 7-16, 59-65
EXPNO 11
PROCNO 1
Date_ 20150314
Time 22.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 311.5 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127572 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

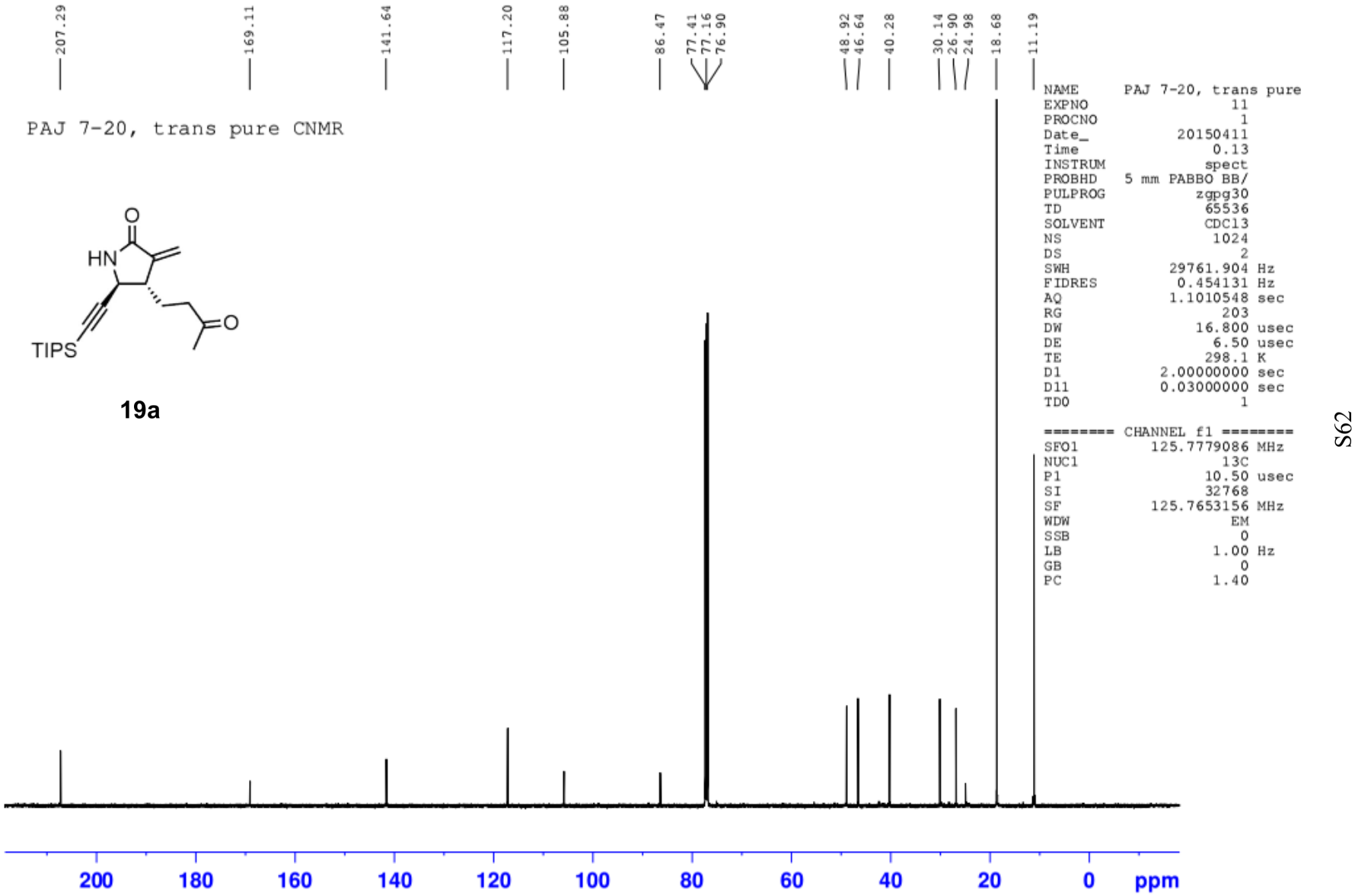

7.402 7.396 7.386 7.380 7.370 7.322 7.313 7.308 7.299 7.294 7.292 7.288 7.251 6.770 6.114 6.105 5.401 5.394 4.696 4.671 4.216 4.200 3.162 3.154 3.145 3.136 3.127 3.119 3.109 3.100 3.091 3.083 2.668 2.661 2.642 2.616 2.144 2.128 2.109 2.087 2.081 2.062 2.056 2.037 2.025 1.923 1.901 1.896 1.875 1.853 1.847 1.825 1.799
PAJ 6-150, plug
6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 ppm
3.0 2.8 2.6 2.4 2.2 2.0 ppm
HN
O
O
Ph
18a
6.4:1 trans:cis
9 8 7 6 5 4 3 2 1 0 ppm
2.18 2.90 1.00 1.04 1.04 0.14 0.89 1.02 2.04 3.27 1.20 1.19
NAME PAJ 6-150, plug
EXPNO 10
PROCNO 1
Date_ 20150120
Time 14.32
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 101
DW 80.800 usec
DE 6.50 usec
TE -929.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 300.2318540 MHz
NUC1 1H
P1 12.71 usec
SI 32768
SF 300.2300109 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

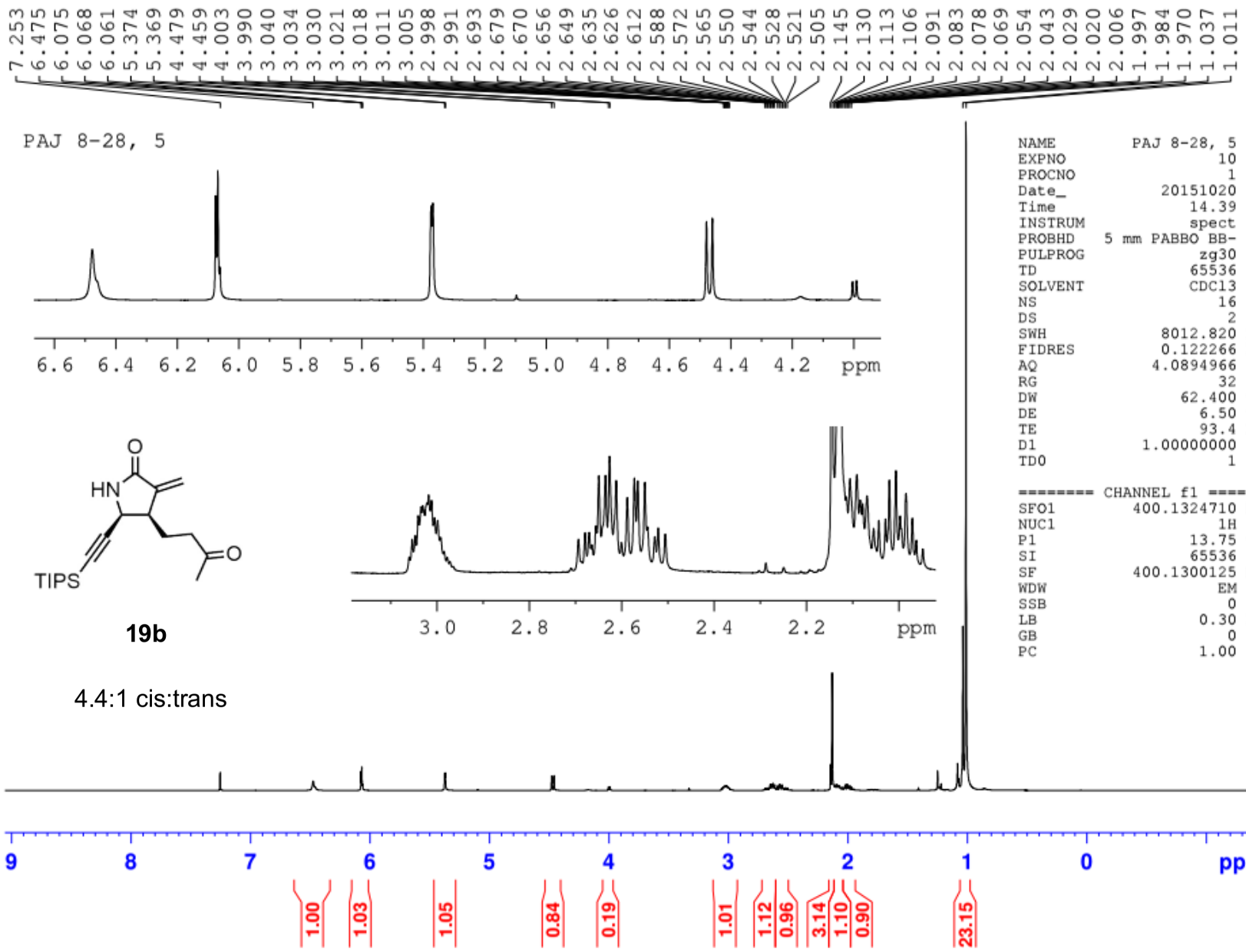

207.92
207.49
170.14
169.47
141.77
131.82
128.93
128.51
122.07
117.40
87.43
87.01
84.86
84.65
77.47
77.15
76.83
48.83
47.50
46.12
41.61
40.52
39.94
30.25
30.18
26.99
24.39
PAJ 6-150, plug
NAME PAJ 6-150, plug
EXPNO 10
PROCNO 1
Date_ 20150122
Time 2.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 94.8 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127585 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HN
O
O
Ph
18a
6.4:1 trans:cis
200
180
160
140
120
100
80
60
40
20
0
ppm

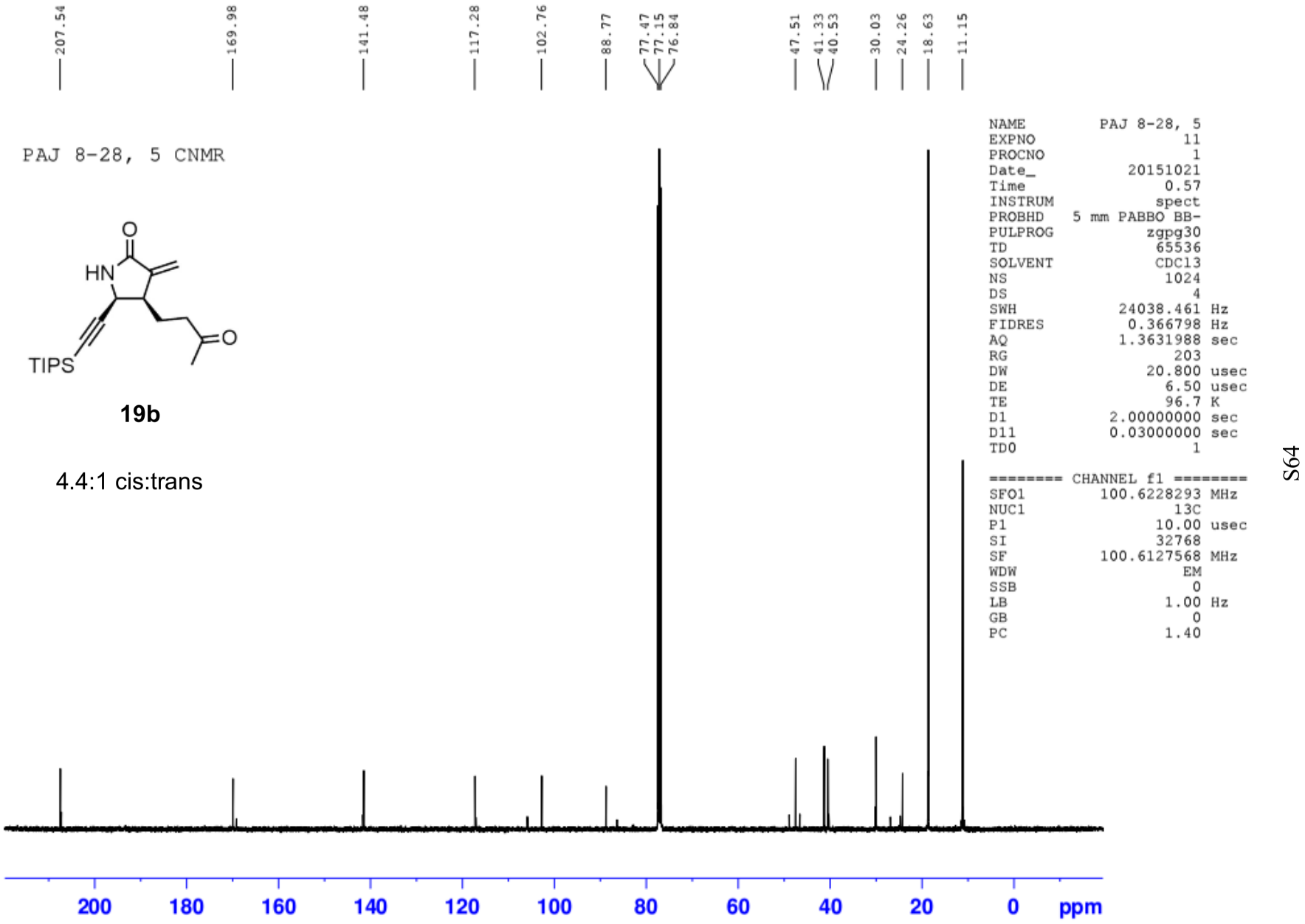

7.388
7.385
7.377
7.373
7.369
7.337
7.330
7.327
7.323
7.319
7.304
7.260
6.393
6.137
6.132
5.423
5.419
4.706
4.691
3.172
3.167
3.162
3.156
3.151
3.146
3.140
3.135
3.130
2.722
2.710
2.705
2.692
2.688
2.675
2.671
2.658
2.646
2.633
2.629
2.616
2.598
2.594
2.581
2.163
2.149
2.136
2.124
2.119
2.107
2.102
2.090
PAJ 7-46, 31-36
6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 ppm
3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 ppm
HN
O
O
Ph
18b
9.8:1 cis:trans
NAME PAJ 7-46, 31-36
EXPNO 10
PROCNO 1
Date_ 20150417
Time 17.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 161
DW 50.000 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600127 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
2.30 3.22 1.00 1.06 1.00 0.88 0.09 0.97 2.31 2.89 2.21

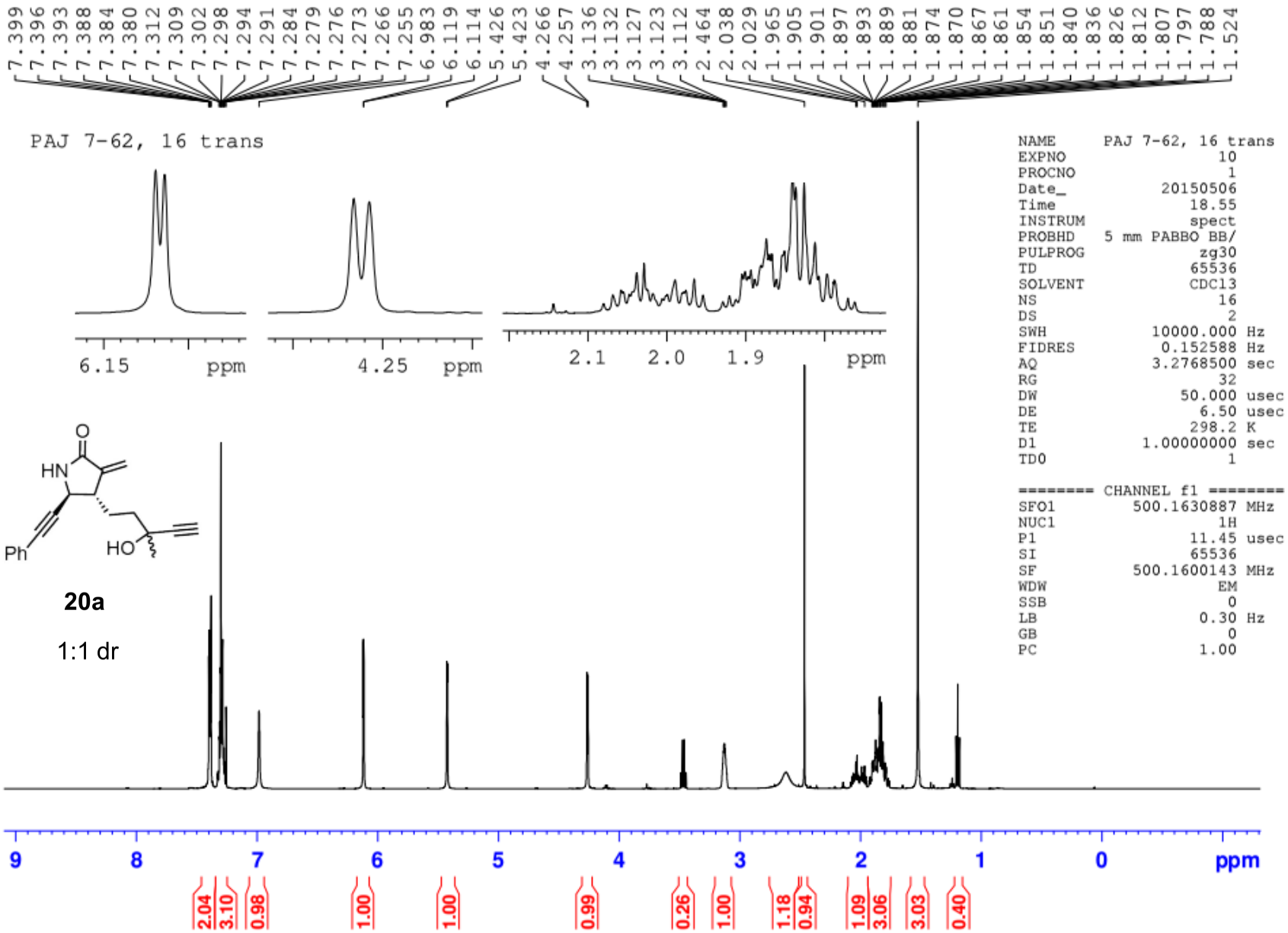

207.876
169.946
141.470
131.865
129.027
128.579
122.000
117.597
87.161
84.619
77.417
77.162
76.908
47.485
41.677
40.546
30.196
24.482
PAJ 7-46, 31-36 CNMR
HN
O
O
Ph
18b
9.8:1 cis:trans
NAME PAJ 7-46, 31-36
EXPNO 11
PROCNO 1
Date_ 20150418
Time 6.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2500
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

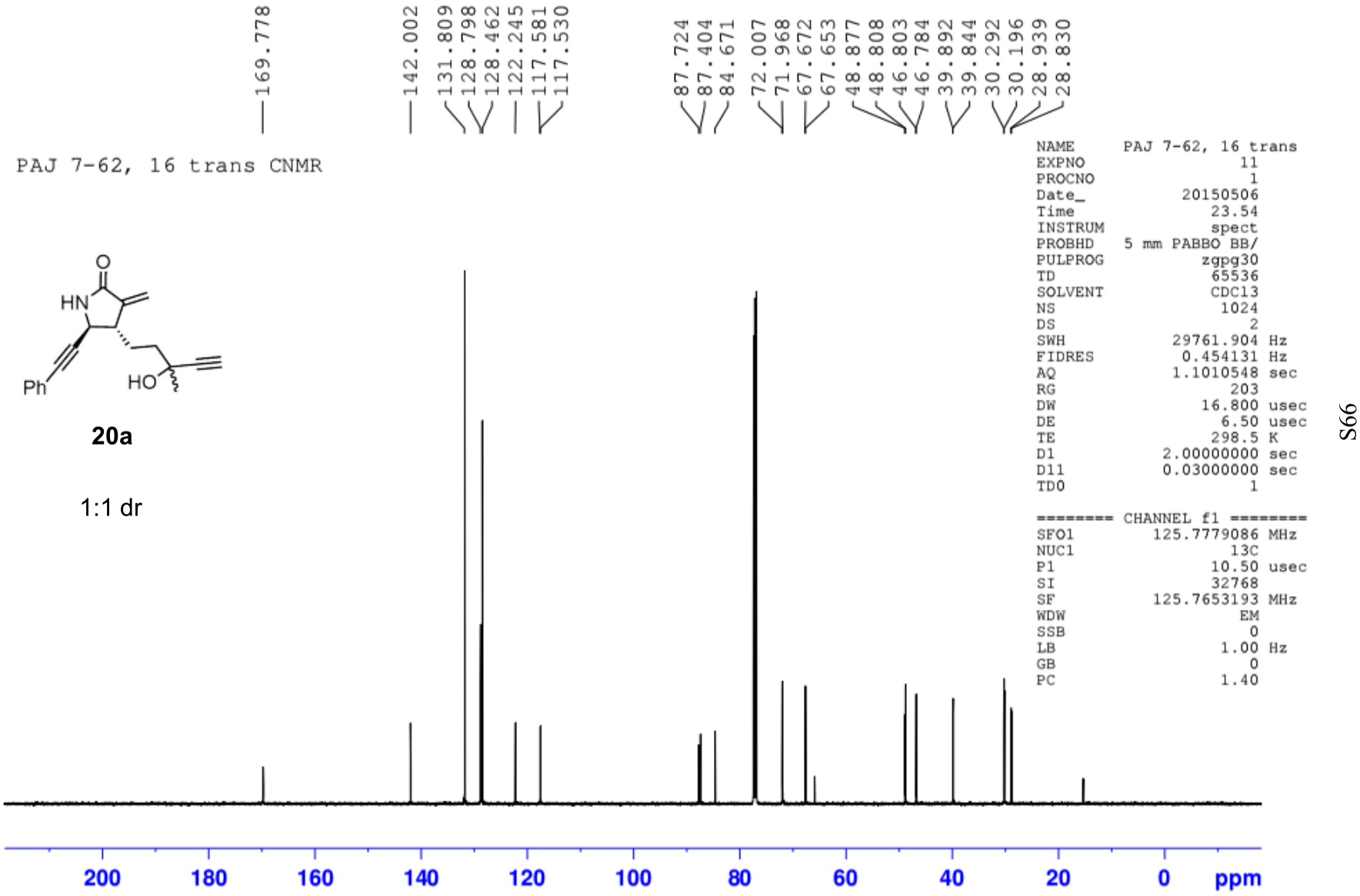

7.257 6.259 6.081 6.074 5.385 5.383 5.379 5.377 4.010 3.998 3.025 3.019 3.012 3.008 3.002 2.996 2.989 2.983 2.970 2.679 2.672 2.657 2.648 2.634 2.630 2.623 2.608 2.601 2.578 2.162 2.153 2.139 2.132 2.128 2.121 2.116 2.108 2.093 2.085 2.080 2.073 2.070 2.057 1.845 1.830 1.822 1.808 1.800 1.794 1.786 1.772 1.764 1.749 1.735 1.045
PAJ 7-20, trans
6.2 6.0 5.8 5.6 ppm
4.1 ppm
HN
O
O
TIPS
19a
3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 ppm
NAME PAJ 7-20, trans
EXPNO 10
PROCNO 1
Date_ 20150408
Time 15.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 57
DW 60.800 usec
DE 6.50 usec
TE 92.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
0.96 1.05 1.00 0.97 1.10 2.13 3.32 1.06 1.47 0.47 22.42

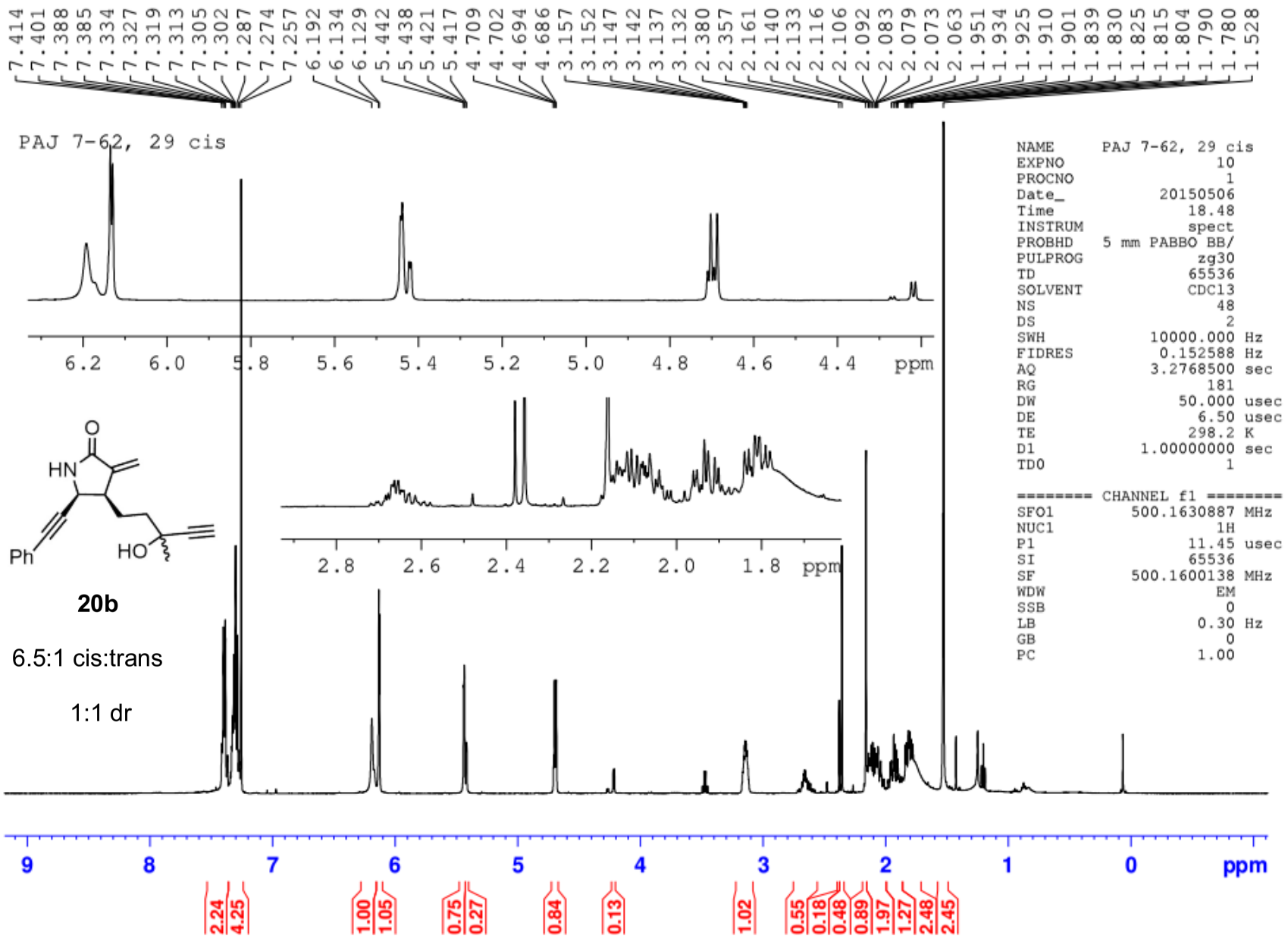

207.29
169.11
141.64
117.20
105.88
86.47
77.41
77.16
76.90
48.92
46.64
40.28
30.14
26.90
24.98
18.68
11.19
PAJ 7-20, trans pure CNMR
HN
O
O
TIPS
19a
NAME PAJ 7-20, trans pure
EXPNO 11
PROCNO 1
Date_ 20150411
Time 0.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653156 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

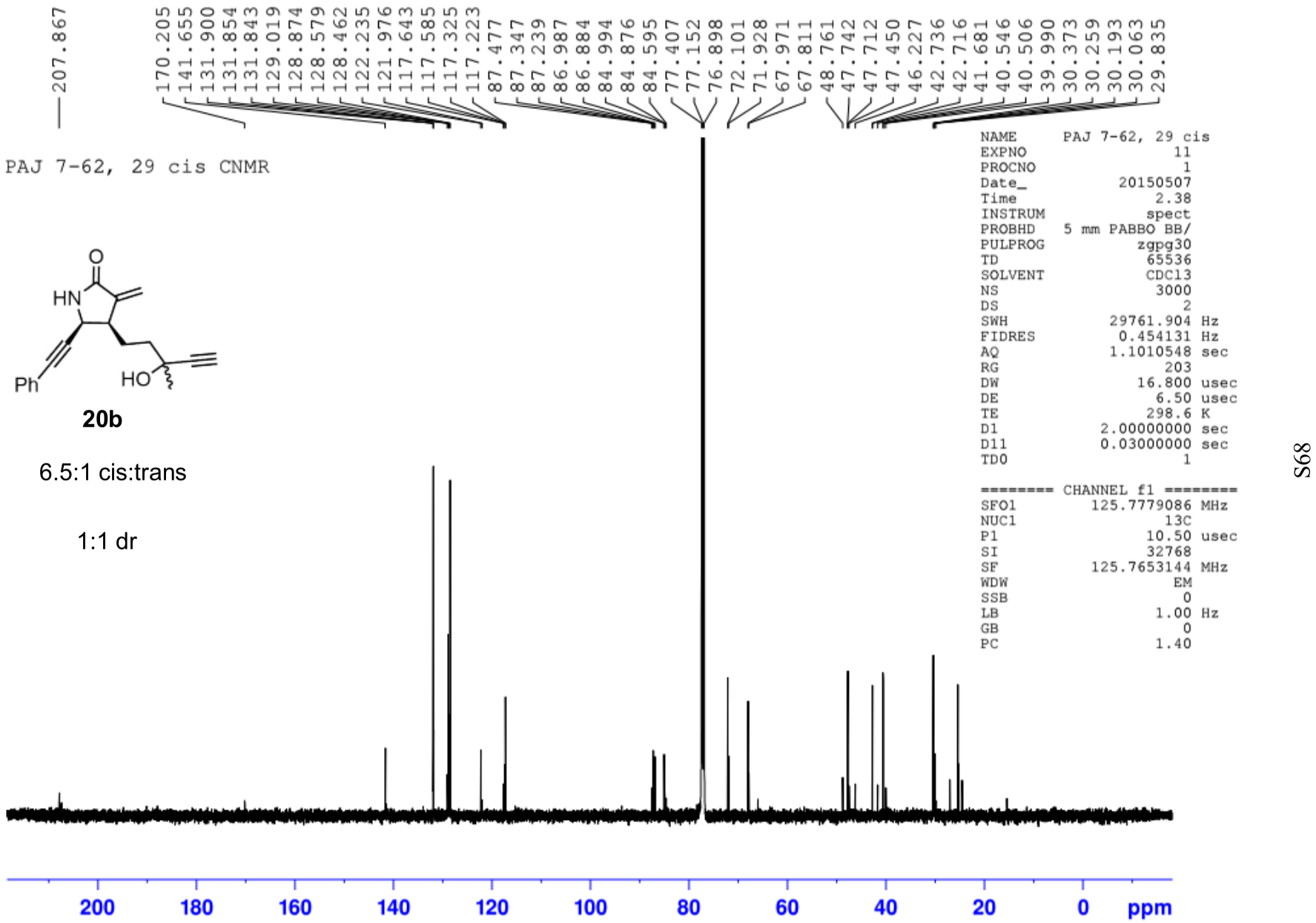

7.253 6.475 6.075 6.068 6.061 5.374 5.369 4.479 4.459 4.003 3.990 3.040 3.034 3.030 3.021 3.018 3.011 3.005 2.998 2.991 2.693 2.679 2.670 2.656 2.649 2.635 2.626 2.612 2.588 2.572 2.565 2.550 2.544 2.528 2.521 2.505 2.145 2.130 2.113 2.106 2.091 2.083 2.078 2.069 2.054 2.043 2.029 2.020 2.006 1.997 1.984 1.970 1.037 1.011
PAJ 8-28, 5
6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 ppm
3.0 2.8 2.6 2.4 2.2 ppm
HN
O
O
TIPS
19b
4.4:1 cis:trans
NAME PAJ 8-28, 5
EXPNO 10
PROCNO 1
Date_ 20151020
Time 14.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 32
DW 62.400 usec
DE 6.50 usec
TE 93.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.00 1.03 1.05 0.84 0.19 1.01 1.12 0.96 3.14 1.10 0.90 23.15

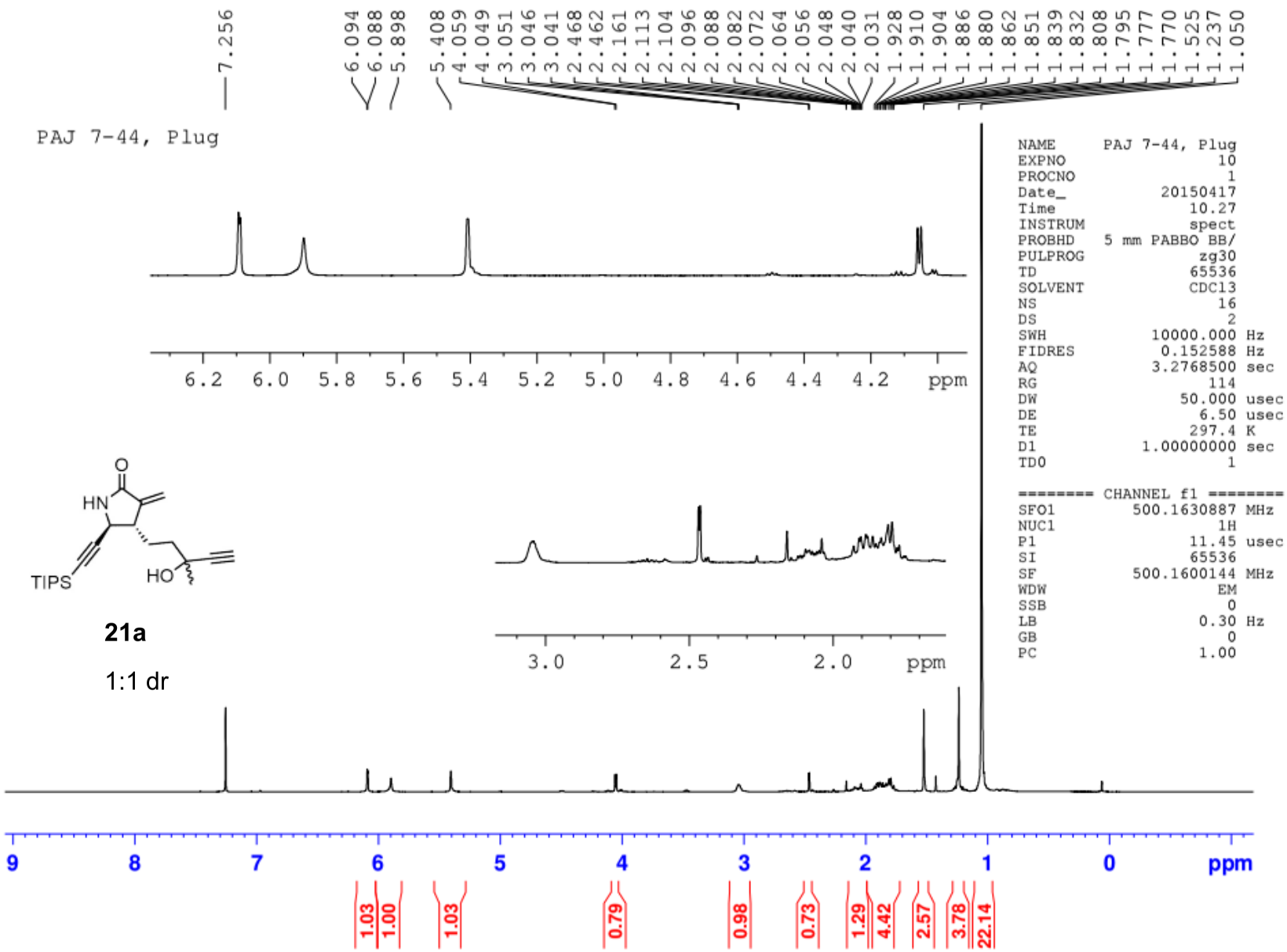

207.54
169.98
141.48
117.28
102.76
88.77
77.47
77.15
76.84
47.51
41.33
40.53
30.03
24.26
18.63
11.15
PAJ 8-28, 5 CNMR
HN
O
O
TIPS
19b
4.4:1 cis:trans
NAME PAJ 8-28, 5
EXPNO 11
PROCNO 1
Date_ 20151021
Time 0.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 96.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127568 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

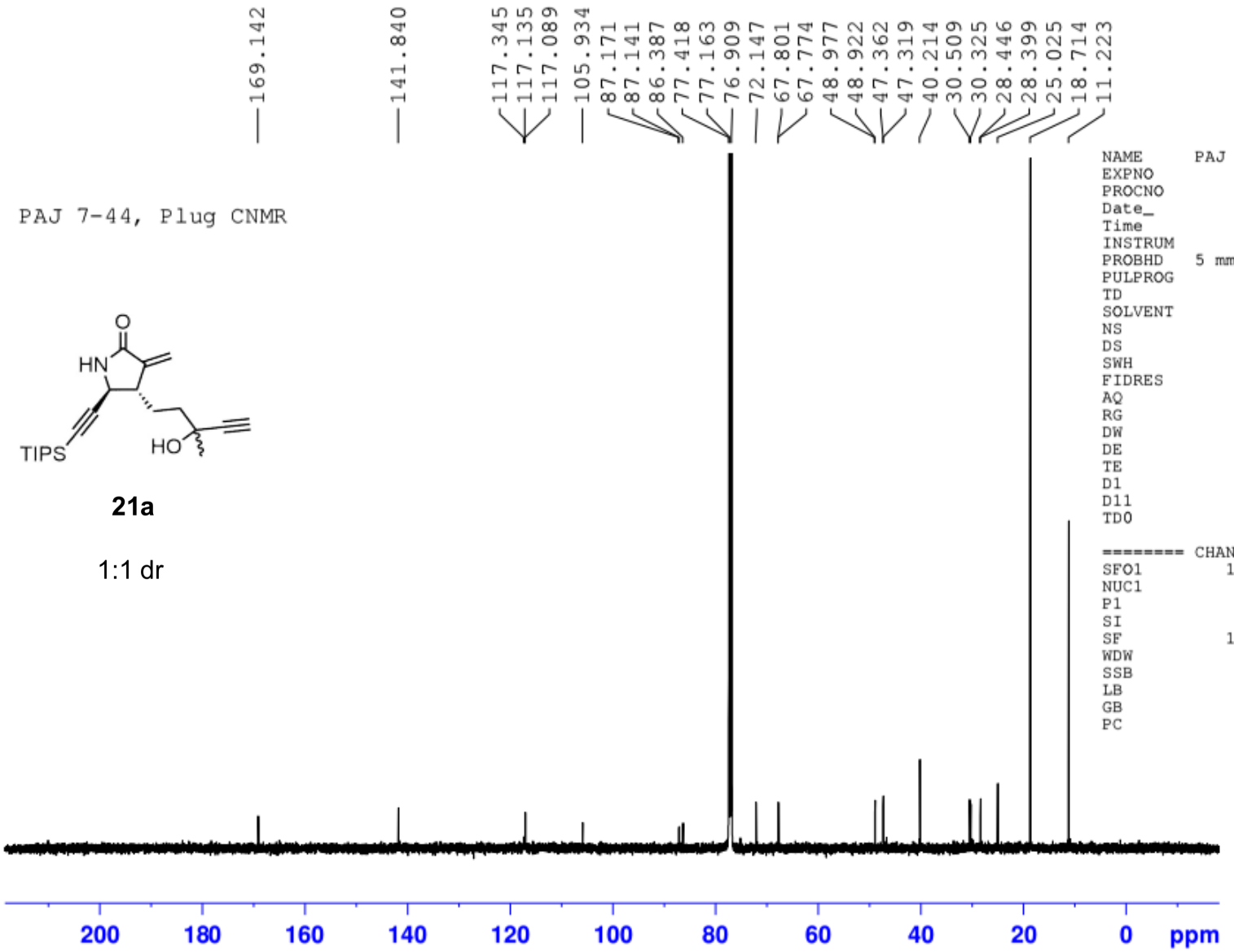

7.399 7.396 7.393 7.388 7.384 7.380 7.312 7.309 7.302 7.298 7.294 7.291 7.284 7.279 7.276 7.273 7.266 7.255 6.983 6.119 6.114 5.426 5.423 4.266 4.257 3.136 3.132 3.127 3.123 3.112 2.464 2.038 2.029 1.965 1.905 1.901 1.897 1.893 1.889 1.881 1.874 1.870 1.867 1.861 1.854 1.851 1.840 1.836 1.826 1.812 1.807 1.797 1.788 1.524
PAJ 7-62, 16 trans
6.15 ppm
4.25 ppm
2.1 2.0 1.9 ppm
NAME PAJ 7-62, 16 trans
EXPNO 10
PROCNO 1
Date_ 20150506
Time 18.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 32
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600143 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HN
O
Ph
HO
20a
1:1 dr
9 8 7 6 5 4 3 2 1 0 ppm
2.04 3.10 0.98 1.00 1.00 0.99 0.26 1.00 1.18 0.94 1.09 3.06 3.03 0.40

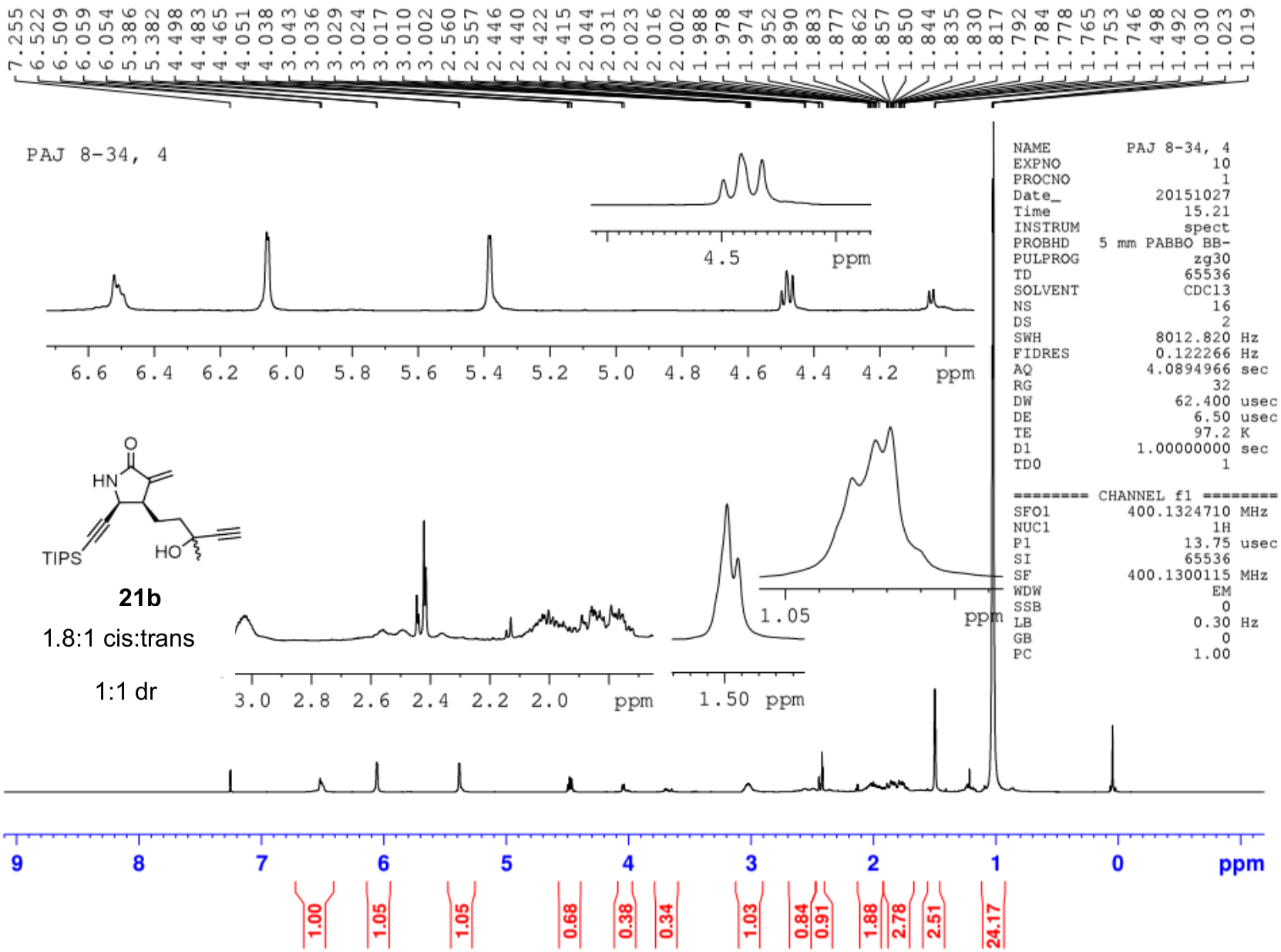

169.778
142.002
131.809
128.798
128.462
122.245
117.581
117.530
87.724
87.404
84.671
72.007
71.968
67.672
67.653
48.877
48.808
46.803
46.784
39.892
39.844
30.292
30.196
28.939
28.830
PAJ 7-62, 16 trans CNMR
NAME PAJ 7-62, 16 trans
EXPNO 11
PROCNO 1
Date_ 20150506
Time 23.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HN
O
Ph
HO
20a
1:1 dr
200
180
160
140
120
100
80
60
40
20
0
ppm

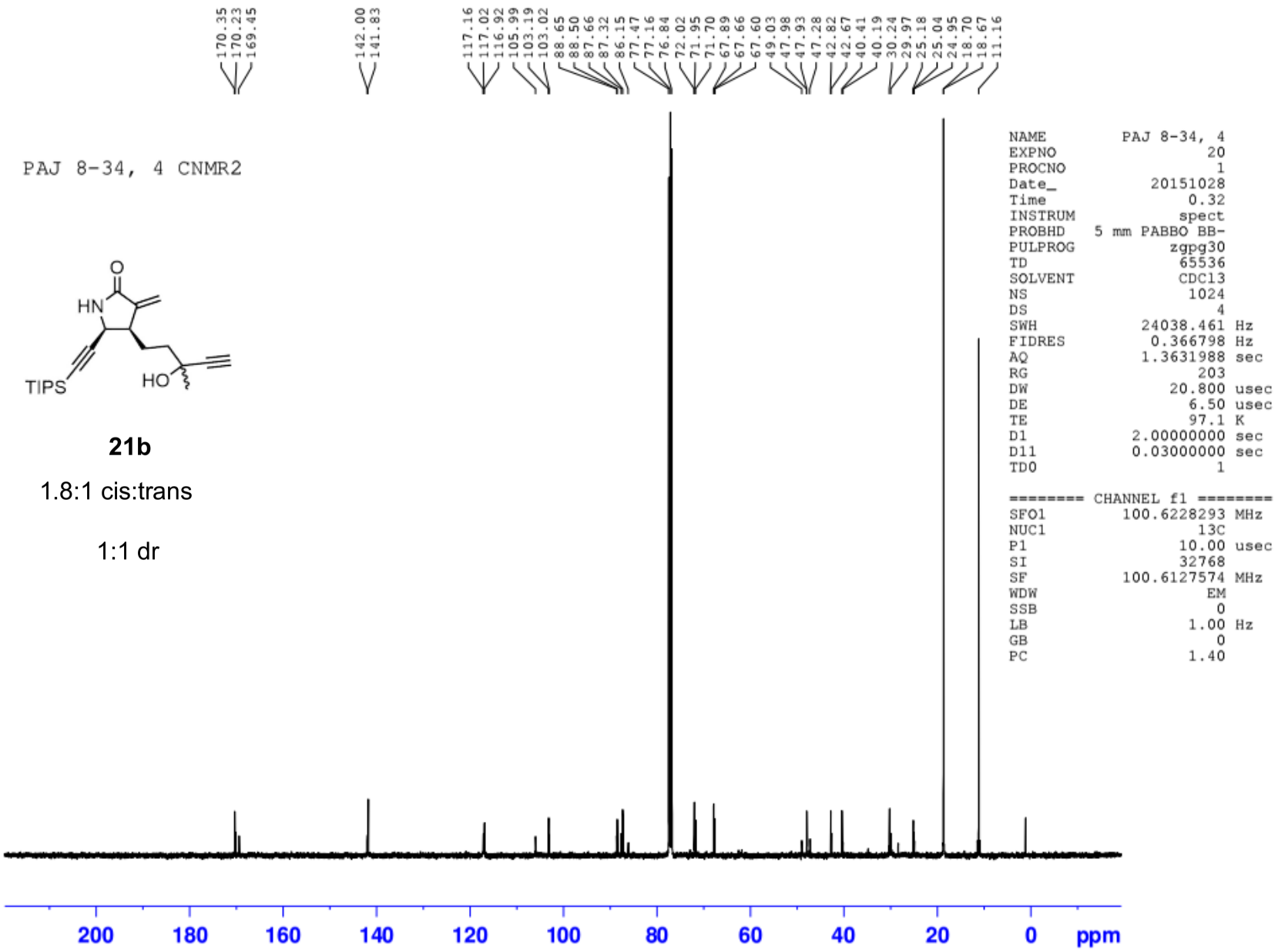

7.414 7.401 7.388 7.385 7.334 7.327 7.319 7.313 7.305 7.302 7.287 7.274 7.257 6.192 6.134 6.129 5.442 5.438 5.421 5.417 4.709 4.702 4.694 4.686 3.157 3.152 3.147 3.142 3.137 3.132 2.380 2.357 2.161 2.140 2.133 2.116 2.106 2.092 2.083 2.079 2.073 2.063 1.951 1.934 1.925 1.910 1.901 1.839 1.830 1.825 1.815 1.804 1.790 1.780 1.528
PAJ 7-62, 29 cis
6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 ppm
2.8 2.6 2.4 2.2 2.0 1.8 ppm
NAME PAJ 7-62, 29 cis
EXPNO 10
PROCNO 1
Date_ 20150506
Time 18.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 48
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 181
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HN
O
Ph
HO
20b
6.5:1 cis:trans
1:1 dr
9 8 7 6 5 4 3 2 1 0 ppm
2.24 4.25 1.00 1.05 0.75 0.27 0.84 0.13 1.02 0.55 0.18 0.48 0.89 1.97 1.27 2.48 2.45


207.867
170.205
141.655
131.900
131.854
131.843
129.019
128.874
128.579
128.462
122.235
121.976
117.643
117.585
117.325
117.223
87.477
87.347
87.239
86.987
86.884
84.994
84.876
84.595
77.407
77.152
76.898
72.101
71.928
67.971
67.811
48.761
47.742
47.712
47.450
46.227
42.736
42.716
41.681
40.546
40.506
39.990
30.373
30.259
30.193
30.063
29.835
PAJ 7-62, 29 cis CNMR
HN
O
Ph
HO
20b
6.5:1 cis:trans
1:1 dr
NAME PAJ 7-62, 29 cis
EXPNO 11
PROCNO 1
Date_ 20150507
Time 2.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 3000
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653144 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


7.256
6.094
6.088
5.898
5.408
4.059
4.049
3.051
3.046
3.041
2.468
2.462
2.161
2.113
2.104
2.096
2.088
2.082
2.072
2.064
2.056
2.048
2.040
2.031
1.928
1.910
1.904
1.886
1.880
1.862
1.851
1.839
1.832
1.808
1.795
1.777
1.770
1.525
1.237
1.050
PAJ 7-44, Plug
6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 ppm
3.0 2.5 2.0 ppm
HN
O
HO
TIPS
21a
1:1 dr
NAME PAJ 7-44, Plug
EXPNO 10
PROCNO 1
Date_ 20150417
Time 10.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 114
DW 50.000 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600144 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.03
1.00
1.03
0.79
0.98
0.73
1.29
4.42
2.57
3.78
22.14


169.142
141.840
117.345
117.135
117.089
105.934
87.171
87.141
86.387
77.418
77.163
76.909
72.147
67.801
67.774
48.977
48.922
47.362
47.319
40.214
30.509
30.325
28.446
28.399
25.025
18.714
11.223
PAJ 7-44, Plug CNMR
HN
O
TIPS
HO
21a
1:1 dr
NAME PAJ 7-44, Plug
EXPNO 11
PROCNO 1
Date_ 20150417
Time 22.53
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2048
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653130 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

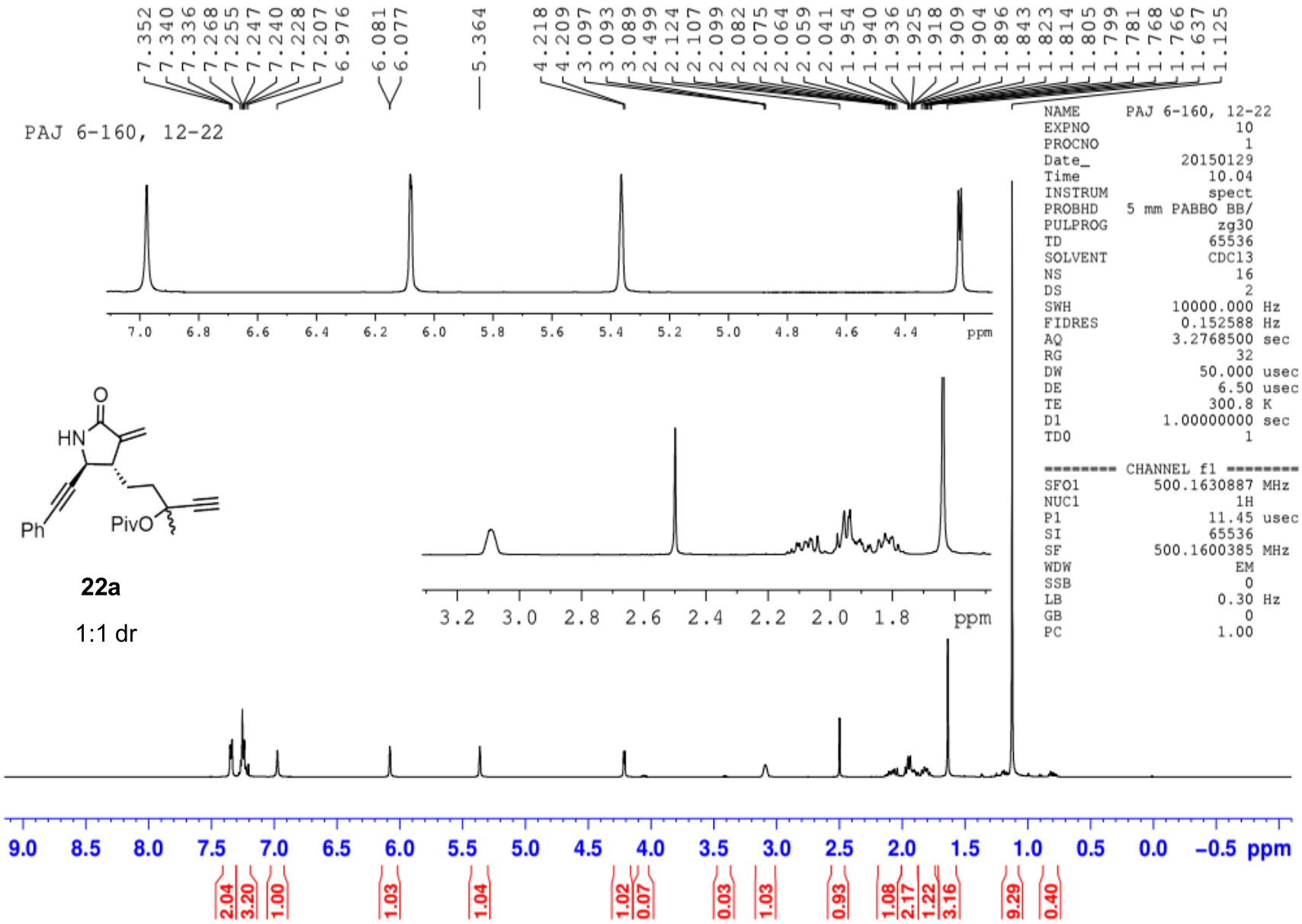

7.255 6.522 6.509 6.059 6.054 5.386 5.382 4.498 4.483 4.465 4.051 4.038 3.043 3.036 3.029 3.024 3.017 3.010 3.002 2.560 2.557 2.446 2.440 2.422 2.415 2.044 2.031 2.023 2.016 2.002 1.988 1.978 1.974 1.952 1.890 1.883 1.877 1.862 1.857 1.850 1.844 1.835 1.830 1.817 1.792 1.784 1.778 1.765 1.753 1.746 1.498 1.492 1.030 1.023 1.019
PAJ 8-34, 4
NAME PAJ 8-34, 4
EXPNO 10
PROCNO 1
Date_ 20151027
Time 15.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 32
DW 62.400 usec
DE 6.50 usec
TE 97.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300115 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HN
O
TIPS
HO
21b
1.8:1 cis:trans
1:1 dr
4.5 ppm
6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 ppm
1.05 ppm
3.0 2.8 2.6 2.4 2.2 2.0 ppm
1.50 ppm
9 8 7 6 5 4 3 2 1 0 ppm
1.00 1.05 1.05 0.68 0.38 0.34 1.03 0.84 0.91 1.88 2.78 2.51 24.17



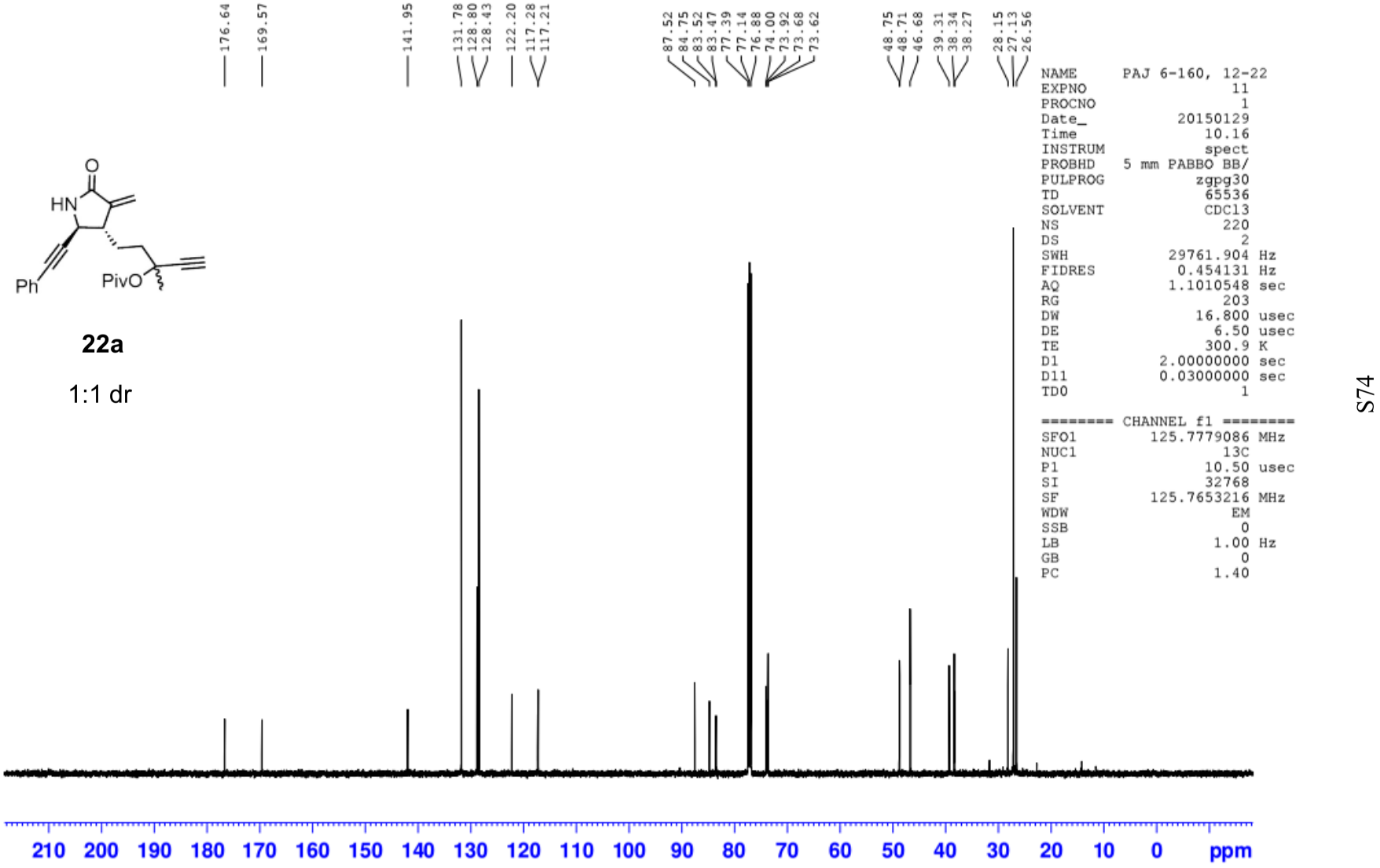

**21b**

1.8:1 cis:trans

1:1 dr

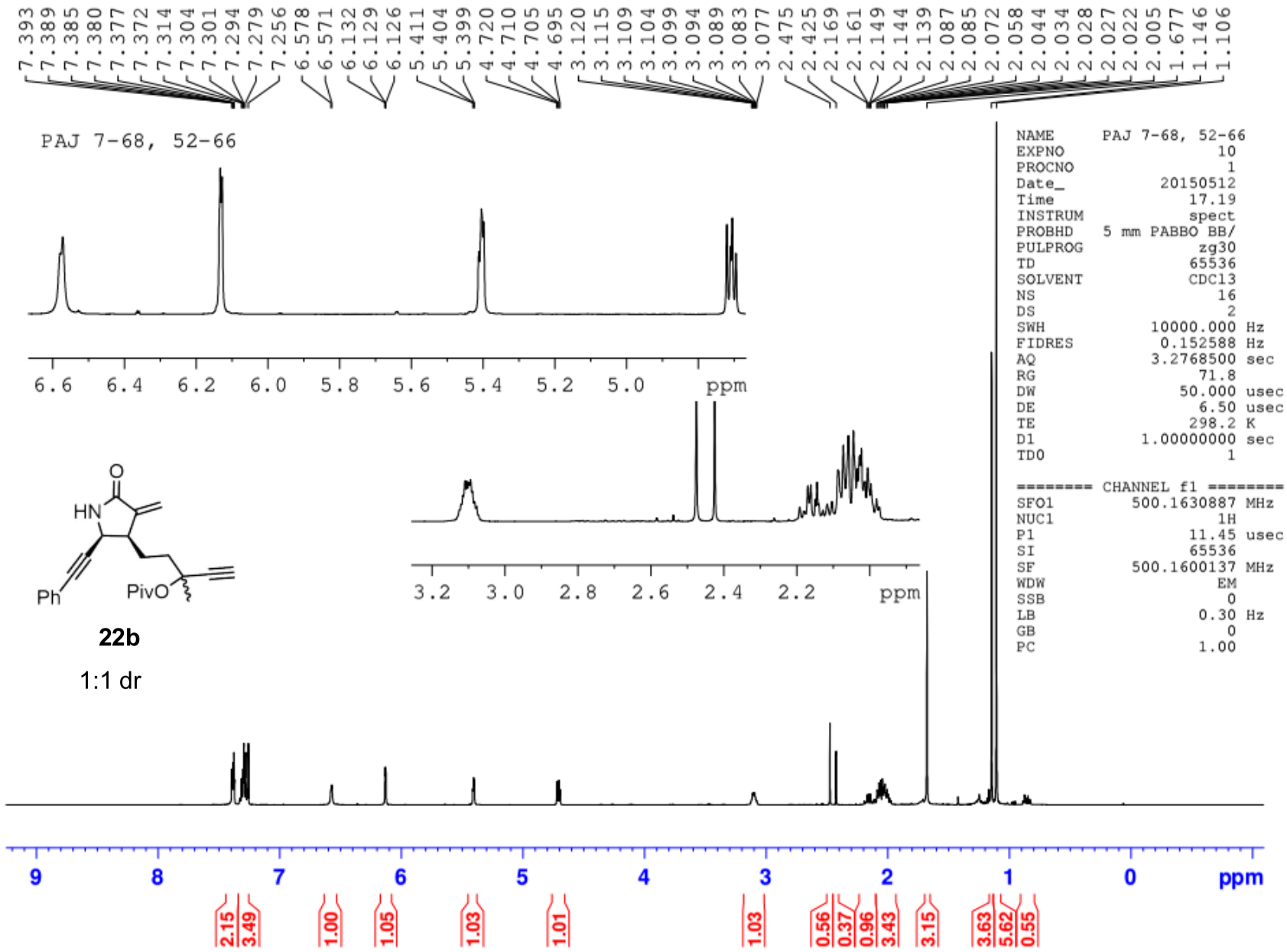

7.352
7.340
7.336
7.268
7.255
7.247
7.240
7.228
7.207
6.976
6.081
6.077
5.364
4.218
4.209
3.097
3.093
3.089
2.499
2.124
2.107
2.099
2.082
2.075
2.064
2.059
2.041
1.954
1.940
1.936
1.925
1.918
1.909
1.904
1.896
1.843
1.823
1.814
1.805
1.799
1.781
1.768
1.766
1.637
1.125
PAJ 6-160, 12-22
NAME PAJ 6-160, 12-22
EXPNO 10
PROCNO 1
Date_ 20150129
Time 10.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 32
DW 50.000 usec
DE 6.50 usec
TE 300.8 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600385 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HN
O
Ph
PivO
22a
1:1 dr
ppm
2.04
3.20
1.00
1.03
1.04
1.02
0.07
0.03
1.03
0.93
1.08
2.17
1.22
3.16
9.29
0.40

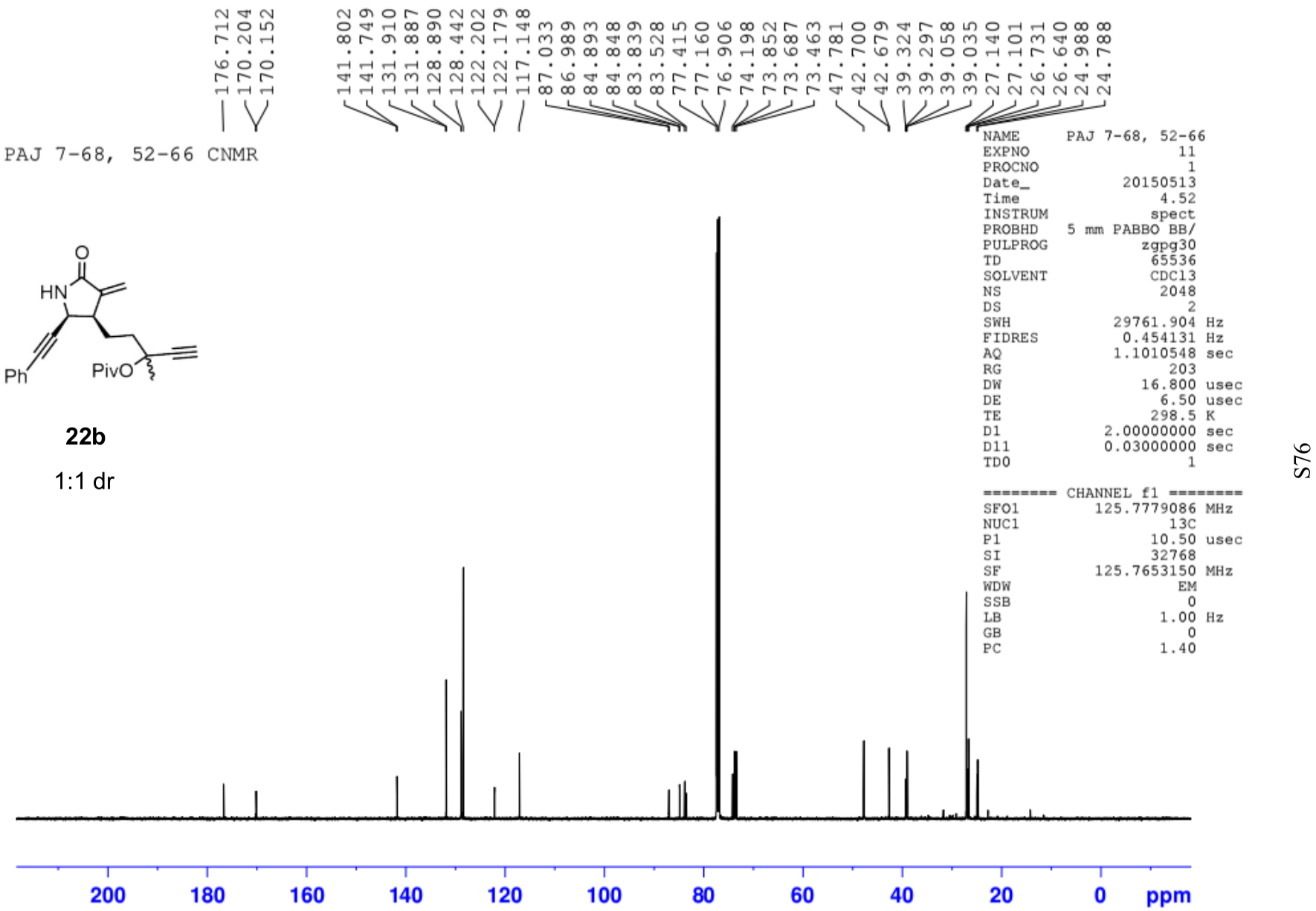
PAJ 6-160, 12-22 CNMR




**22a**

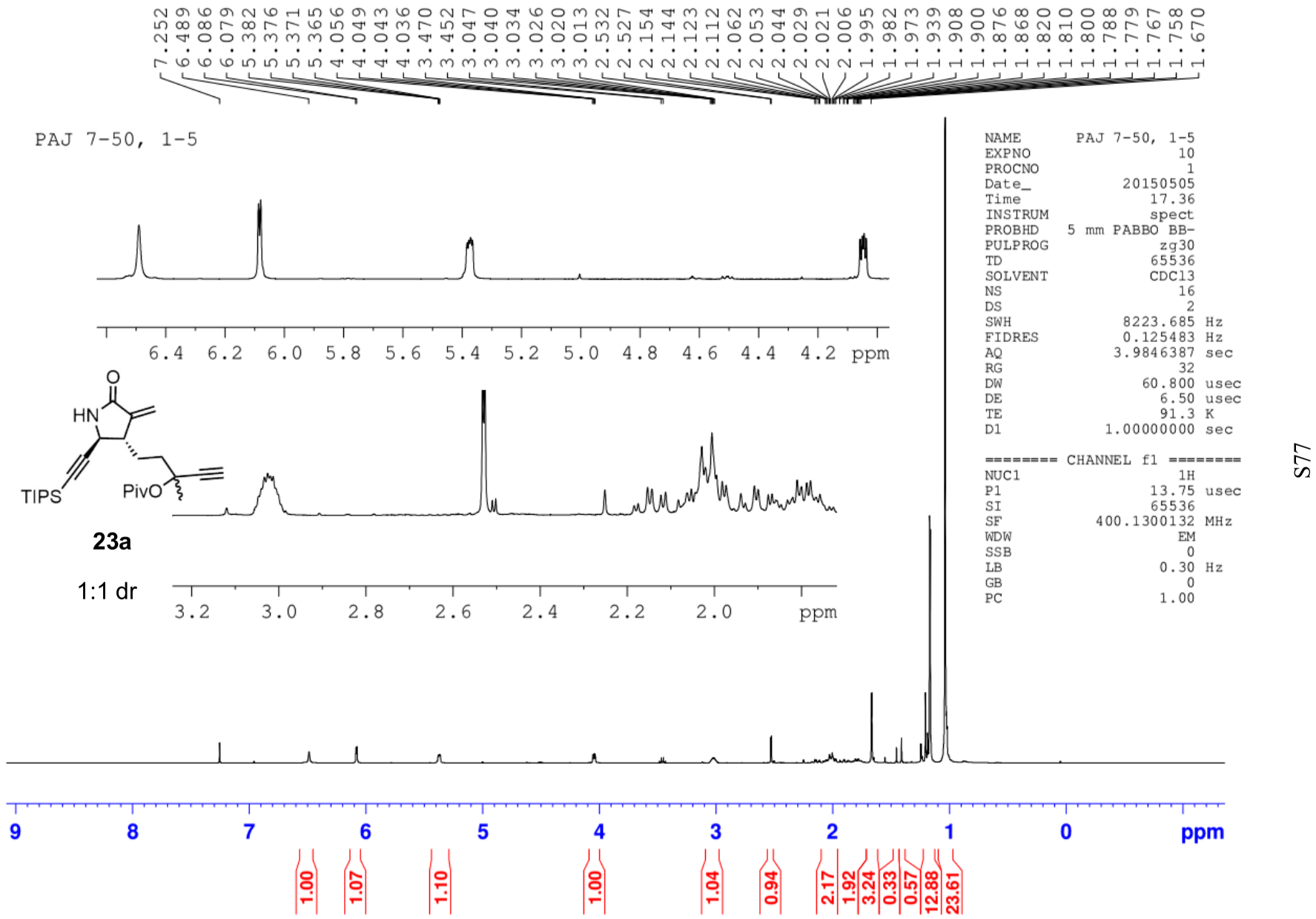
1:1 dr

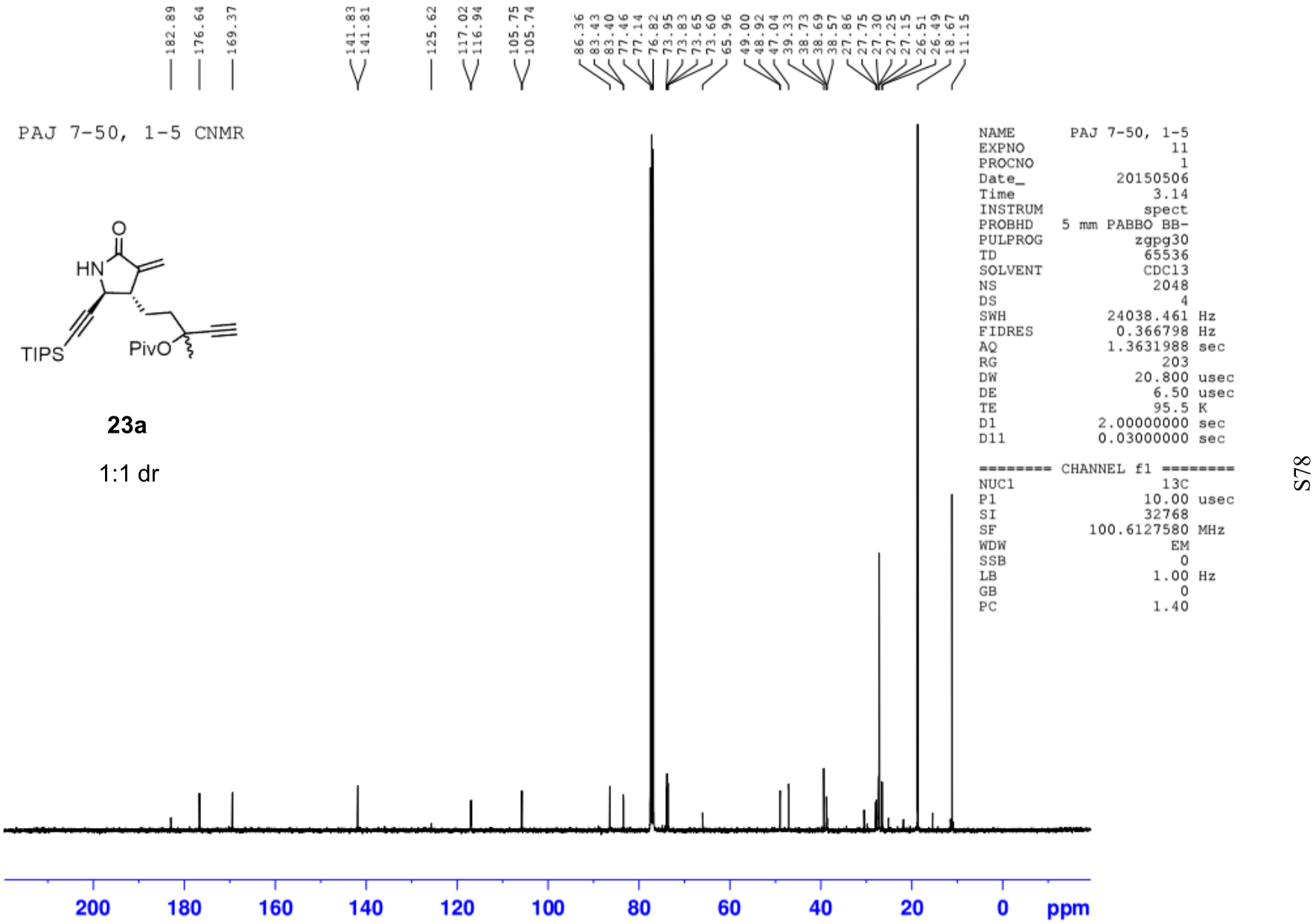

7.393 7.389 7.385 7.380 7.377 7.372 7.314 7.304 7.301 7.294 7.279 7.256 6.578 6.571 6.132 6.129 6.126 5.411 5.404 5.399 4.720 4.710 4.705 4.695 3.120 3.115 3.109 3.104 3.099 3.094 3.089 3.083 3.077 2.475 2.425 2.169 2.161 2.149 2.144 2.139 2.087 2.085 2.072 2.058 2.044 2.034 2.028 2.027 2.022 2.005 1.677 1.146 1.106
PAJ 7-68, 52-66
6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 ppm
3.2 3.0 2.8 2.6 2.4 2.2 ppm
NAME PAJ 7-68, 52-66
EXPNO 10
PROCNO 1
Date_ 20150512
Time 17.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 71.8
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600137 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HN
O
Ph
PivO
22b
1:1 dr
9 8 7 6 5 4 3 2 1 0 ppm
2.15 3.49 1.00 1.05 1.03 1.01 1.03 0.56 0.37 0.96 3.43 3.15 3.63 5.62 0.55

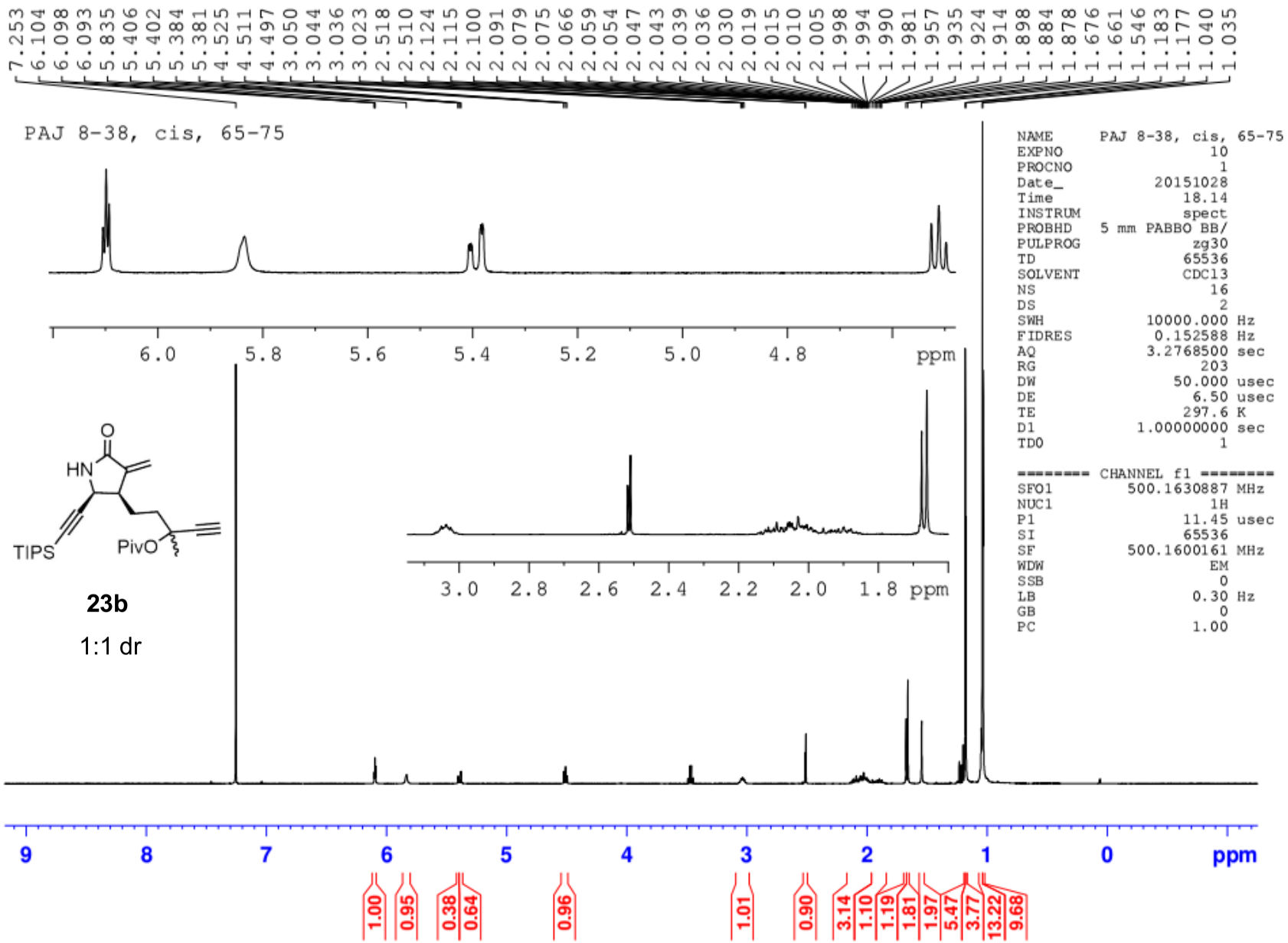

176.712
170.204
170.152
141.802
141.749
131.910
131.887
128.890
128.442
122.202
122.179
117.148
87.033
86.989
84.893
84.848
83.839
83.528
77.415
77.160
76.906
74.198
73.852
73.687
73.463
47.781
42.700
42.679
39.324
39.297
39.058
39.035
27.140
27.101
26.731
26.640
24.988
24.788
PAJ 7-68, 52-66 CNMR
NAME PAJ 7-68, 52-66
EXPNO 11
PROCNO 1
Date_ 20150513
Time 4.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653150 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HN
O
Ph
PivO
200
180
160
140
120
100
80
60
40
20
0
ppm

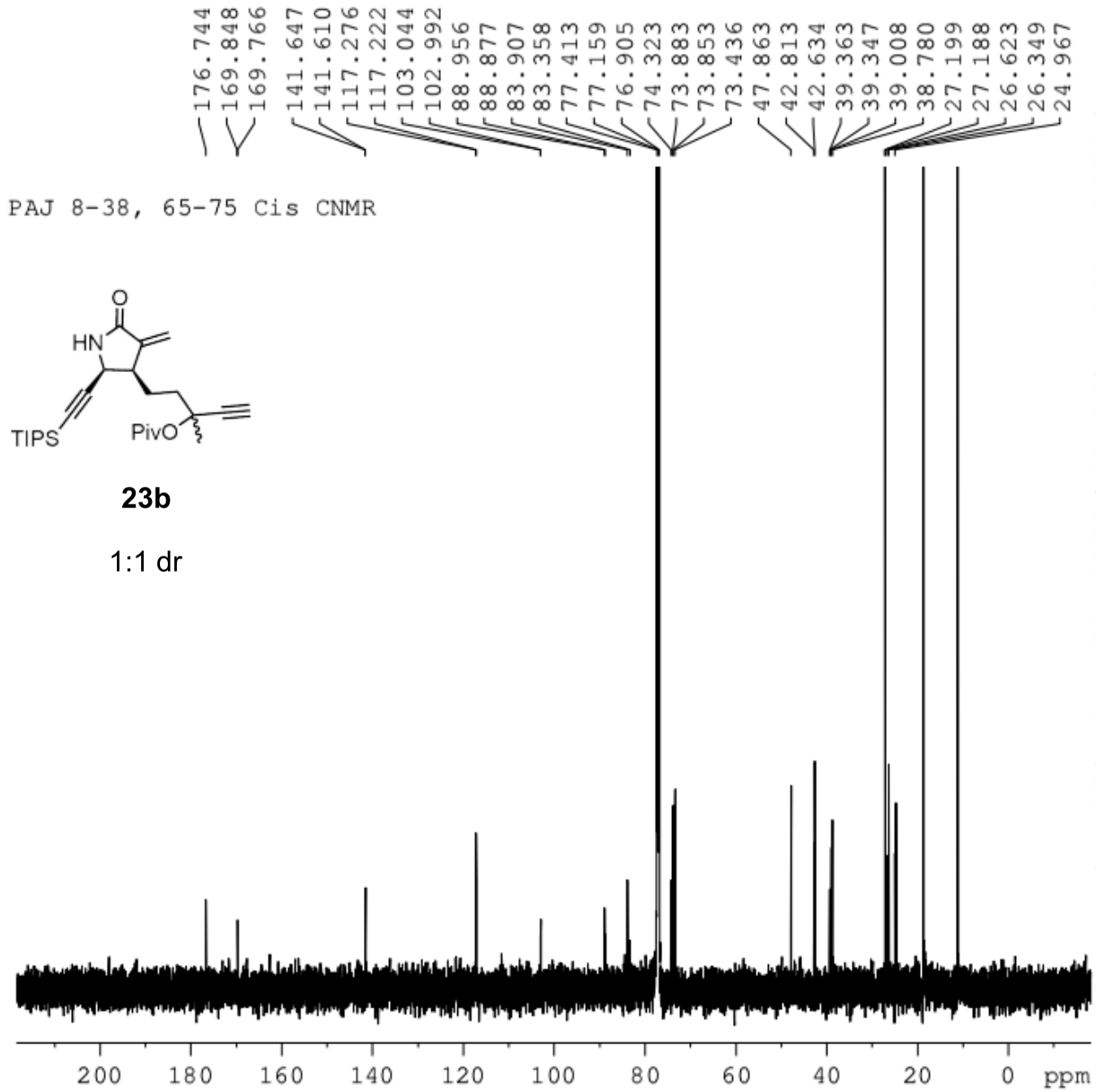
**22b**

1:1 dr


7.252
6.489
6.086
6.079
5.382
5.376
5.371
5.365
4.056
4.049
4.043
4.036
3.470
3.452
3.047
3.040
3.034
3.026
3.020
3.013
2.532
2.527
2.154
2.144
2.123
2.112
2.062
2.053
2.044
2.029
2.021
2.006
1.995
1.982
1.973
1.939
1.908
1.900
1.876
1.868
1.820
1.810
1.800
1.788
1.779
1.767
1.758
1.670
PAJ 7-50, 1-5
6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 ppm
HN
O
TIPS
PivO
23a
1:1 dr
3.2 3.0 2.8 2.6 2.4 2.2 2.0 ppm
NAME PAJ 7-50, 1-5
EXPNO 10
PROCNO 1
Date_ 20150505
Time 17.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 32
DW 60.800 usec
DE 6.50 usec
TE 91.3 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300132 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.00 1.07 1.10 1.00 1.04 0.94 2.17 1.92 3.24 0.33 0.57 12.88 23.61


182.89
176.64
169.37
141.83
141.81
125.62
117.02
116.94
105.75
105.74
86.36
83.43
83.40
77.46
77.14
76.82
73.95
73.83
73.65
73.60
65.96
49.00
48.92
47.04
39.33
38.73
38.69
38.57
27.86
27.75
27.30
27.25
27.15
26.51
26.49
18.67
11.15
PAJ 7-50, 1-5 CNMR
O
HN
TIPS
PivO
23a
1:1 dr
NAME PAJ 7-50, 1-5
EXPNO 11
PROCNO 1
Date_ 20150506
Time 3.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 95.5 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127580 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

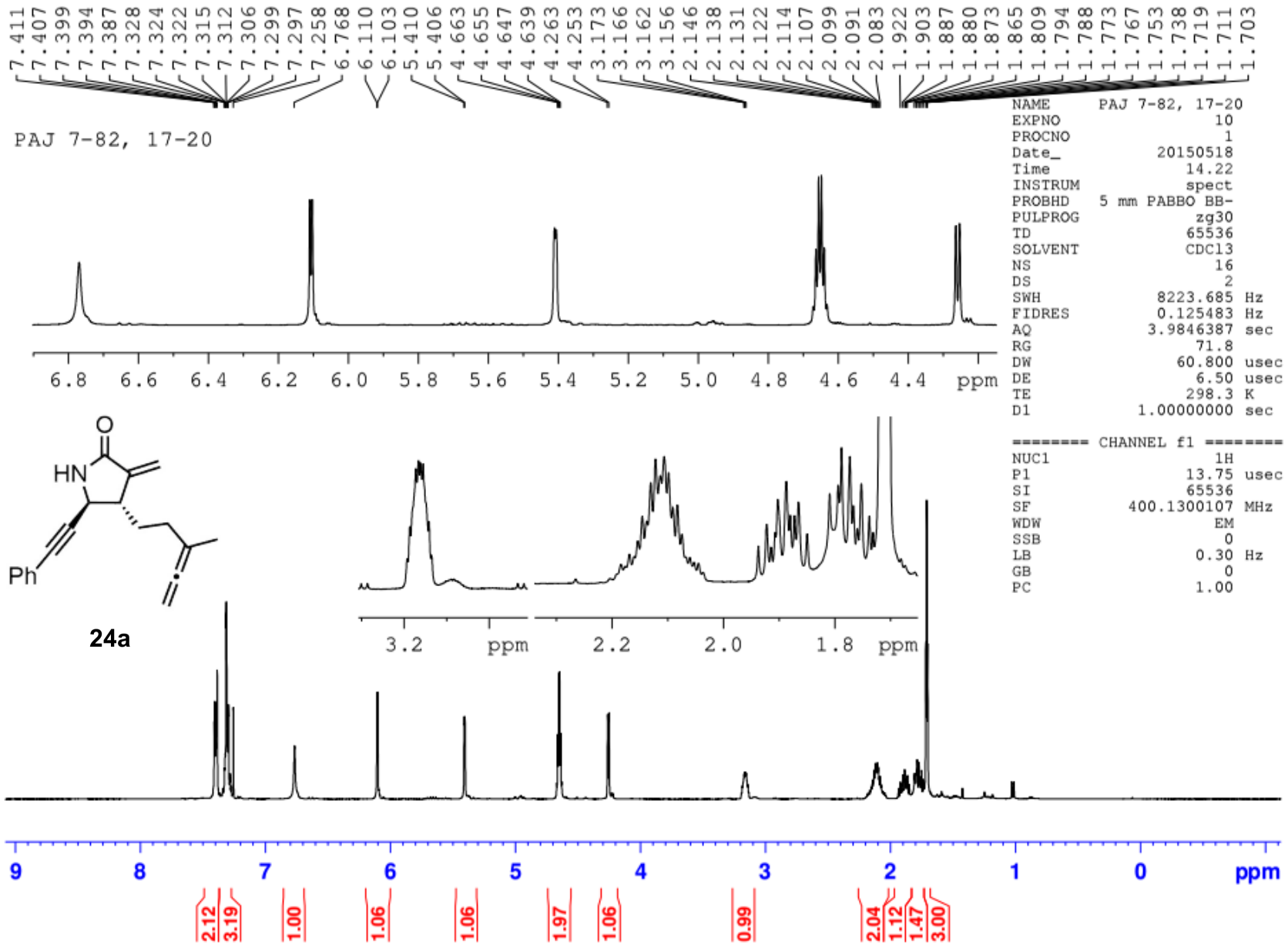

7.253
6.104
6.098
6.093
5.835
5.406
5.402
5.384
5.381
4.525
4.511
4.497
3.050
3.044
3.036
3.023
2.518
2.510
2.124
2.115
2.100
2.091
2.079
2.075
2.066
2.059
2.054
2.047
2.043
2.039
2.036
2.030
2.019
2.015
2.010
2.005
1.998
1.994
1.990
1.981
1.957
1.935
1.924
1.914
1.898
1.884
1.878
1.676
1.661
1.546
1.183
1.177
1.040
1.035
PAJ 8-38, cis, 65-75
6.0
5.8
5.6
5.4
5.2
5.0
4.8
ppm
3.0
2.8
2.6
2.4
2.2
2.0
1.8 ppm
HN
O
TIPS
PivO
23b
1:1 dr
NAME PAJ 8-38, cis, 65-75
EXPNO 10
PROCNO 1
Date_ 20151028
Time 18.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600161 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.00
0.95
0.38
0.64
0.96
1.01
0.90
3.14
1.10
1.19
1.81
1.97
5.47
3.77
13.22
9.68



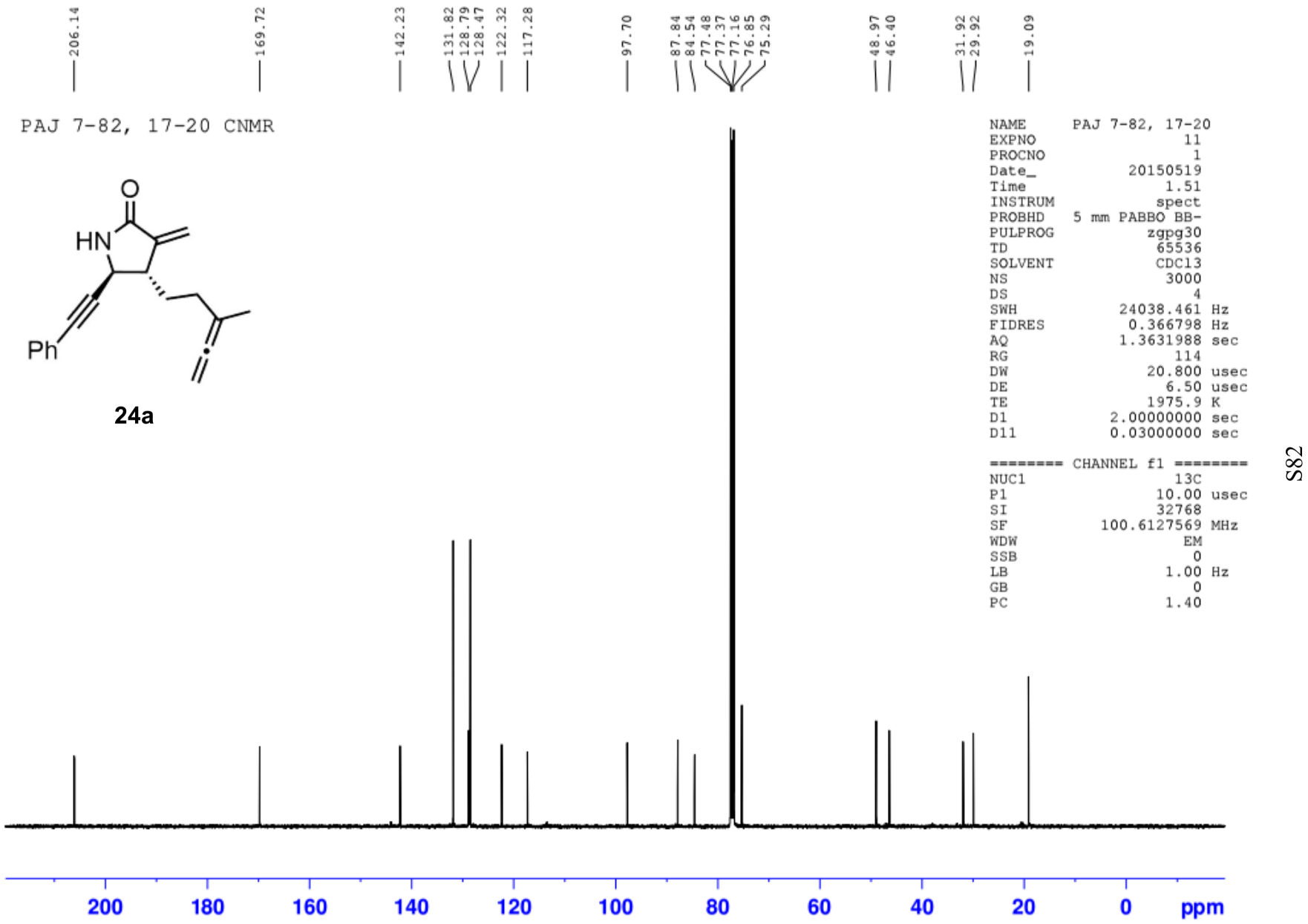

**23b**

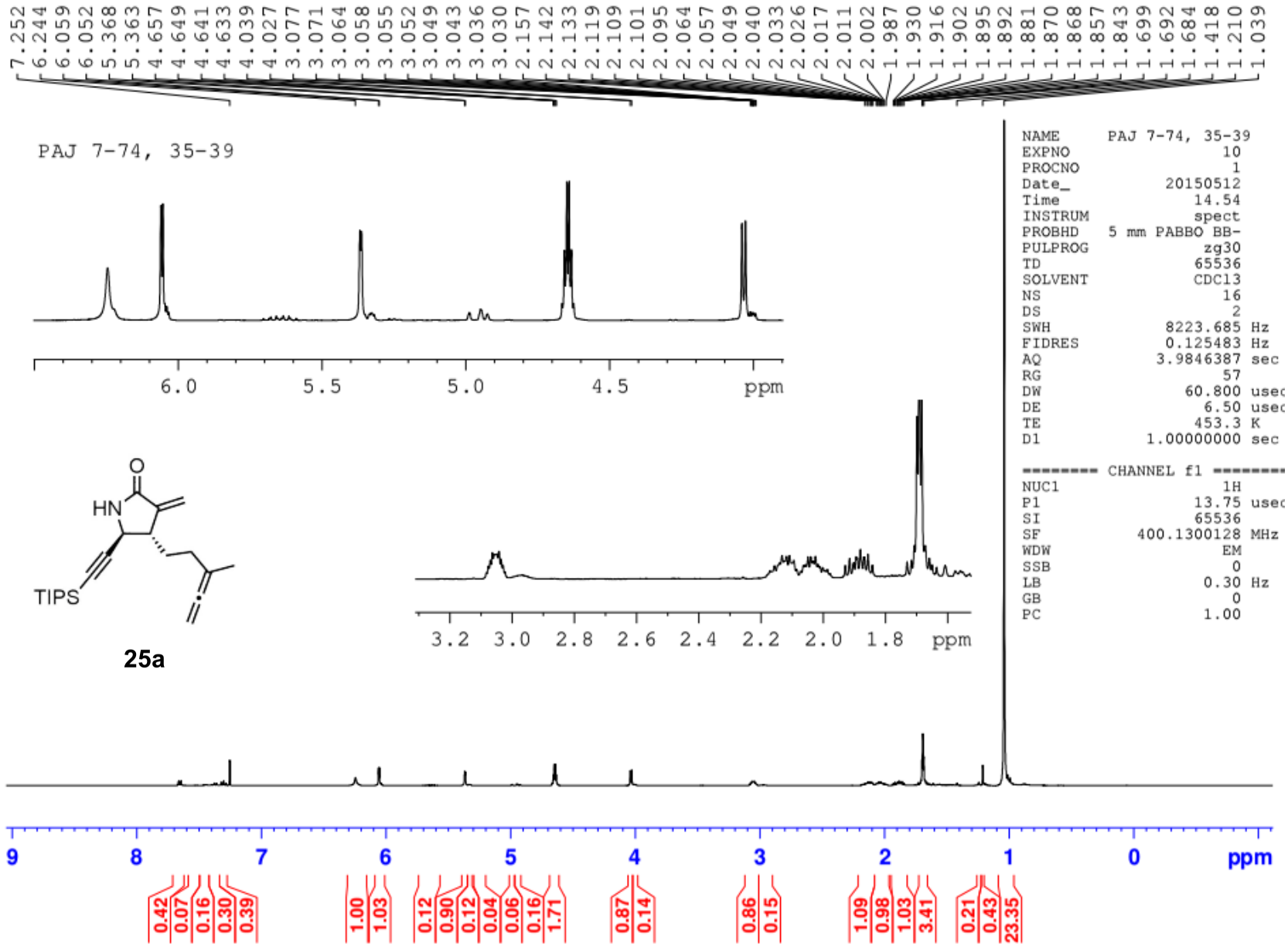
1:1 dr

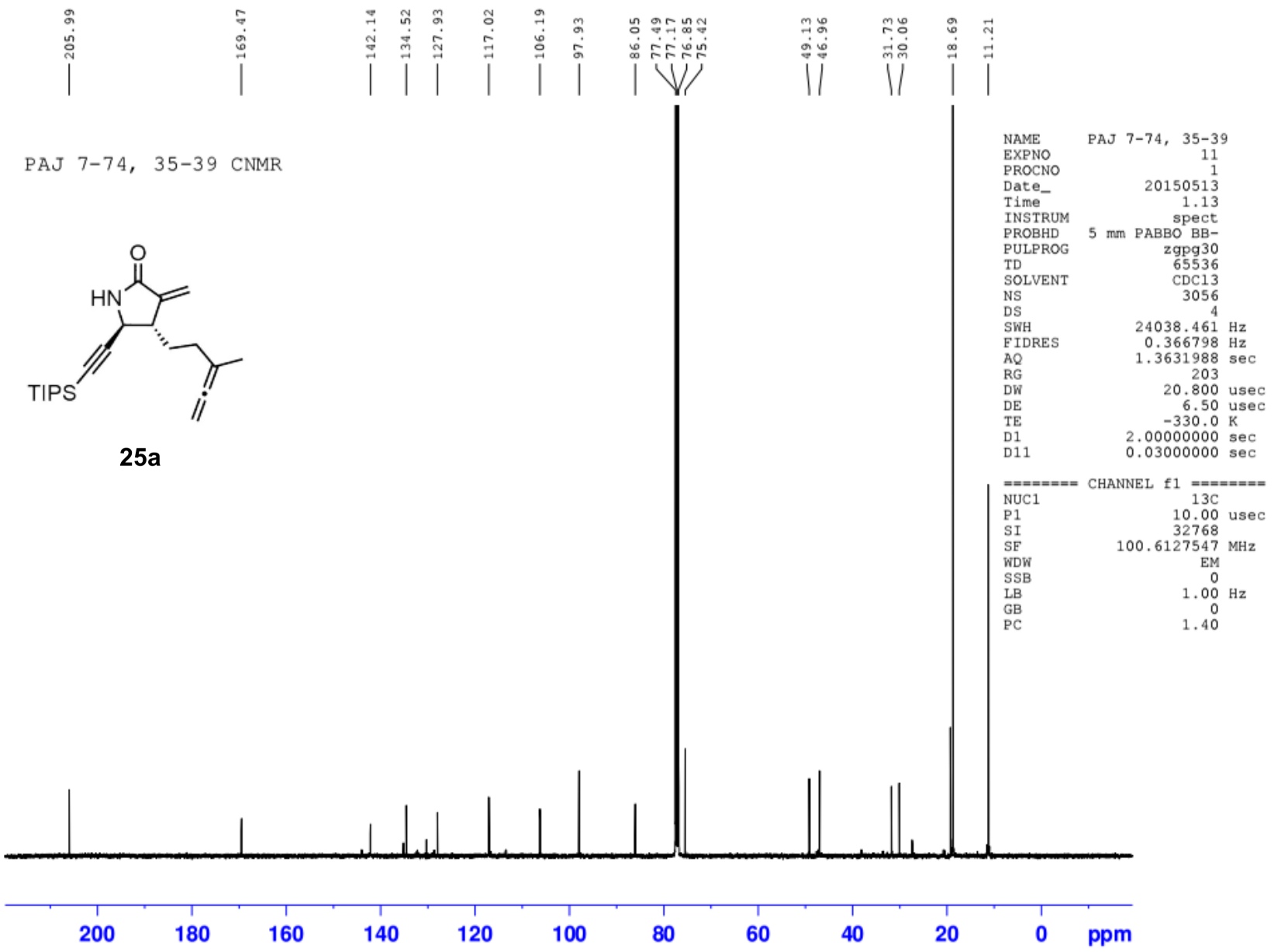
| | |
|---|---|
| NAME | PAJ 8-38, 65-75 Cis |
| EXPNO | 10 |
| PROCNO | 1 |
| Date_ | 20151107 |
| Time | 4.07 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB/ |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 3000 |
| DS | 2 |
| SWH | 29761.904 Hz |
| FIDRES | 0.454131 Hz |
| AQ | 1.1010548 sec |
| RG | 203 |
| DW | 16.800 usec |
| DE | 6.50 usec |
| TE | 298.5 K |
| D1 | 2.00000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 1 |
| ======== | CHANNEL f1 ======== |
| SFO1 | 125.7779086 MHz |
| NUC1 | 13C |
| P1 | 10.50 usec |
| SI | 32768 |
| SF | 125.7653129 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |

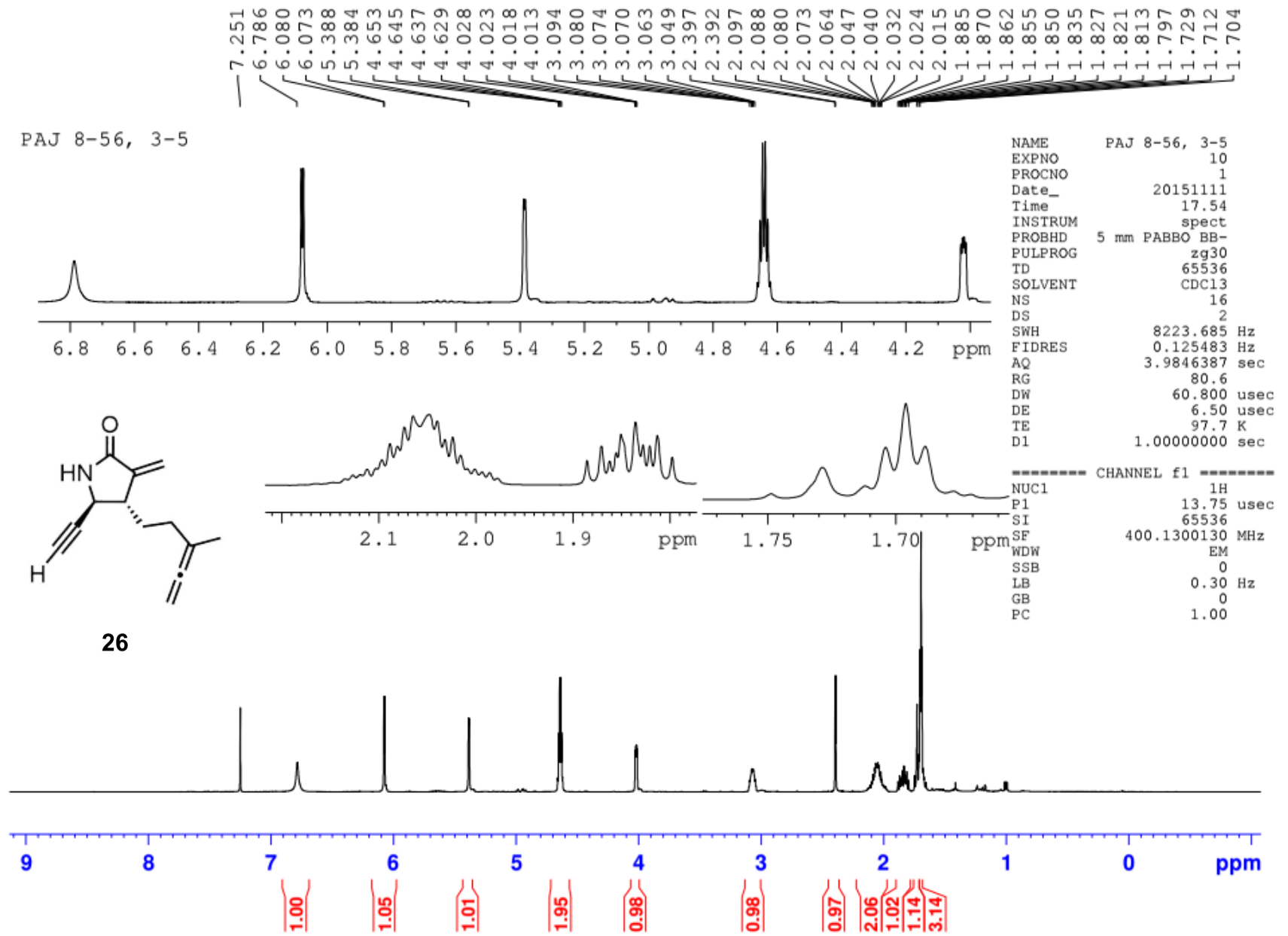



| | |
|---|---|
| NAME | PAJ 7-82, 17-20 |
| EXPNO | 10 |
| PROCNO | 1 |
| Date_ | 20150518 |
| Time | 14.22 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB- |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 16 |
| DS | 2 |
| SWH | 8223.685 Hz |
| FIDRES | 0.125483 Hz |
| AQ | 3.9846387 sec |
| RG | 71.8 |
| DW | 60.800 usec |
| DE | 6.50 usec |
| TE | 298.3 K |
| D1 | 1.00000000 sec |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 13.75 usec |
| SI | 65536 |
| SF | 400.1300107 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

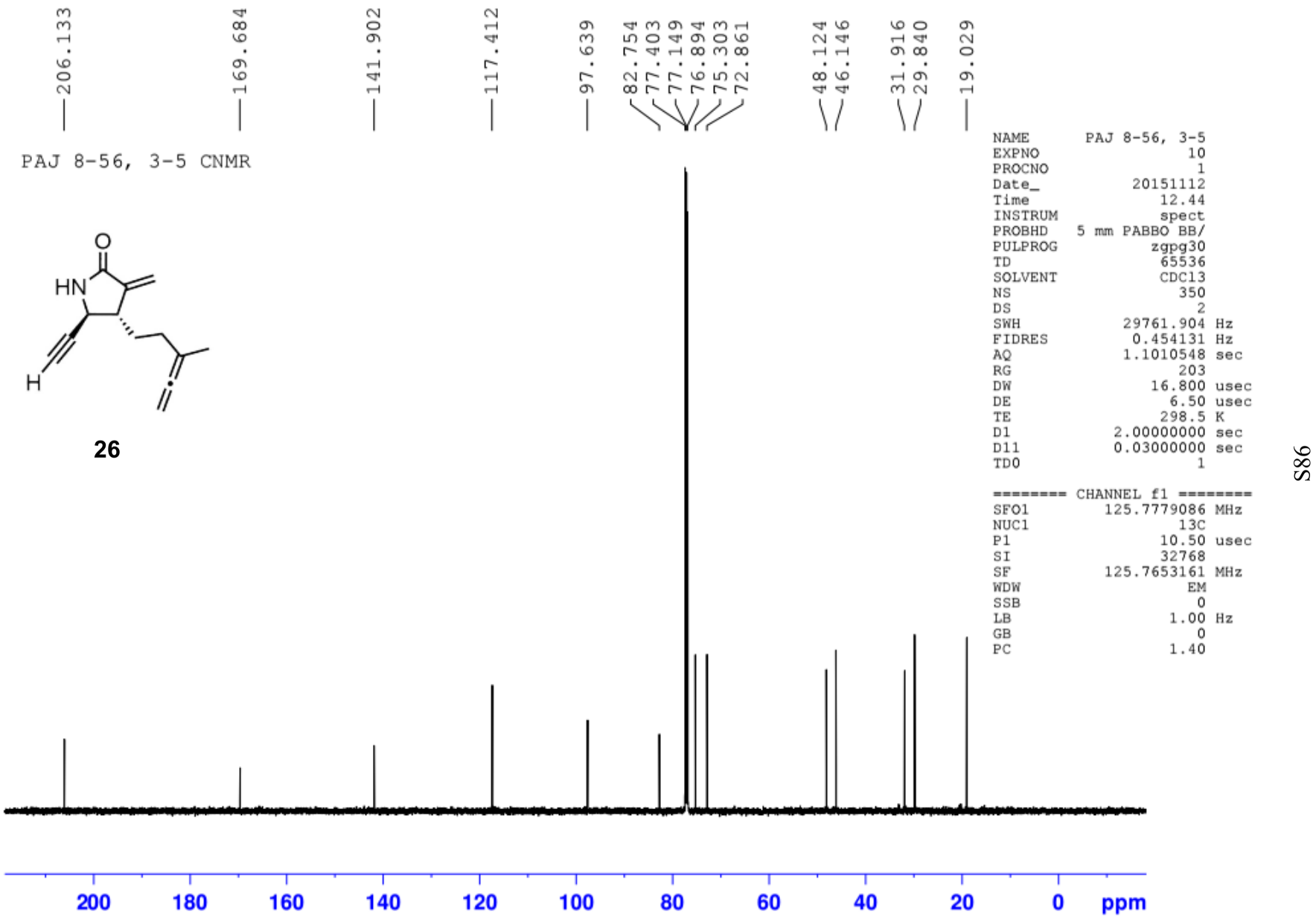

206.14
169.72
142.23
131.82
128.79
128.47
122.32
117.28
97.70
87.84
84.54
77.48
77.37
77.16
76.85
75.29
48.97
46.40
31.92
29.92
19.09
PAJ 7-82, 17-20 CNMR
HN
O
Ph
24a
NAME PAJ 7-82, 17-20
EXPNO 11
PROCNO 1
Date_ 20150519
Time 1.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 3000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 114
DW 20.800 usec
DE 6.50 usec
TE 1975.9 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127569 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

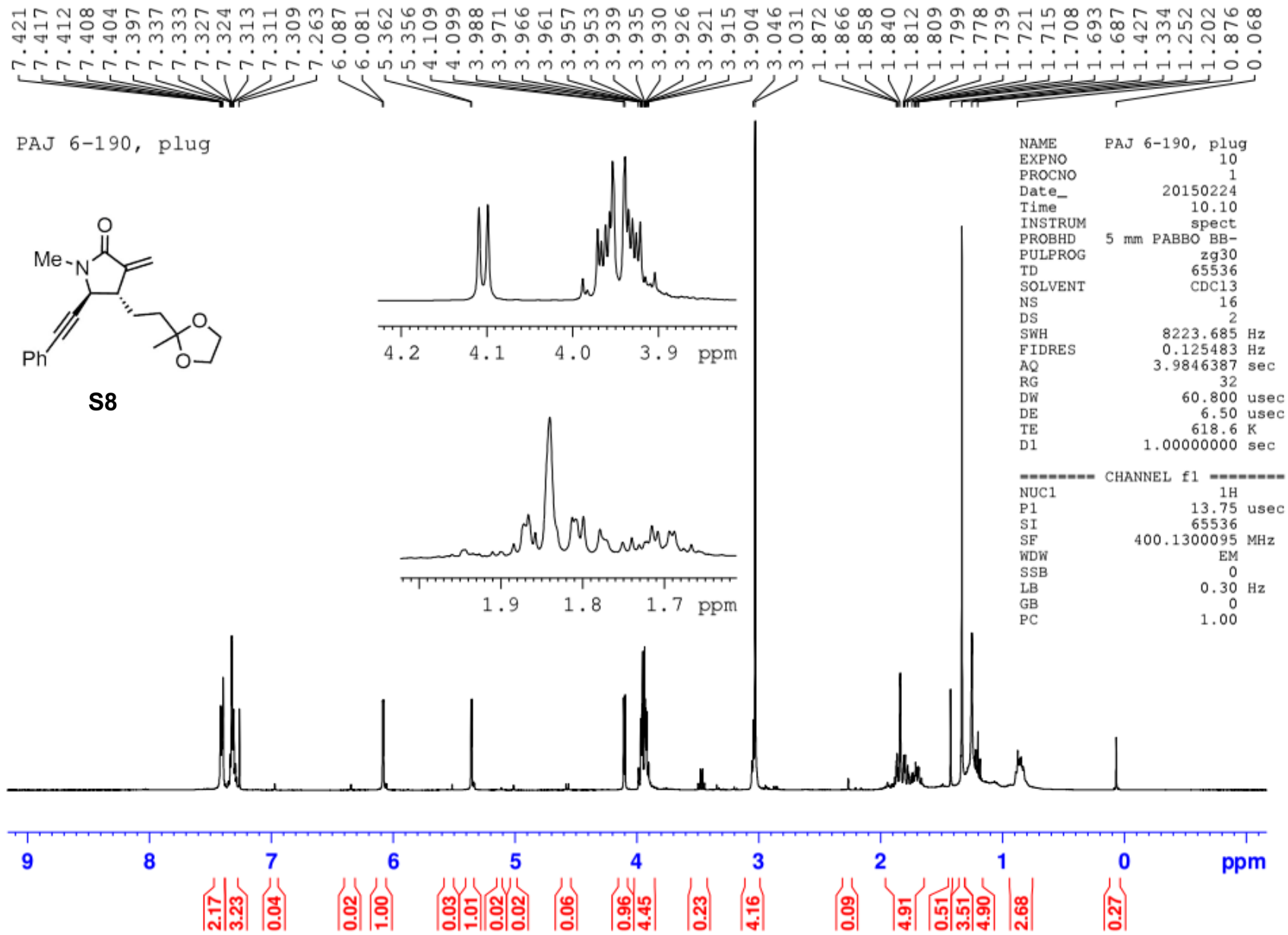

7.252 6.244 6.059 6.052 5.368 5.363 4.657 4.649 4.641 4.633 4.039 4.027 3.077 3.071 3.064 3.058 3.055 3.052 3.049 3.043 3.036 3.030 2.157 2.142 2.133 2.119 2.109 2.101 2.095 2.064 2.057 2.049 2.040 2.033 2.026 2.017 2.011 2.002 1.987 1.930 1.916 1.902 1.895 1.892 1.881 1.870 1.868 1.857 1.843 1.699 1.692 1.684 1.418 1.210 1.039
PAJ 7-74, 35-39
6.0 5.5 5.0 4.5 ppm
3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 ppm
HN
O
TIPS
25a
NAME PAJ 7-74, 35-39
EXPNO 10
PROCNO 1
Date_ 20150512
Time 14.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 57
DW 60.800 usec
DE 6.50 usec
TE 453.3 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
0.42 0.07 0.16 0.30 0.39 1.00 1.03 0.12 0.90 0.12 0.04 0.06 0.16 1.71 0.87 0.14 0.86 0.15 1.09 0.98 1.03 3.41 0.21 0.43 23.35

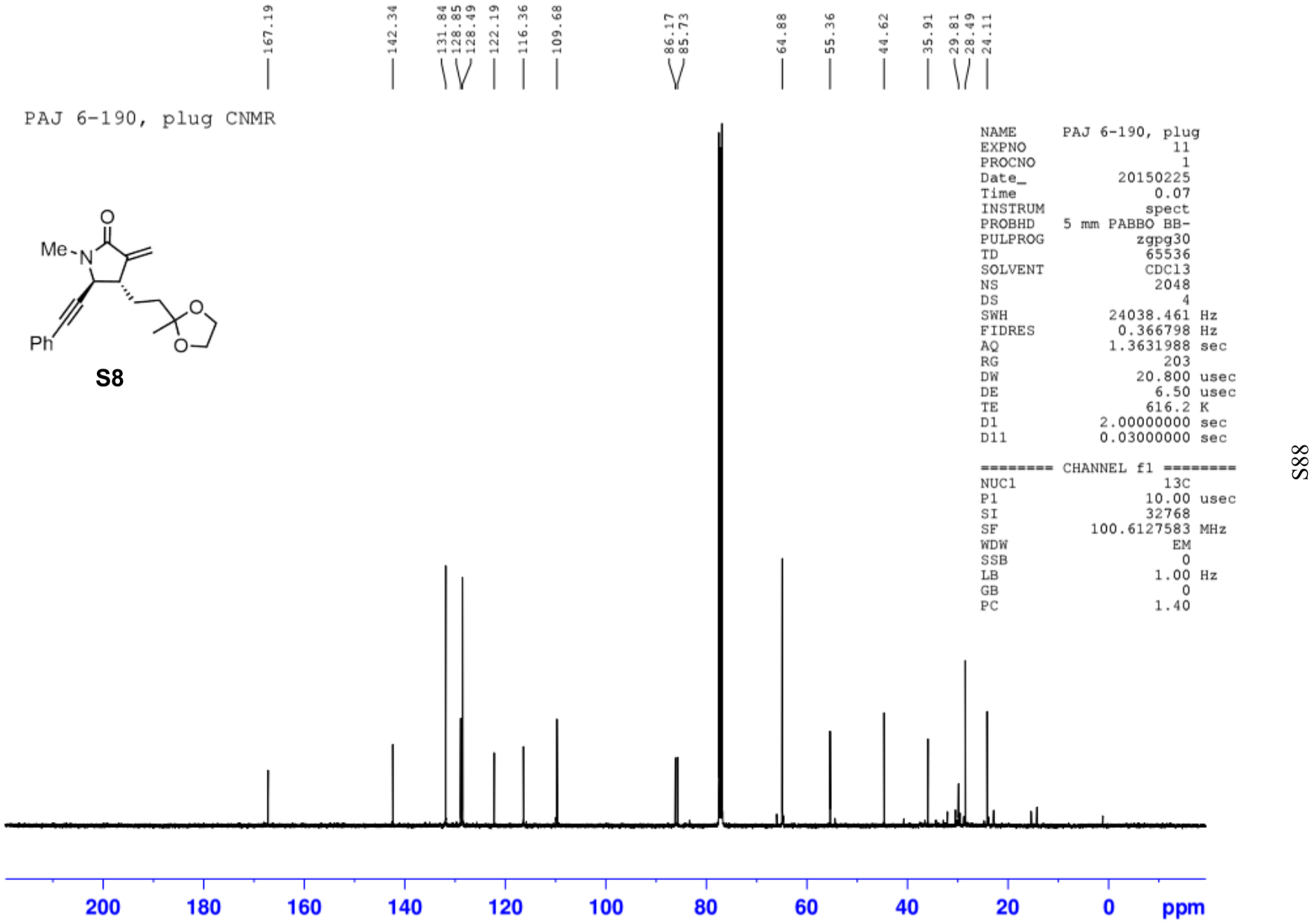

205.99
169.47
142.14
134.52
127.93
117.02
106.19
97.93
86.05
77.49
77.17
76.85
75.42
49.13
46.96
31.73
30.06
18.69
11.21
PAJ 7-74, 35-39 CNMR
O
HN
TIPS
25a
NAME PAJ 7-74, 35-39
EXPNO 11
PROCNO 1
Date_ 20150513
Time 1.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3056
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE -330.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127547 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


7.251
6.786
6.080
6.073
5.388
5.384
4.653
4.645
4.637
4.629
4.028
4.023
4.018
4.013
3.094
3.080
3.074
3.070
3.063
3.049
2.397
2.392
2.097
2.088
2.080
2.073
2.064
2.047
2.040
2.032
2.024
2.015
1.885
1.870
1.862
1.855
1.850
1.835
1.827
1.821
1.813
1.797
1.729
1.712
1.704
PAJ 8-56, 3-5
6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 ppm
2.1 2.0 1.9 ppm
1.75 1.70 ppm
NAME PAJ 8-56, 3-5
EXPNO 10
PROCNO 1
Date_ 20151111
Time 17.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 97.7 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HN
O
H
9 8 7 6 5 4 3 2 1 0 ppm
1.00 1.05 1.01 1.95 0.98 0.98 0.97 2.06 1.02 1.14 3.14

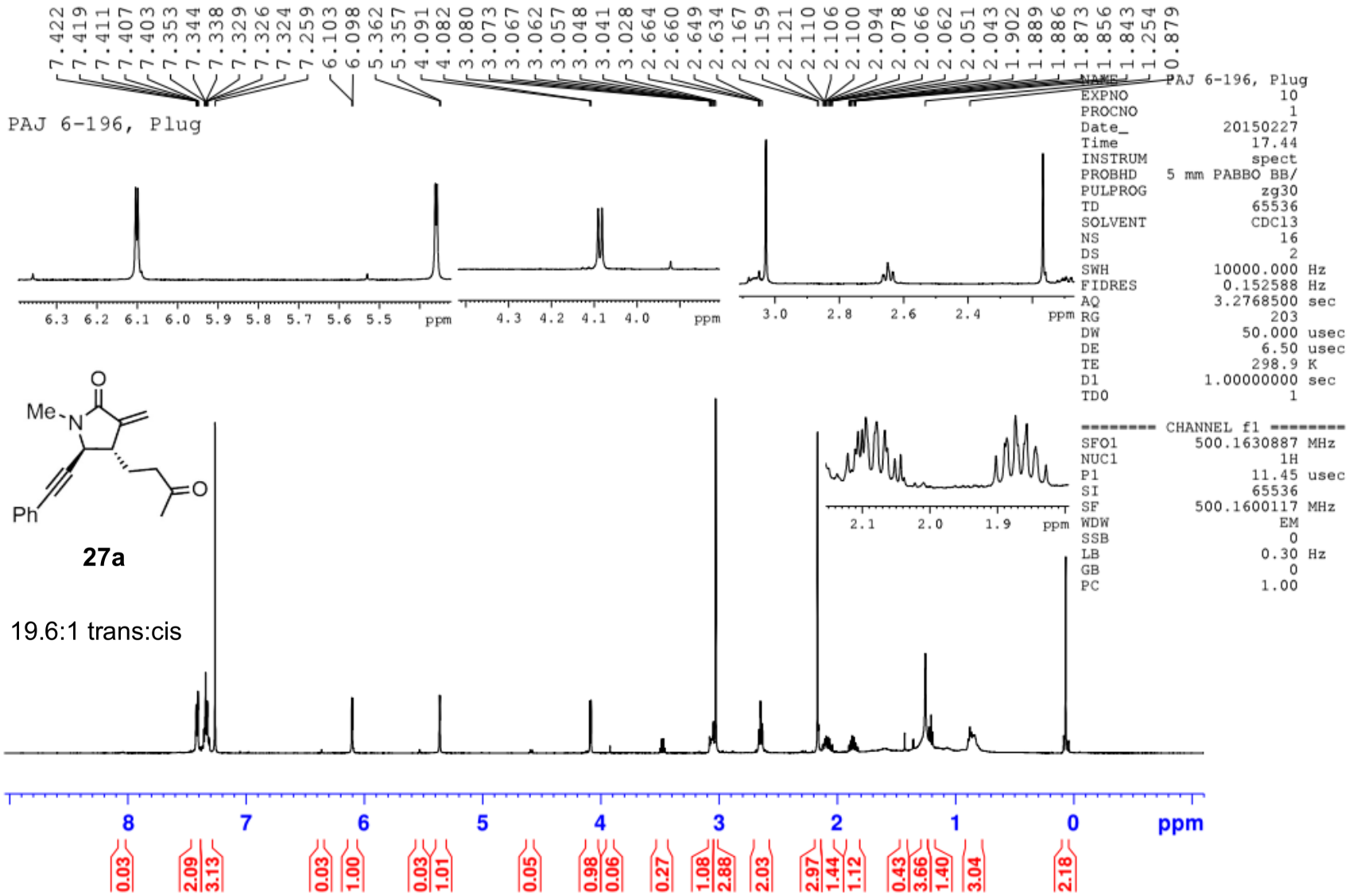
**26**

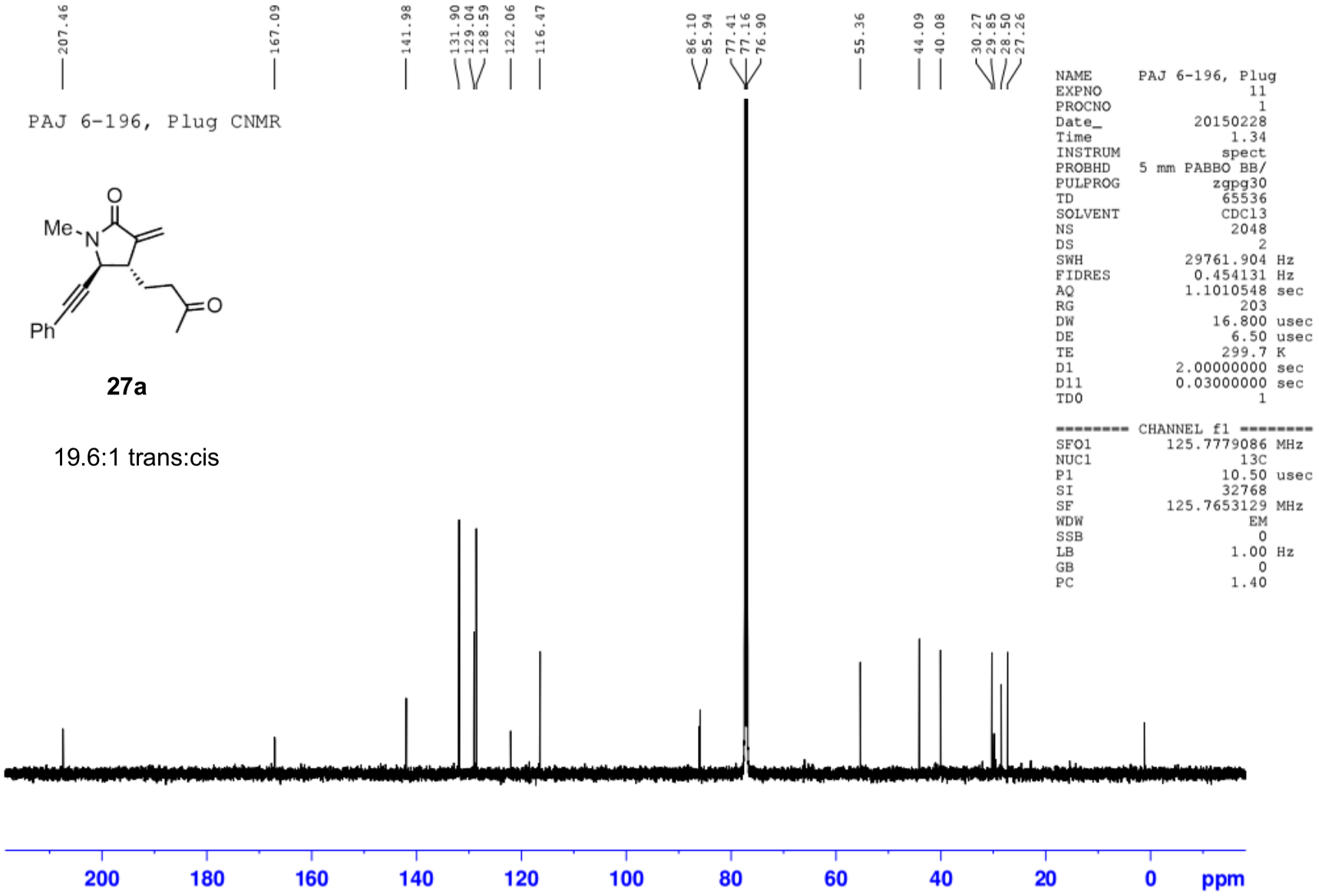

206.133
169.684
141.902
117.412
97.639
82.754
77.403
77.149
76.894
75.303
72.861
48.124
46.146
31.916
29.840
19.029
PAJ 8-56, 3-5 CNMR
O
HN
H
26
NAME PAJ 8-56, 3-5
EXPNO 10
PROCNO 1
Date_ 20151112
Time 12.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 350
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653161 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

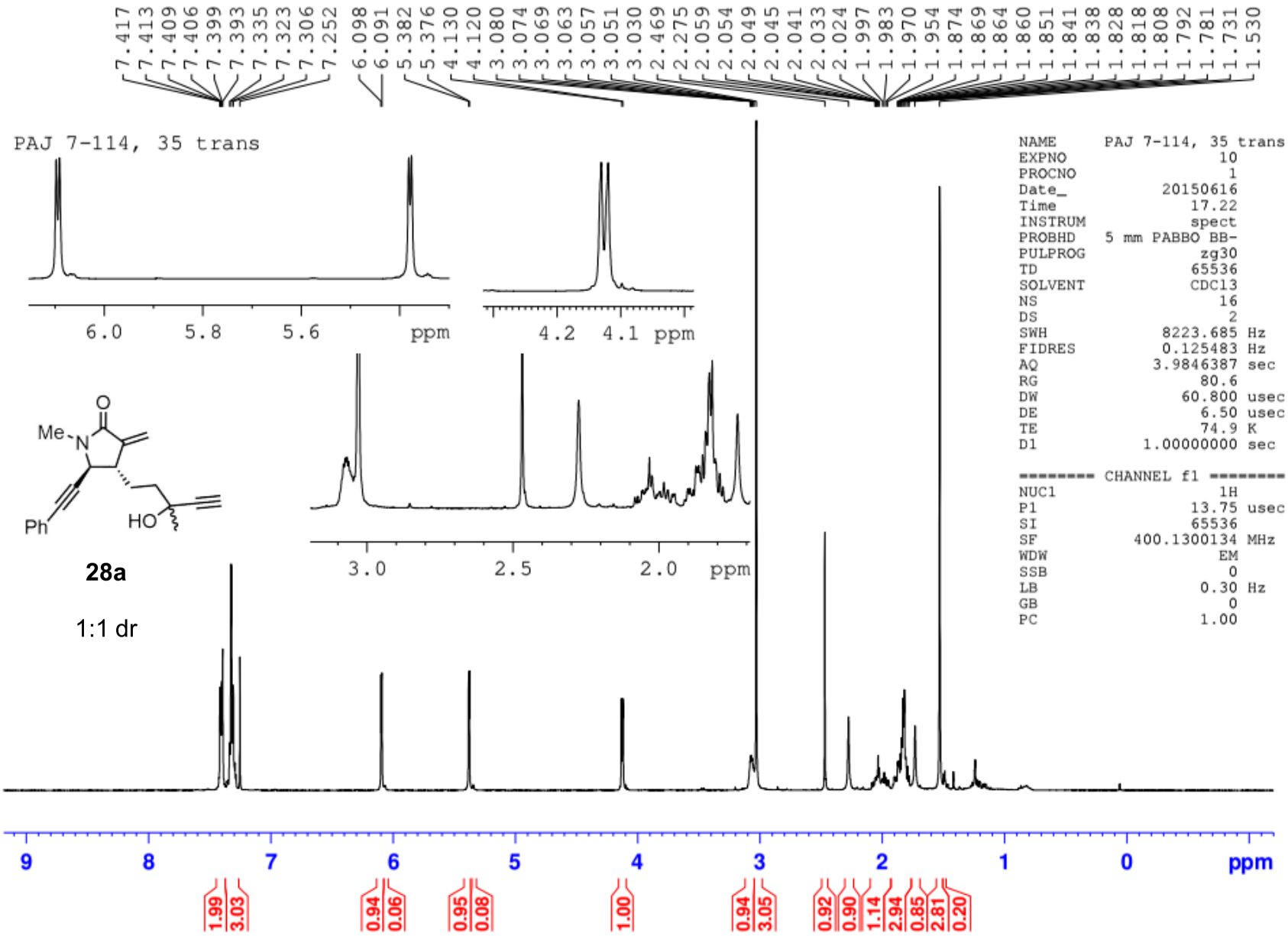

7.421 7.417 7.412 7.408 7.404 7.397 7.337 7.333 7.327 7.324 7.313 7.311 7.309 7.263
6.087 6.081 5.362 5.356 4.109 4.099 3.988 3.971 3.966 3.961 3.957 3.953 3.939 3.935 3.930 3.926 3.921 3.915 3.904 3.046 3.031
1.872 1.866 1.858 1.840 1.812 1.809 1.799 1.778 1.739 1.721 1.715 1.708 1.693 1.687 1.427 1.334 1.252 1.202 0.876 0.068
PAJ 6-190, plug
Me
N
O
Ph
O
O
S8
4.2 4.1 4.0 3.9 ppm
1.9 1.8 1.7 ppm
NAME PAJ 6-190, plug
EXPNO 10
PROCNO 1
Date_ 20150224
Time 10.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 32
DW 60.800 usec
DE 6.50 usec
TE 618.6 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
2.17 3.23 0.04 0.02 1.00 0.03 1.01 0.02 0.02 0.06 0.96 4.45 0.23 4.16 0.09 4.91 0.51 3.51 4.90 2.68 0.27

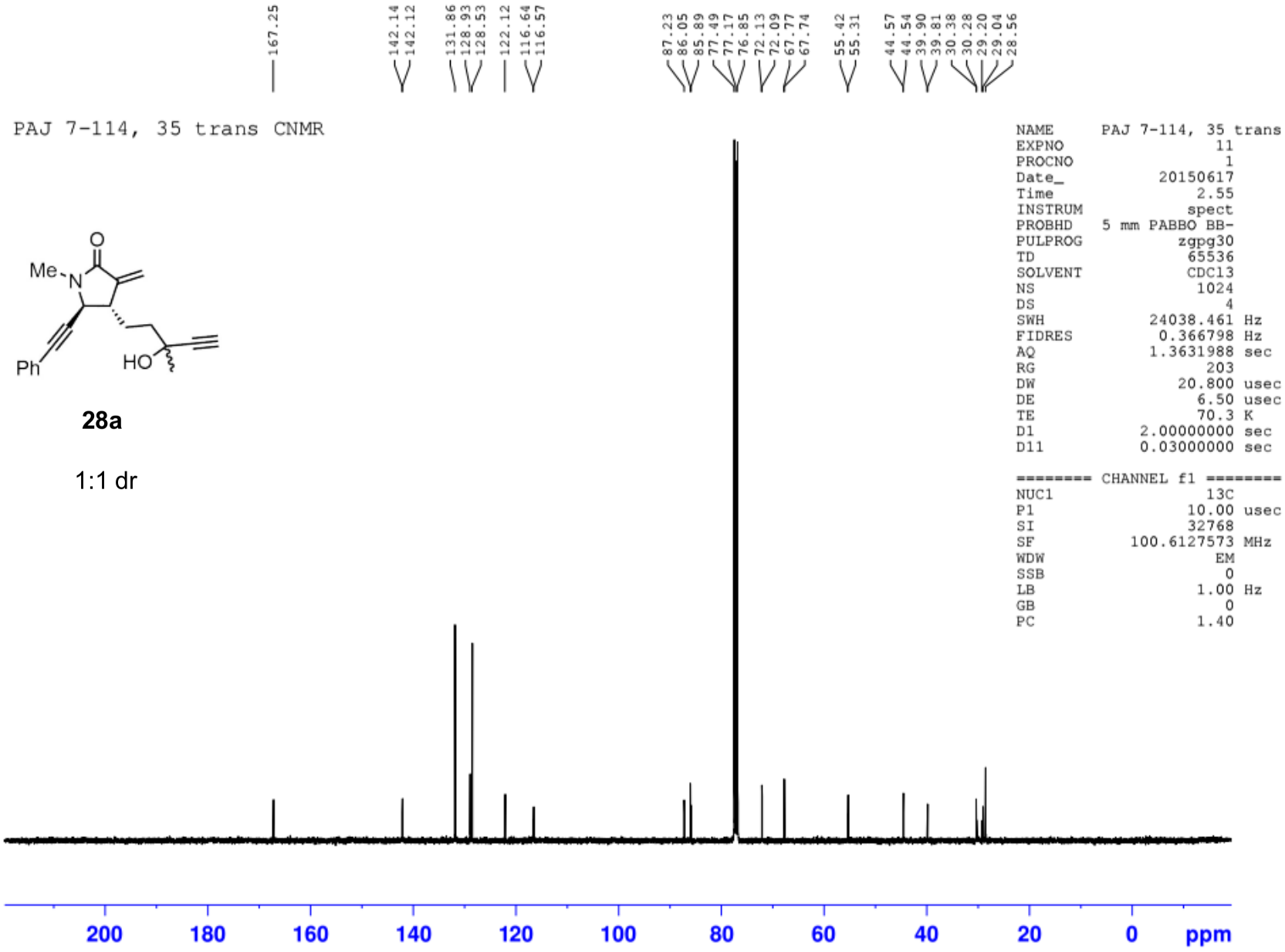

167.19
142.34
131.84
128.85
128.49
122.19
116.36
109.68
86.17
85.73
64.88
55.36
44.62
35.91
29.81
28.49
24.11
PAJ 6-190, plug CNMR
Me
N
O
Ph
O
O
NAME PAJ 6-190, plug
EXPNO 11
PROCNO 1
Date_ 20150225
Time 0.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 616.2 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127583 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

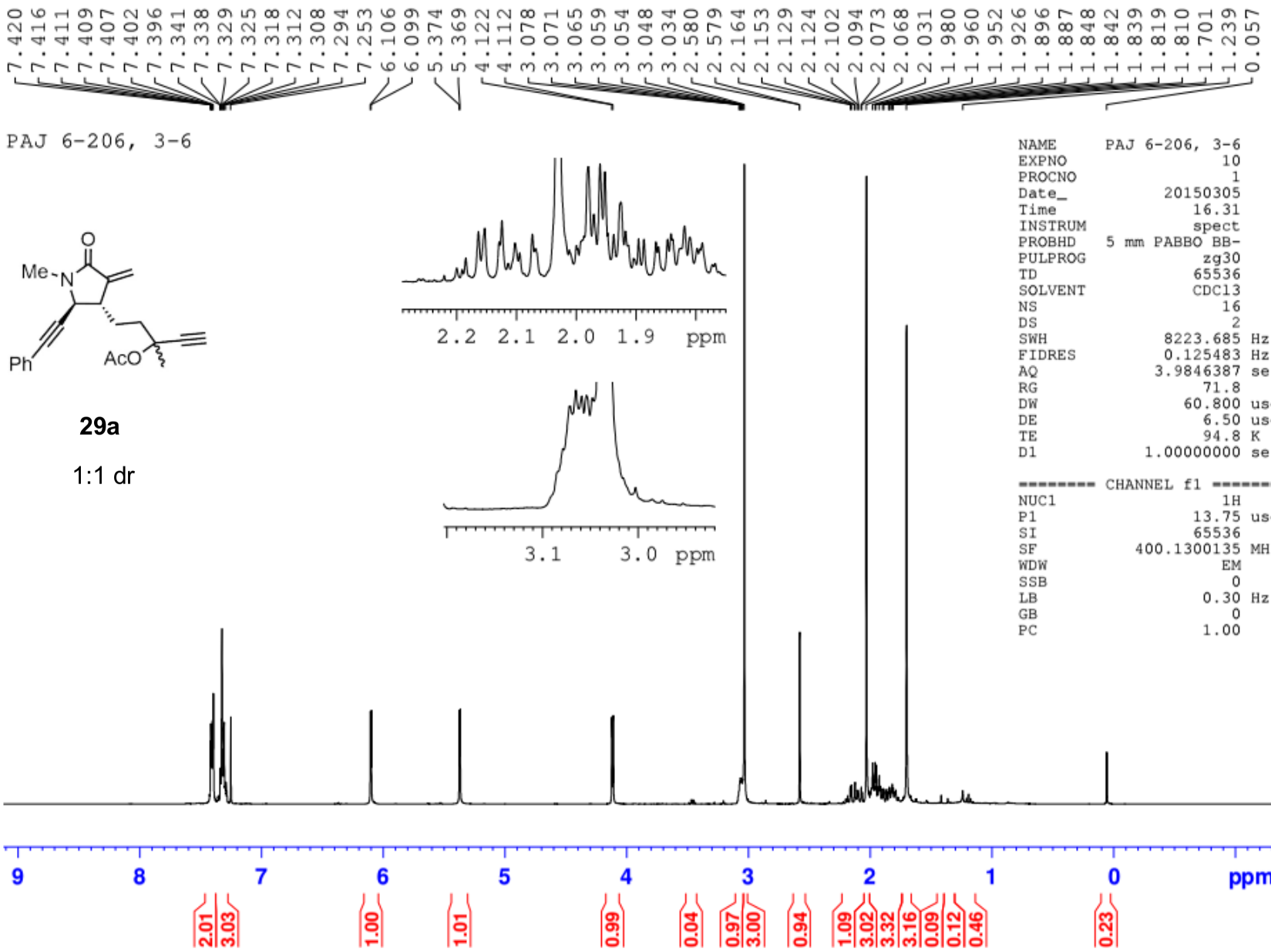
**S8**

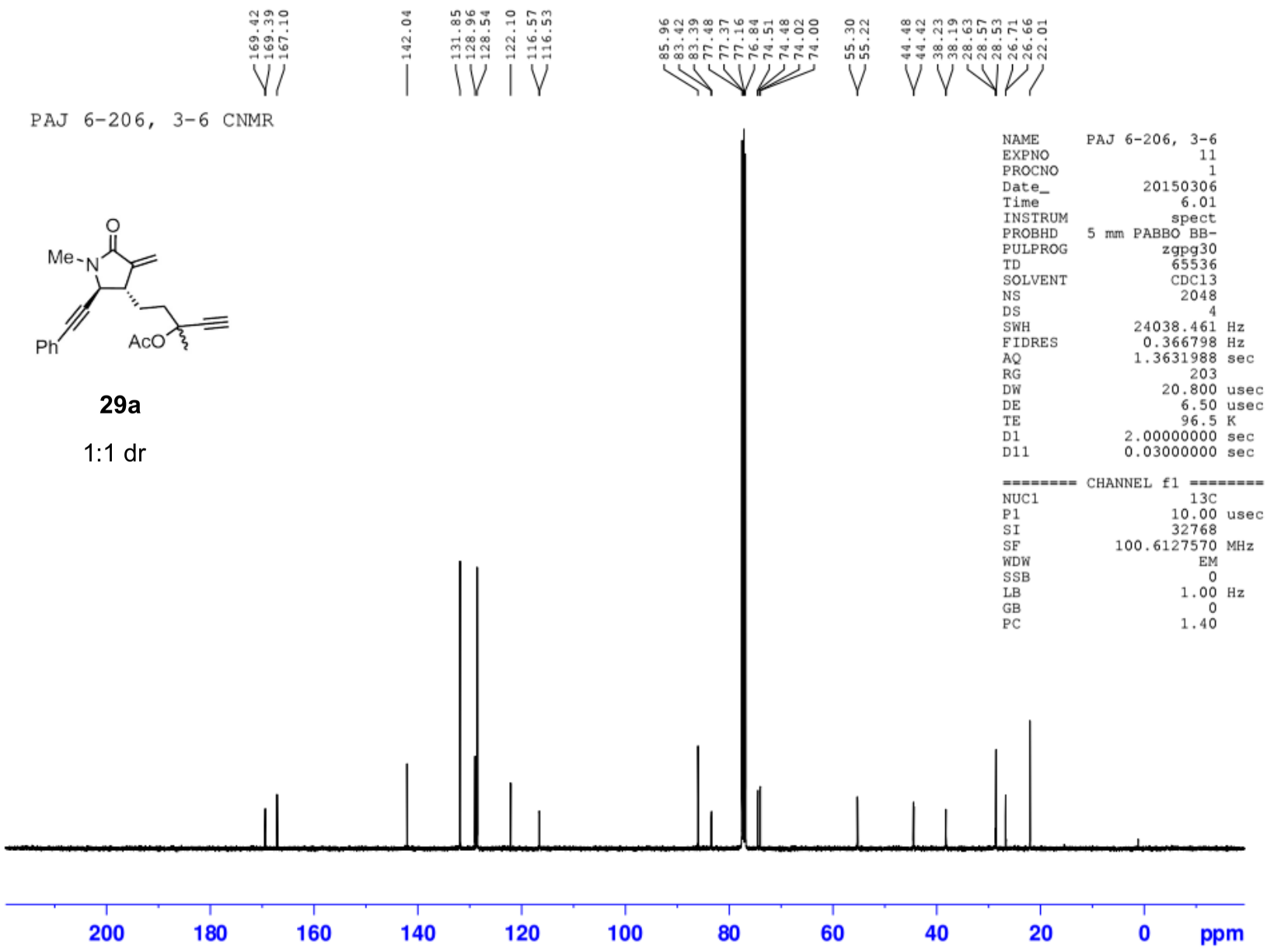

7.422 7.419 7.411 7.407 7.403 7.353 7.344 7.338 7.329 7.326 7.324 7.259 6.103 6.098 5.362 5.357 4.091 4.082 3.080 3.073 3.067 3.062 3.057 3.048 3.041 3.028 2.664 2.660 2.649 2.634 2.167 2.159 2.121 2.110 2.106 2.100 2.094 2.078 2.066 2.062 2.051 2.043 1.902 1.889 1.886 1.873 1.856 1.843 1.254 0.879
PAJ 6-196, Plug
NAME PAJ 6-196, Plug
EXPNO 10
PROCNO 1
Date_ 20150227
Time 17.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 298.9 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600117 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
Ph
O
27a
19.6:1 trans:cis
ppm
0.03 2.09 3.13 0.03 1.00 0.03 1.01 0.05 0.98 0.06 0.27 1.08 2.88 2.03 2.97 1.44 1.12 0.43 3.66 1.40 3.04 2.18

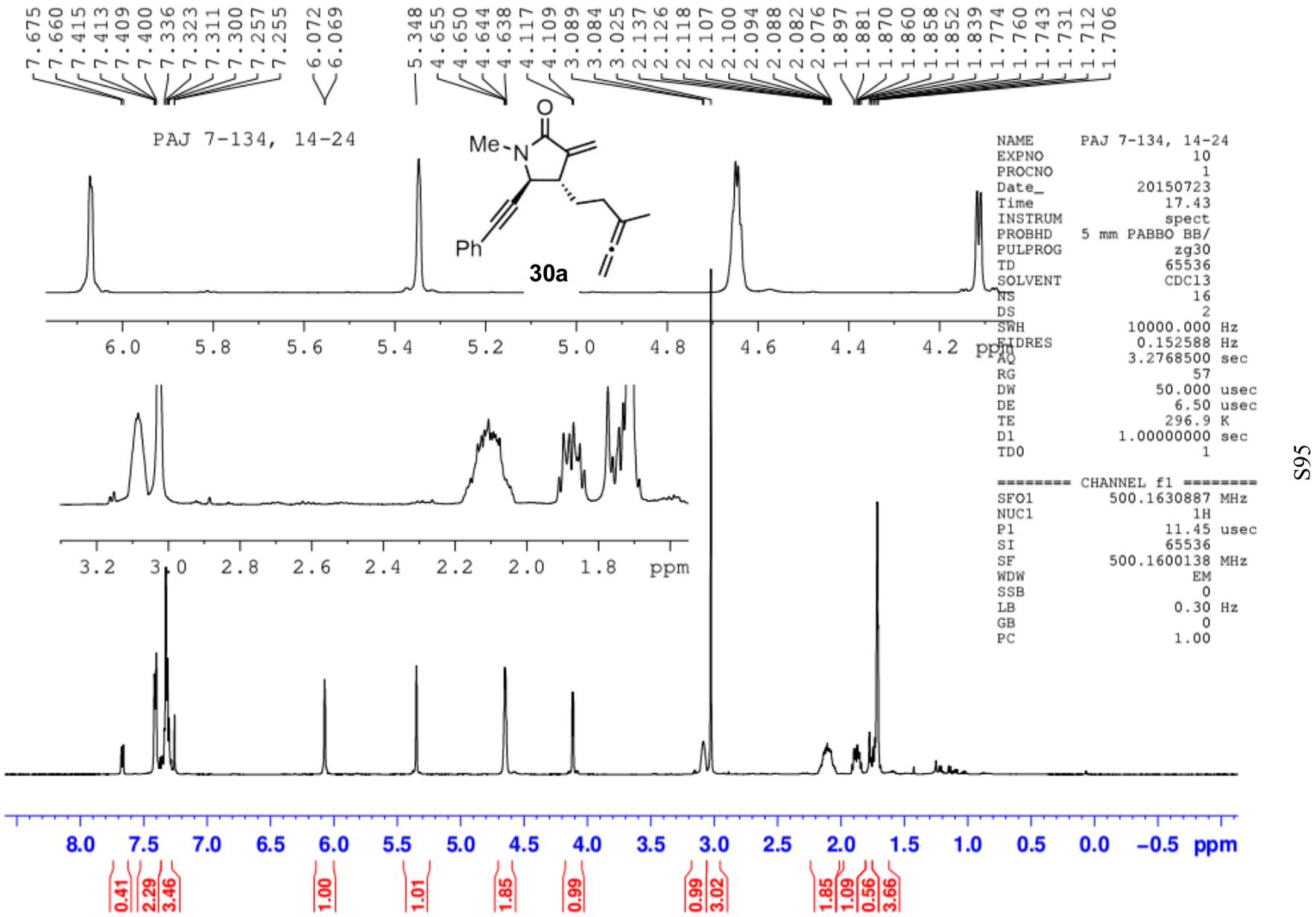

207.46
167.09
141.98
131.90
129.04
128.59
122.06
116.47
86.10
85.94
77.41
77.16
76.90
55.36
44.09
40.08
30.27
29.85
28.50
27.26
PAJ 6-196, Plug CNMR
Me
N
O
O
Ph
27a
19.6:1 trans:cis
NAME PAJ 6-196, Plug
EXPNO 11
PROCNO 1
Date_ 20150228
Time 1.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653129 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

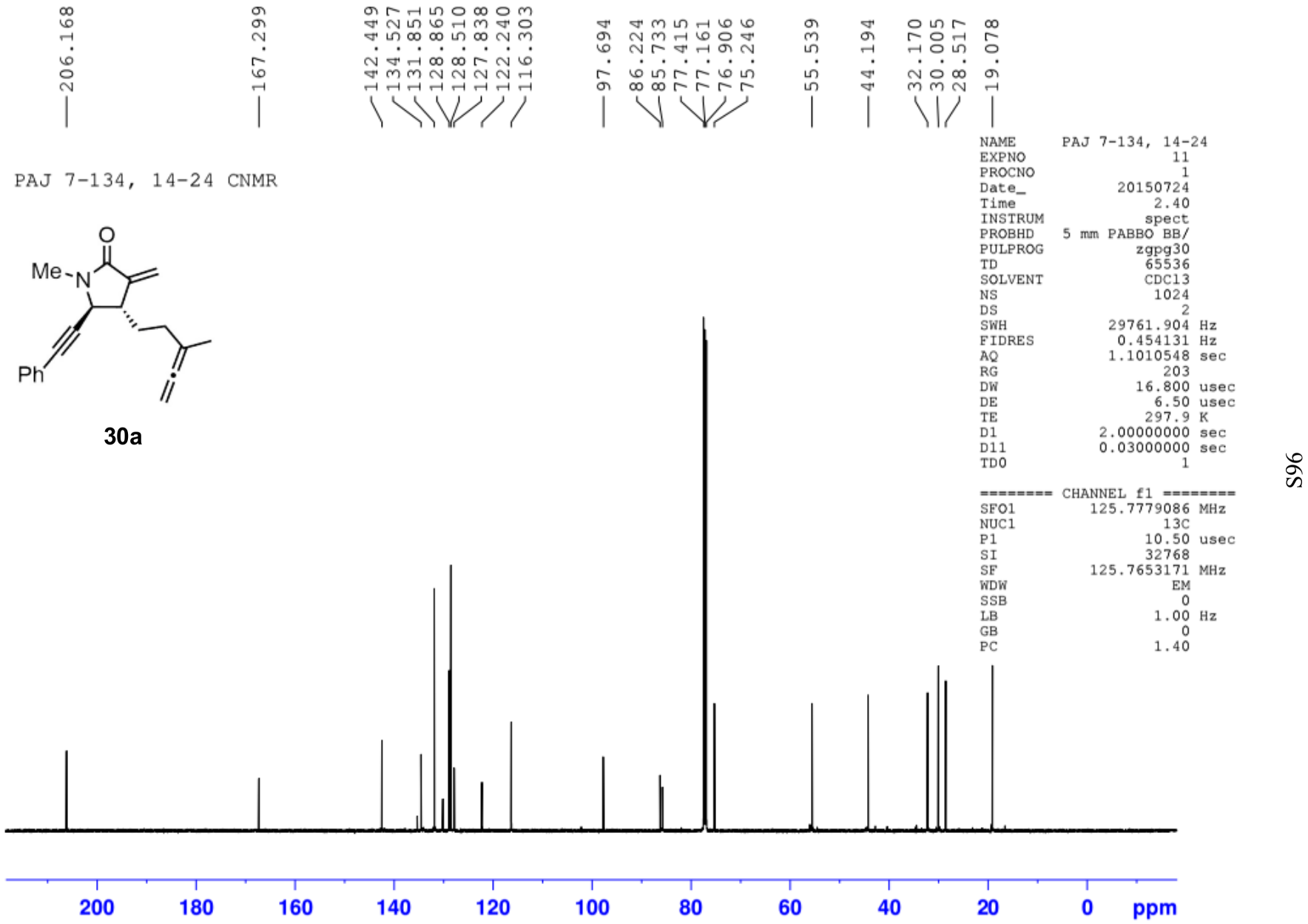

7.417 7.413 7.409 7.406 7.399 7.393 7.335 7.323 7.306 7.252 6.098 6.091 5.382 5.376 4.130 4.120 3.080 3.074 3.069 3.063 3.057 3.051 3.030 2.469 2.275 2.059 2.054 2.049 2.045 2.041 2.033 2.024 1.997 1.983 1.970 1.954 1.874 1.869 1.864 1.860 1.851 1.841 1.838 1.828 1.818 1.808 1.792 1.781 1.731 1.530
PAJ 7-114, 35 trans
6.0 5.8 5.6 ppm
4.2 4.1 ppm
3.0 2.5 2.0 ppm
Me N O Ph HO
28a
1:1 dr
NAME PAJ 7-114, 35 trans
EXPNO 10
PROCNO 1
Date_ 20150616
Time 17.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 74.9 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300134 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.99 3.03 0.94 0.06 0.95 0.08 1.00 0.94 3.05 0.92 0.90 1.14 2.94 0.85 2.81 0.20

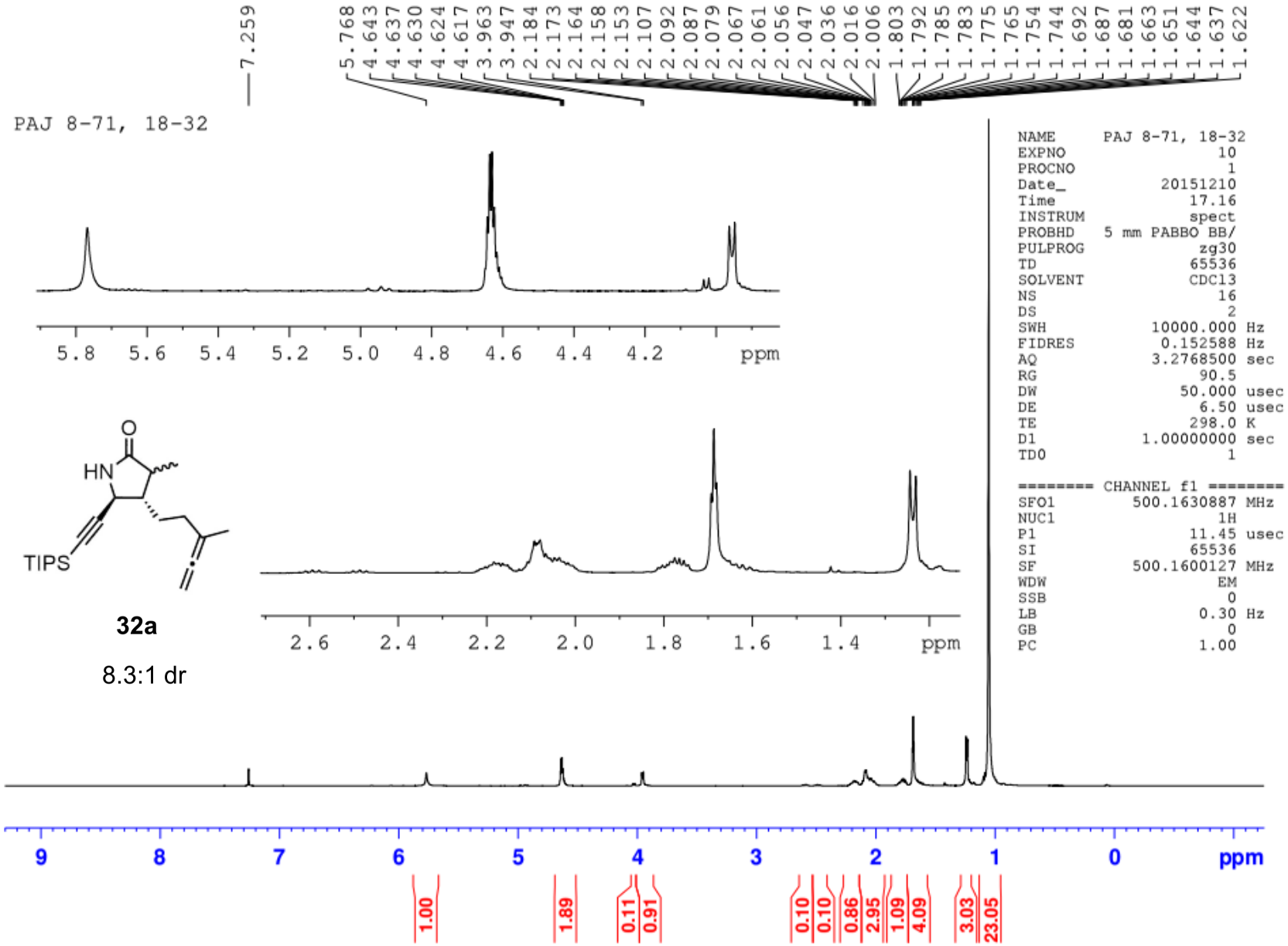

167.25
142.14
142.12
131.86
128.93
128.53
122.12
116.64
116.57
87.23
86.05
85.89
77.49
77.17
76.85
72.13
72.09
67.77
67.74
55.42
55.31
44.57
44.54
39.90
39.81
30.38
30.28
29.20
29.04
28.56
PAJ 7-114, 35 trans CNMR
Me
N
O
Ph
HO
28a
1:1 dr
NAME PAJ 7-114, 35 trans
EXPNO 11
PROCNO 1
Date_ 20150617
Time 2.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 70.3 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127573 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


7.420 7.416 7.411 7.409 7.407 7.402 7.396 7.341 7.338 7.329 7.325 7.318 7.312 7.308 7.294 7.253 6.106 6.099 5.374 5.369 4.122 4.112 3.078 3.071 3.065 3.059 3.054 3.048 3.034 2.580 2.579 2.164 2.153 2.129 2.124 2.102 2.094 2.073 2.068 2.031 1.980 1.960 1.952 1.926 1.896 1.887 1.848 1.842 1.839 1.819 1.810 1.701 1.239 0.057
PAJ 6-206, 3-6
Me
N
O
Ph
AcO
29a
1:1 dr
2.2 2.1 2.0 1.9 ppm
3.1 3.0 ppm
NAME PAJ 6-206, 3-6
EXPNO 10
PROCNO 1
Date_ 20150305
Time 16.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 71.8
DW 60.800 usec
DE 6.50 usec
TE 94.8 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
2.01 3.03 1.00 1.01 0.99 0.04 0.97 3.00 0.94 1.09 3.02 3.32 3.16 0.09 0.12 0.46 0.23


169.42
169.39
167.10
142.04
131.85
128.96
128.54
122.10
116.57
116.53
85.96
83.42
83.39
77.48
77.37
77.16
76.84
74.51
74.48
74.02
74.00
55.30
55.22
44.48
44.42
38.23
38.19
28.63
28.57
28.53
26.71
26.66
22.01
PAJ 6-206, 3-6 CNMR
Me
N
O
Ph
AcO
29a
1:1 dr
NAME PAJ 6-206, 3-6
EXPNO 11
PROCNO 1
Date_ 20150306
Time 6.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 96.5 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127570 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

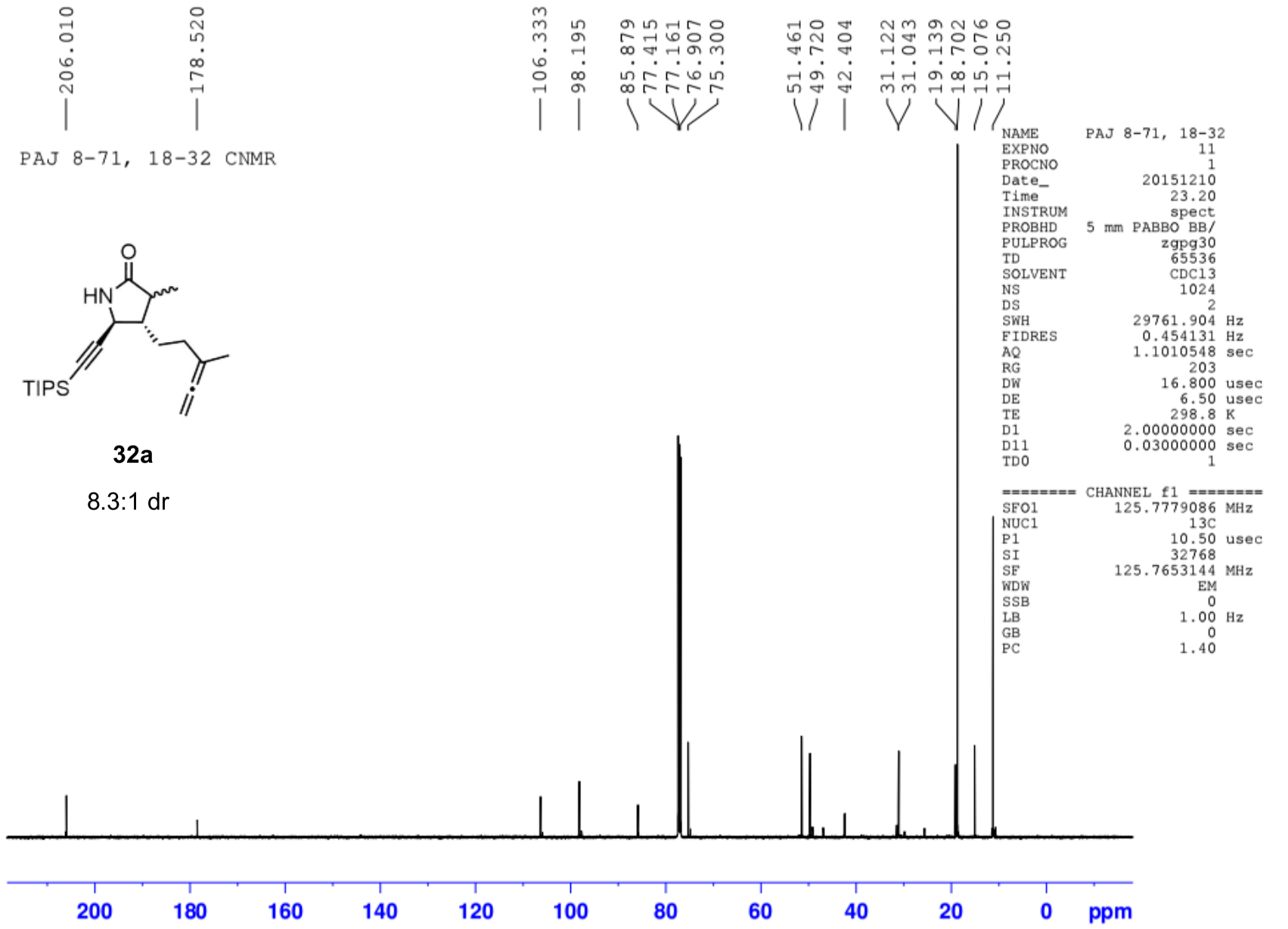

7.675
7.660
7.415
7.413
7.409
7.400
7.336
7.323
7.311
7.300
7.257
7.255
6.072
6.069
5.348
4.655
4.650
4.644
4.638
4.117
4.109
3.089
3.084
3.025
2.137
2.126
2.118
2.107
2.100
2.094
2.088
2.082
2.076
1.897
1.881
1.870
1.860
1.858
1.852
1.839
1.774
1.760
1.743
1.731
1.712
1.706
PAJ 7-134, 14-24
Me
N
O
Ph
30a
NAME PAJ 7-134, 14-24
EXPNO 10
PROCNO 1
Date_ 20150723
Time 17.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 57
DW 50.000 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
0.41
2.29
3.46
1.00
1.01
1.85
0.99
0.99
3.02
1.85
1.09
0.56
3.66

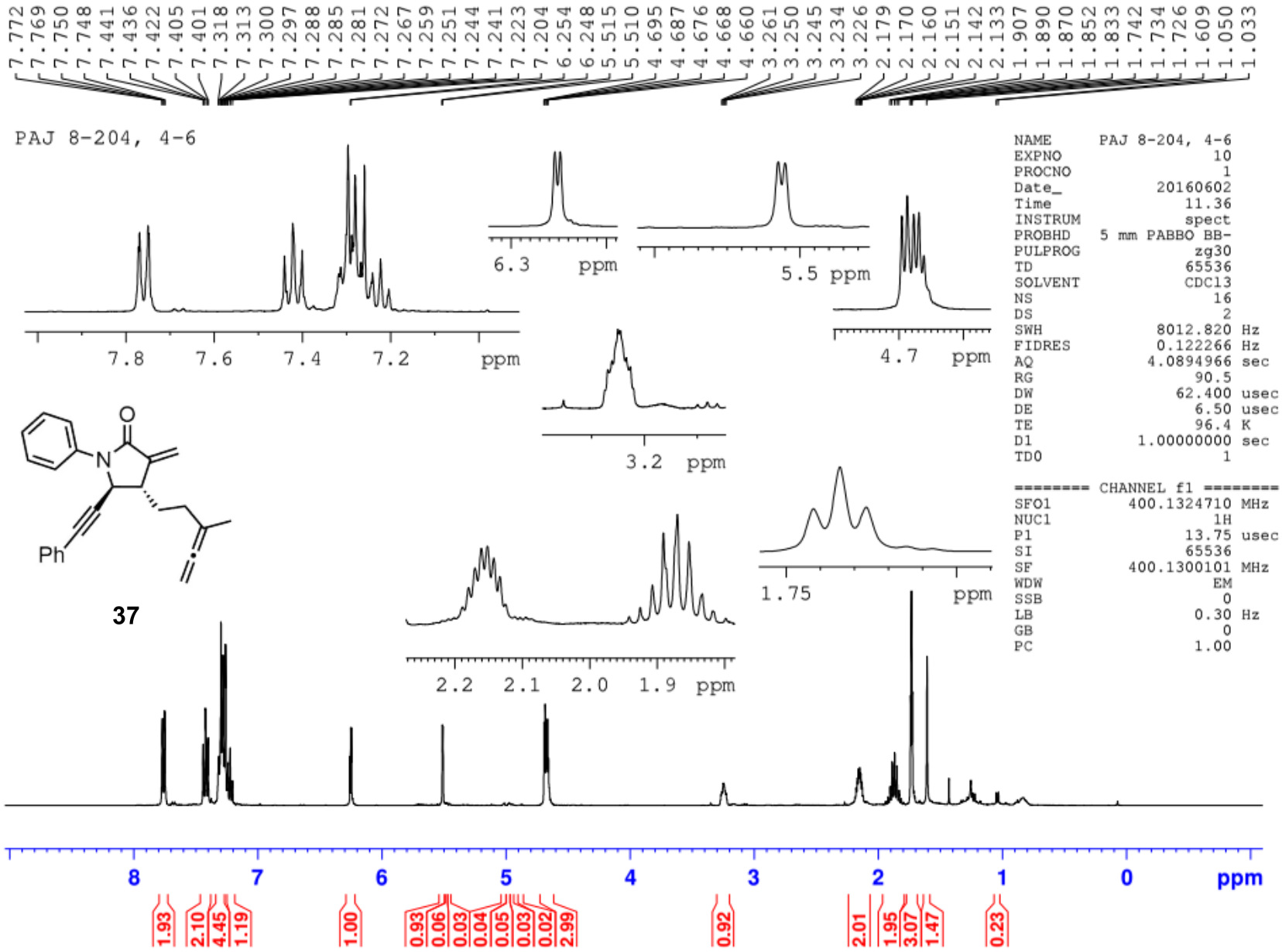

206.168
167.299
142.449
134.527
131.851
128.865
128.510
127.838
122.240
116.303
97.694
86.224
85.733
77.415
77.161
76.906
75.246
55.539
44.194
32.170
30.005
28.517
19.078
PAJ 7-134, 14-24 CNMR
Me
N
O
Ph
30a
NAME PAJ 7-134, 14-24
EXPNO 11
PROCNO 1
Date_ 20150724
Time 2.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653171 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

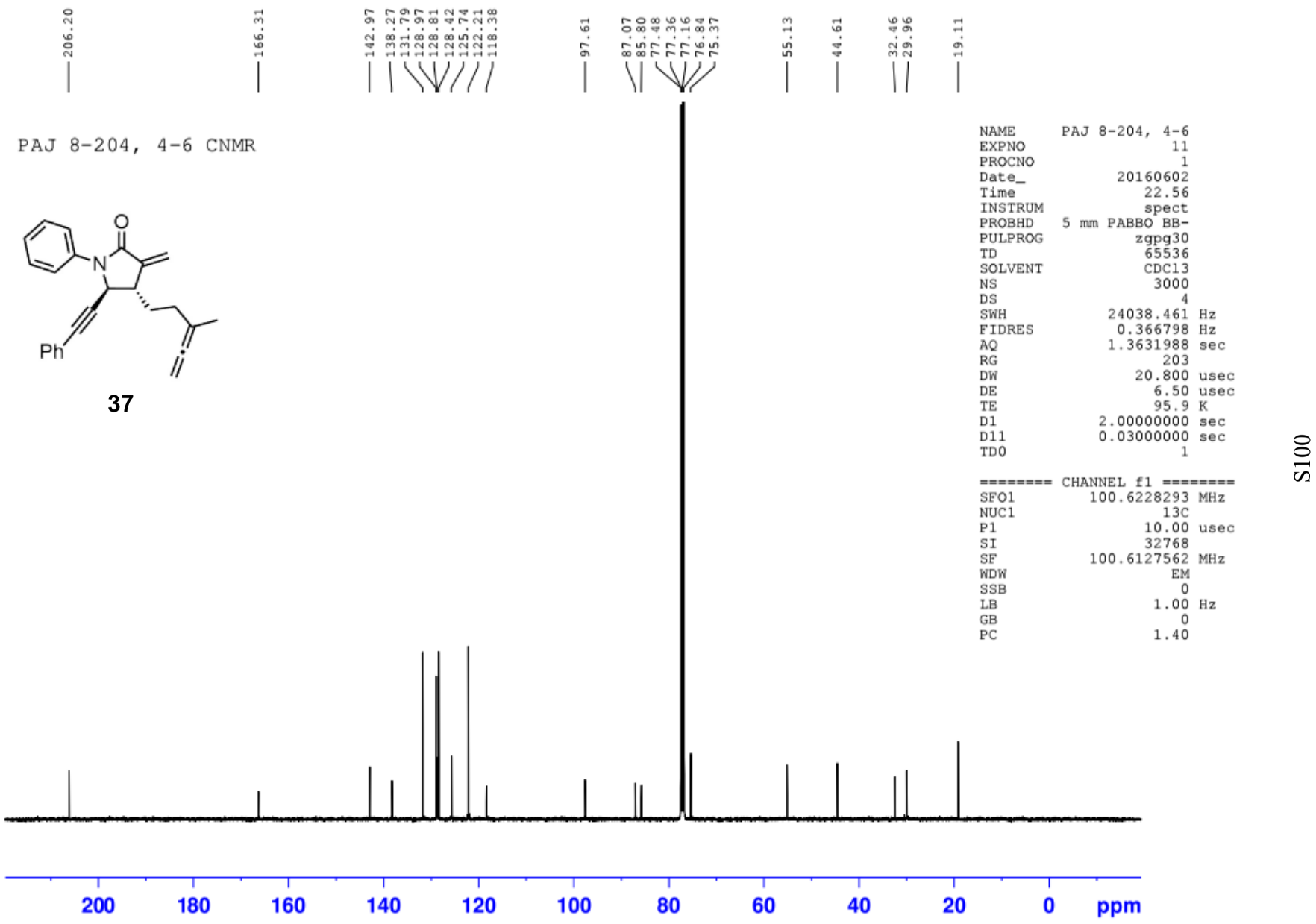

7.259
5.768
4.643
4.637
4.630
4.624
4.617
3.963
3.947
2.184
2.173
2.164
2.158
2.153
2.107
2.092
2.087
2.079
2.067
2.061
2.056
2.047
2.036
2.016
2.006
1.803
1.792
1.785
1.783
1.775
1.765
1.754
1.744
1.692
1.687
1.681
1.663
1.651
1.644
1.637
1.622
PAJ 8-71, 18-32
5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 ppm
HN
TIPS
2.6 2.4 2.2 2.0 1.8 1.6 1.4 ppm
NAME PAJ 8-71, 18-32
EXPNO 10
PROCNO 1
Date_ 20151210
Time 17.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 90.5
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600127 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.00 1.89 0.11 0.91 0.10 0.10 0.86 2.95 1.09 4.09 3.03 23.05

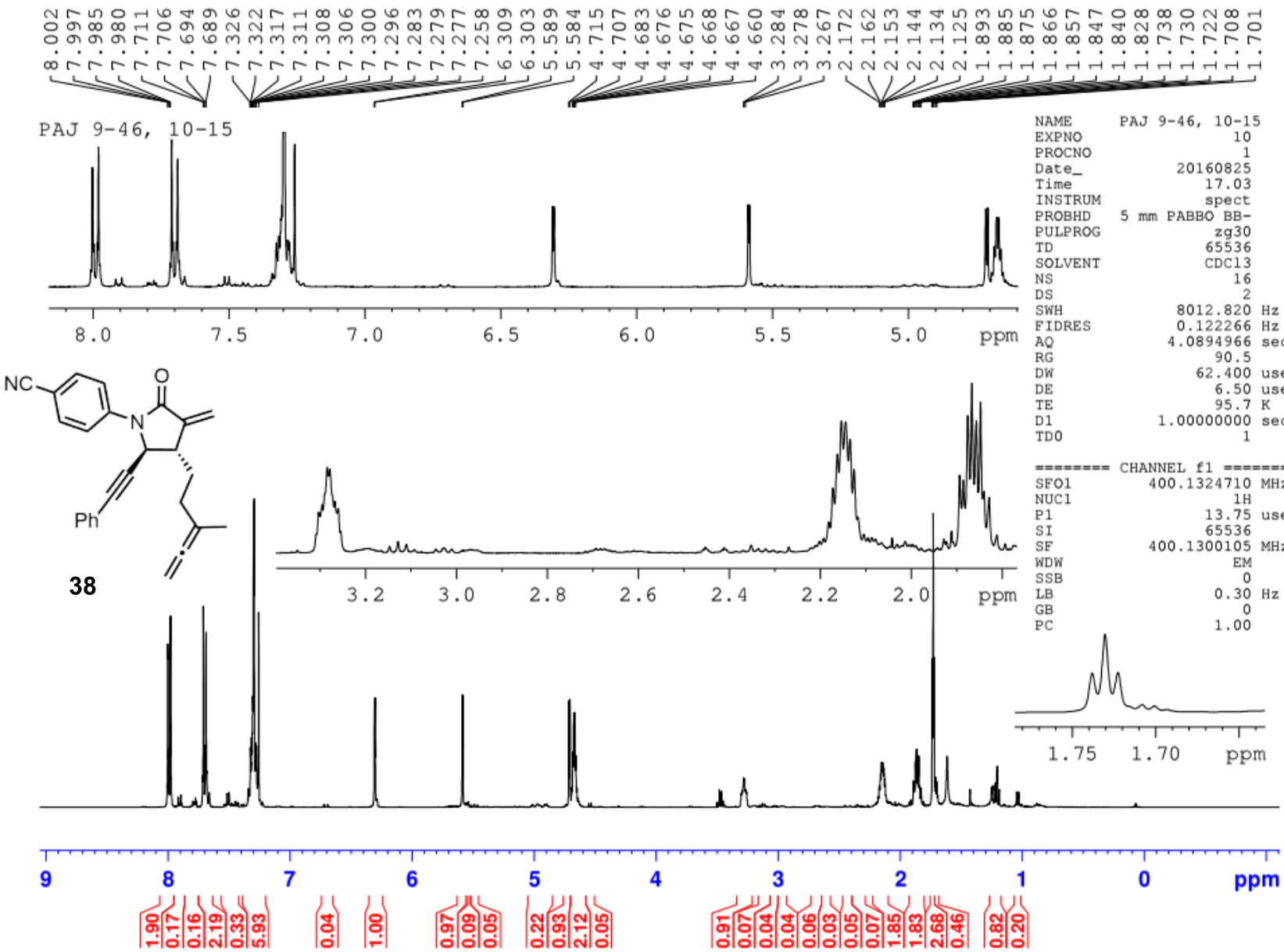
**32a**

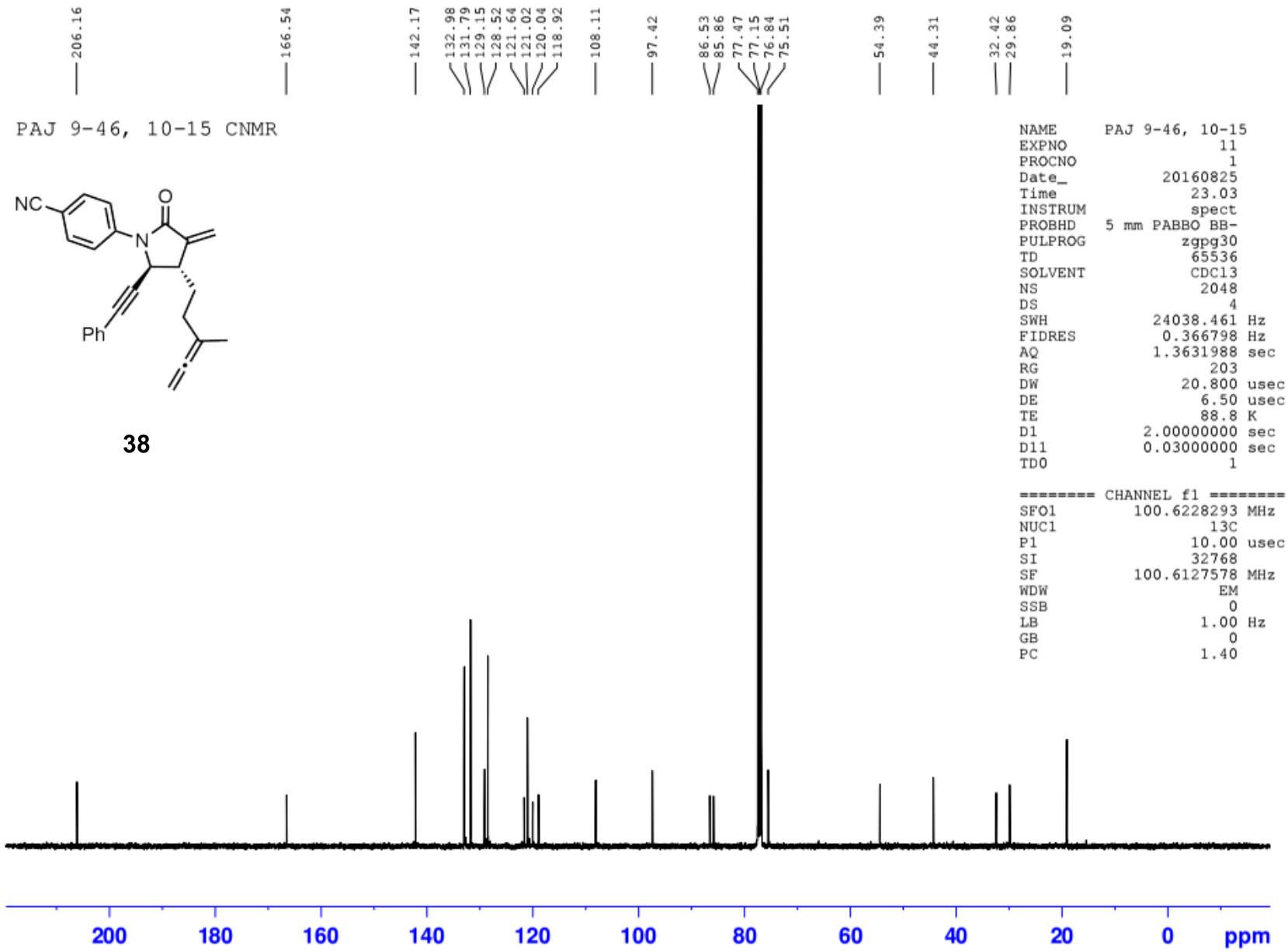
8.3:1 dr

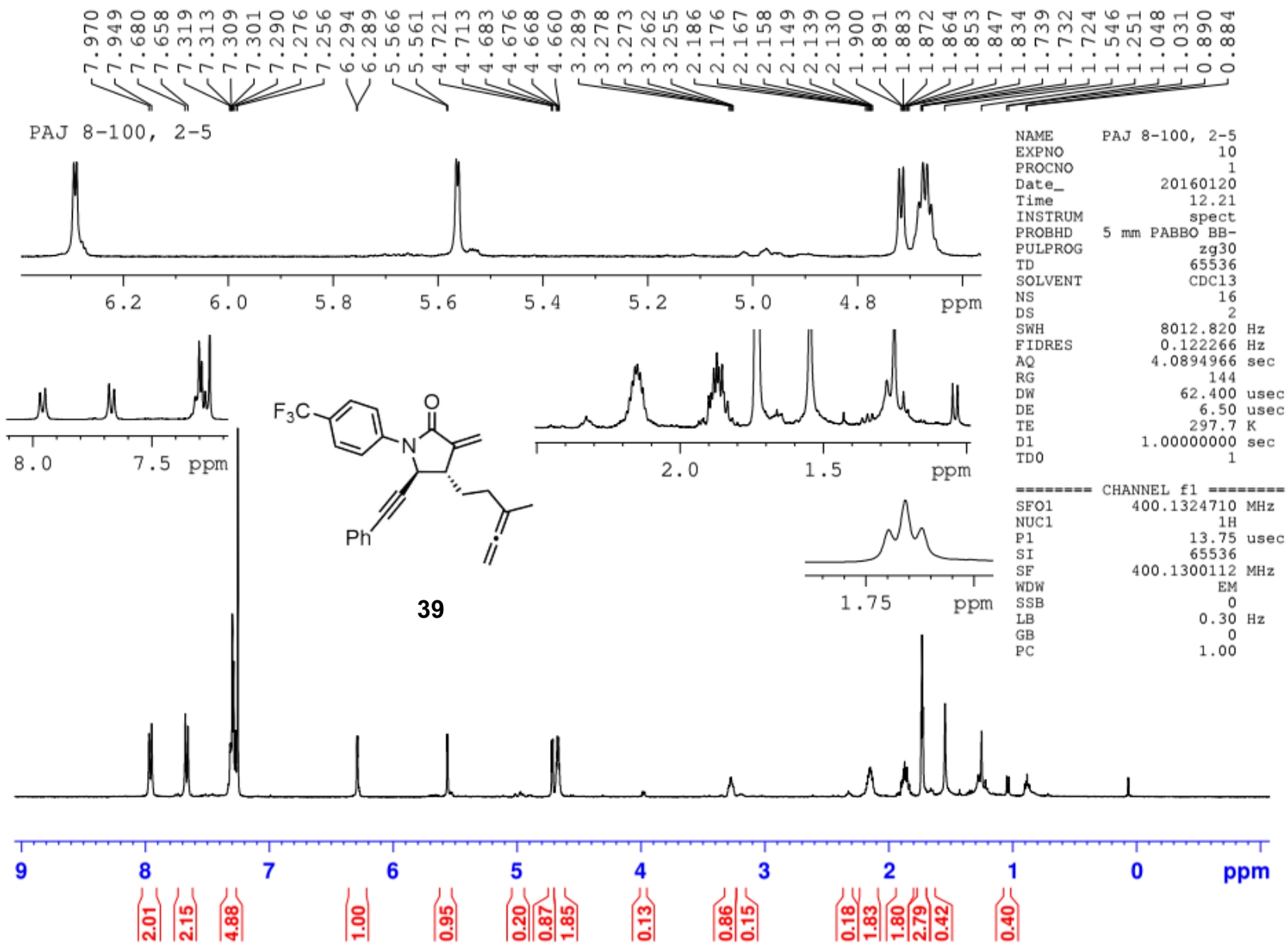

206.010
178.520
106.333
98.195
85.879
77.415
77.161
76.907
75.300
51.461
49.720
42.404
31.122
31.043
19.139
18.702
15.076
11.250
PAJ 8-71, 18-32 CNMR
HN
O
TIPS
32a
8.3:1 dr
NAME PAJ 8-71, 18-32
EXPNO 11
PROCNO 1
Date_ 20151210
Time 23.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653144 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

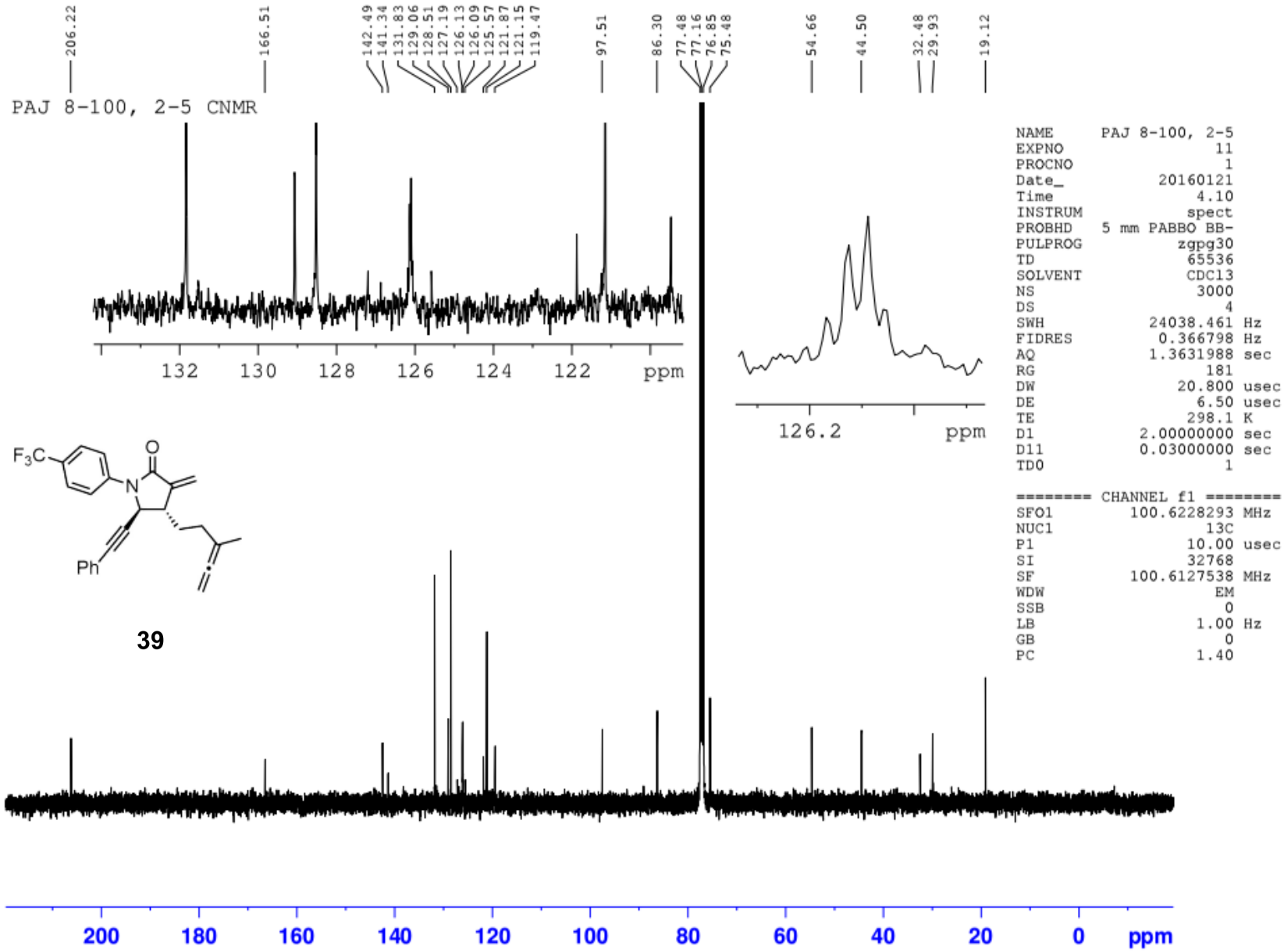

7.772 7.769 7.750 7.748 7.441 7.436 7.422 7.405 7.401 7.318 7.313 7.300 7.297 7.288 7.285 7.281 7.272 7.267 7.259 7.251 7.244 7.241 7.223 7.204 6.254 6.248 5.515 5.510 4.695 4.687 4.676 4.668 4.660 3.261 3.250 3.245 3.234 3.226 2.179 2.170 2.160 2.151 2.142 2.133 1.907 1.890 1.870 1.852 1.833 1.742 1.734 1.726 1.609 1.050 1.033
PAJ 8-204, 4-6
7.8 7.6 7.4 7.2 ppm
6.3 ppm
5.5 ppm
4.7 ppm
3.2 ppm
1.75 ppm
2.2 2.1 2.0 1.9 ppm
Ph
37
NAME PAJ 8-204, 4-6
EXPNO 10
PROCNO 1
Date_ 20160602
Time 11.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 90.5
DW 62.400 usec
DE 6.50 usec
TE 96.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8 7 6 5 4 3 2 1 0 ppm
1.93 2.10 4.45 1.19 1.00 0.93 0.06 0.03 0.04 0.05 0.03 0.02 2.99 0.92 2.01 1.95 3.07 1.47 0.23

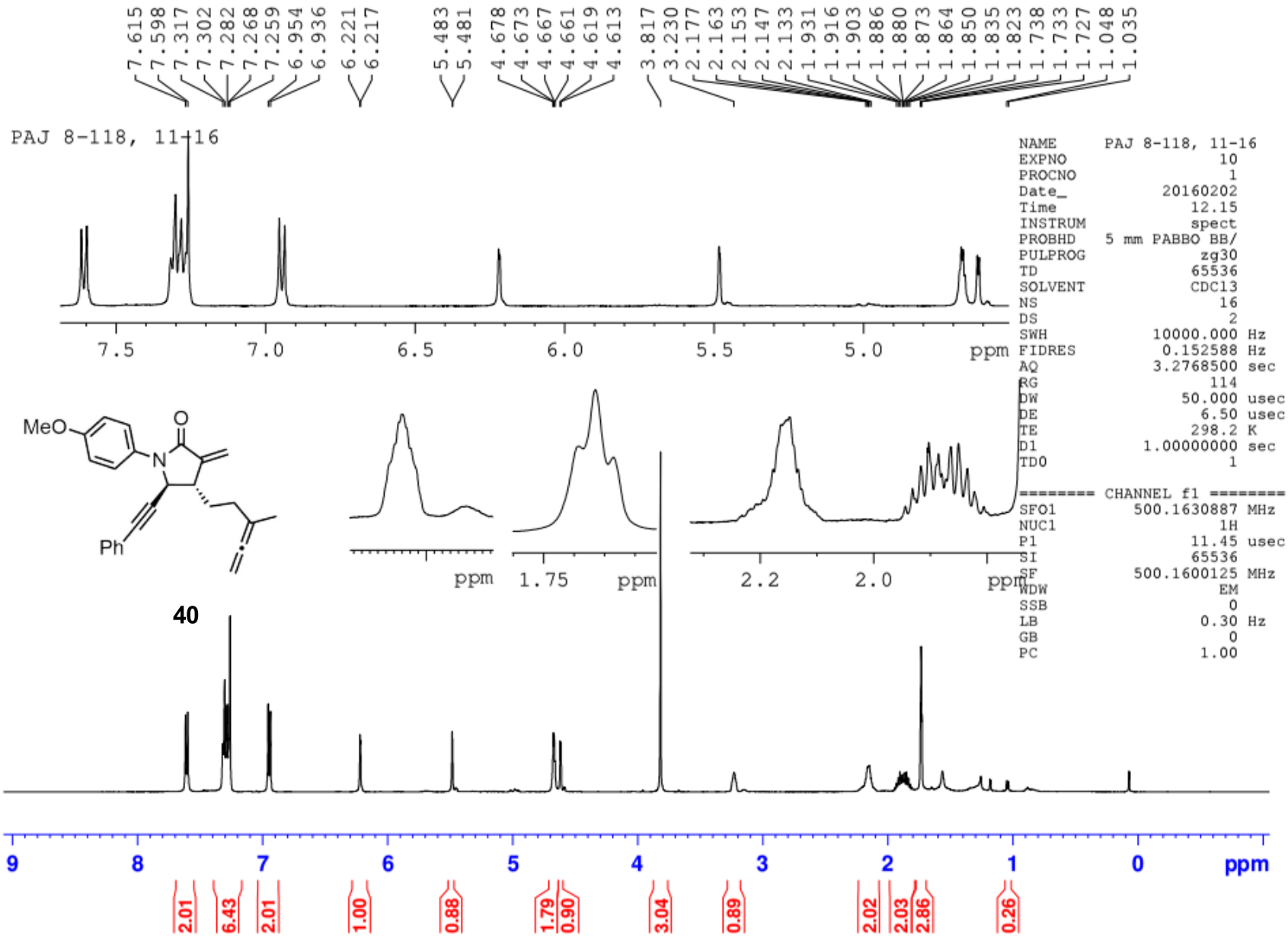

206.20
166.31
142.97
138.27
131.79
128.97
128.81
128.42
125.74
122.21
118.38
97.61
87.07
85.80
77.48
77.36
77.16
76.84
75.37
55.13
44.61
32.46
29.96
19.11
PAJ 8-204, 4-6 CNMR
O
N
Ph
37
NAME PAJ 8-204, 4-6
EXPNO 11
PROCNO 1
Date_ 20160602
Time 22.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 95.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127562 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

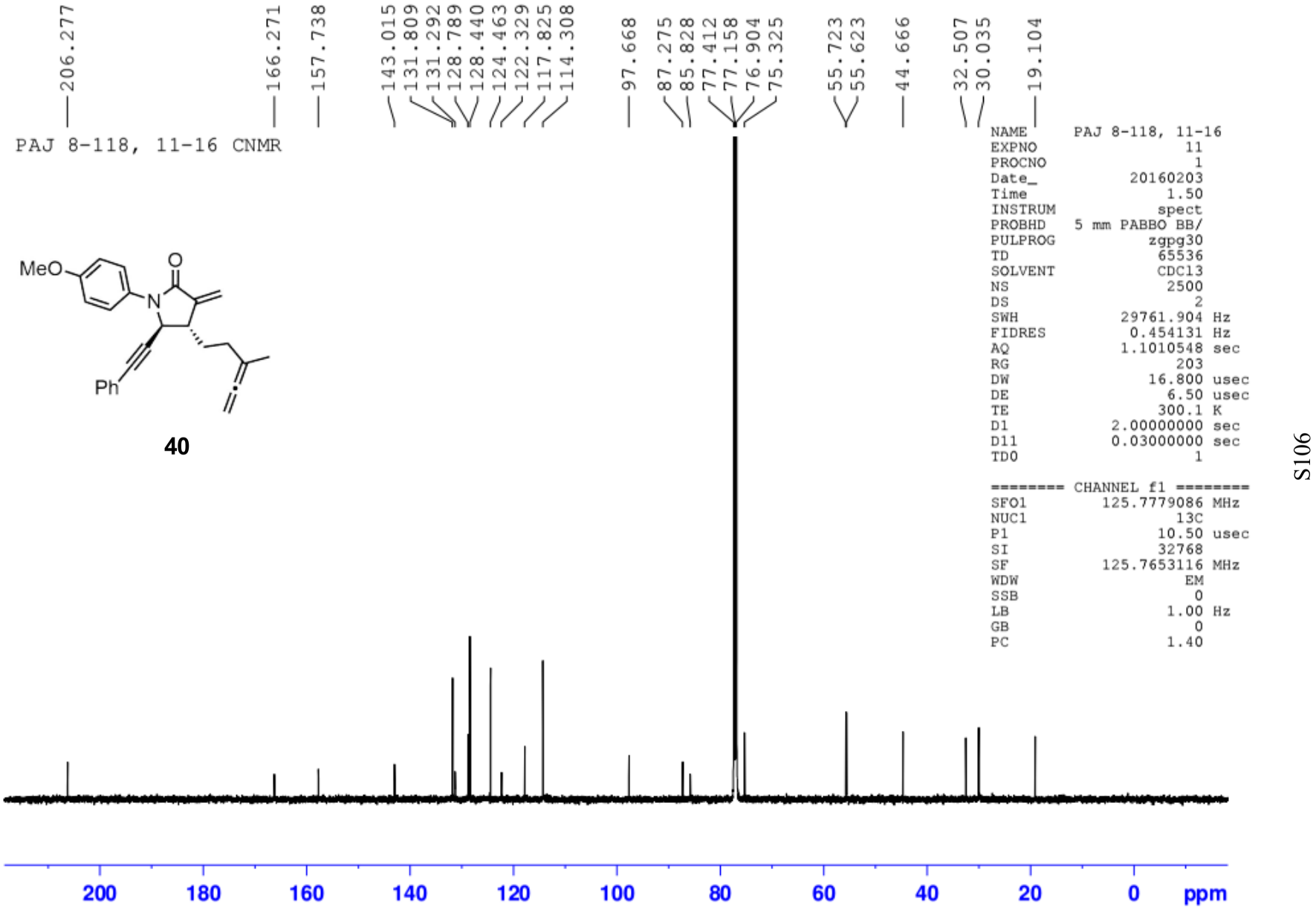

8.002
7.997
7.985
7.980
7.711
7.706
7.694
7.689
7.326
7.322
7.317
7.311
7.308
7.306
7.300
7.296
7.283
7.279
7.277
7.258
6.309
6.303
5.589
5.584
4.715
4.707
4.683
4.676
4.675
4.668
4.667
4.660
3.284
3.278
3.267
2.172
2.162
2.153
2.144
2.134
2.125
1.893
1.885
1.875
1.866
1.857
1.847
1.840
1.828
1.738
1.730
1.722
1.708
1.701
PAJ 9-46, 10-15
NAME PAJ 9-46, 10-15
EXPNO 10
PROCNO 1
Date_ 20160825
Time 17.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 90.5
DW 62.400 usec
DE 6.50 usec
TE 95.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300105 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
ppm
NC
O
N
Ph
38
3.2
3.0
2.8
2.6
2.4
2.2
2.0
ppm
1.75
1.70
ppm
9
8
7
6
5
4
3
2
1
0
ppm
1.90
0.17
0.16
2.19
0.33
5.93
0.04
1.00
0.97
0.09
0.05
0.22
0.93
2.12
0.05
0.91
0.07
0.04
0.04
0.06
0.03
0.05
0.07
1.85
1.83
2.68
0.46
0.82
0.20


206.16
166.54
142.17
132.98
131.79
129.15
128.52
121.64
121.02
120.04
118.92
108.11
97.42
86.53
85.86
77.47
77.15
76.84
75.51
54.39
44.31
32.42
29.86
19.09
PAJ 9-46, 10-15 CNMR
NC
O
N
Ph
38
NAME PAJ 9-46, 10-15
EXPNO 11
PROCNO 1
Date_ 20160825
Time 23.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 88.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127578 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

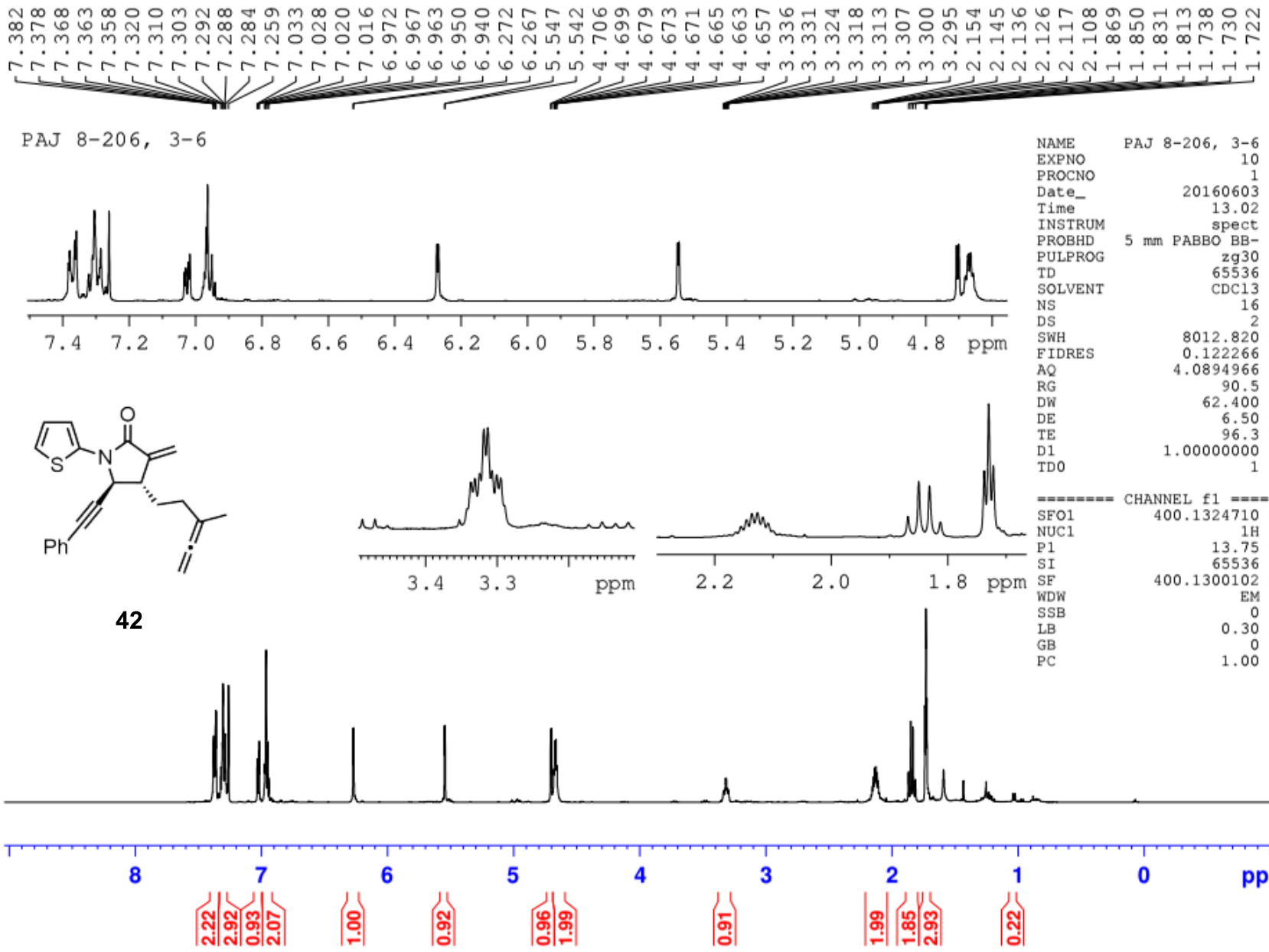

7.970 7.949 7.680 7.658 7.319 7.313 7.309 7.301 7.290 7.276 7.256 6.294 6.289 5.566 5.561 4.721 4.713 4.683 4.676 4.668 4.660 3.289 3.278 3.273 3.262 3.255 2.186 2.176 2.167 2.158 2.149 2.139 2.130 1.900 1.891 1.883 1.872 1.864 1.853 1.847 1.834 1.739 1.732 1.724 1.546 1.251 1.048 1.031 0.890 0.884
PAJ 8-100, 2-5
6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 ppm
8.0 7.5 ppm
2.0 1.5 ppm
1.75 ppm
F3C
N
O
Ph
39
NAME PAJ 8-100, 2-5
EXPNO 10
PROCNO 1
Date_ 20160120
Time 12.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 144
DW 62.400 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
2.01 2.15 4.88 1.00 0.95 0.20 0.87 1.85 0.13 0.86 0.15 0.18 1.83 1.80 2.79 0.42 0.40

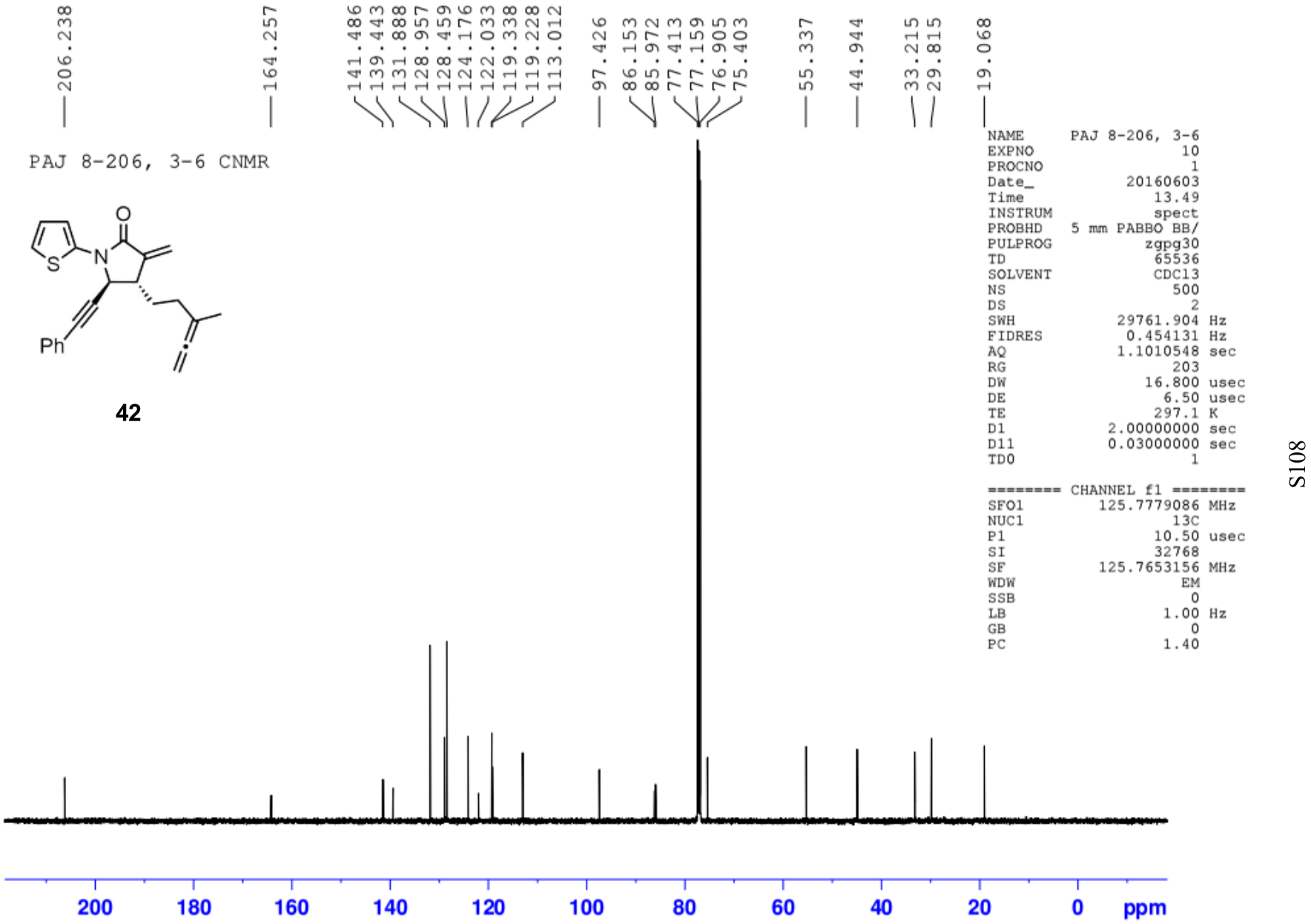

206.22
166.51
142.49
141.34
131.83
129.06
128.51
127.19
126.13
126.09
125.57
121.87
121.15
119.47
97.51
86.30
77.48
77.16
76.85
75.48
54.66
44.50
32.48
29.93
19.12
PAJ 8-100, 2-5 CNMR
132
130
128
126
124
122
ppm
126.2
ppm
F3C
O
N
Ph
39
NAME PAJ 8-100, 2-5
EXPNO 11
PROCNO 1
Date_ 20160121
Time 4.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127538 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


7.615
7.598
7.317
7.302
7.282
7.268
7.259
6.954
6.936
6.221
6.217
5.483
5.481
4.678
4.673
4.667
4.661
4.619
4.613
3.817
3.230
2.177
2.163
2.153
2.147
2.133
1.931
1.916
1.903
1.886
1.880
1.873
1.864
1.850
1.835
1.823
1.738
1.733
1.727
1.048
1.035
PAJ 8-118, 11-16
NAME PAJ 8-118, 11-16
EXPNO 10
PROCNO 1
Date_ 20160202
Time 12.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 114
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
N
O
Ph
40
ppm
9
8
7
6
5
4
3
2
1
0
2.01
6.43
2.01
1.00
0.88
1.79
0.90
3.04
0.89
2.02
2.03
2.86
0.26

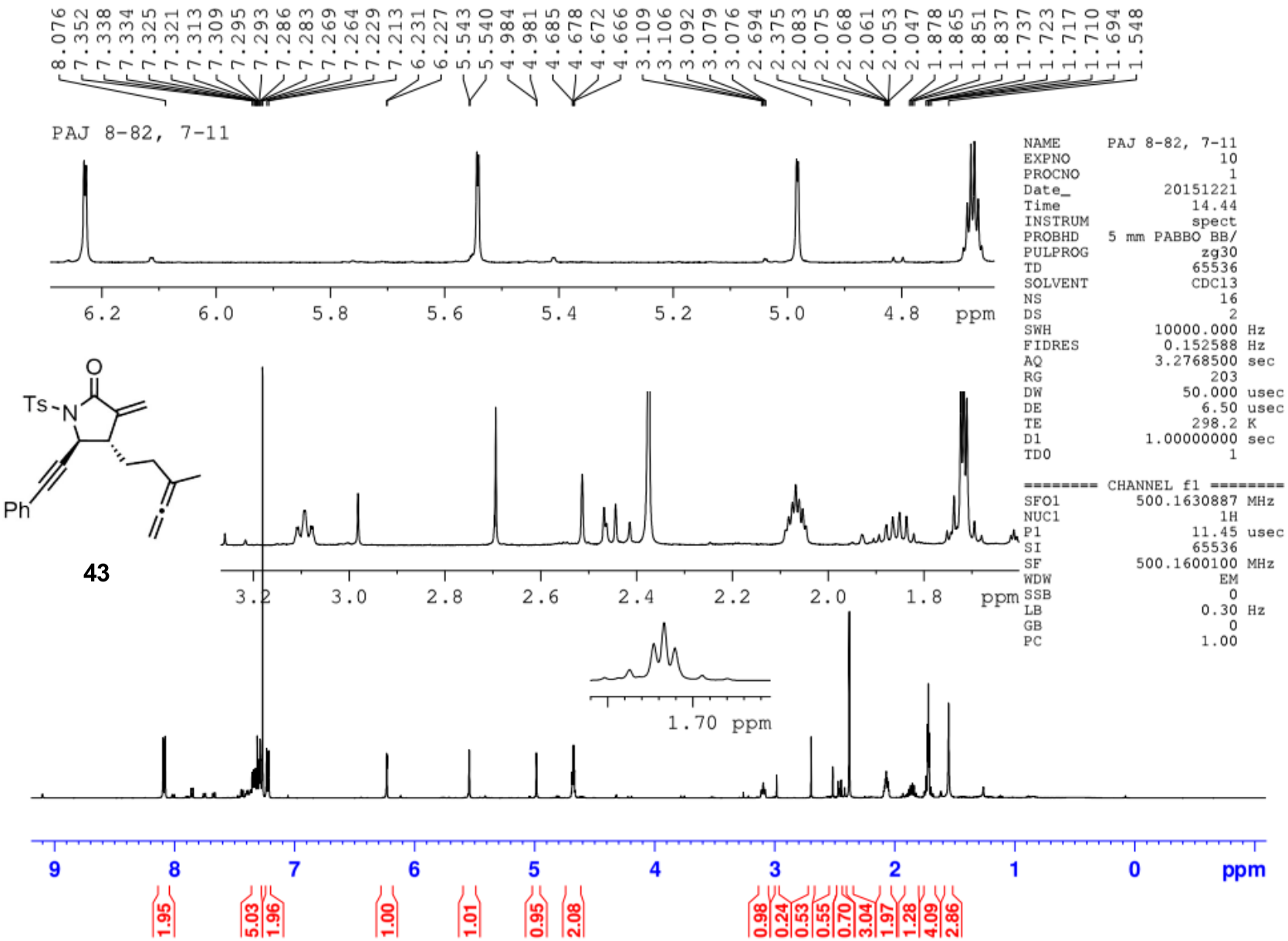

206.277
166.271
157.738
143.015
131.809
131.292
128.789
128.440
124.463
122.329
117.825
114.308
97.668
87.275
85.828
77.412
77.158
76.904
75.325
55.723
55.623
44.666
32.507
30.035
19.104
PAJ 8-118, 11-16 CNMR
MeO
O
N
Ph
NAME PAJ 8-118, 11-16
EXPNO 11
PROCNO 1
Date_ 20160203
Time 1.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2500
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653116 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

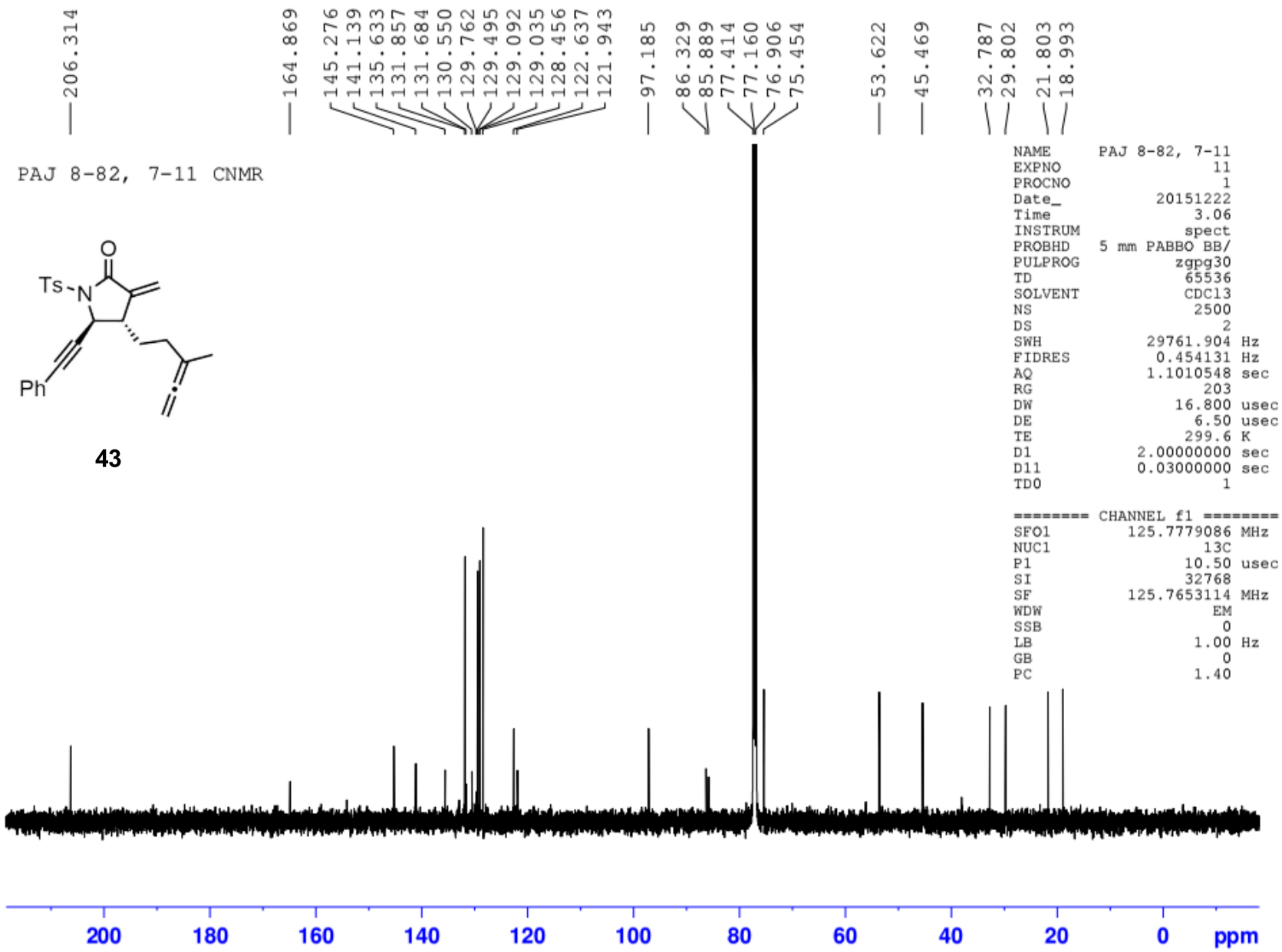
**40**

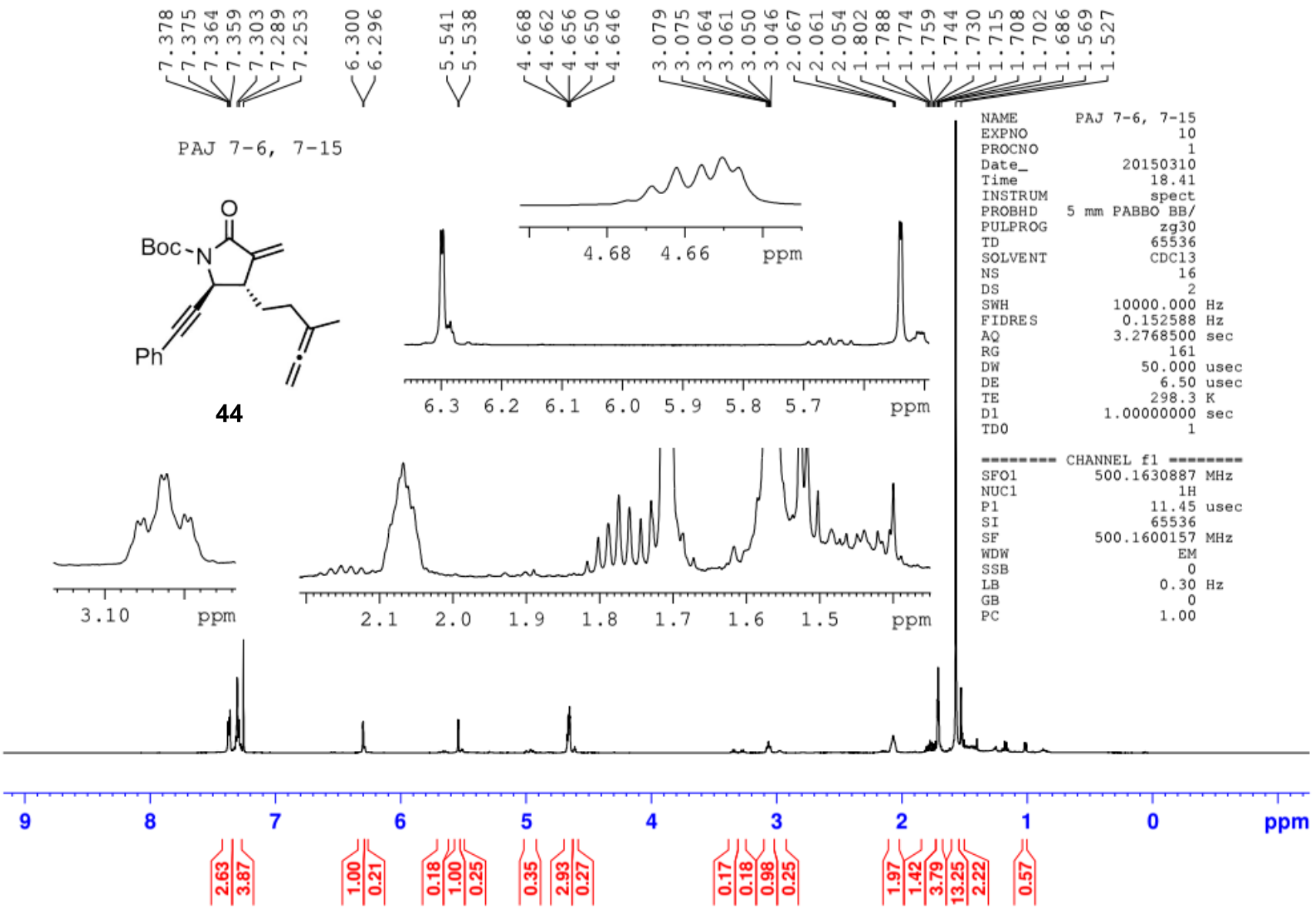

7.382
7.378
7.368
7.363
7.358
7.320
7.310
7.303
7.292
7.288
7.284
7.259
7.033
7.028
7.020
7.016
6.972
6.967
6.963
6.950
6.940
6.272
6.267
5.547
5.542
4.706
4.699
4.679
4.673
4.671
4.665
4.663
4.657
3.336
3.331
3.324
3.318
3.313
3.307
3.300
3.295
2.154
2.145
2.136
2.126
2.117
2.108
1.869
1.850
1.831
1.813
1.738
1.730
1.722
PAJ 8-206, 3-6
NAME PAJ 8-206, 3-6
EXPNO 10
PROCNO 1
Date_ 20160603
Time 13.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 90.5
DW 62.400 usec
DE 6.50 usec
TE 96.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 ppm
3.4 3.3 ppm
2.2 2.0 1.8 ppm
S
N
O
Ph
42
8 7 6 5 4 3 2 1 0 ppm
2.22
2.92
0.93
2.07
1.00
0.92
0.96
1.99
0.91
1.99
1.85
2.93
0.22

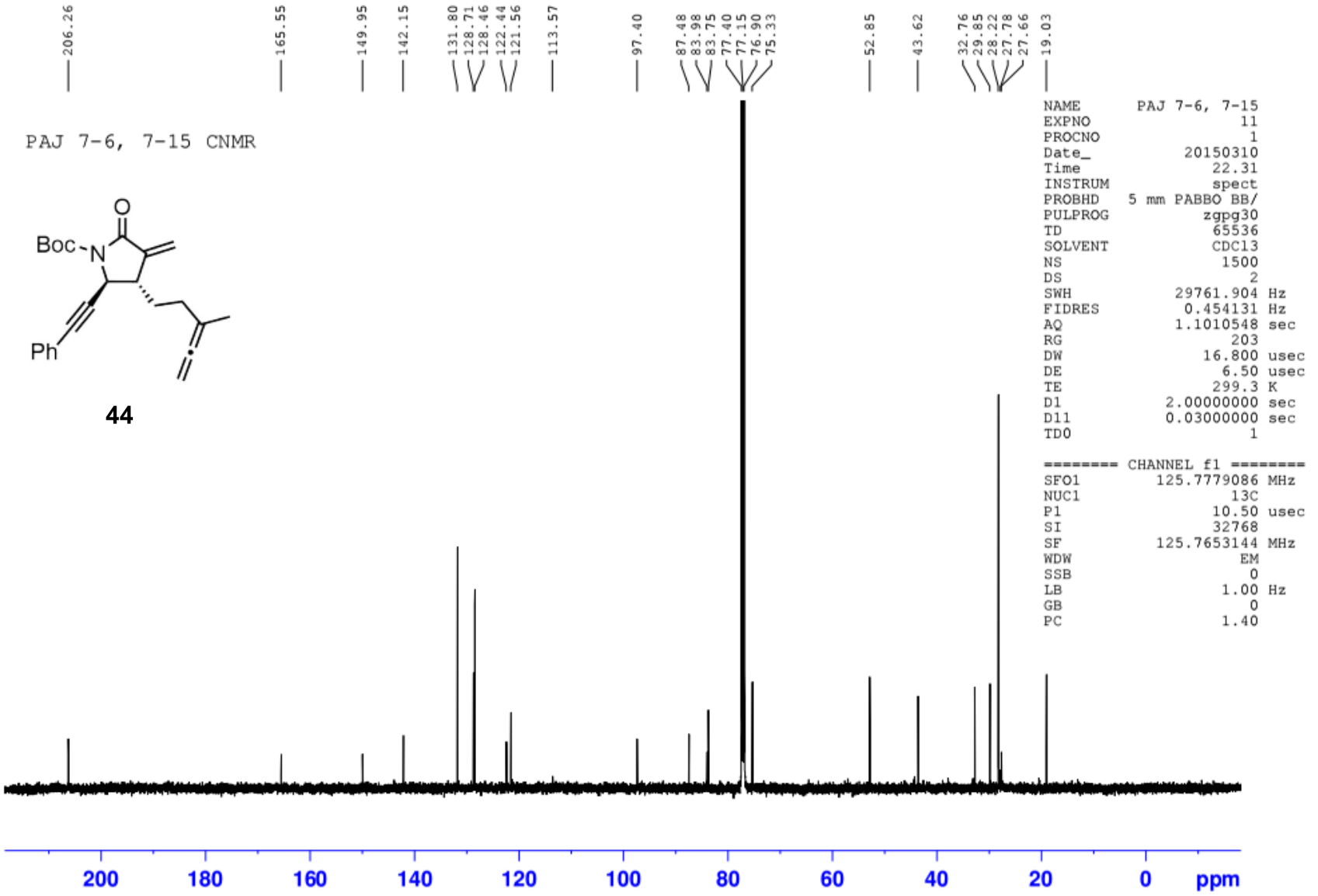

206.238
164.257
141.486
139.443
131.888
128.957
128.459
124.176
122.033
119.338
119.228
113.012
97.426
86.153
85.972
77.413
77.159
76.905
75.403
55.337
44.944
33.215
29.815
19.068
PAJ 8-206, 3-6 CNMR
NAME PAJ 8-206, 3-6
EXPNO 10
PROCNO 1
Date_ 20160603
Time 13.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 500
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653156 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

**42**

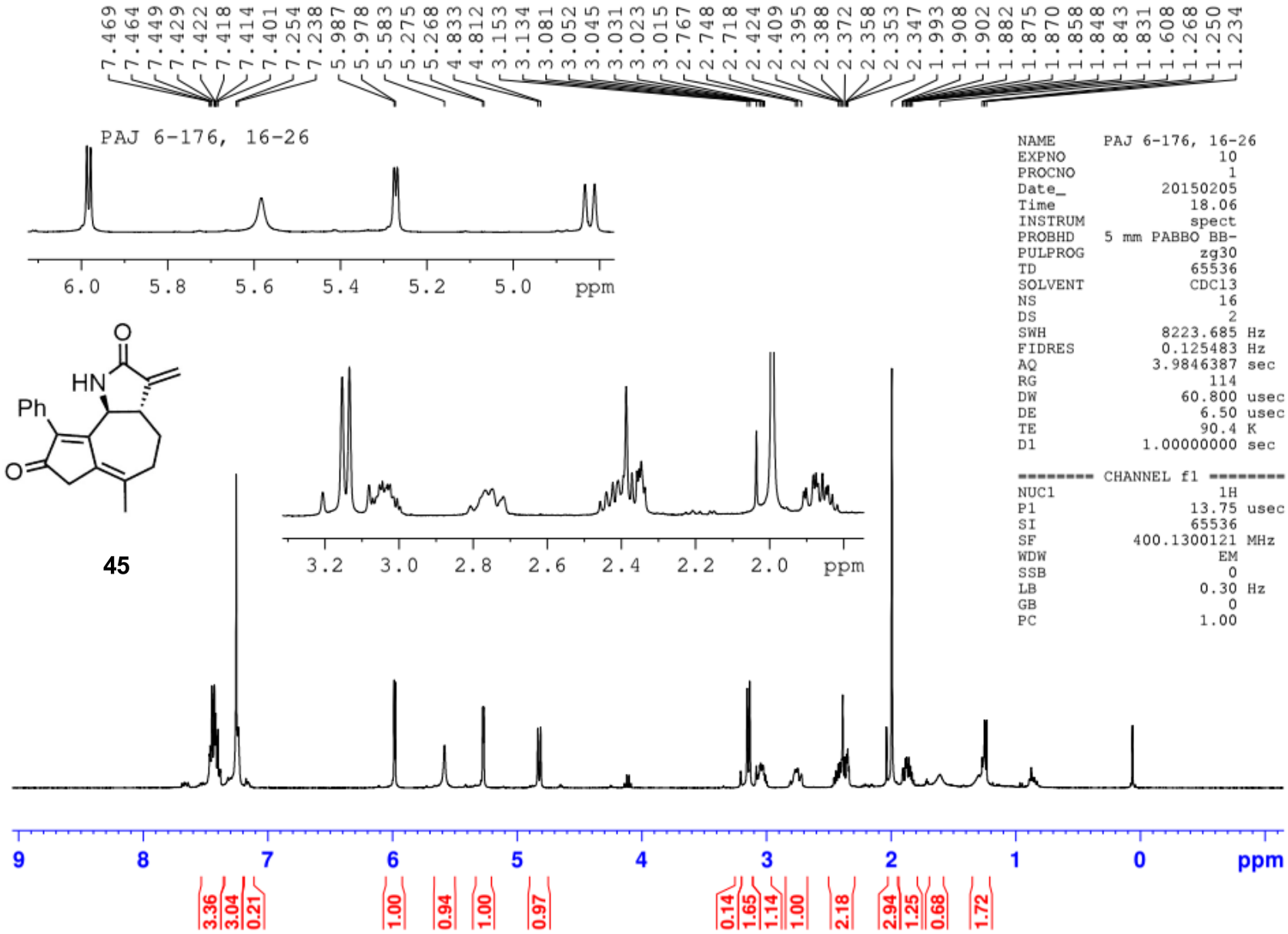

8.076
7.352
7.338
7.334
7.325
7.321
7.313
7.309
7.295
7.293
7.286
7.283
7.269
7.264
7.229
7.213
6.231
6.227
5.543
5.540
4.984
4.981
4.685
4.678
4.672
4.666
3.109
3.106
3.092
3.079
3.076
2.694
2.375
2.083
2.075
2.068
2.061
2.053
2.047
1.878
1.865
1.851
1.837
1.737
1.723
1.717
1.710
1.694
1.548
PAJ 8-82, 7-11
6.2
6.0
5.8
5.6
5.4
5.2
5.0
4.8
ppm
NAME PAJ 8-82, 7-11
EXPNO 10
PROCNO 1
Date_ 20151221
Time 14.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ts
N
O
Ph
43
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
ppm
1.70 ppm
9
8
7
6
5
4
3
2
1
0
ppm
1.95
5.03
1.96
1.00
1.01
0.95
2.08
0.98
0.24
0.53
0.55
0.70
3.04
1.97
1.28
4.09
2.86

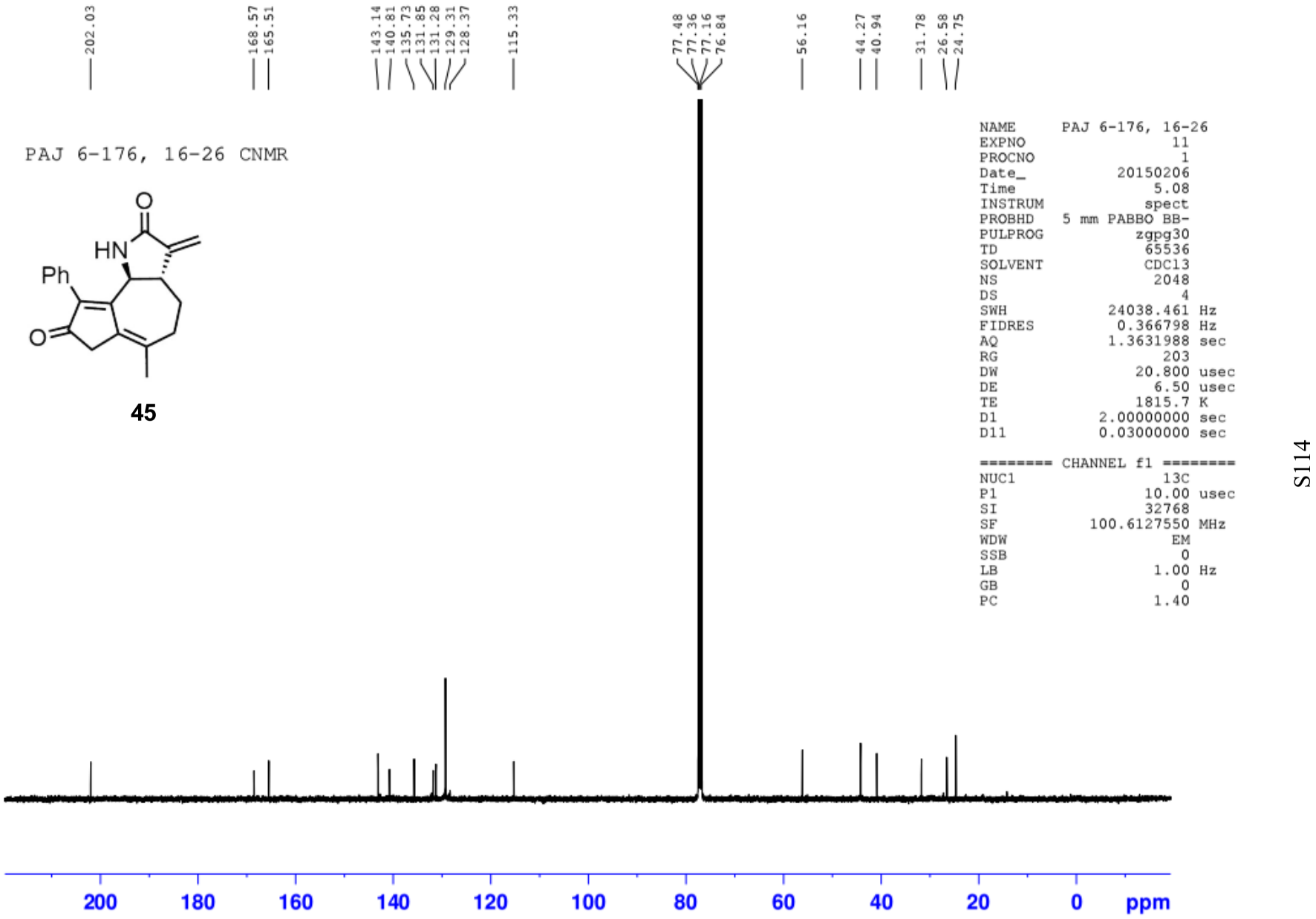

206.314
164.869
145.276
141.139
135.633
131.857
131.684
130.550
129.762
129.495
129.092
129.035
128.456
122.637
121.943
97.185
86.329
85.889
77.414
77.160
76.906
75.454
53.622
45.469
32.787
29.802
21.803
18.993
PAJ 8-82, 7-11 CNMR
Ts
N
O
Ph
43
NAME PAJ 8-82, 7-11
EXPNO 11
PROCNO 1
Date_ 20151222
Time 3.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2500
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653114 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

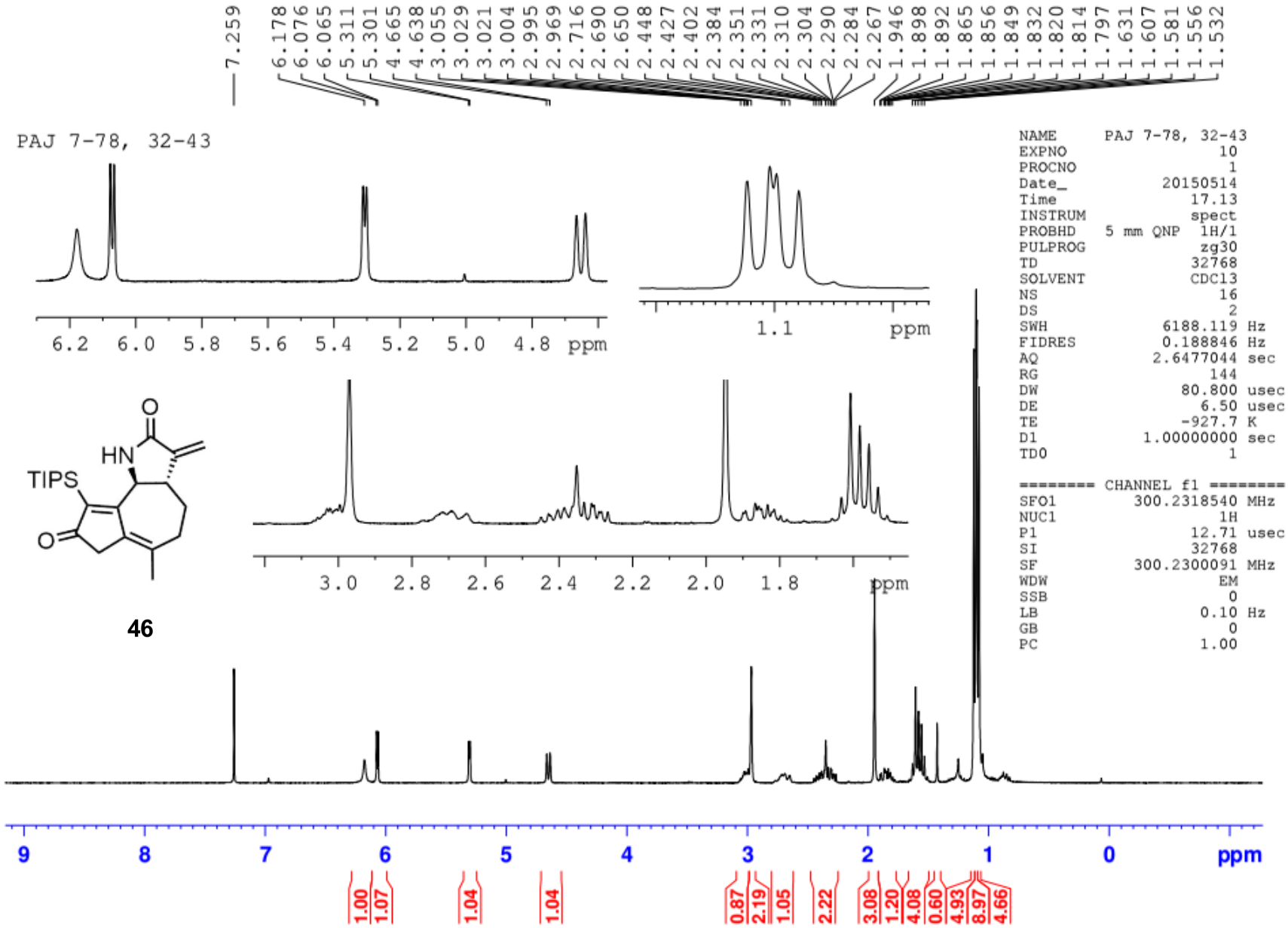

7.378
7.375
7.364
7.359
7.303
7.289
7.253
6.300
6.296
5.541
5.538
4.668
4.662
4.656
4.650
4.646
3.079
3.075
3.064
3.061
3.050
3.046
2.067
2.061
2.054
1.802
1.788
1.774
1.759
1.744
1.730
1.715
1.708
1.702
1.686
1.569
1.527
PAJ 7-6, 7-15
Boc
N
O
Ph
44
4.68 4.66 ppm
6.3 6.2 6.1 6.0 5.9 5.8 5.7 ppm
3.10 ppm
2.1 2.0 1.9 1.8 1.7 1.6 1.5 ppm
NAME PAJ 7-6, 7-15
EXPNO 10
PROCNO 1
Date_ 20150310
Time 18.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 161
DW 50.000 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600157 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
2.63 3.87 1.00 0.21 0.18 1.00 0.25 0.35 2.93 0.27 0.17 0.18 0.98 0.25 1.97 1.42 3.79 13.25 2.22 0.57

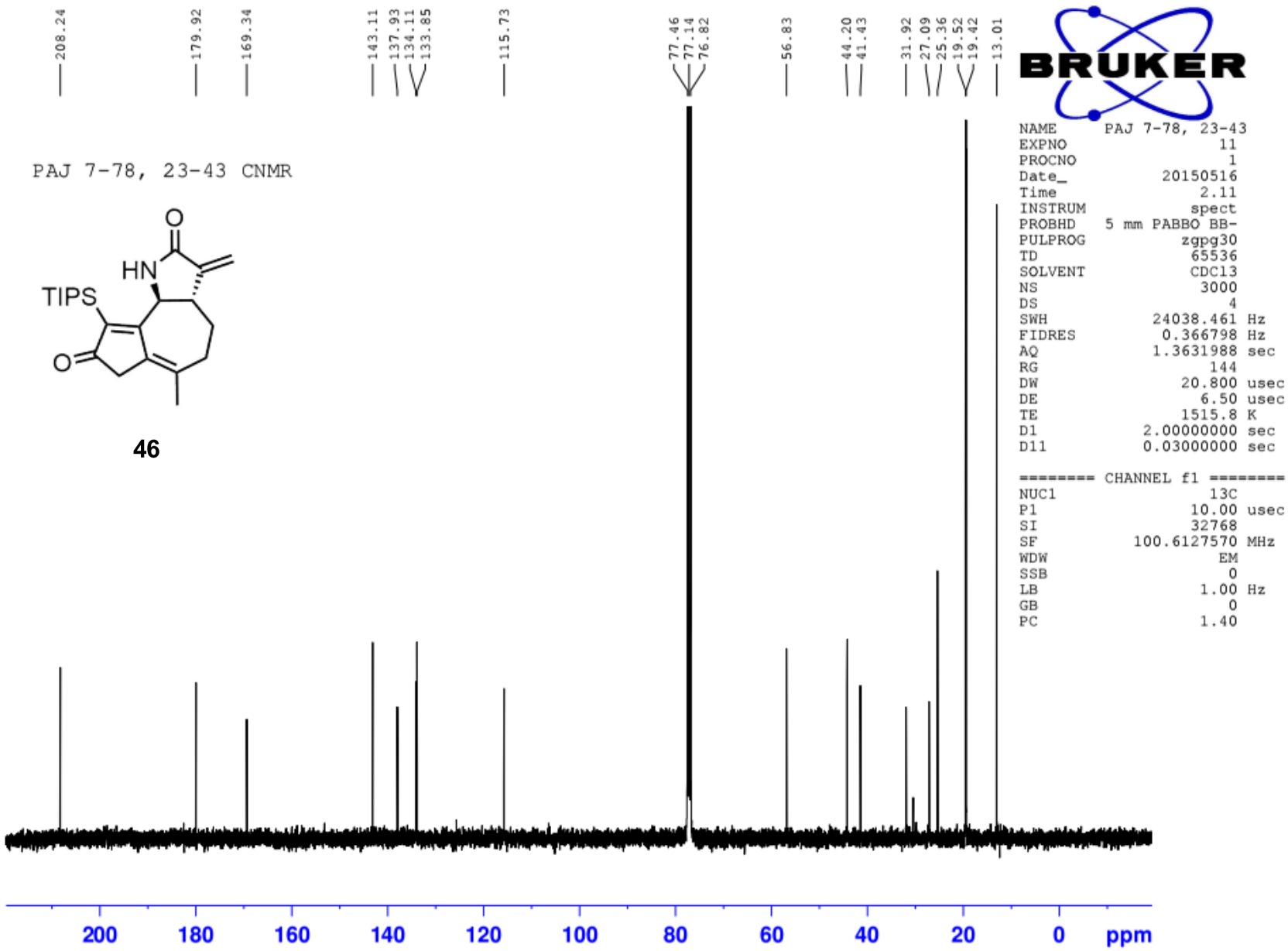

206.26
165.55
149.95
142.15
131.80
128.71
128.46
122.44
121.56
113.57
97.40
87.48
83.98
83.75
77.40
77.15
76.90
75.33
52.85
43.62
32.76
29.85
28.22
27.78
27.66
19.03
PAJ 7-6, 7-15 CNMR
Boc
N
O
Ph
NAME PAJ 7-6, 7-15
EXPNO 11
PROCNO 1
Date_ 20150310
Time 22.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1500
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653144 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

**44**

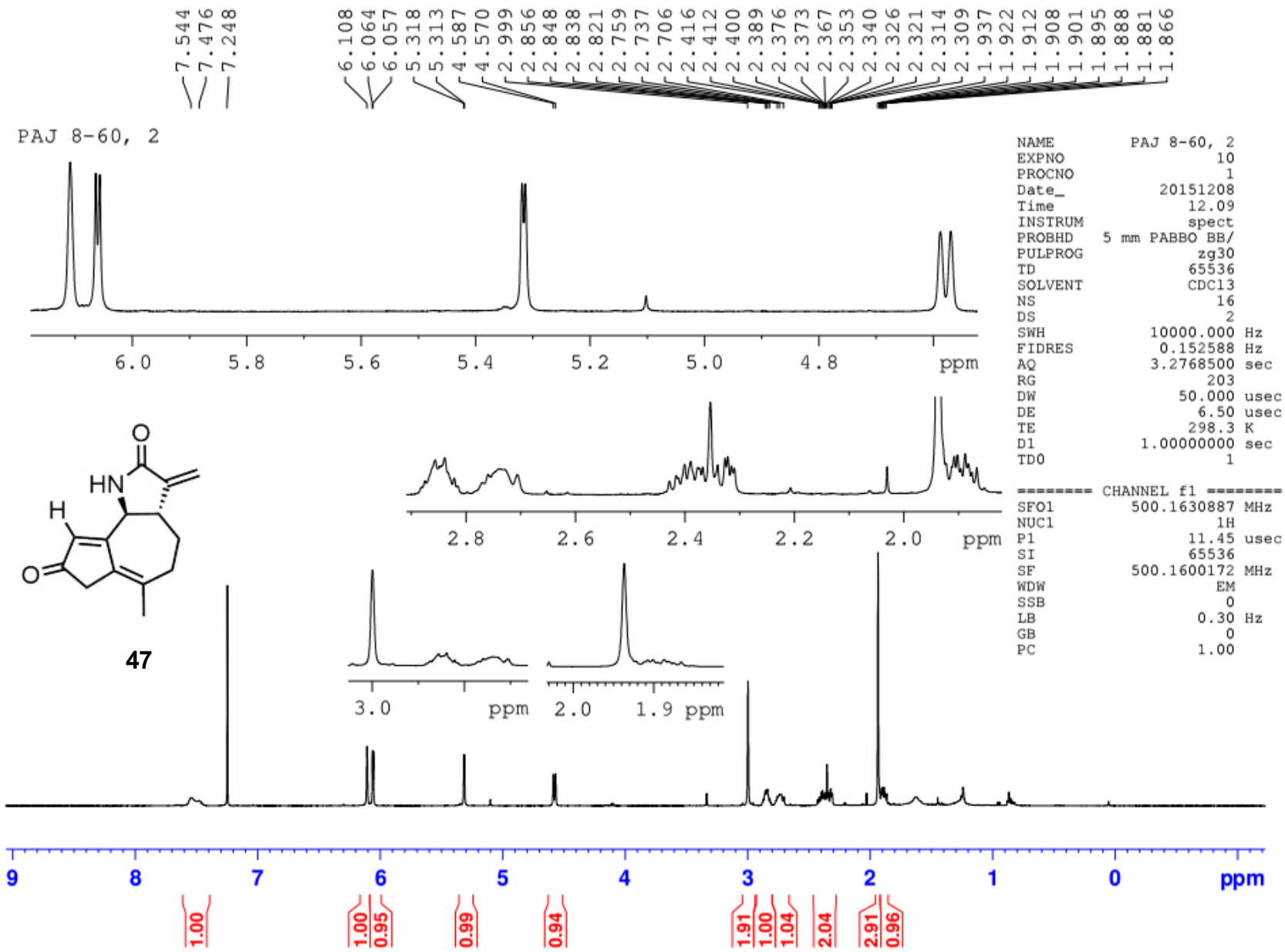

7.469
7.464
7.449
7.429
7.422
7.418
7.414
7.401
7.254
7.238
5.987
5.978
5.583
5.275
5.268
4.833
4.812
3.153
3.134
3.081
3.052
3.045
3.031
3.023
3.015
2.767
2.748
2.718
2.424
2.409
2.395
2.388
2.372
2.358
2.353
2.347
1.993
1.908
1.902
1.882
1.875
1.870
1.858
1.848
1.843
1.831
1.608
1.268
1.250
1.234
PAJ 6-176, 16-26
6.0
5.8
5.6
5.4
5.2
5.0
ppm
NAME PAJ 6-176, 16-26
EXPNO 10
PROCNO 1
Date_ 20150205
Time 18.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 114
DW 60.800 usec
DE 6.50 usec
TE 90.4 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HN
Ph
O
O
45
3.2
3.0
2.8
2.6
2.4
2.2
2.0
ppm
9
8
7
6
5
4
3
2
1
0
ppm
3.36
3.04
0.21
1.00
0.94
1.00
0.97
0.14
1.65
1.14
1.00
2.18
2.94
1.25
0.68
1.72

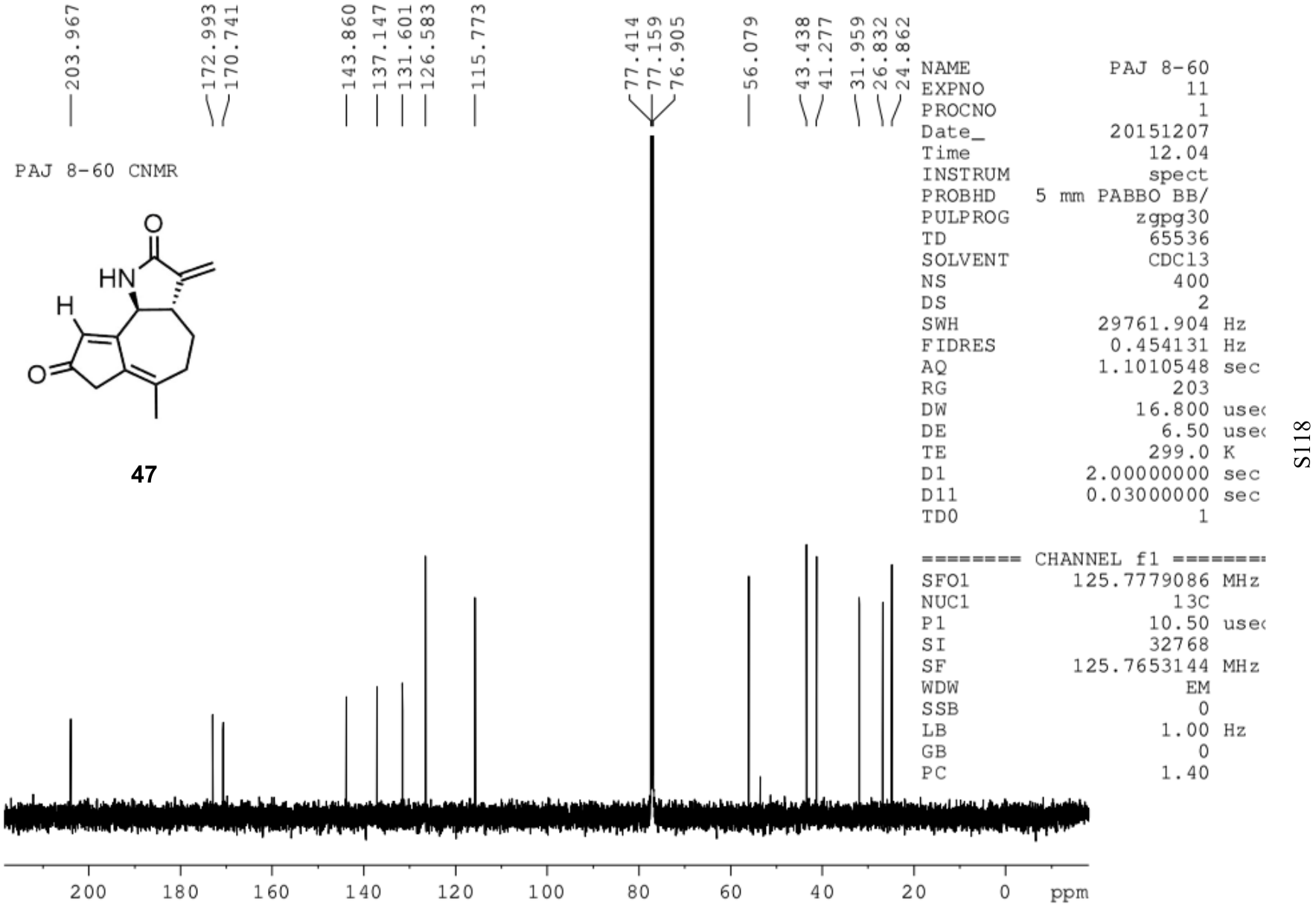

202.03
168.57
165.51
143.14
140.81
135.73
131.85
131.28
129.31
128.37
115.33
77.48
77.36
77.16
76.84
56.16
44.27
40.94
31.78
26.58
24.75
PAJ 6-176, 16-26 CNMR
HN
O
Ph
O
45
NAME PAJ 6-176, 16-26
EXPNO 11
PROCNO 1
Date_ 20150206
Time 5.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 1815.7 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127550 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

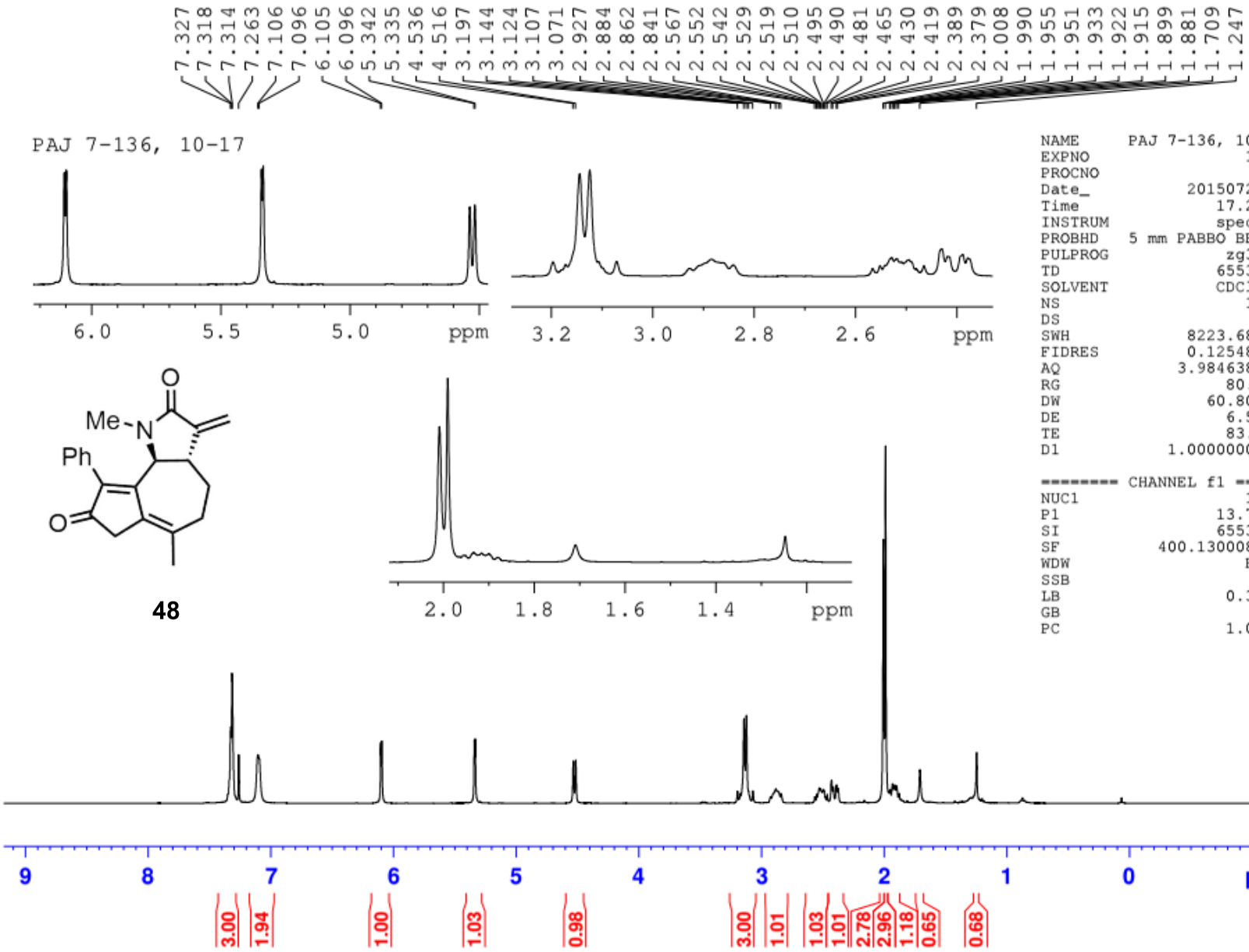

7.259
6.178
6.076
6.065
5.311
5.301
4.665
4.638
3.055
3.029
3.021
3.004
2.995
2.969
2.716
2.690
2.650
2.448
2.427
2.402
2.384
2.351
2.331
2.310
2.304
2.290
2.284
2.267
1.946
1.898
1.892
1.865
1.856
1.849
1.832
1.820
1.814
1.797
1.631
1.607
1.581
1.556
1.532
PAJ 7-78, 32-43
6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 ppm
1.1 ppm
3.0 2.8 2.6 2.4 2.2 2.0 1.8 ppm
HN
TIPS
O
O
46
NAME PAJ 7-78, 32-43
EXPNO 10
PROCNO 1
Date_ 20150514
Time 17.13
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 144
DW 80.800 usec
DE 6.50 usec
TE -927.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 300.2318540 MHz
NUC1 1H
P1 12.71 usec
SI 32768
SF 300.2300091 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.00 1.07 1.04 1.04 0.87 2.19 1.05 2.22 3.08 1.20 4.08 0.60 4.93 8.97 4.66

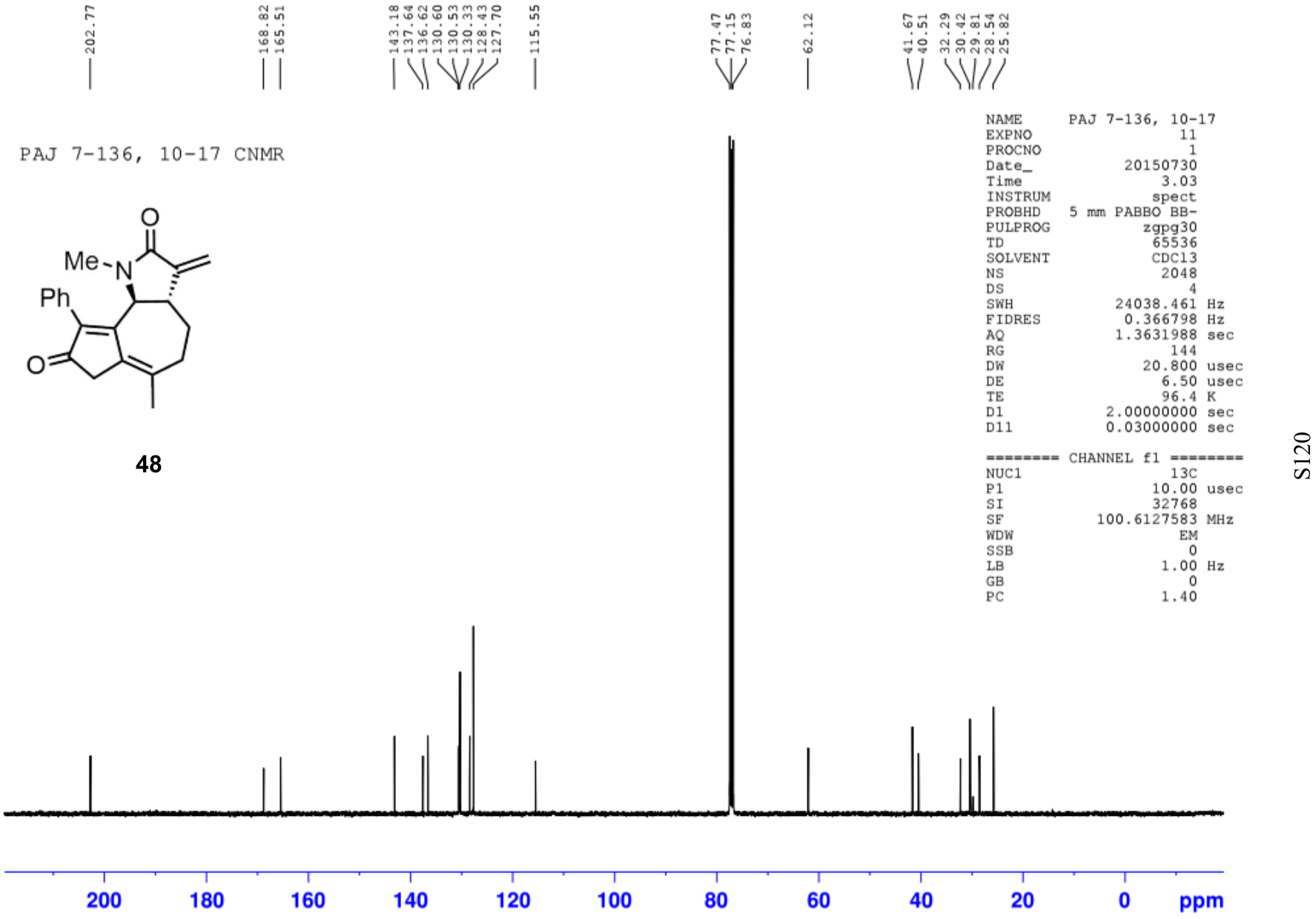

208.24
179.92
169.34
143.11
137.93
134.11
133.85
115.73
77.46
77.14
76.82
56.83
44.20
41.43
31.92
27.09
25.36
19.52
19.42
13.01
PAJ 7-78, 23-43 CNMR
O
HN
TIPS
O
NAME PAJ 7-78, 23-43
EXPNO 11
PROCNO 1
Date_ 20150516
Time 2.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 3000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 144
DW 20.800 usec
DE 6.50 usec
TE 1515.8 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127570 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

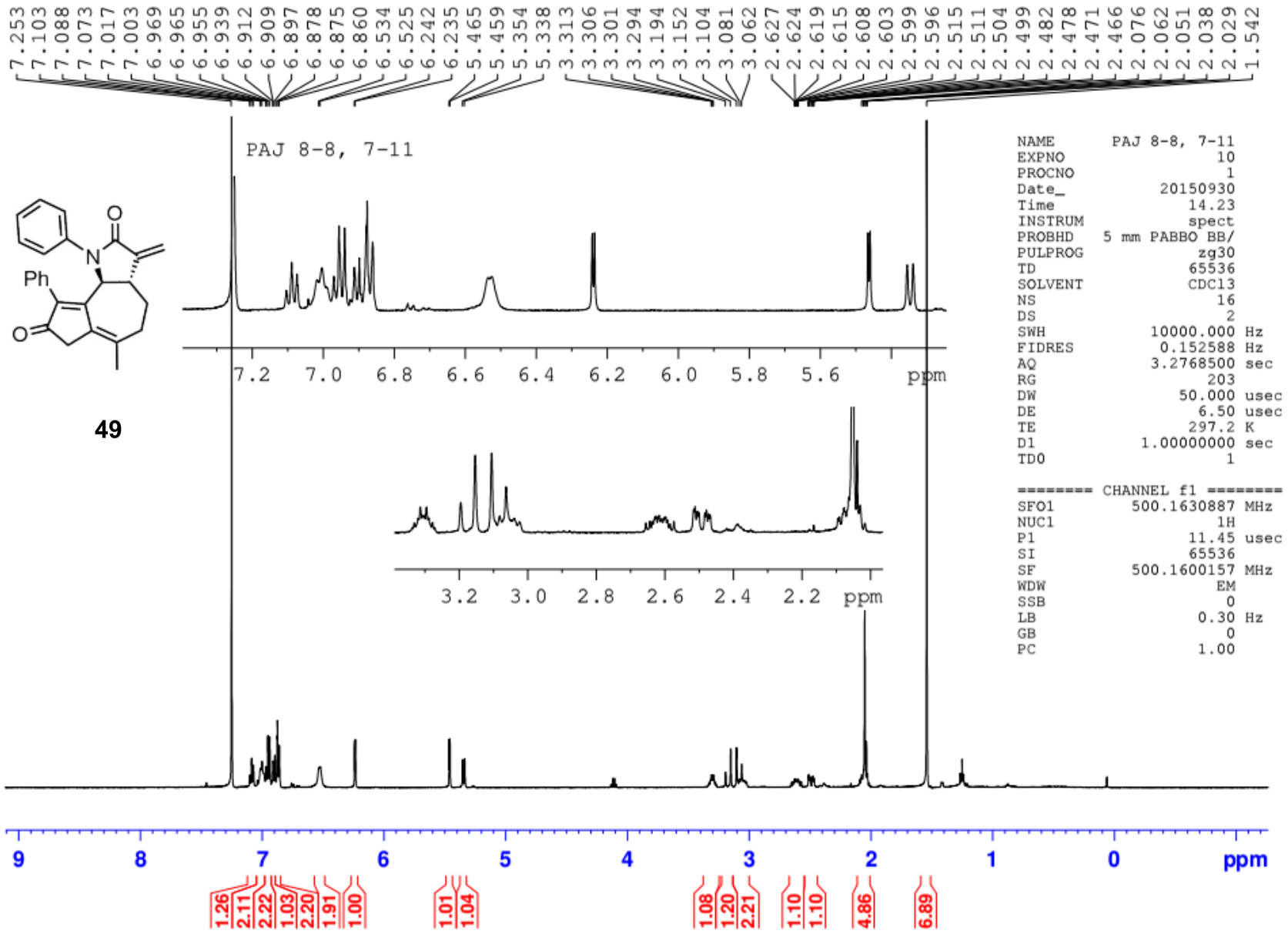
**46**


7.544
7.476
7.248
6.108
6.064
6.057
5.318
5.313
4.587
4.570
2.999
2.856
2.848
2.838
2.821
2.759
2.737
2.706
2.416
2.412
2.400
2.389
2.376
2.373
2.367
2.353
2.340
2.326
2.321
2.314
2.309
1.937
1.922
1.912
1.908
1.901
1.895
1.888
1.881
1.866
PAJ 8-60, 2
NAME PAJ 8-60, 2
EXPNO 10
PROCNO 1
Date_ 20151208
Time 12.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
47
ppm
1.00
1.00
0.95
0.99
0.94
1.91
1.00
1.04
2.04
2.91
0.96

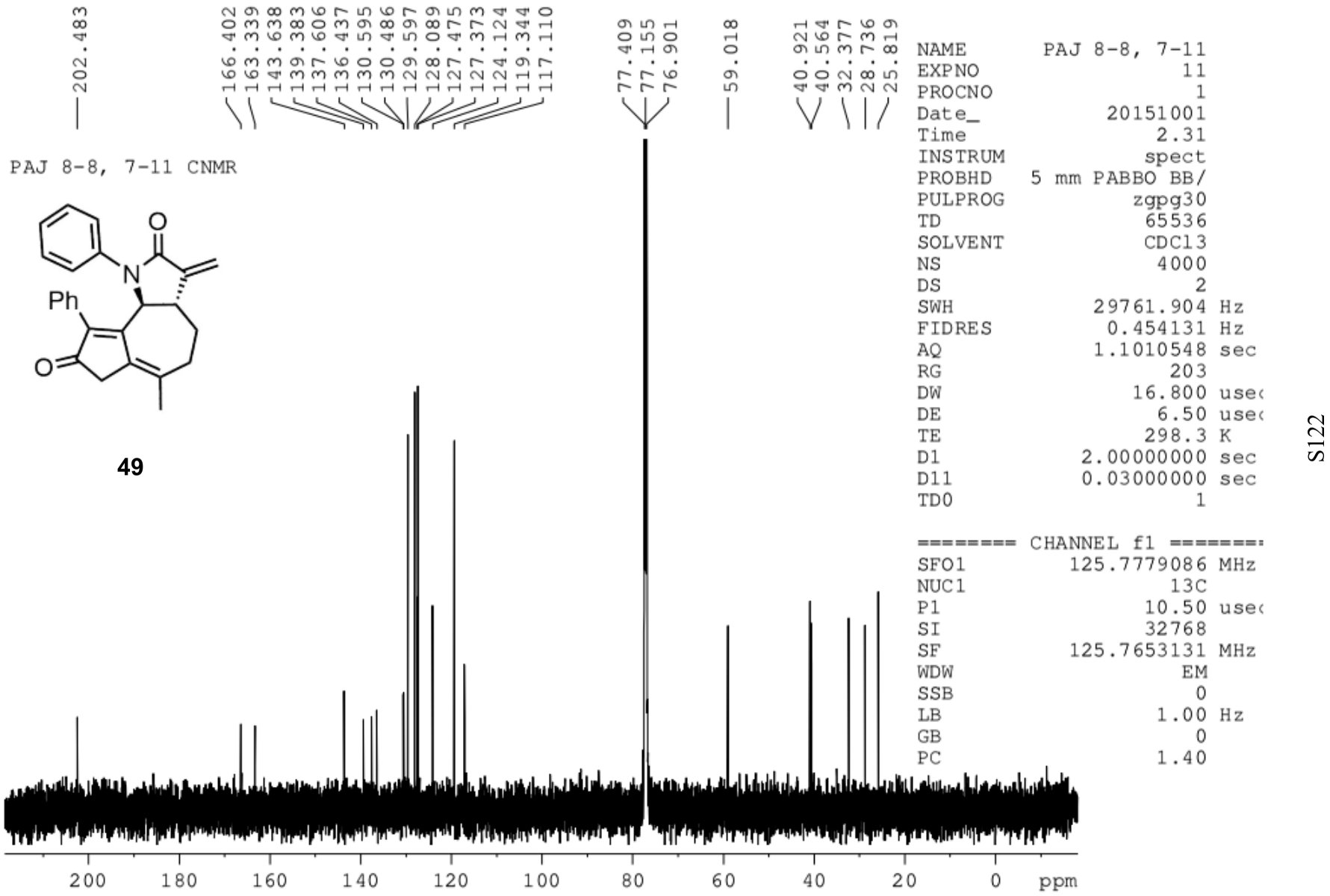

203.967
172.993
170.741
143.860
137.147
131.601
126.583
115.773
77.414
77.159
76.905
56.079
43.438
41.277
31.959
26.832
24.862
PAJ 8-60 CNMR
O
HN
H
O
47
NAME PAJ 8-60
EXPNO 11
PROCNO 1
Date_ 20151207
Time 12.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 use
DE 6.50 use
TE 299.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 use
SI 32768
SF 125.7653144 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


7.327
7.318
7.314
7.263
7.106
7.096
6.105
6.096
5.342
5.335
4.536
4.516
3.197
3.144
3.124
3.107
3.071
2.927
2.884
2.862
2.841
2.567
2.552
2.542
2.529
2.519
2.510
2.495
2.490
2.481
2.465
2.430
2.419
2.389
2.379
2.008
1.990
1.955
1.951
1.933
1.922
1.915
1.899
1.881
1.709
1.247
PAJ 7-136, 10-17
6.0
5.5
5.0
ppm
3.2
3.0
2.8
2.6
ppm
Me
N
Ph
O
O
48
2.0
1.8
1.6
1.4
ppm
NAME PAJ 7-136, 10-17
EXPNO 10
PROCNO 1
Date_ 20150729
Time 17.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 83.2 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
3.00
1.94
1.00
1.03
0.98
3.00
1.01
1.03
1.01
2.78
2.96
1.18
0.65
0.68

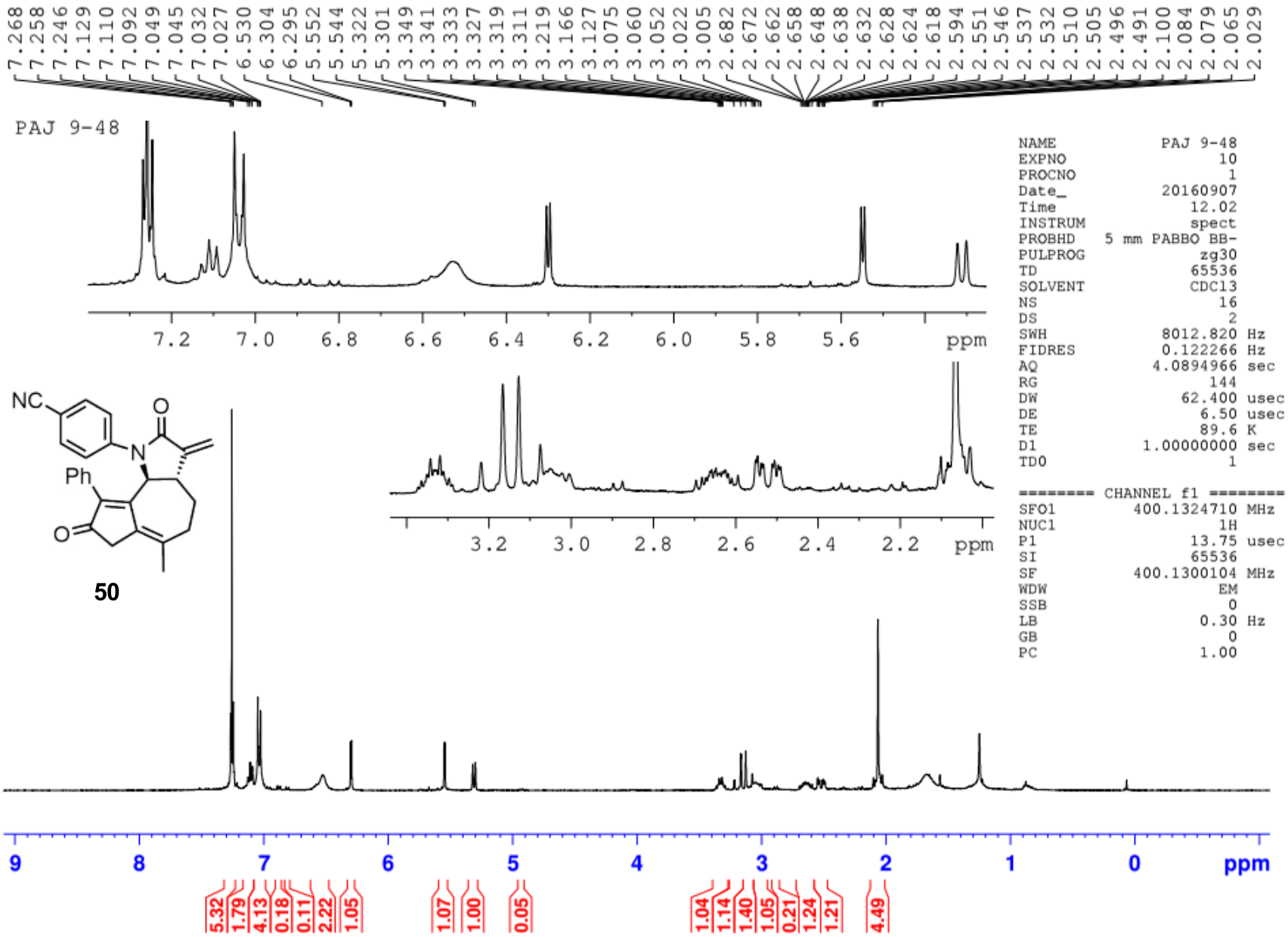

202.77
168.82
165.51
143.18
137.64
136.62
130.60
130.53
130.33
128.43
127.70
115.55
77.47
77.15
76.83
62.12
41.67
40.51
32.29
30.42
29.81
28.54
25.82
PAJ 7-136, 10-17 CNMR
Me
N
Ph
O
O
48
NAME PAJ 7-136, 10-17
EXPNO 11
PROCNO 1
Date_ 20150730
Time 3.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 144
DW 20.800 usec
DE 6.50 usec
TE 96.4 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127583 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

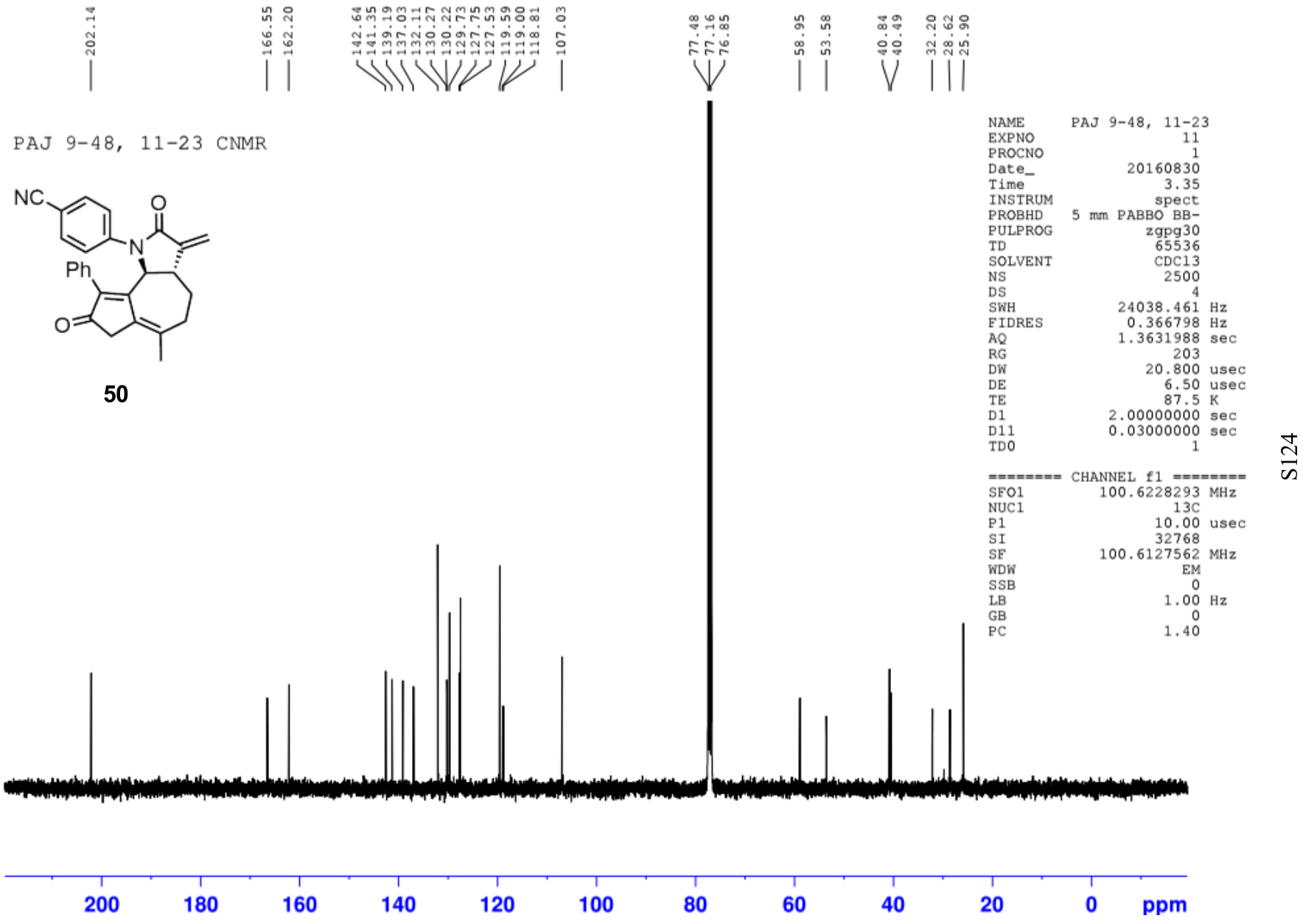

7.253 7.103 7.088 7.073 7.017 7.003 6.969 6.965 6.955 6.939 6.912 6.909 6.897 6.878 6.875 6.860 6.534 6.525 6.242 6.235 5.465 5.459 5.354 5.338 3.313 3.306 3.301 3.294 3.194 3.152 3.104 3.081 3.062 2.627 2.624 2.619 2.615 2.608 2.603 2.599 2.596 2.515 2.511 2.504 2.499 2.482 2.478 2.471 2.466 2.076 2.062 2.051 2.038 2.029 1.542
PAJ 8-8, 7-11
Ph
O
N
O
7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 ppm
3.2 3.0 2.8 2.6 2.4 2.2 ppm
NAME PAJ 8-8, 7-11
EXPNO 10
PROCNO 1
Date_ 20150930
Time 14.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600157 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.26 2.11 2.22 1.03 2.20 1.91 1.00 1.01 1.04 1.08 1.20 2.21 1.10 1.10 4.86 6.89

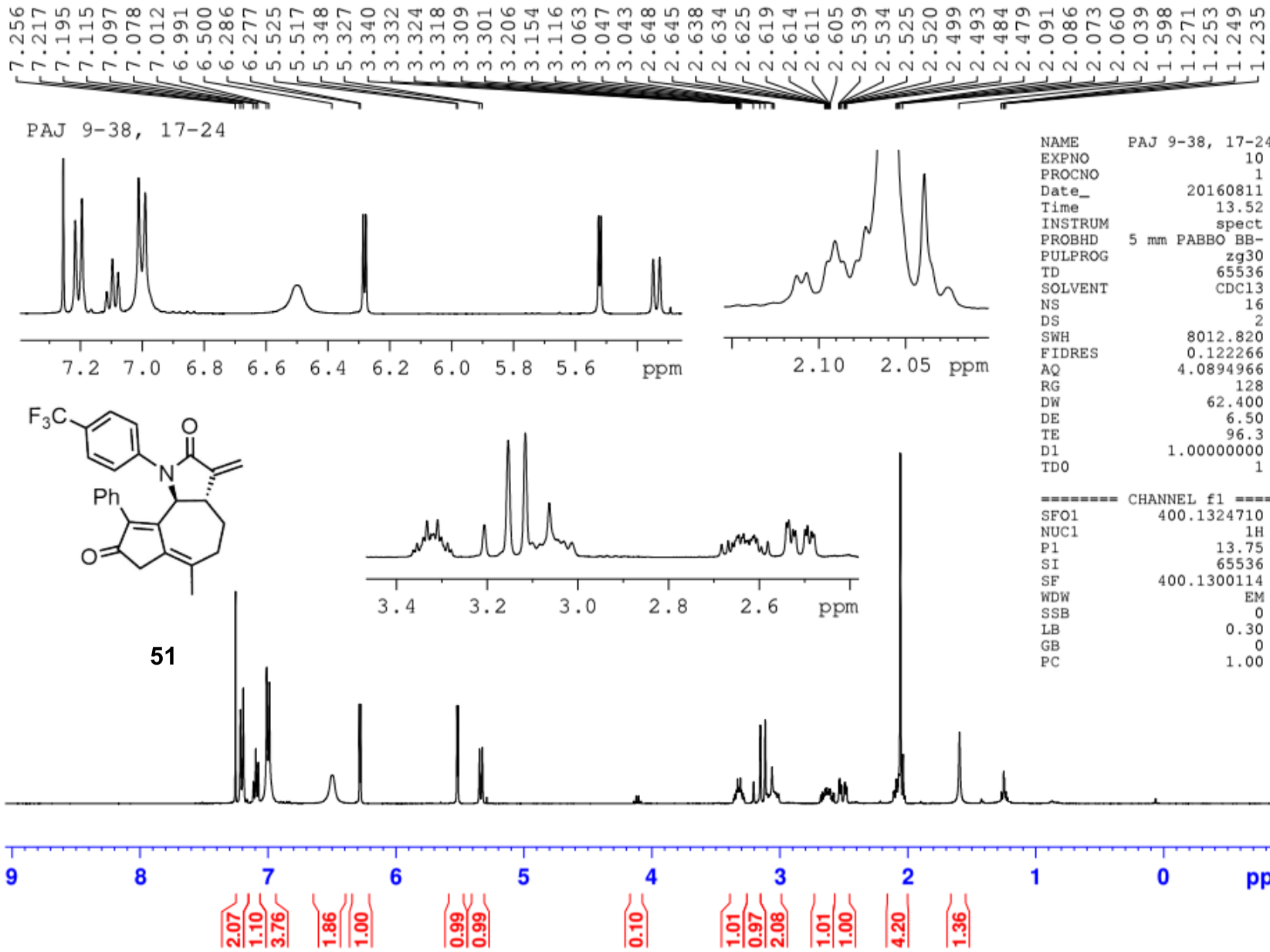
**49**

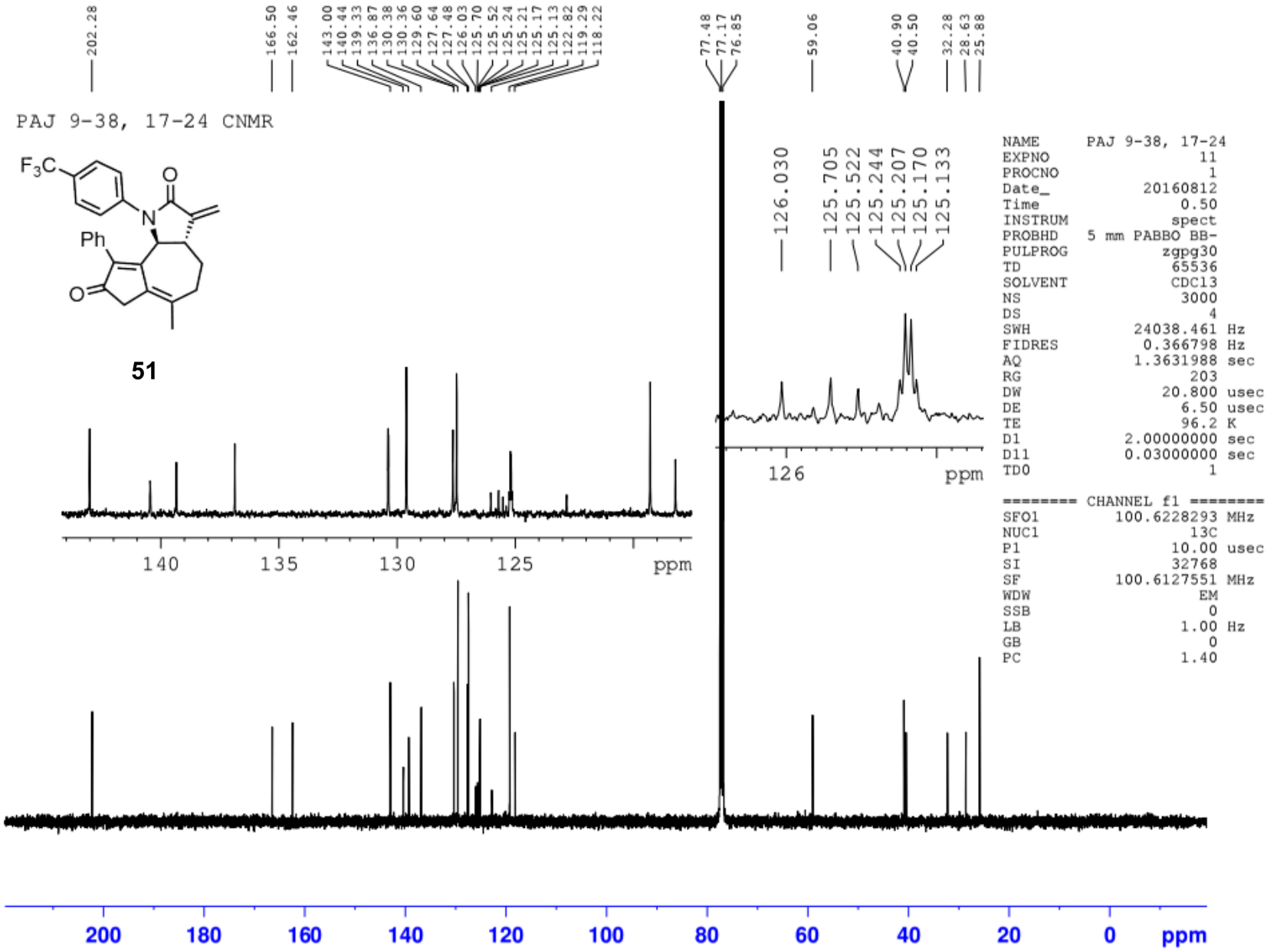

202.483
166.402
163.339
143.638
139.383
137.606
136.437
130.595
130.486
129.597
128.089
127.475
127.373
124.124
119.344
117.110
77.409
77.155
76.901
59.018
40.921
40.564
32.377
28.736
25.819
PAJ 8-8, 7-11 CNMR
Ph
O
N
O
NAME PAJ 8-8, 7-11
EXPNO 11
PROCNO 1
Date_ 20151001
Time 2.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4000
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 use
DE 6.50 use
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 use
SI 32768
SF 125.7653131 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

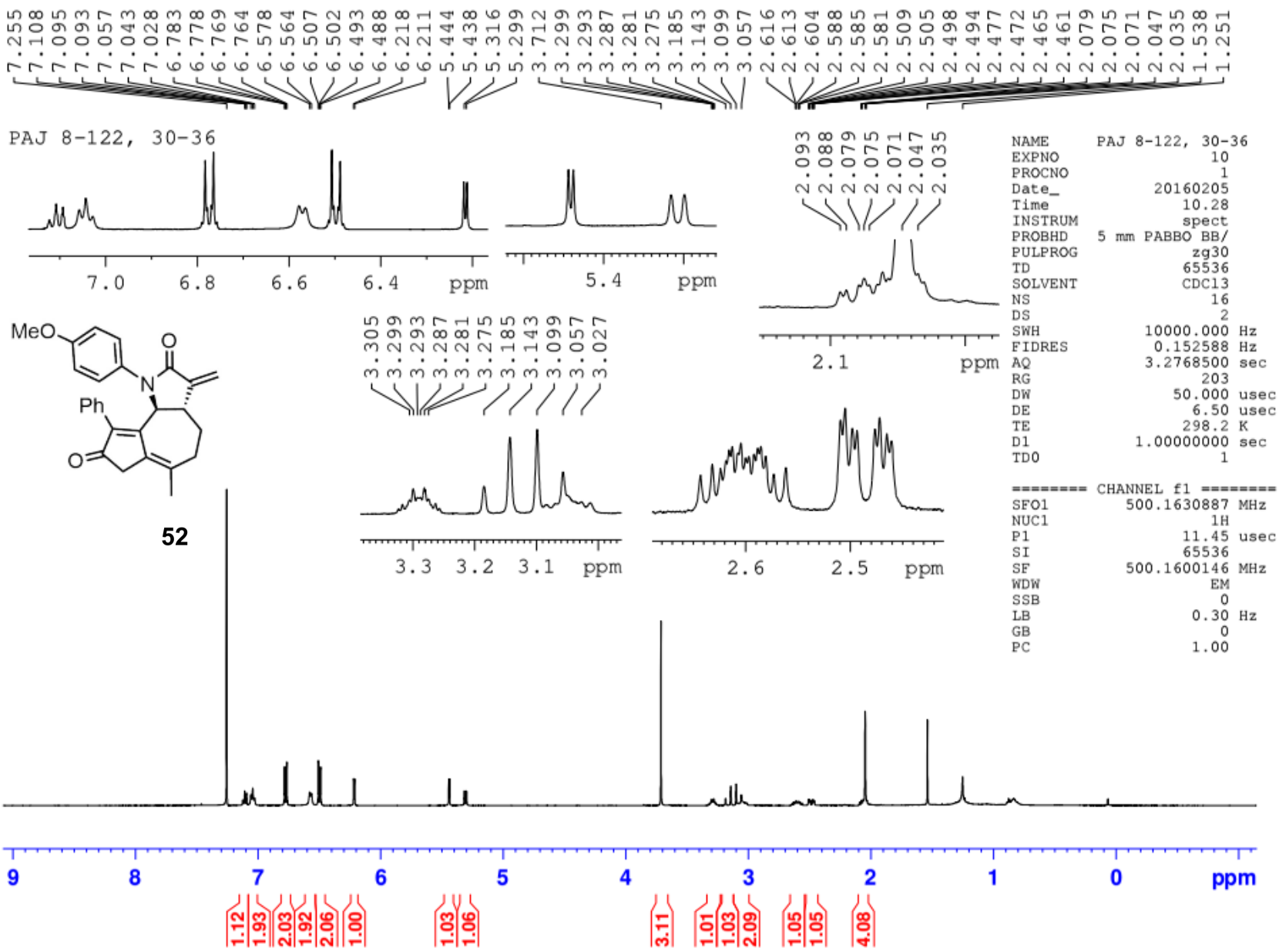
**49**

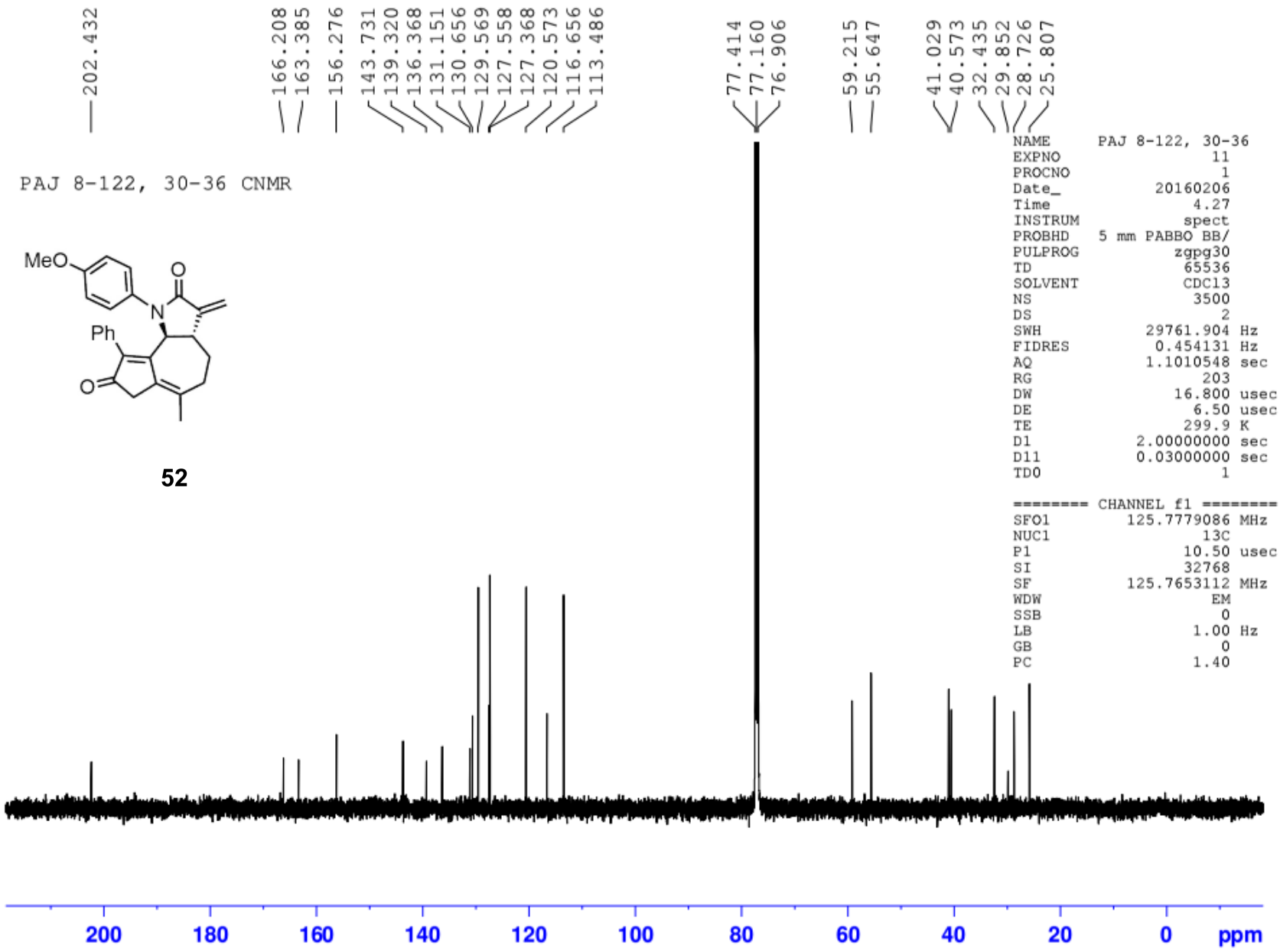

7.268 7.258 7.246 7.129 7.110 7.092 7.049 7.045 7.032 7.027 6.530 6.304 6.295 5.552 5.544 5.322 5.301 3.349 3.341 3.333 3.327 3.319 3.311 3.219 3.166 3.127 3.075 3.060 3.052 3.022 3.005 2.682 2.672 2.662 2.658 2.648 2.638 2.632 2.628 2.624 2.618 2.594 2.551 2.546 2.537 2.532 2.510 2.505 2.496 2.491 2.100 2.084 2.079 2.065 2.029
PAJ 9-48
7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 ppm
3.2 3.0 2.8 2.6 2.4 2.2 ppm
NC
O
N
Ph
O
50
NAME PAJ 9-48
EXPNO 10
PROCNO 1
Date_ 20160907
Time 12.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 144
DW 62.400 usec
DE 6.50 usec
TE 89.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300104 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
5.32 1.79 4.13 0.18 0.11 2.22 1.05 1.07 1.00 0.05 1.04 1.14 1.40 1.05 0.21 1.24 1.21 4.49

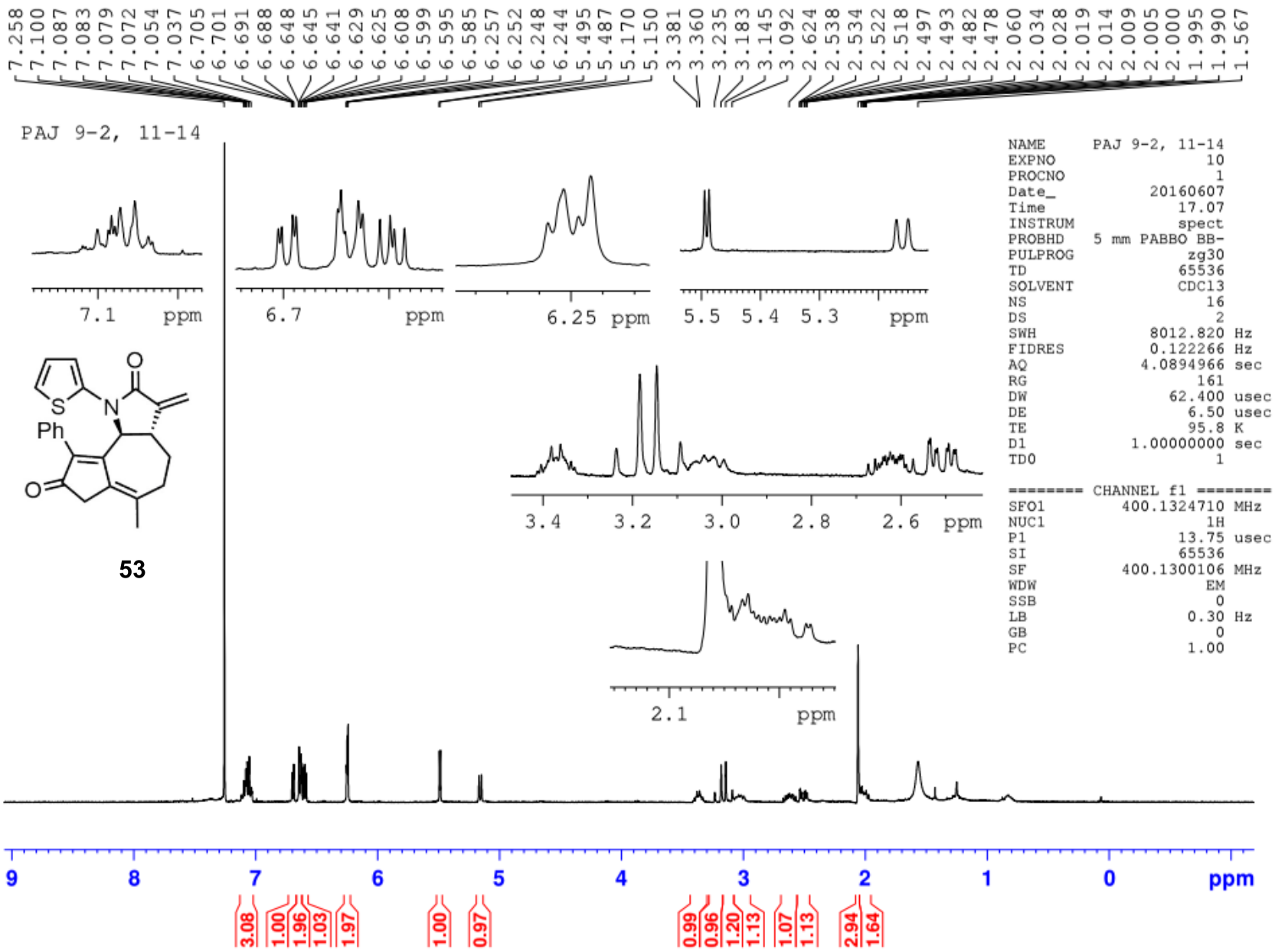

202.14
166.55
162.20
142.64
141.35
139.19
137.03
132.11
130.27
130.22
129.73
127.75
127.53
119.59
119.00
118.81
107.03
77.48
77.16
76.85
58.95
53.58
40.84
40.49
32.20
28.62
25.90
PAJ 9-48, 11-23 CNMR
NC
O
N
Ph
O
50
NAME PAJ 9-48, 11-23
EXPNO 11
PROCNO 1
Date_ 20160830
Time 3.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2500
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 87.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127562 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

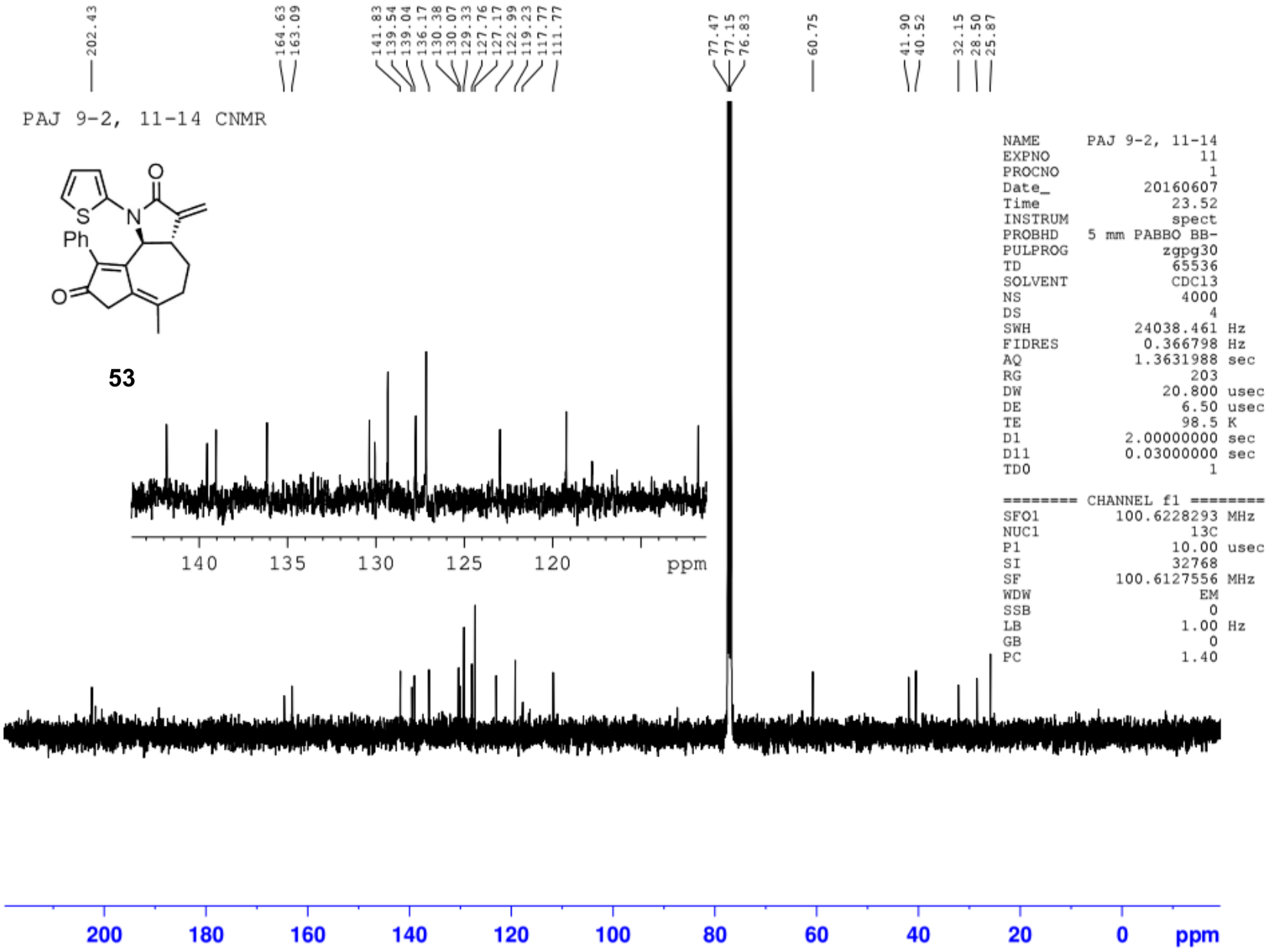

7.256 7.217 7.195 7.115 7.097 7.078 7.012 6.991 6.500 6.286 6.277 5.525 5.517 5.348 5.327 3.340 3.332 3.324 3.318 3.309 3.301 3.206 3.154 3.116 3.063 3.047 3.043 2.648 2.645 2.638 2.634 2.625 2.619 2.614 2.611 2.605 2.539 2.534 2.525 2.520 2.499 2.493 2.484 2.479 2.091 2.086 2.073 2.060 2.039 1.598 1.271 1.253 1.249 1.235
PAJ 9-38, 17-24
7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 ppm
2.10 2.05 ppm
3.4 3.2 3.0 2.8 2.6 ppm
F3C
O
N
Ph
O
51
NAME PAJ 9-38, 17-24
EXPNO 10
PROCNO 1
Date_ 20160811
Time 13.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 128
DW 62.400 usec
DE 6.50 usec
TE 96.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300114 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
2.07 1.10 3.76 1.86 1.00 0.99 0.99 0.10 1.01 0.97 2.08 1.01 1.00 4.20 1.36

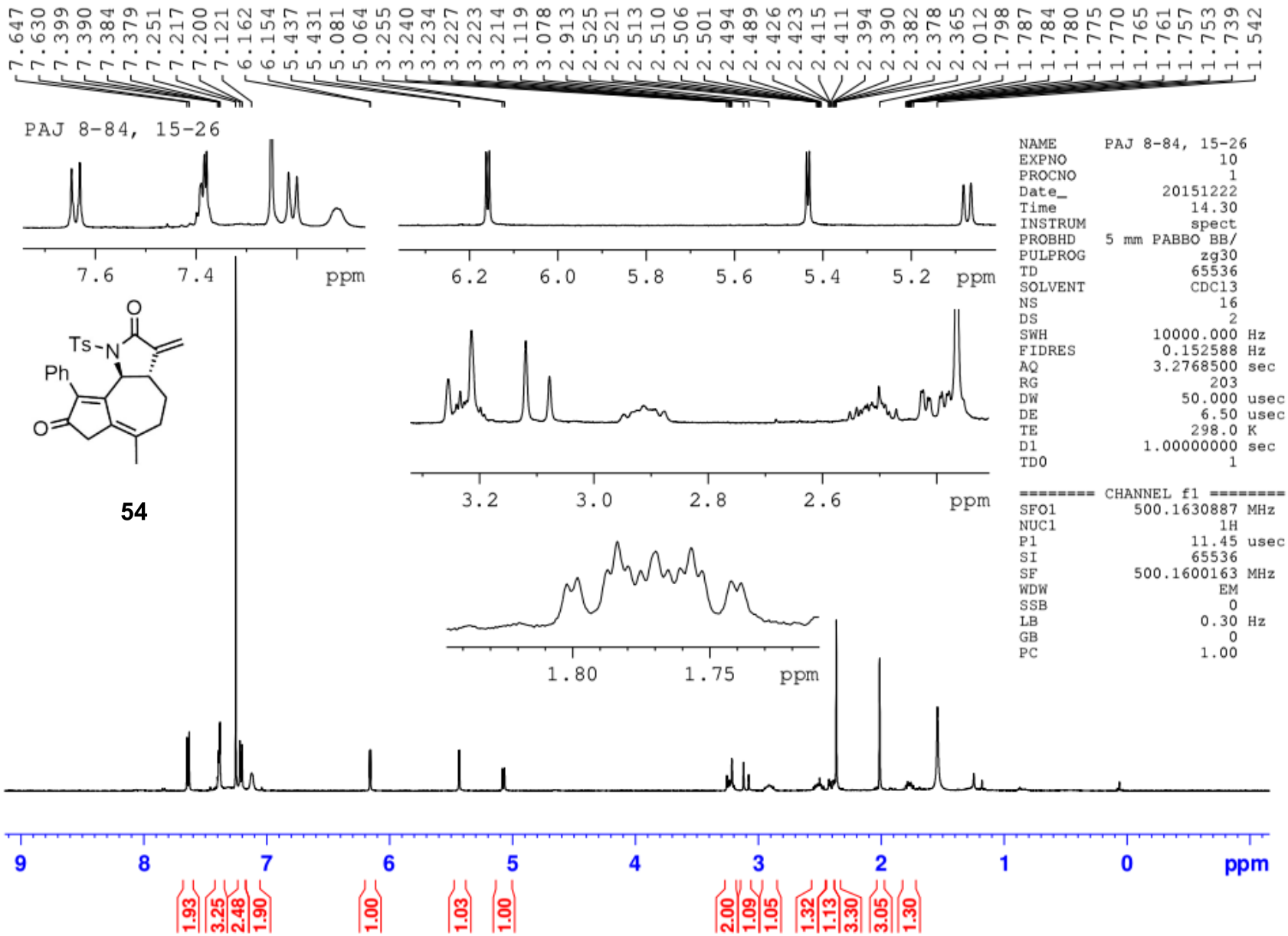

PAJ 9-38, 17-24 CNMR
202.28
166.50
162.46
143.00
140.44
139.33
136.87
130.38
130.36
129.60
127.64
127.48
126.03
125.70
125.52
125.24
125.21
125.17
125.13
122.82
119.29
118.22
77.48
77.17
76.85
59.06
40.90
40.50
32.28
28.63
25.88
126.030
125.705
125.522
125.244
125.207
125.170
125.133
F3C
O
N
Ph
O
51
140
135
130
125
ppm
126
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm
NAME PAJ 9-38, 17-24
EXPNO 11
PROCNO 1
Date_ 20160812
Time 0.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 96.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127551 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

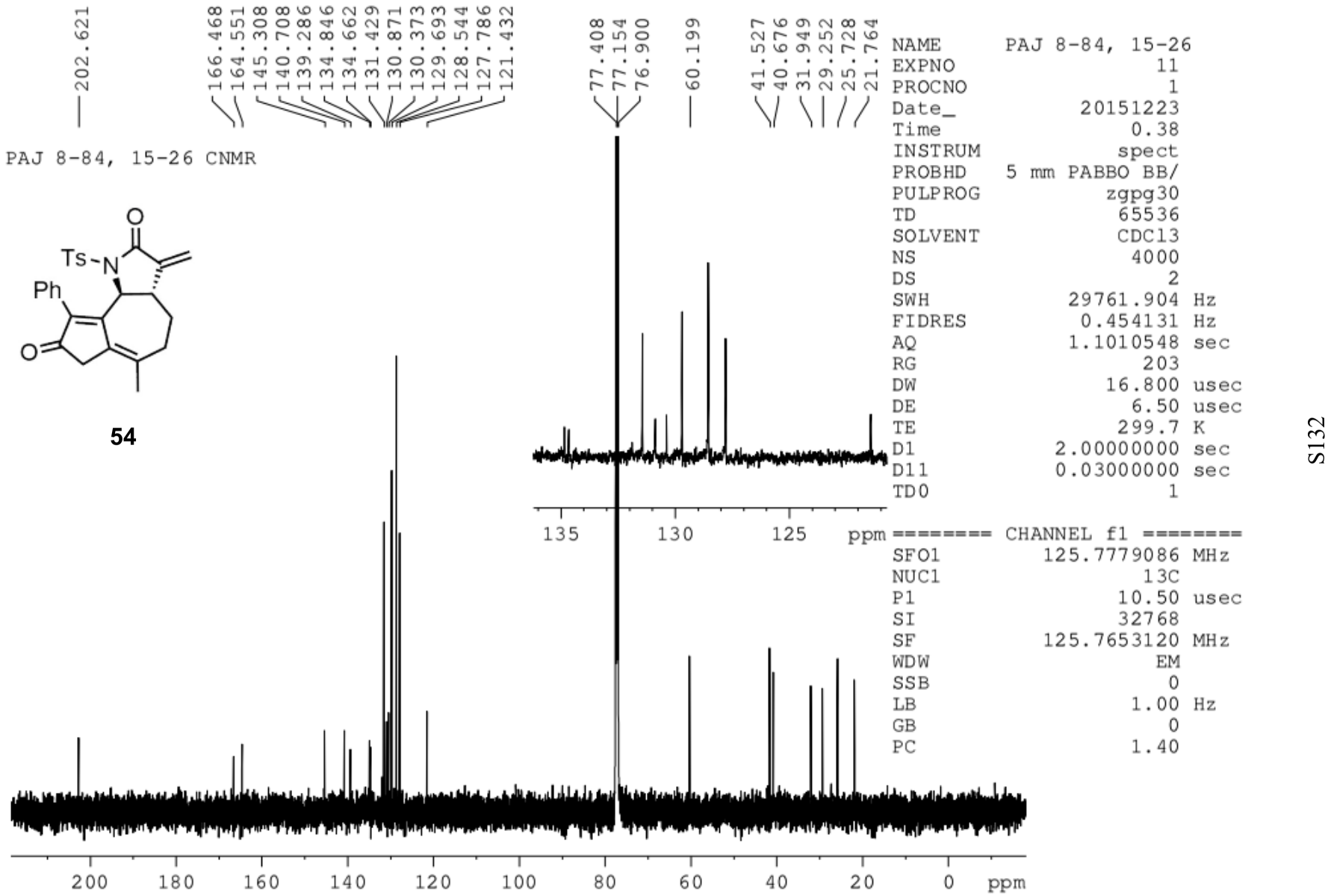

7.255 7.108 7.095 7.093 7.057 7.043 7.028 6.783 6.778 6.769 6.764 6.578 6.564 6.507 6.502 6.493 6.488 6.218 6.211 5.444 5.438 5.316 5.299 3.712 3.299 3.293 3.287 3.281 3.275 3.185 3.143 3.099 3.057 2.616 2.613 2.604 2.588 2.585 2.581 2.509 2.505 2.498 2.494 2.477 2.472 2.465 2.461 2.079 2.075 2.071 2.047 2.035 1.538 1.251
PAJ 8-122, 30-36
7.0 6.8 6.6 6.4 ppm
5.4 ppm
2.093 2.088 2.079 2.075 2.071 2.047 2.035
2.1 ppm
MeO
O
N
Ph
O
52
3.305 3.299 3.293 3.287 3.281 3.275 3.185 3.143 3.099 3.057 3.027
3.3 3.2 3.1 ppm
2.6 2.5 ppm
NAME PAJ 8-122, 30-36
EXPNO 10
PROCNO 1
Date_ 20160205
Time 10.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.12 1.93 2.03 1.92 2.06 1.00 1.03 1.06 3.11 1.01 1.03 2.09 1.05 1.05 4.08

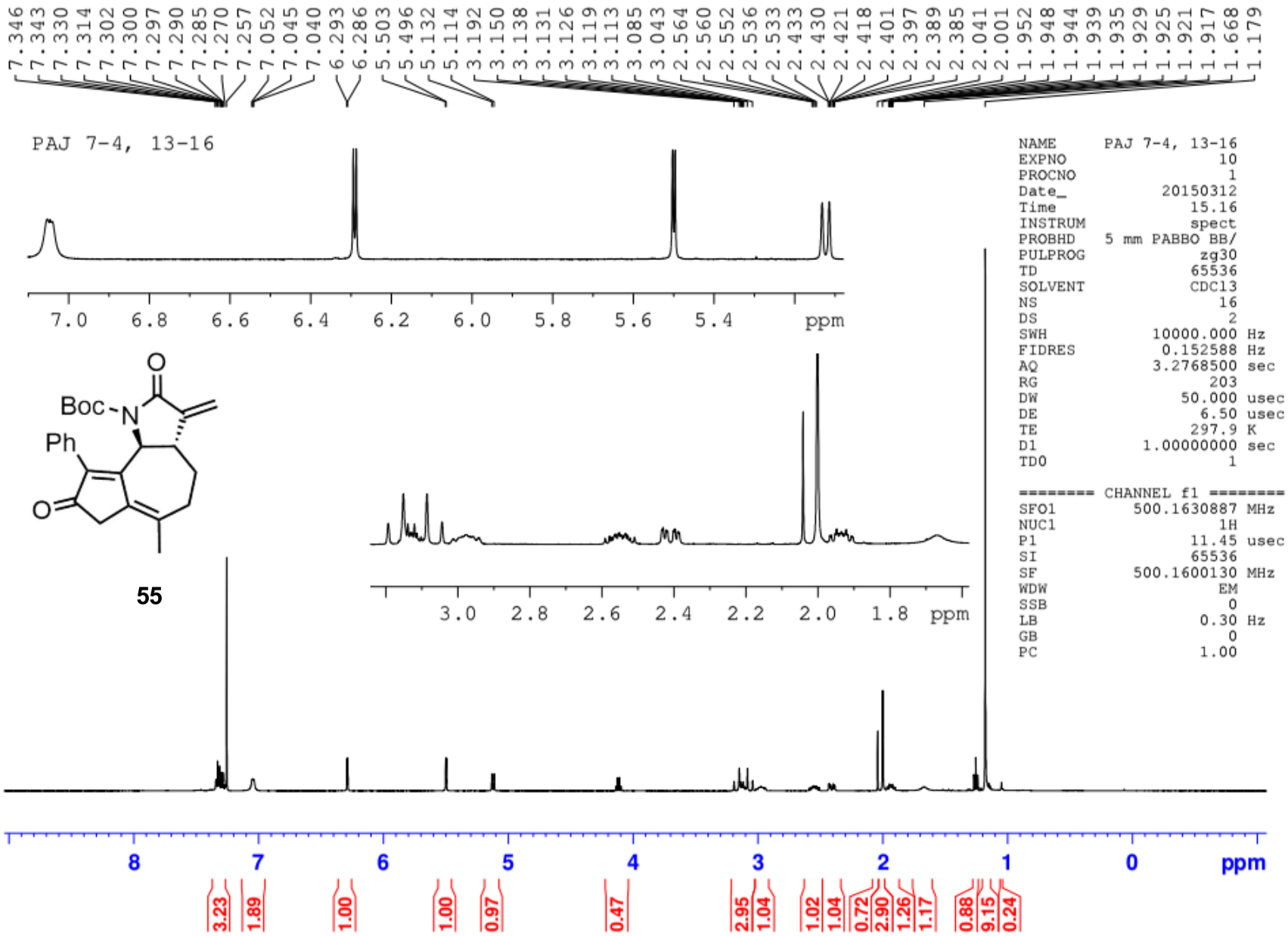

202.432
166.208
163.385
156.276
143.731
139.320
136.368
131.151
130.656
129.569
127.558
127.368
120.573
116.656
113.486
77.414
77.160
76.906
59.215
55.647
41.029
40.573
32.435
29.852
28.726
25.807
PAJ 8-122, 30-36 CNMR
MeO
O
N
Ph
O
52
NAME PAJ 8-122, 30-36
EXPNO 11
PROCNO 1
Date_ 20160206
Time 4.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 3500
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653112 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

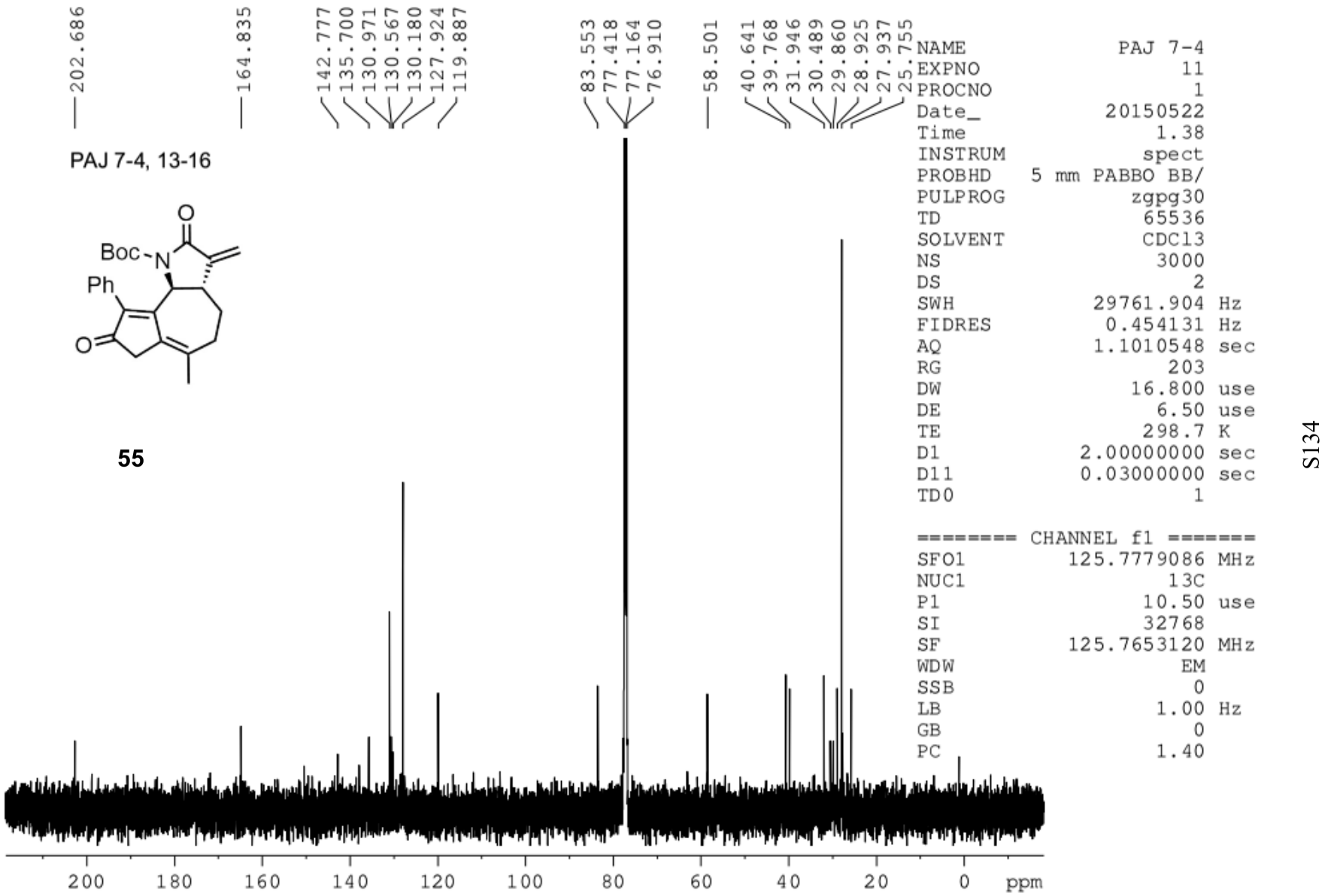

7.258 7.100 7.087 7.083 7.079 7.072 7.054 7.037 6.705 6.701 6.691 6.688 6.648 6.645 6.641 6.629 6.625 6.608 6.599 6.595 6.585 6.257 6.252 6.248 6.244 5.495 5.487 5.170 5.150 3.381 3.360 3.235 3.183 3.145 3.092 2.624 2.538 2.534 2.522 2.518 2.497 2.493 2.482 2.478 2.060 2.034 2.028 2.019 2.014 2.009 2.005 2.000 1.995 1.990 1.567
PAJ 9-2, 11-14
7.1 ppm
6.7 ppm
6.25 ppm
5.5 5.4 5.3 ppm
3.4 3.2 3.0 2.8 2.6 ppm
2.1 ppm
S
N
O
Ph
O
53
NAME PAJ 9-2, 11-14
EXPNO 10
PROCNO 1
Date_ 20160607
Time 17.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 161
DW 62.400 usec
DE 6.50 usec
TE 95.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
3.08 1.00 1.96 1.03 1.97 1.00 0.97 0.99 0.96 1.20 1.13 1.07 1.13 2.94 1.64

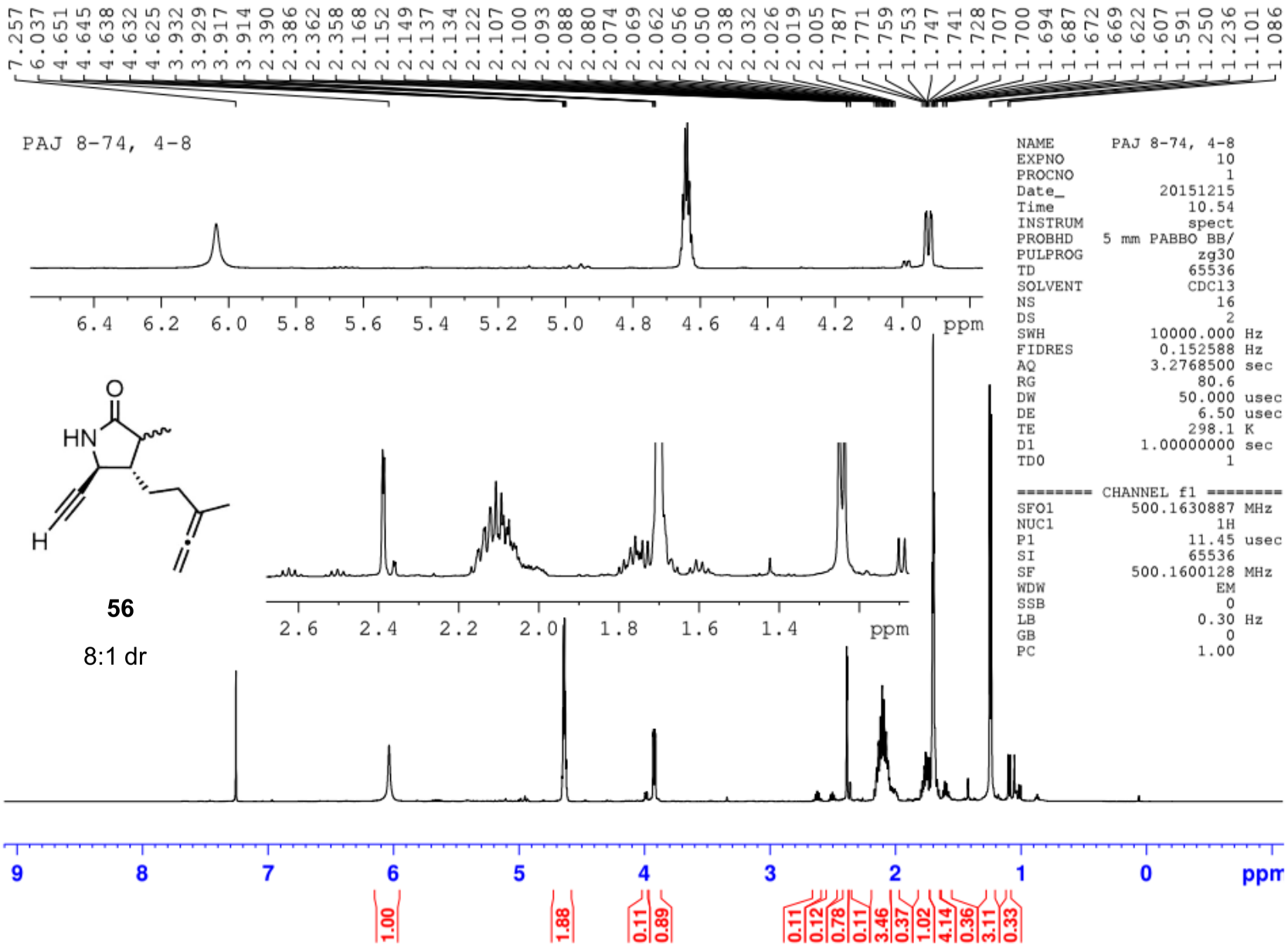

202.43
164.63
163.09
141.83
139.54
139.04
136.17
130.38
130.07
129.33
127.76
127.17
122.99
119.23
117.77
111.77
77.47
77.15
76.83
60.75
41.90
40.52
32.15
28.50
25.87
PAJ 9-2, 11-14 CNMR
O
N
S
Ph
O
53
140
135
130
125
120
ppm
NAME PAJ 9-2, 11-14
EXPNO 11
PROCNO 1
Date_ 20160607
Time 23.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 4000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 98.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127556 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


7.647 7.630 7.399 7.390 7.384 7.379 7.251 7.217 7.200 7.121 6.162 6.154 5.437 5.431 5.081 5.064 3.255 3.240 3.234 3.227 3.223 3.214 3.119 3.078 2.913 2.525 2.521 2.513 2.510 2.506 2.501 2.494 2.489 2.426 2.423 2.415 2.411 2.394 2.390 2.382 2.378 2.365 2.012 1.798 1.787 1.784 1.780 1.775 1.770 1.765 1.761 1.757 1.753 1.739 1.542
PAJ 8-84, 15-26
7.6 7.4 ppm
6.2 6.0 5.8 5.6 5.4 5.2 ppm
3.2 3.0 2.8 2.6 ppm
1.80 1.75 ppm
Ts-N
Ph
O
O
54
NAME PAJ 8-84, 15-26
EXPNO 10
PROCNO 1
Date_ 20151222
Time 14.30
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600163 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.93 3.25 2.48 1.90 1.00 1.03 1.00 2.00 1.09 1.05 1.32 1.13 3.30 3.05 1.30


202.621
166.468
164.551
145.308
140.708
139.286
134.846
134.662
131.429
130.871
130.373
129.693
128.544
127.786
121.432
77.408
77.154
76.900
60.199
41.527
40.676
31.949
29.252
25.728
21.764
PAJ 8-84, 15-26 CNMR
Ts
N
Ph
O
O
54
NAME PAJ 8-84, 15-26
EXPNO 11
PROCNO 1
Date_ 20151223
Time 0.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 4000
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653120 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
135
130
125
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm

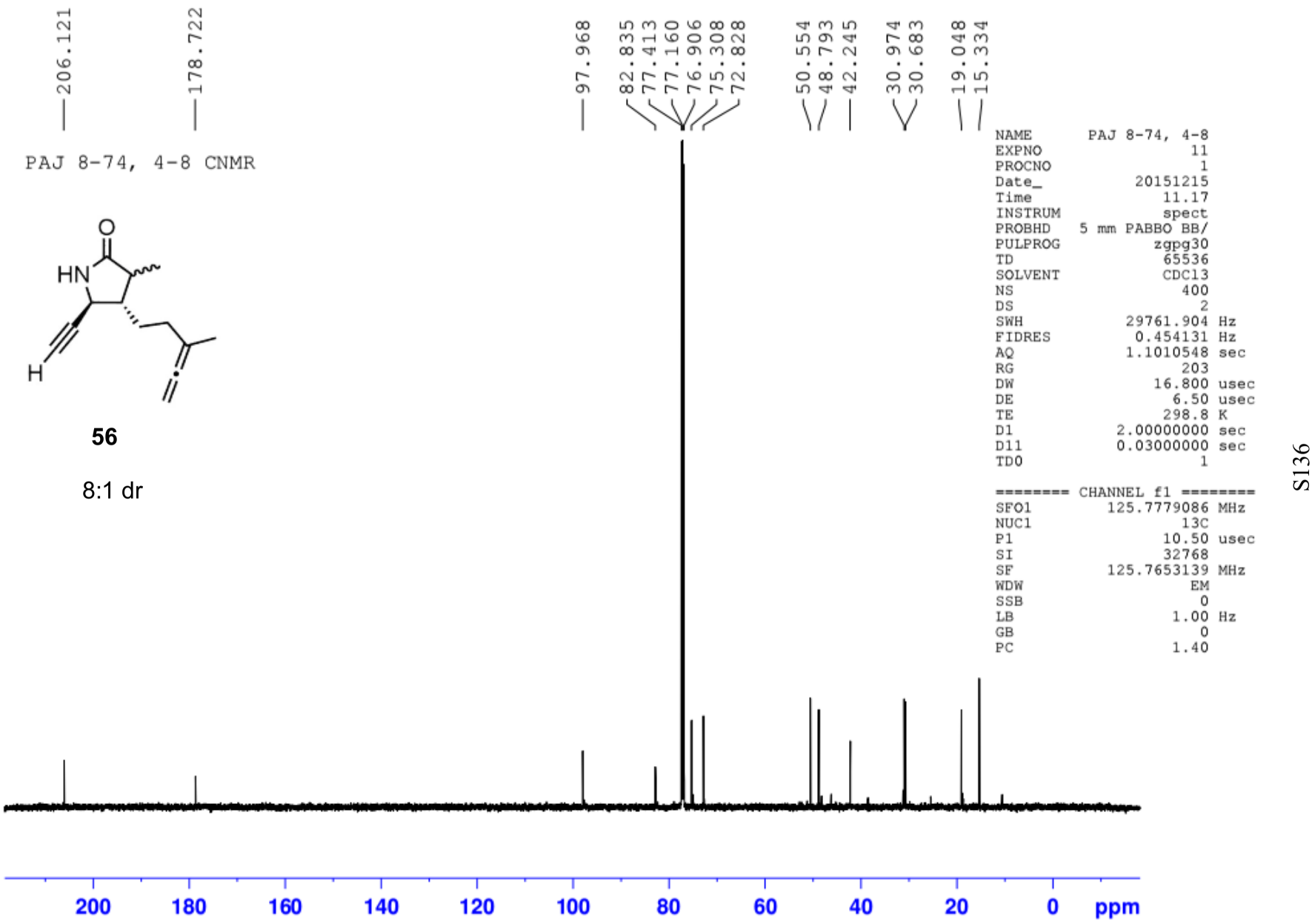

7.346
7.343
7.330
7.314
7.302
7.300
7.297
7.290
7.285
7.270
7.257
7.052
7.045
7.040
6.293
6.286
5.503
5.496
5.132
5.114
3.192
3.150
3.138
3.131
3.126
3.119
3.113
3.085
3.043
2.564
2.560
2.552
2.536
2.533
2.433
2.430
2.421
2.418
2.401
2.397
2.389
2.385
2.041
2.001
1.952
1.948
1.944
1.939
1.935
1.929
1.925
1.921
1.917
1.668
1.179
PAJ 7-4, 13-16
7.0
6.8
6.6
6.4
6.2
6.0
5.8
5.6
5.4
ppm
Boc
N
Ph
O
O
55
3.0
2.8
2.6
2.4
2.2
2.0
1.8
ppm
NAME PAJ 7-4, 13-16
EXPNO 10
PROCNO 1
Date_ 20150312
Time 15.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
0
ppm
3.23
1.89
1.00
1.00
0.97
0.47
2.95
1.04
1.02
1.04
0.72
2.90
1.26
1.17
0.88
9.15
0.24


202.686
164.835
142.777
135.700
130.971
130.567
130.180
127.924
119.887
83.553
77.418
77.164
76.910
58.501
40.641
39.768
31.946
30.489
29.860
28.925
27.937
25.755
PAJ 7-4, 13-16
Boc
N
Ph
O
O
55
NAME PAJ 7-4
EXPNO 11
PROCNO 1
Date_ 20150522
Time 1.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3000
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 use
DE 6.50 use
TE 298.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 use
SI 32768
SF 125.7653120 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


7.257 6.037 4.651 4.645 4.638 4.632 4.625 3.932 3.929 3.917 3.914 3.390 2.386 2.362 2.358 2.168 2.152 2.149 2.137 2.134 2.122 2.107 2.100 2.093 2.088 2.080 2.074 2.069 2.062 2.056 2.050 2.038 2.032 2.026 2.019 2.005 1.787 1.771 1.759 1.753 1.747 1.741 1.728 1.707 1.700 1.694 1.687 1.672 1.669 1.622 1.607 1.591 1.250 1.236 1.101 1.086
PAJ 8-74, 4-8
6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 ppm
NAME PAJ 8-74, 4-8
EXPNO 10
PROCNO 1
Date_ 20151215
Time 10.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 80.6
DW 50.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HN
O
H
2.6 2.4 2.2 2.0 1.8 1.6 1.4 ppm
9 8 7 6 5 4 3 2 1 0 ppm
1.00 1.88 0.11 0.89 0.11 0.12 0.78 0.11 3.46 0.37 1.02 4.14 0.36 3.11 0.33

**56**

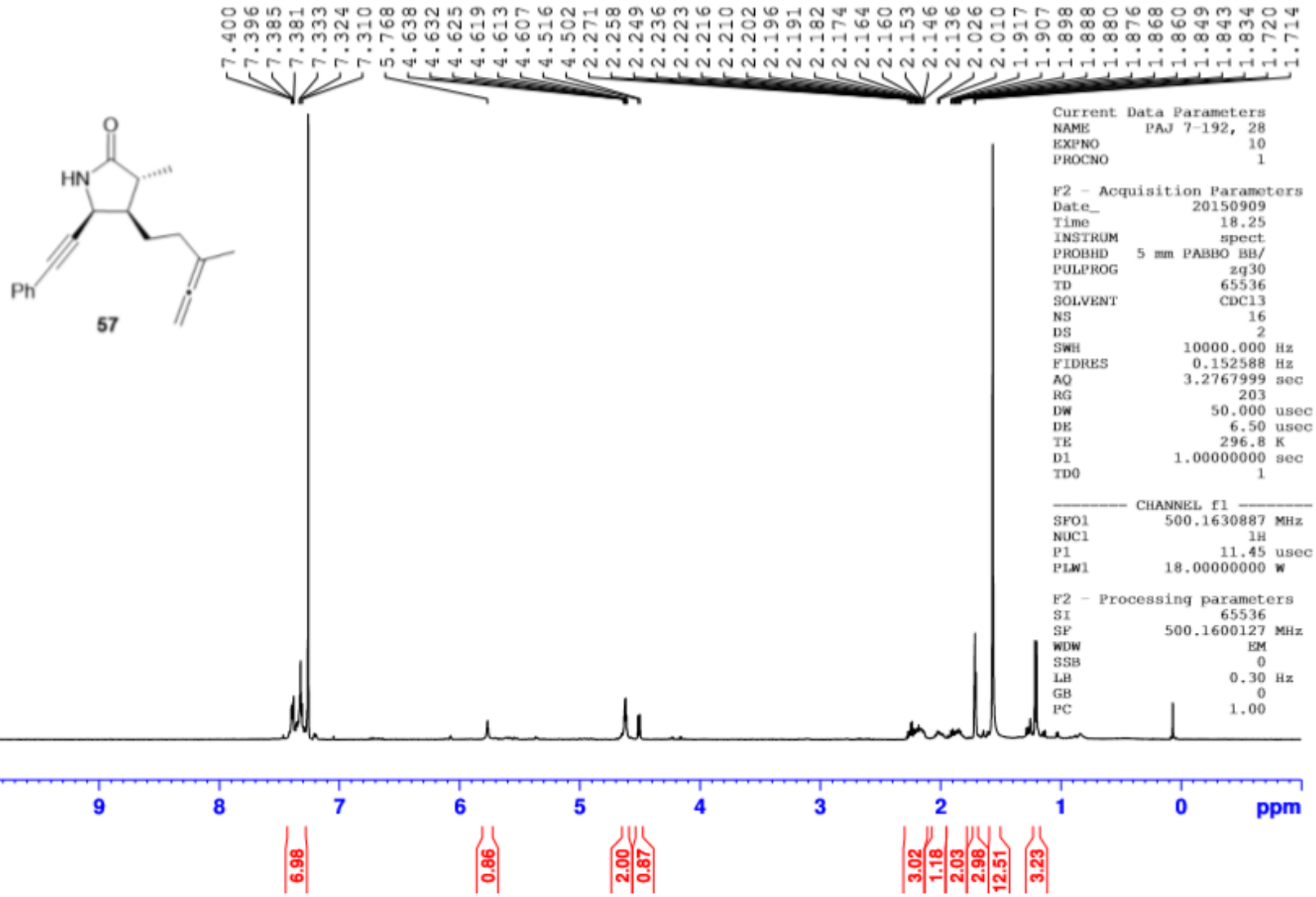
8:1 dr


206.121
178.722
97.968
82.835
77.413
77.160
76.906
75.308
72.828
50.554
48.793
42.245
30.974
30.683
19.048
15.334
PAJ 8-74, 4-8 CNMR
HN
O
H
NAME PAJ 8-74, 4-8
EXPNO 11
PROCNO 1
Date_ 20151215
Time 11.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653139 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

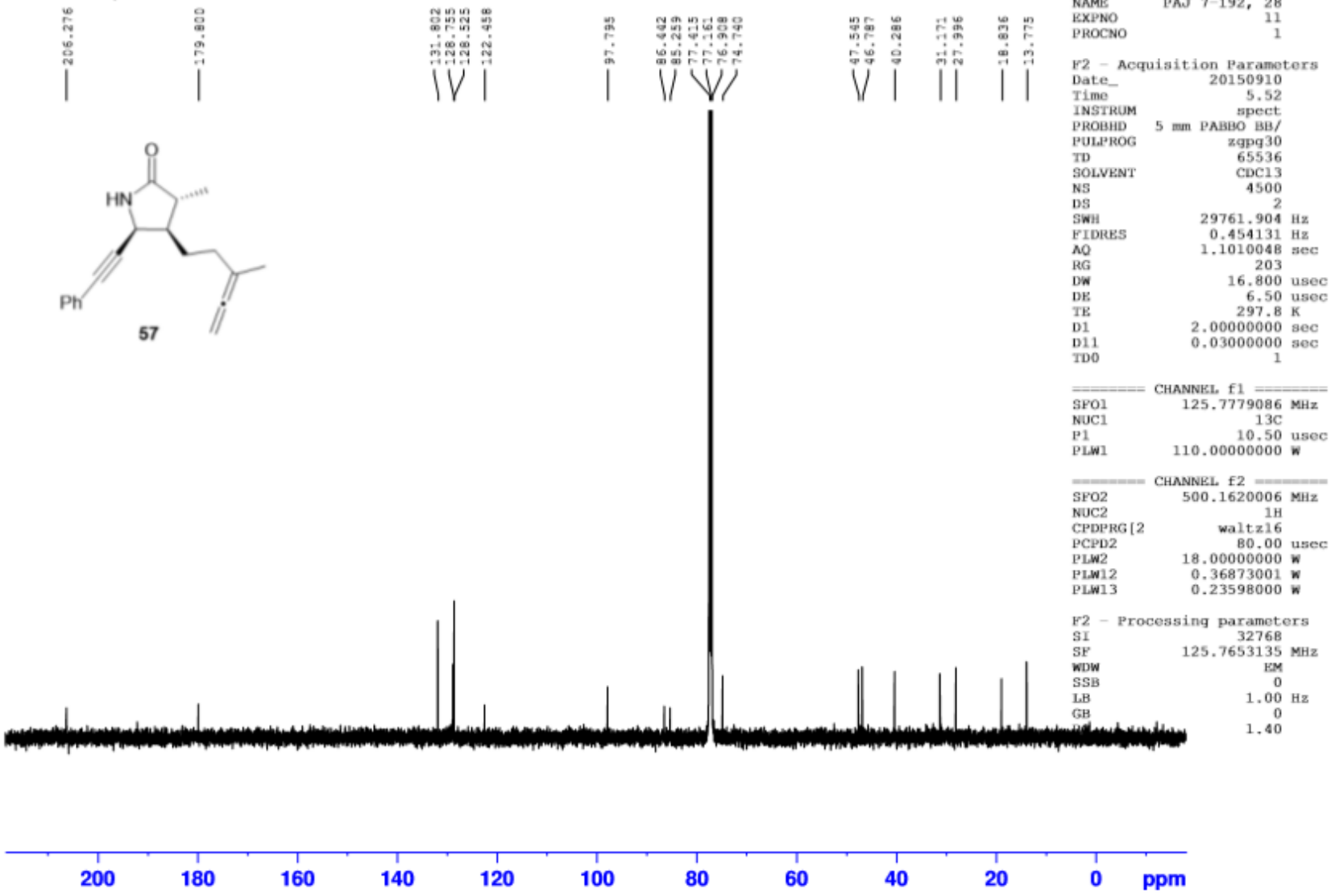
**56**

8:1 dr

PAJ 7-192, 28


7.400
7.396
7.385
7.381
7.333
7.324
7.310
5.768
4.638
4.632
4.625
4.619
4.613
4.607
4.516
4.502
2.271
2.258
2.249
2.236
2.223
2.216
2.210
2.202
2.196
2.191
2.182
2.174
2.164
2.160
2.153
2.146
2.136
2.026
2.010
1.917
1.907
1.898
1.888
1.880
1.876
1.868
1.860
1.849
1.843
1.834
1.720
1.714
O
HN
Ph
57
Current Data Parameters
NAME PAJ 7-192, 28
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150909
Time 18.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
PLW1 18.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1600127 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
6.98
0.86
2.00
0.87
3.02
1.16
2.03
2.98
12.51
3.23

PAJ 7-192, 28 CNMR




Current Data Parameters

| | |
|---|---|
| NAME | PAJ 7-192, 28 |
| EXPNO | 11 |
| PROCNO | 1 |

F2 - Acquisition Parameters

| | |
|---|---|
| Date_ | 20150910 |
| Time | 5.52 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB/ |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 4500 |
| DS | 2 |
| SWH | 29761.904 Hz |
| FIDRES | 0.454131 Hz |
| AQ | 1.1010048 sec |
| RG | 203 |
| DW | 16.800 usec |
| DE | 6.50 usec |
| TE | 297.8 K |
| D1 | 2.00000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 1 |

======== CHANNEL f1 ========

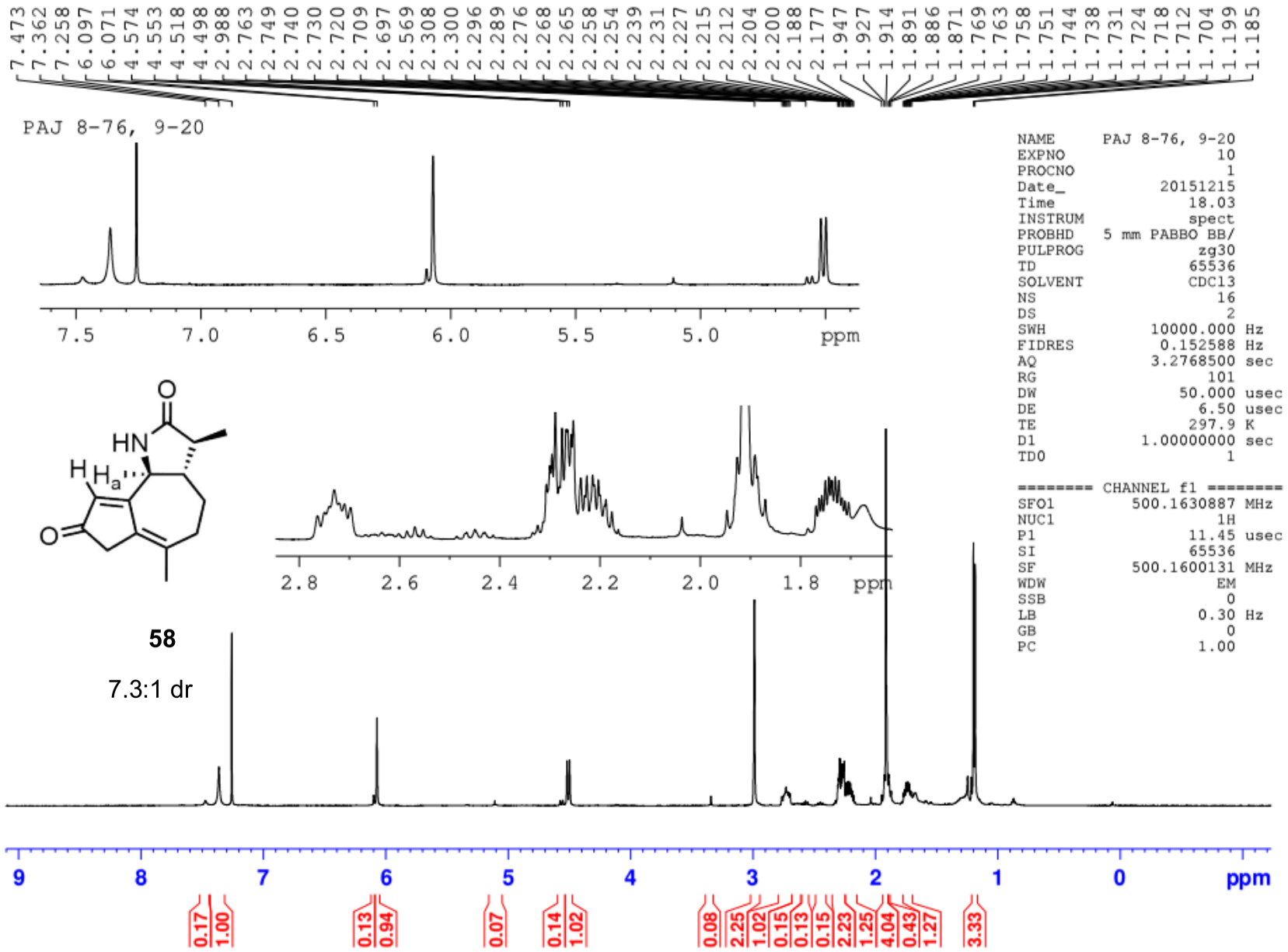
| | |
|---|---|
| SFO1 | 125.7779086 MHz |
| NUC1 | 13C |
| P1 | 10.50 usec |
| PLW1 | 110.00000000 W |

======== CHANNEL f2 ========

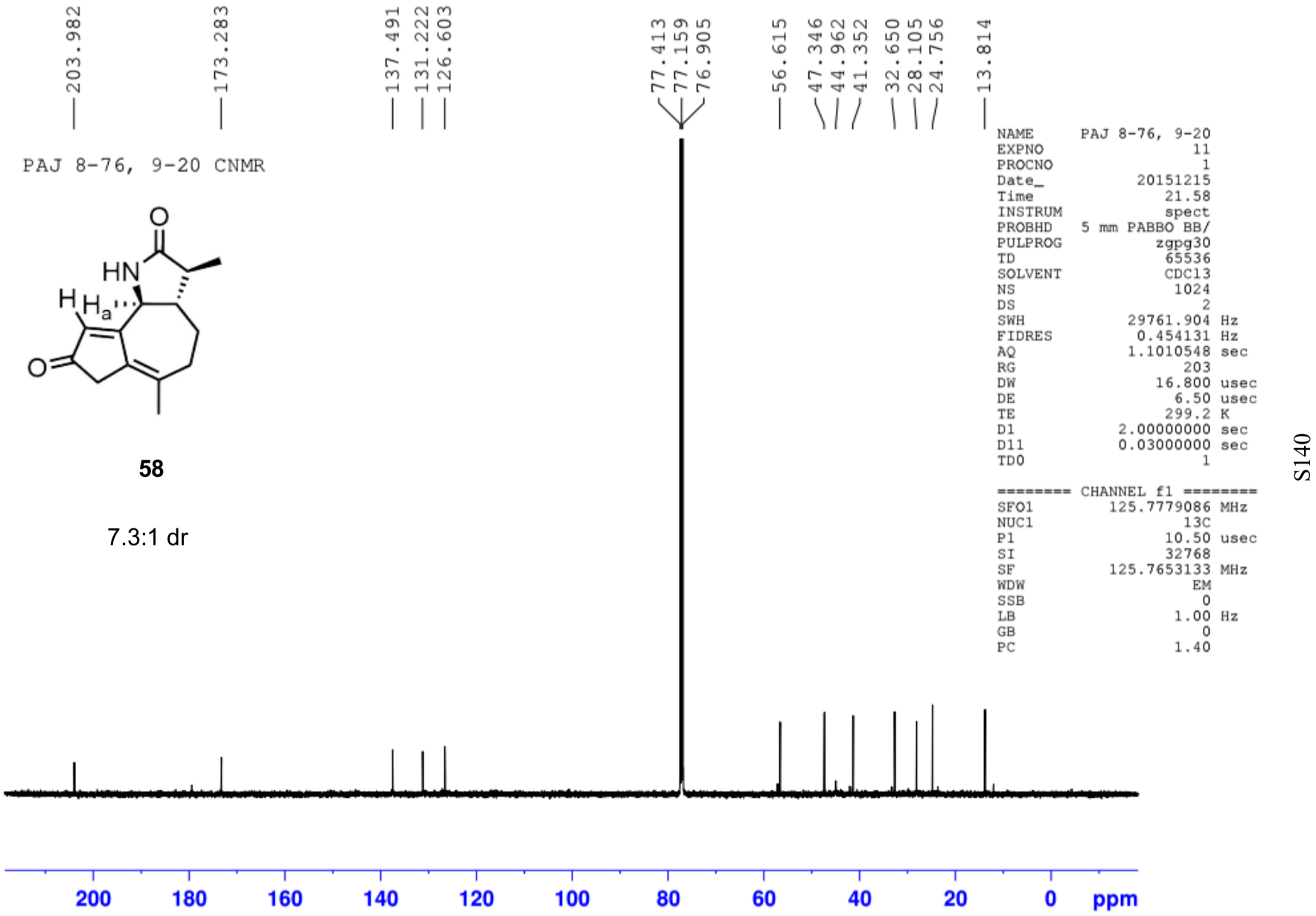
| | |
|---|---|
| SFO2 | 500.1620006 MHz |
| NUC2 | 1H |
| CPDPRG[2 | waltz16 |
| PCPD2 | 80.00 usec |
| PLW2 | 18.00000000 W |
| PLW12 | 0.36873001 W |
| PLW13 | 0.23598000 W |

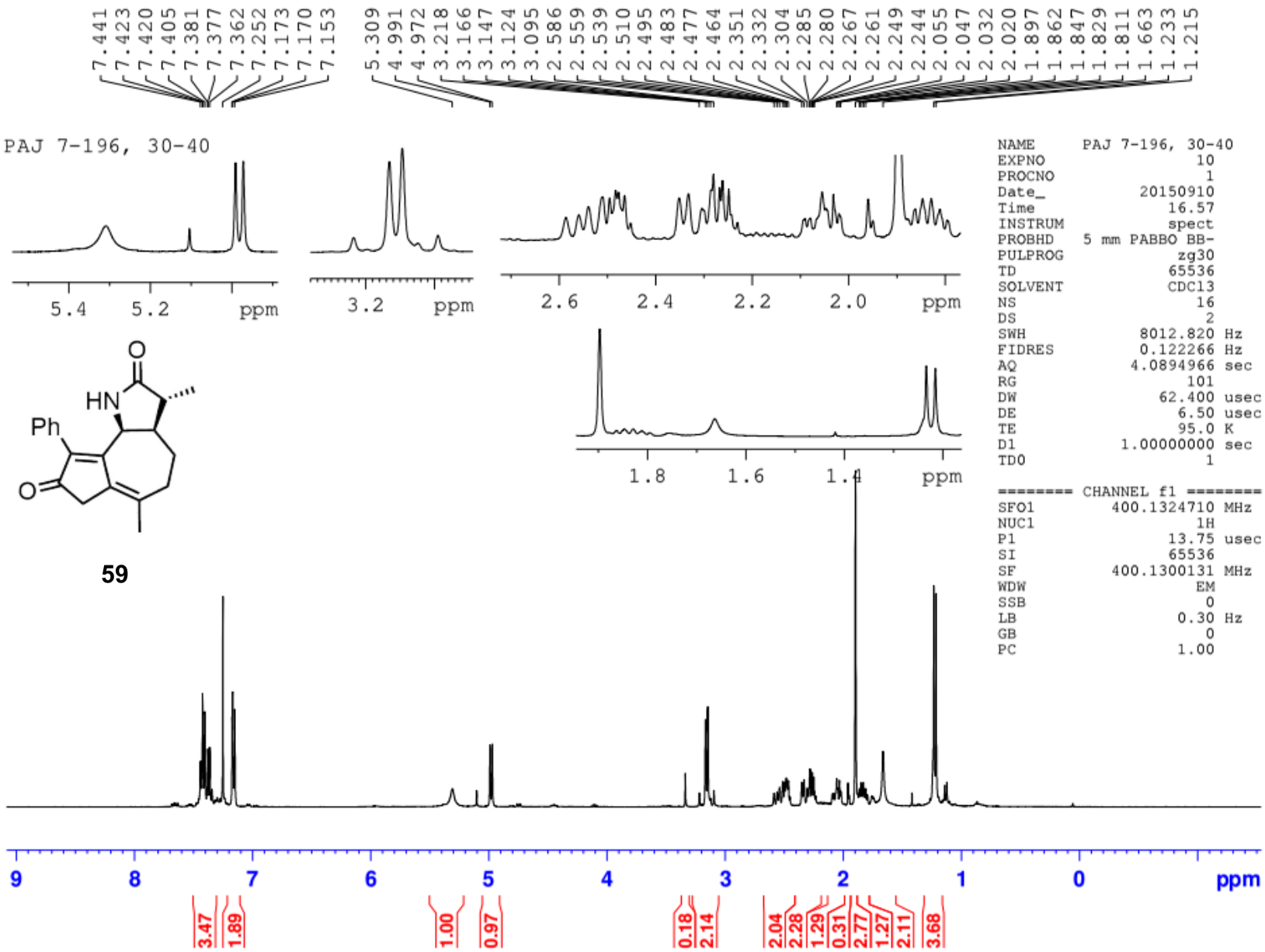
F2 - Processing parameters

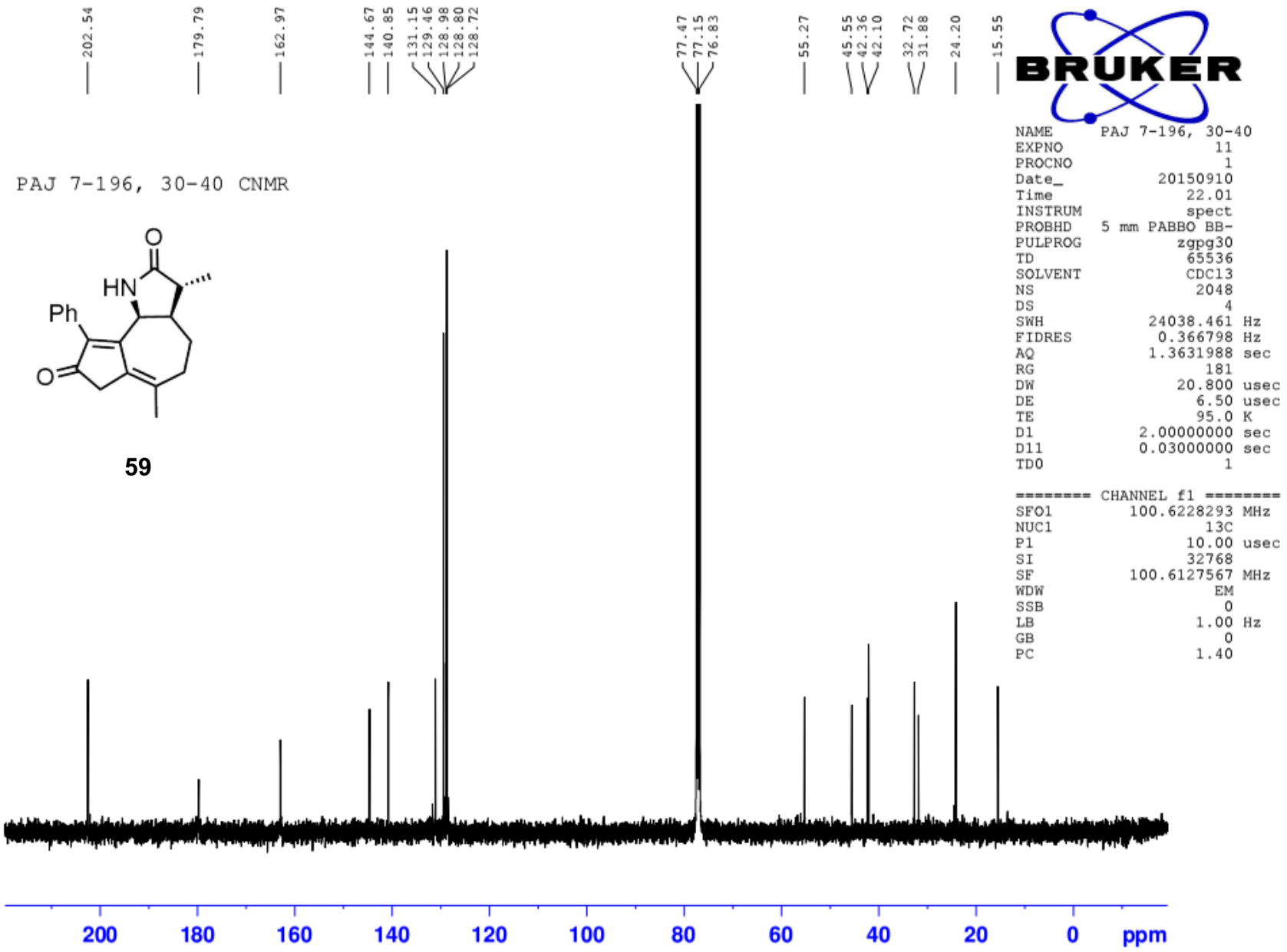
| | |
|---|---|
| SI | 32768 |
| SF | 125.7653135 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| | 1.40 |

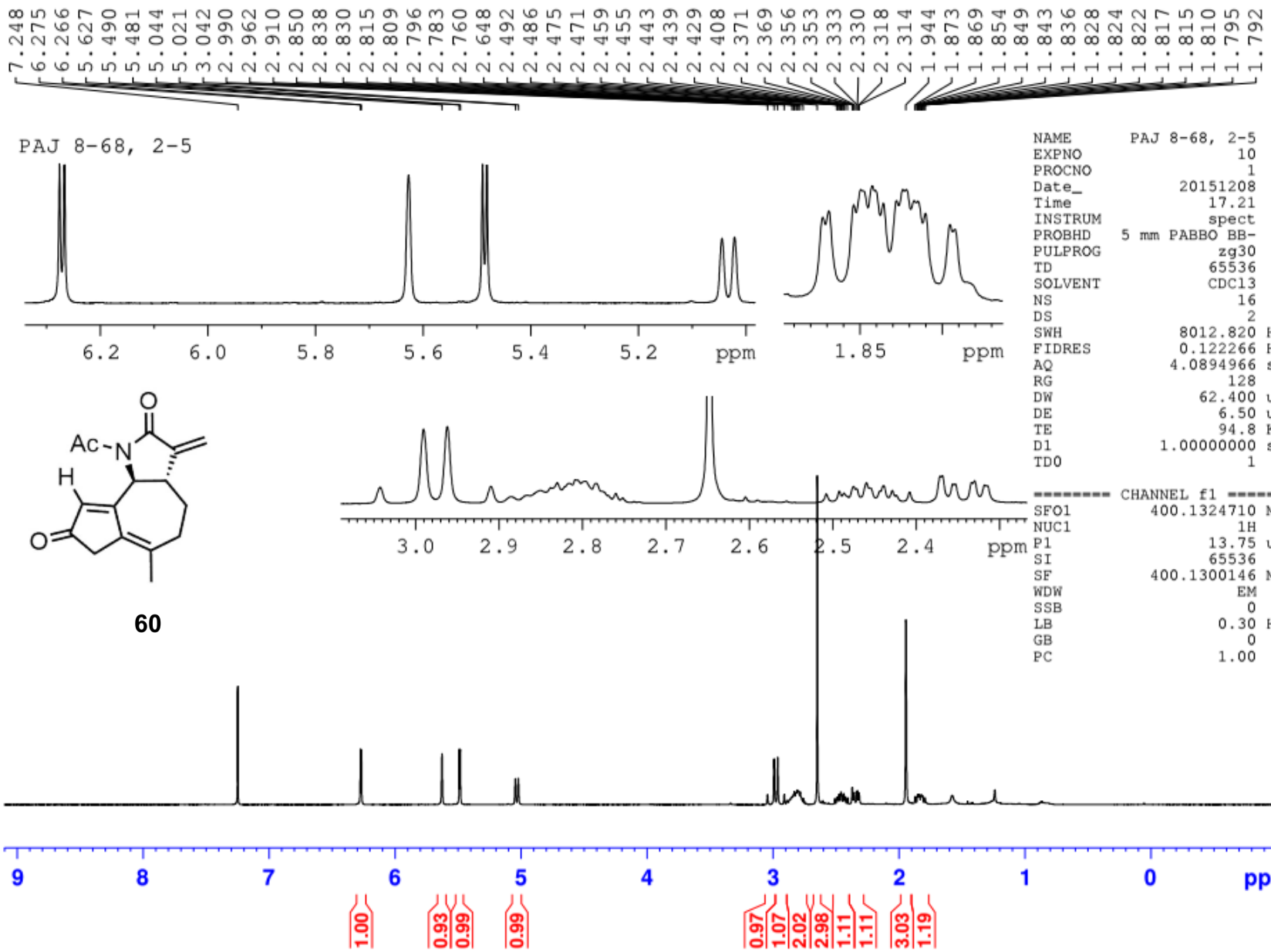


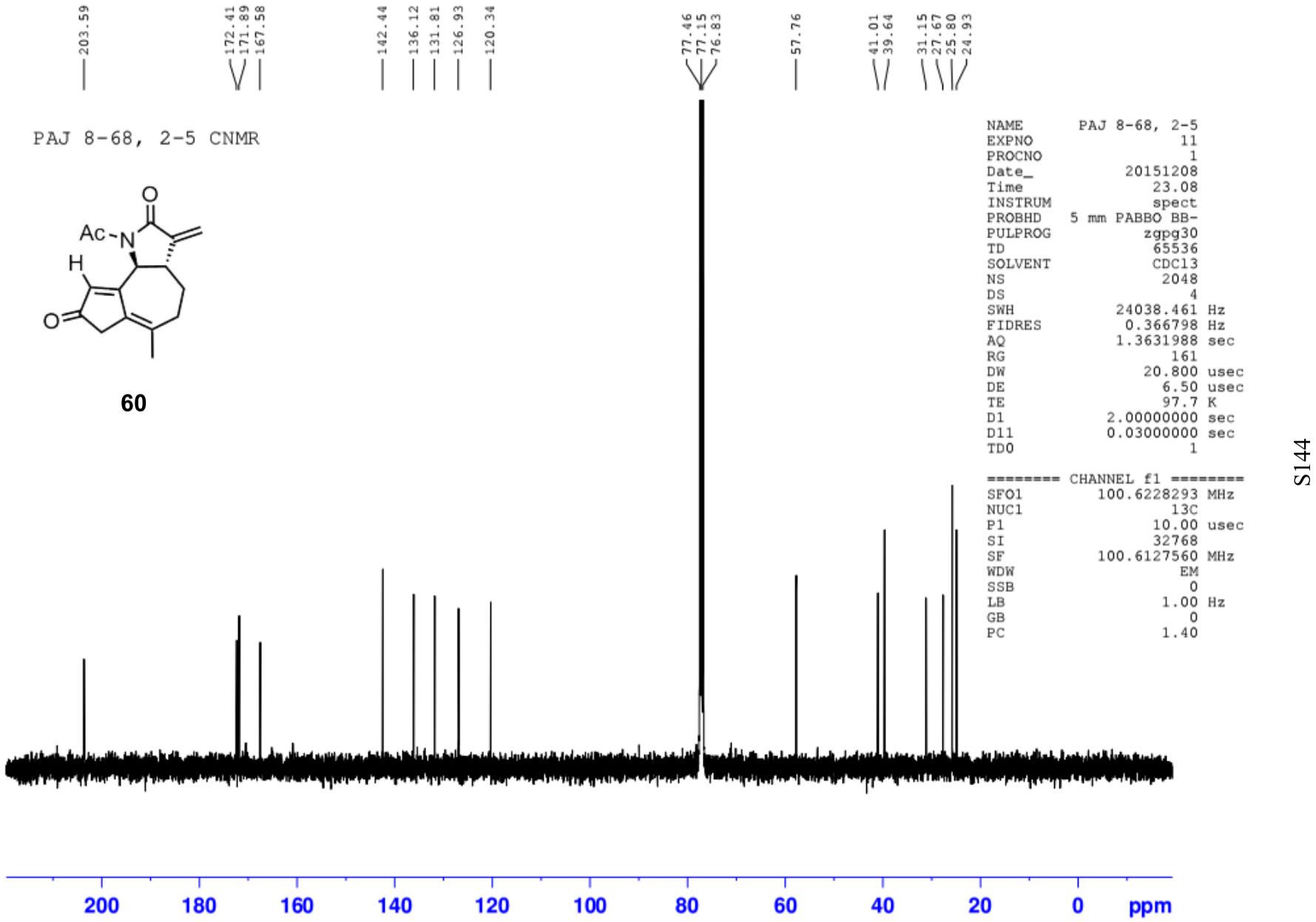

| | |
|---|---|
| NAME | PAJ 8-76, 9-20 |
| EXPNO | 10 |
| PROCNO | 1 |
| Date_ | 20151215 |
| Time | 18.03 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB/ |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 16 |
| DS | 2 |
| SWH | 10000.000 Hz |
| FIDRES | 0.152588 Hz |
| AQ | 3.2768500 sec |
| RG | 101 |
| DW | 50.000 usec |
| DE | 6.50 usec |
| TE | 297.9 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| SFO1 | 500.1630887 MHz |
| NUC1 | 1H |
| P1 | 11.45 usec |
| SI | 65536 |
| SF | 500.1600131 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

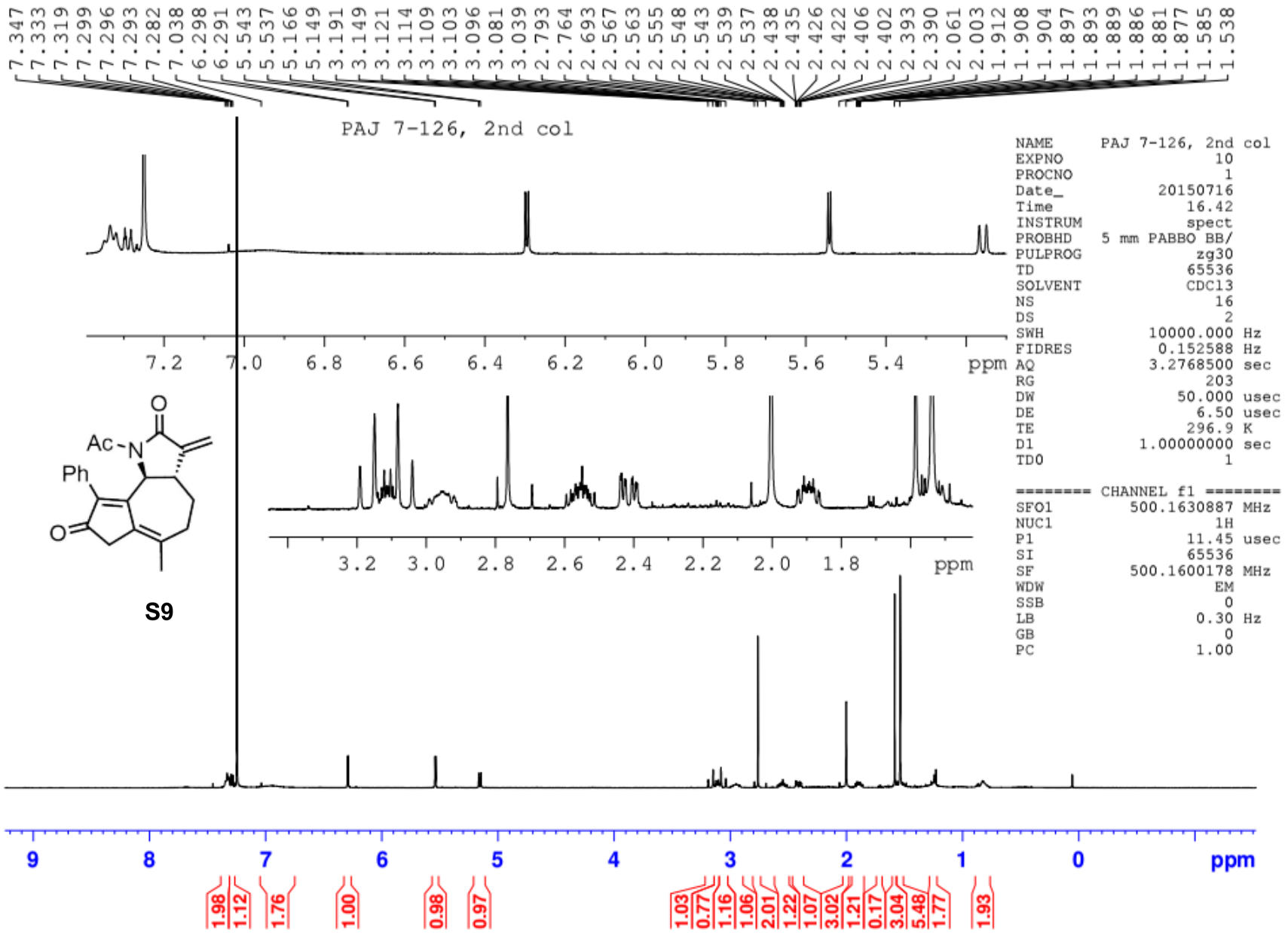

203.982
173.283
137.491
131.222
126.603
77.413
77.159
76.905
56.615
47.346
44.962
41.352
32.650
28.105
24.756
13.814
PAJ 8-76, 9-20 CNMR
HN
H
Ha
O
O
58
7.3:1 dr
NAME PAJ 8-76, 9-20
EXPNO 11
PROCNO 1
Date_ 20151215
Time 21.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7653133 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

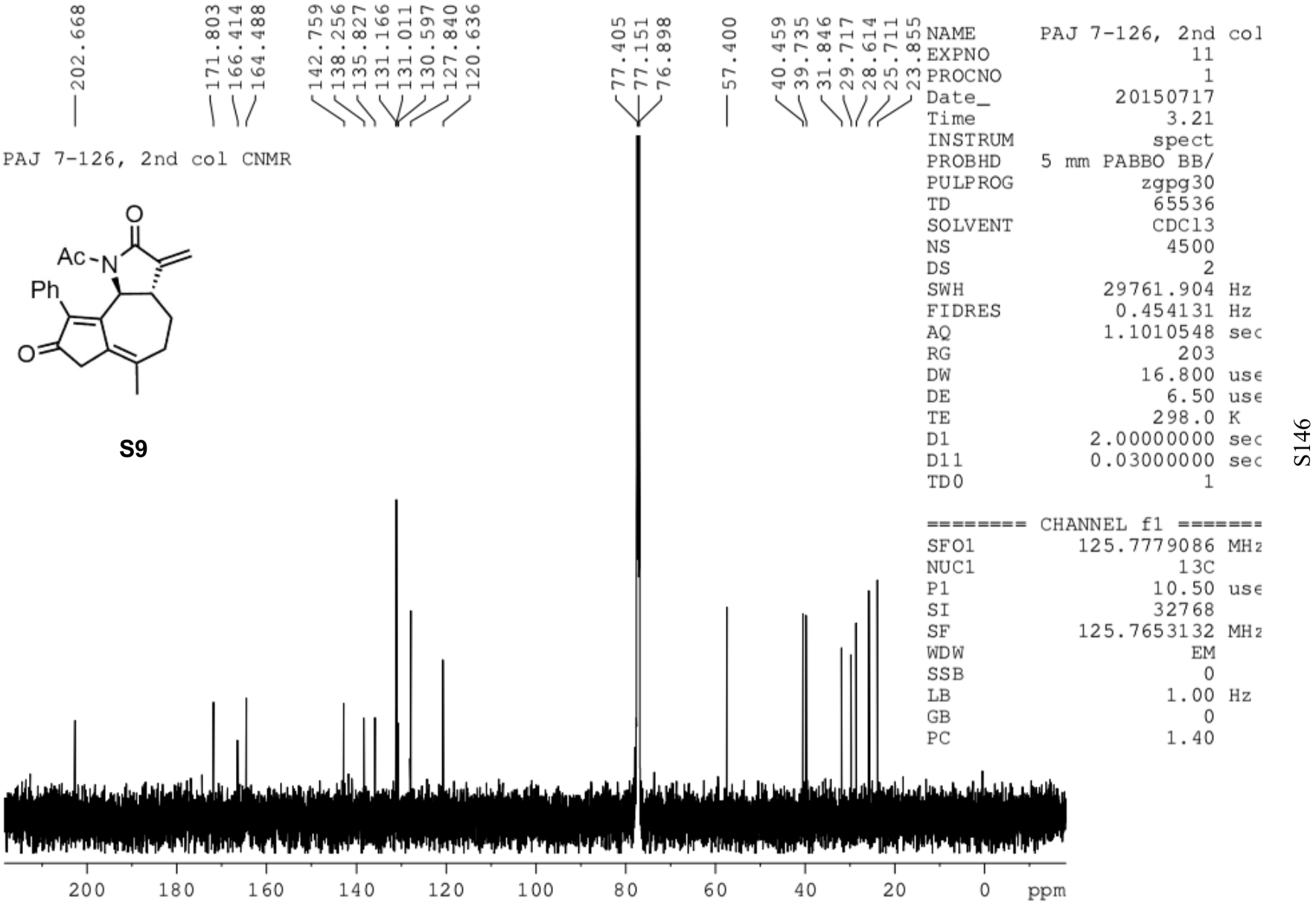

7.441
7.423
7.420
7.405
7.381
7.377
7.362
7.252
7.173
7.170
7.153
5.309
4.991
4.972
3.218
3.166
3.147
3.124
3.095
2.586
2.559
2.539
2.510
2.495
2.483
2.477
2.464
2.351
2.332
2.304
2.285
2.280
2.267
2.261
2.249
2.244
2.055
2.047
2.032
2.020
1.897
1.862
1.847
1.829
1.811
1.663
1.233
1.215
PAJ 7-196, 30-40
5.4
5.2
ppm
3.2
ppm
2.6
2.4
2.2
2.0
ppm
1.8
1.6
1.4
ppm
HN
Ph
O
O
59
NAME PAJ 7-196, 30-40
EXPNO 10
PROCNO 1
Date_ 20150910
Time 16.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 101
DW 62.400 usec
DE 6.50 usec
TE 95.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300131 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
3.47
1.89
1.00
0.97
0.18
2.14
2.04
2.28
1.29
0.31
2.77
1.27
2.11
3.68


202.54
179.79
162.97
144.67
140.85
131.15
129.46
128.98
128.80
128.72
77.47
77.15
76.83
55.27
45.55
42.36
42.10
32.72
31.88
24.20
15.55
BRUKER
PAJ 7-196, 30-40 CNMR
HN
Ph
O
O
59
NAME PAJ 7-196, 30-40
EXPNO 11
PROCNO 1
Date_ 20150910
Time 22.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.50 usec
TE 95.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127567 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

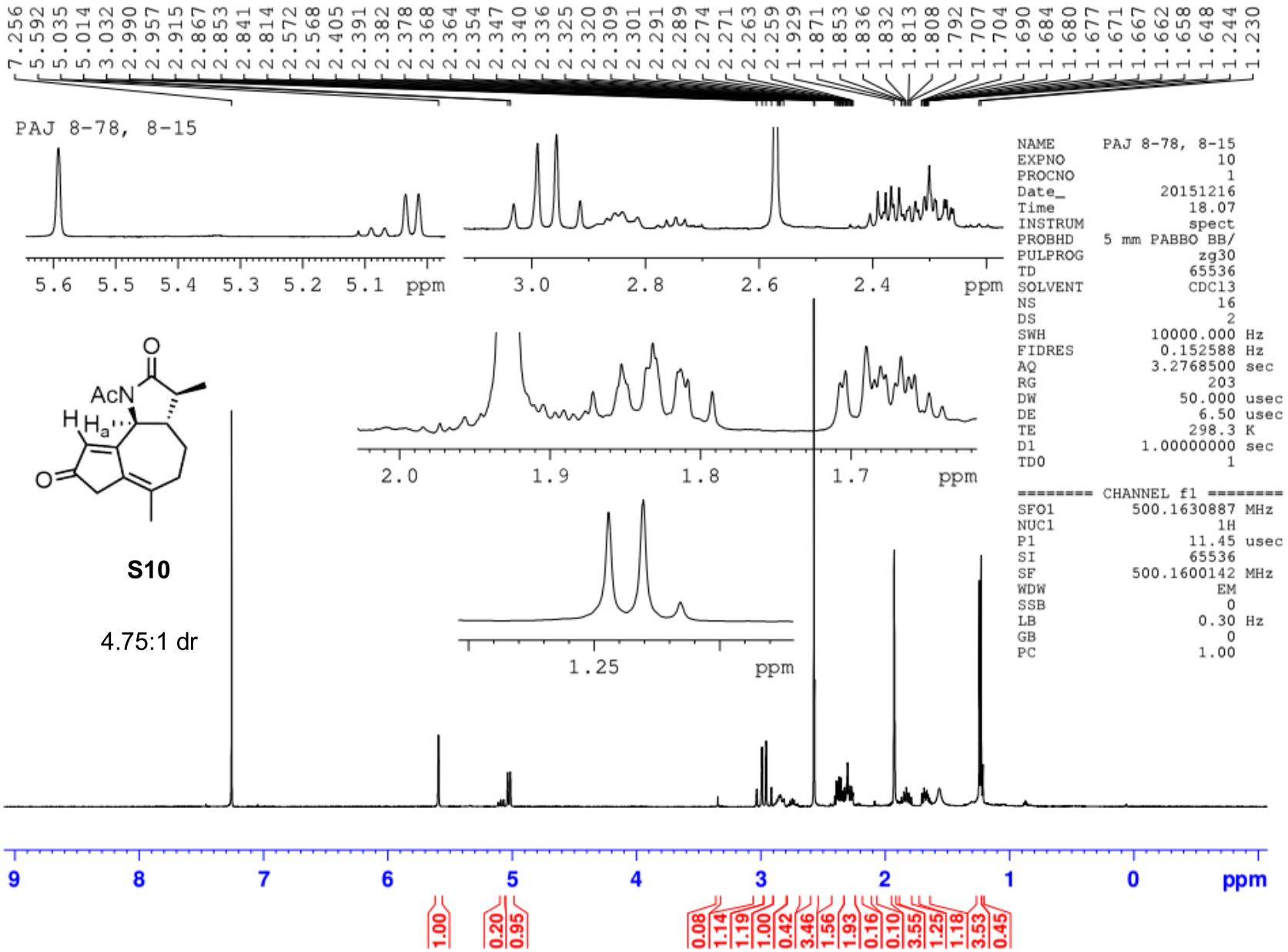

7.248 6.275 6.266 5.627 5.490 5.481 5.044 5.021 3.042 2.990 2.962 2.910 2.850 2.838 2.830 2.815 2.809 2.796 2.783 2.760 2.648 2.492 2.486 2.475 2.471 2.459 2.455 2.443 2.439 2.429 2.408 2.371 2.369 2.356 2.353 2.333 2.330 2.318 2.314 1.944 1.873 1.869 1.854 1.849 1.843 1.836 1.828 1.824 1.822 1.817 1.815 1.810 1.795 1.792
PAJ 8-68, 2-5
6.2 6.0 5.8 5.6 5.4 5.2 ppm
1.85 ppm
3.0 2.9 2.8 2.7 2.6 2.5 2.4 ppm
Ac
N
H
O
O
60
NAME PAJ 8-68, 2-5
EXPNO 10
PROCNO 1
Date_ 20151208
Time 17.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 128
DW 62.400 usec
DE 6.50 usec
TE 94.8 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 400.1324710 MHz
NUC1 1H
P1 13.75 usec
SI 65536
SF 400.1300146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.00 0.93 0.99 0.99 0.97 1.07 2.02 2.98 1.11 1.11 3.03 1.19


203.59
172.41
171.89
167.58
142.44
136.12
131.81
126.93
120.34
77.46
77.15
76.83
57.76
41.01
39.64
31.15
27.67
25.80
24.93
PAJ 8-68, 2-5 CNMR
Ac
N
H
O
O
60
NAME PAJ 8-68, 2-5
EXPNO 11
PROCNO 1
Date_ 20151208
Time 23.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 161
DW 20.800 usec
DE 6.50 usec
TE 97.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127560 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


7.347 7.333 7.319 7.299 7.296 7.293 7.282 7.038 6.298 6.291 6.543 5.537 5.166 5.149 3.191 3.149 3.121 3.114 3.109 3.103 3.096 3.081 3.039 2.793 2.764 2.693 2.567 2.563 2.555 2.548 2.543 2.539 2.537 2.438 2.435 2.426 2.422 2.406 2.402 2.393 2.390 2.061 2.003 1.912 1.908 1.904 1.897 1.893 1.889 1.886 1.881 1.877 1.585 1.538
PAJ 7-126, 2nd col
7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 ppm
3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 ppm
Ac
N
Ph
O
O
S9
NAME PAJ 7-126, 2nd col
EXPNO 10
PROCNO 1
Date_ 20150716
Time 16.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600178 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.98 1.12 1.76 1.00 0.98 0.97 1.03 0.77 1.16 1.06 2.01 1.22 1.07 3.02 1.21 0.17 3.04 5.48 1.77 1.93


202.668
171.803
166.414
164.488
142.759
138.256
135.827
131.166
131.011
130.597
127.840
120.636
77.405
77.151
76.898
57.400
40.459
39.735
31.846
29.717
28.614
25.711
23.855
PAJ 7-126, 2nd col CNMR
Ac
N
Ph
O
O
NAME PAJ 7-126, 2nd col
EXPNO 11
PROCNO 1
Date_ 20150717
Time 3.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4500
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 use
DE 6.50 use
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 use
SI 32768
SF 125.7653132 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

**S9**


7.256 5.592 5.035 5.014 3.032 2.990 2.957 2.915 2.867 2.853 2.841 2.814 2.572 2.568 2.405 2.391 2.382 2.378 2.368 2.364 2.354 2.347 2.340 2.336 2.325 2.320 2.309 2.301 2.291 2.289 2.274 2.271 2.263 2.259 1.929 1.871 1.853 1.836 1.832 1.813 1.808 1.792 1.707 1.704 1.690 1.684 1.680 1.677 1.671 1.667 1.662 1.658 1.648 1.244 1.230
PAJ 8-78, 8-15
5.6 5.5 5.4 5.3 5.2 5.1 ppm
3.0 2.8 2.6 2.4 ppm
2.0 1.9 1.8 1.7 ppm
1.25 ppm
AcN
H
Ha
O
O
S10
4.75:1 dr
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EXPNO 10
PROCNO 1
Date_ 20151216
Time 18.07
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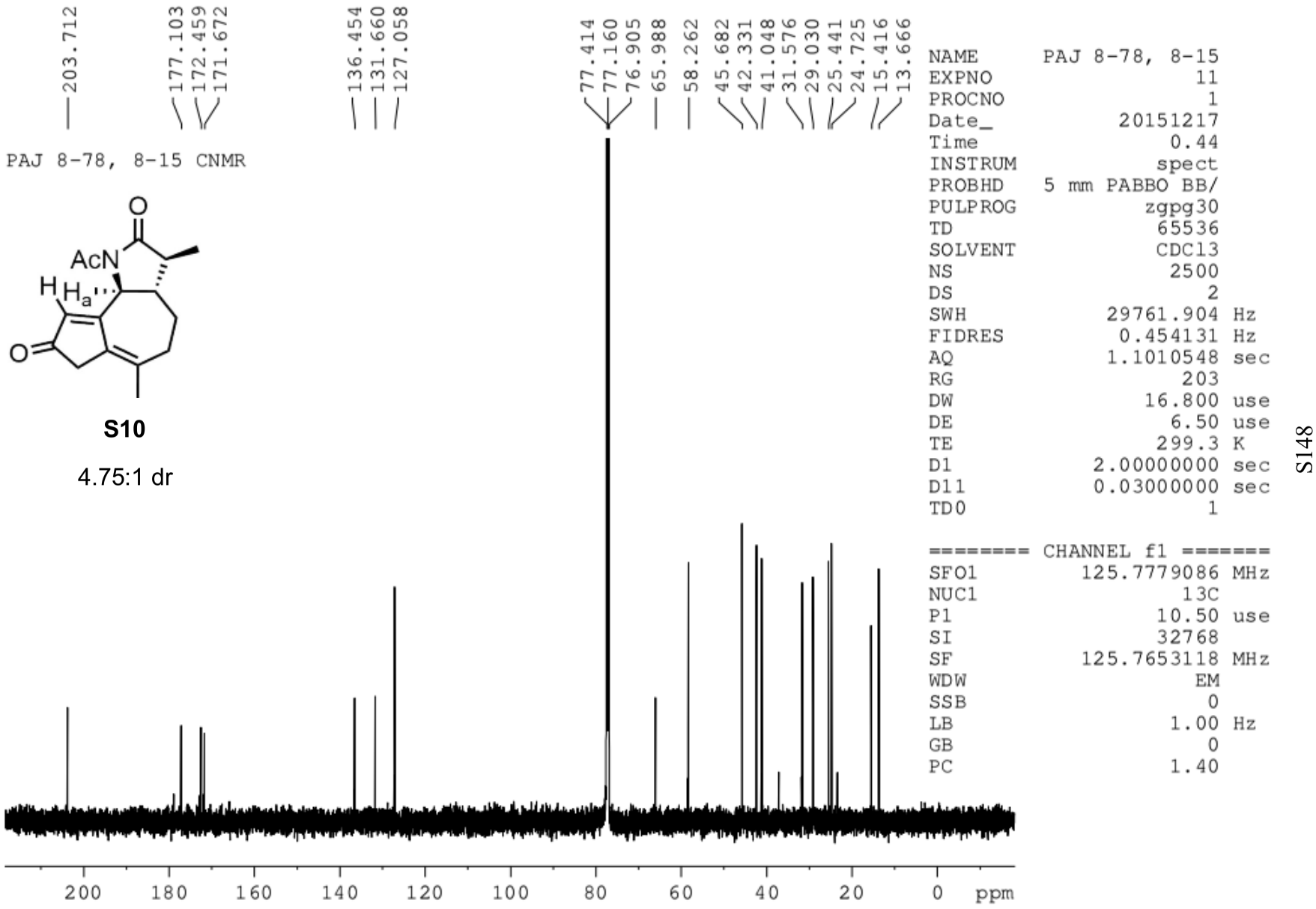
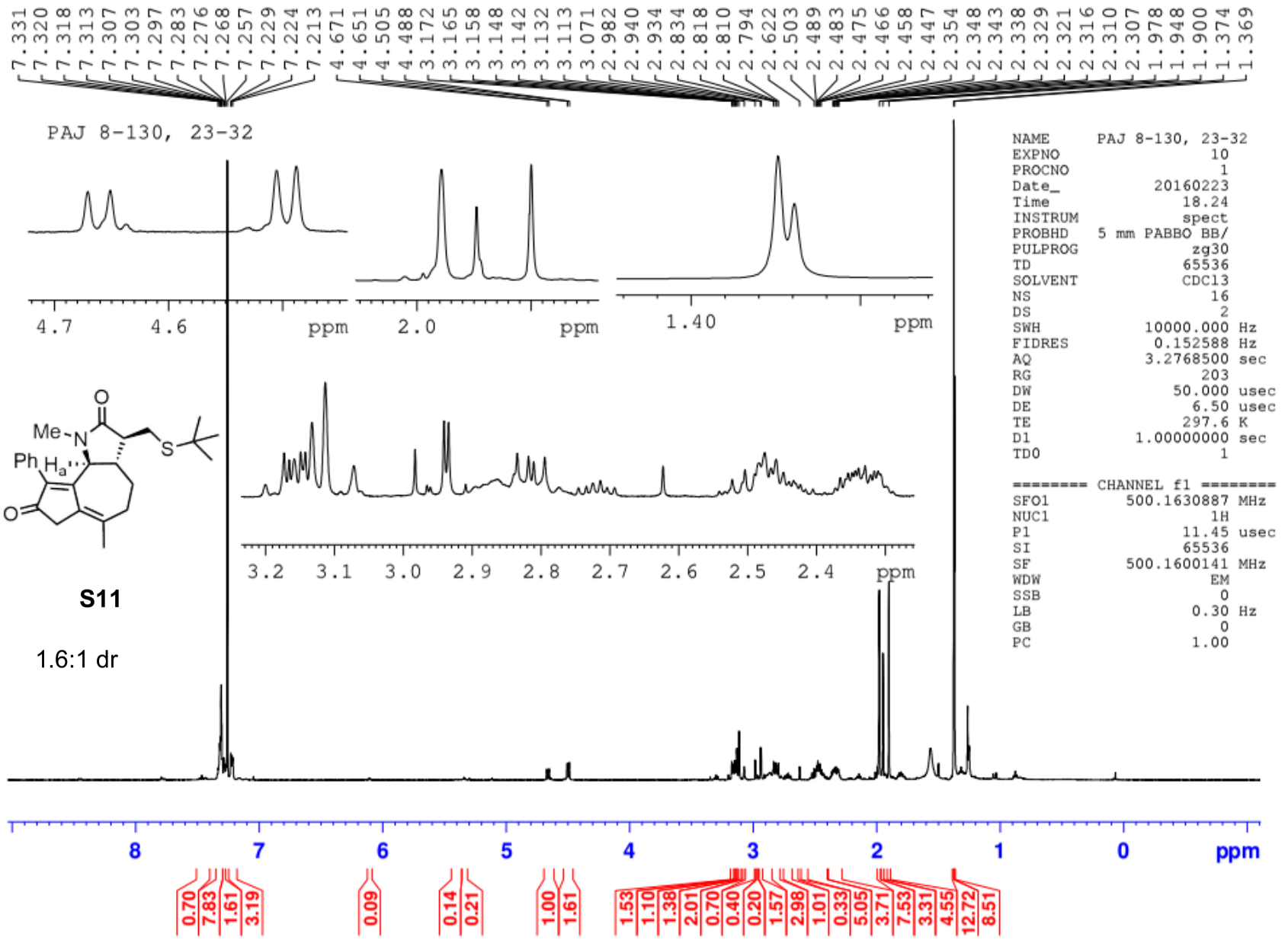
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600142 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.00 0.20 0.95
0.08 1.14 1.19 1.00 0.42 3.46 1.56 1.93 0.16 0.10 3.55 1.25 1.18 3.53 0.45


203.712
177.103
172.459
171.672
136.454
131.660
127.058
77.414
77.160
76.905
65.988
58.262
45.682
42.331
41.048
31.576
29.030
25.441
24.725
15.416
13.666
PAJ 8-78, 8-15 CNMR
AcN
H
Ha
O
O
S10
4.75:1 dr
NAME PAJ 8-78, 8-15
EXPNO 11
PROCNO 1
Date_ 20151217
Time 0.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2500
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 use
DE 6.50 use
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
SFO1 125.7779086 MHz
NUC1 13C
P1 10.50 use
SI 32768
SF 125.7653118 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


7.331 7.320 7.318 7.313 7.307 7.303 7.297 7.283 7.276 7.268 7.257 7.229 7.224 7.213 4.671 4.651 4.505 4.488 3.172 3.165 3.158 3.148 3.142 3.132 3.113 3.071 2.982 2.940 2.934 2.834 2.818 2.810 2.794 2.622 2.503 2.489 2.483 2.475 2.466 2.458 2.447 2.354 2.348 2.343 2.338 2.329 2.321 2.316 2.310 2.307 1.978 1.948 1.900 1.374 1.369
PAJ 8-130, 23-32
4.7 4.6 ppm
2.0 ppm
1.40 ppm
3.2 3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 ppm
Me N
Ph Ha
O
S
S11
1.6:1 dr
NAME PAJ 8-130, 23-32
EXPNO 10
PROCNO 1
Date_ 20160223
Time 18.24
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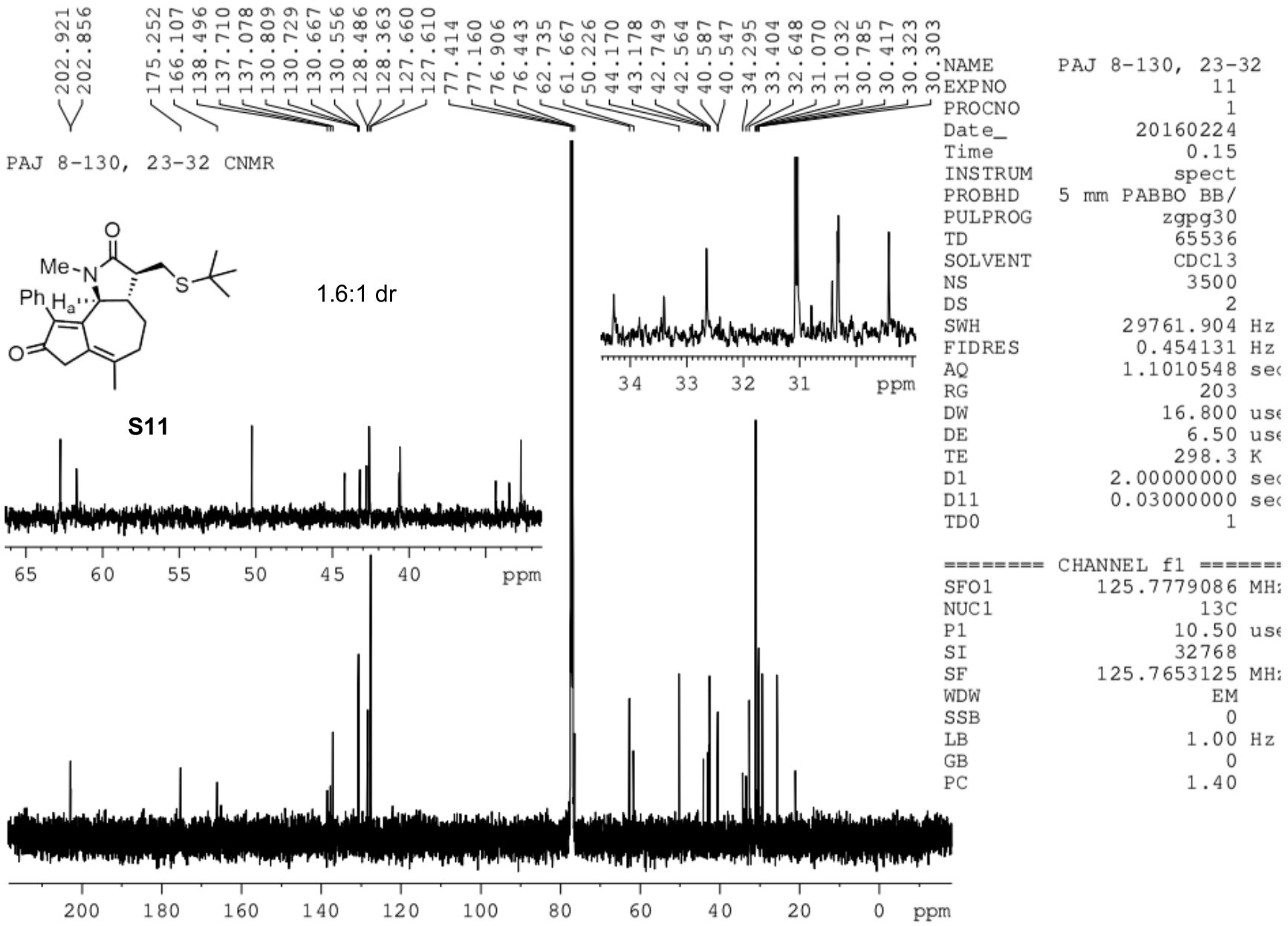
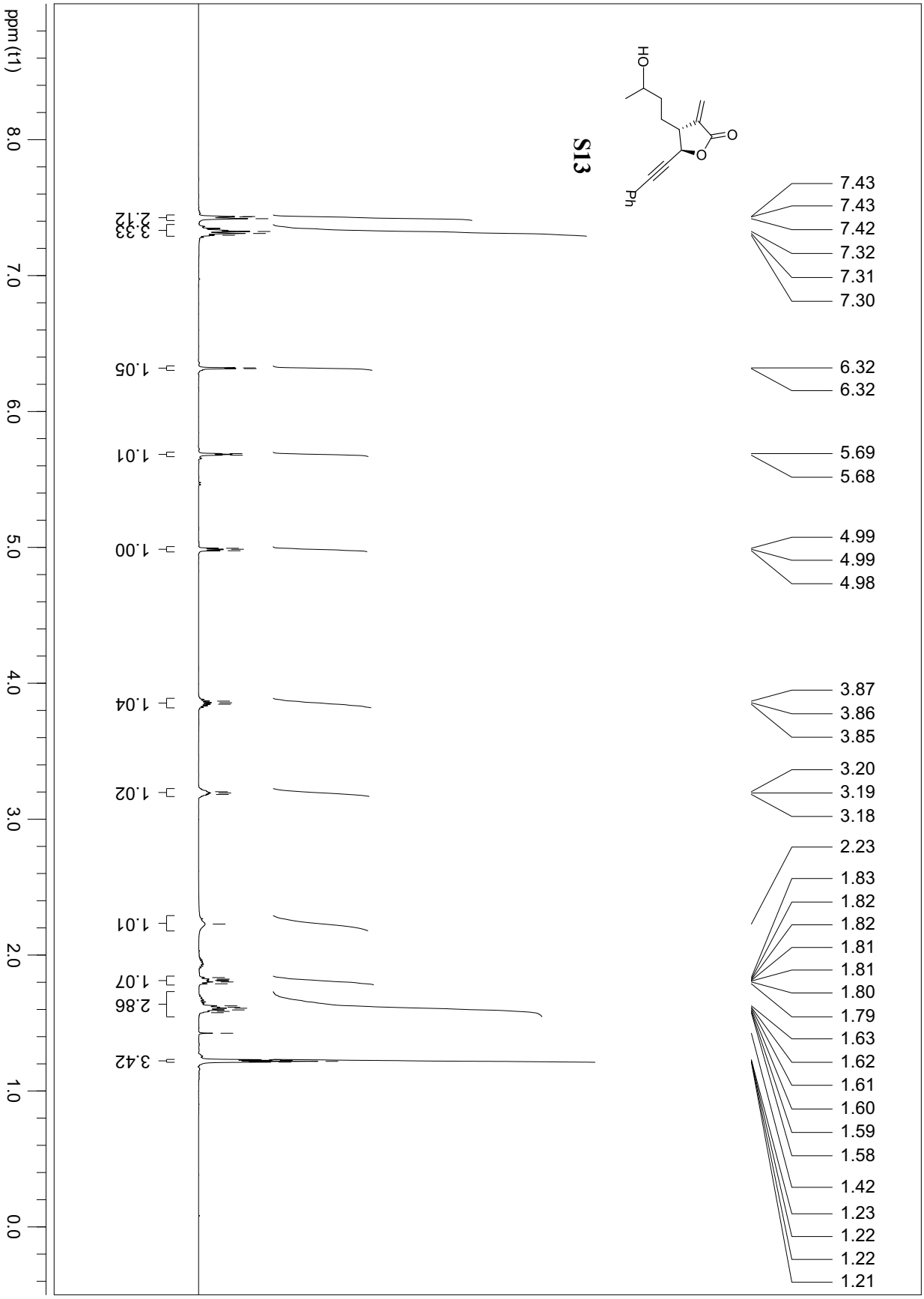
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1630887 MHz
NUC1 1H
P1 11.45 usec
SI 65536
SF 500.1600141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8 7 6 5 4 3 2 1 0 ppm
0.70 7.83 1.61 3.19 0.09 0.14 0.21 1.00 1.61 1.53 1.10 1.38 2.01 0.70 0.40 0.20 1.57 2.98 1.01 0.33 5.05 3.71 7.53 3.31 4.55 12.72 8.51


202.921
202.856
175.252
166.107
138.496
137.710
137.078
130.809
130.729
130.667
130.556
128.486
128.363
127.660
127.610
77.414
77.160
76.906
76.443
62.735
61.667
50.226
44.170
43.178
42.749
42.564
40.587
40.547
34.295
33.404
32.648
31.070
31.032
30.785
30.417
30.323
30.303
PAJ 8-130, 23-32 CNMR
Me
N
Ph
Ha
S
O
O
1.6:1 dr
S11
34 33 32 31 ppm
65 60 55 50 45 40 ppm
200 180 160 140 120 100 80 60 40 20 0 ppm
NAME PAJ 8-130, 23-32
EXPNO 11
PROCNO 1
Date_ 20160224
Time 0.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3500
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 use
DE 6.50 use
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ======
SFO1 125.7779086 MH
NUC1 13C
P1 10.50 use
SI 32768
SF 125.7653125 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


S13
HO
O
O
Ph
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
2.12
2.33
1.05
1.01
1.00
1.04
1.02
1.01
1.07
2.86
3.42
7.43
7.43
7.42
7.32
7.31
7.30
6.32
6.32
5.69
5.68
4.99
4.99
4.98
3.87
3.86
3.85
3.20
3.19
3.18
2.23
1.83
1.82
1.82
1.81
1.81
1.80
1.79
1.63
1.62
1.61
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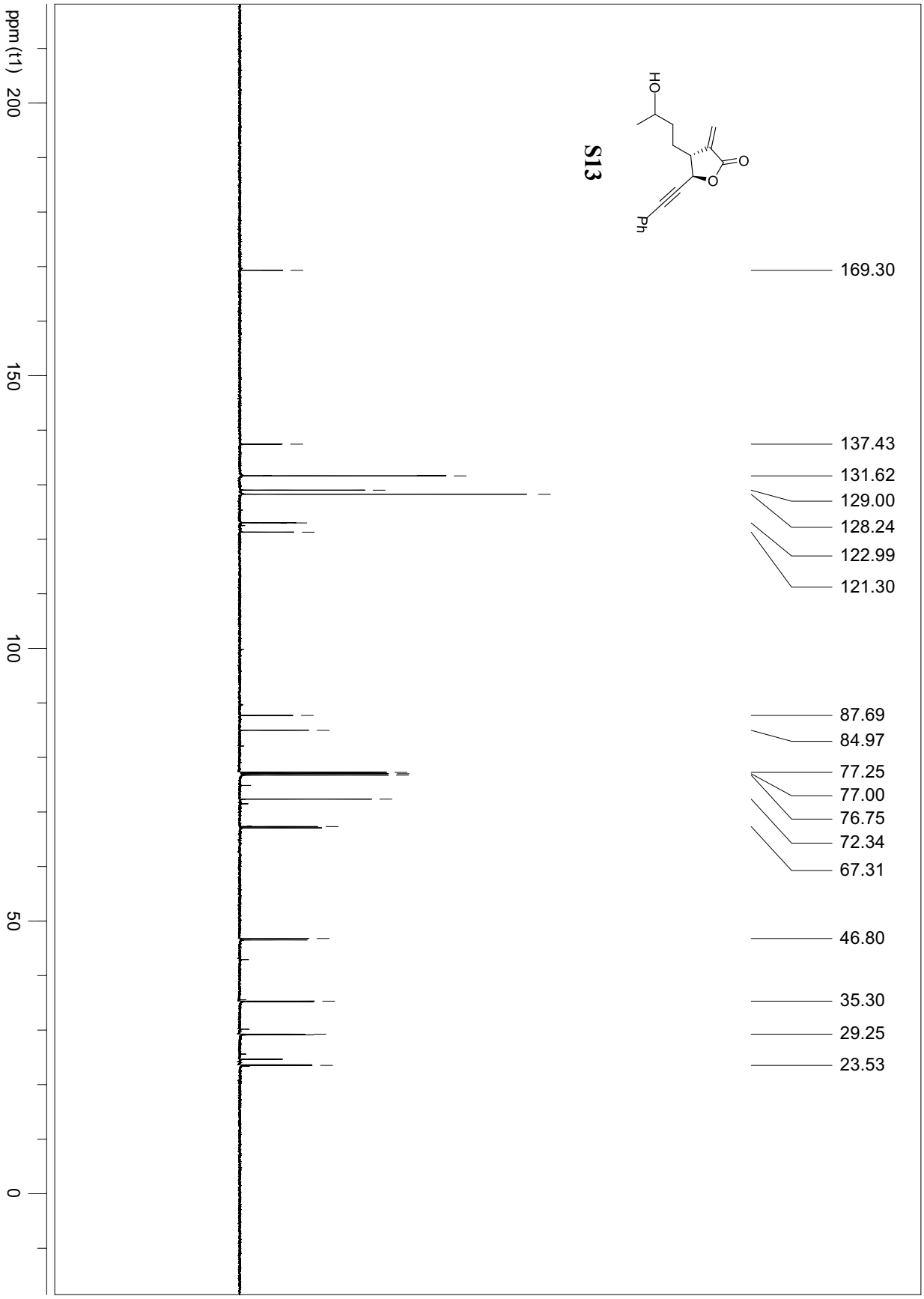
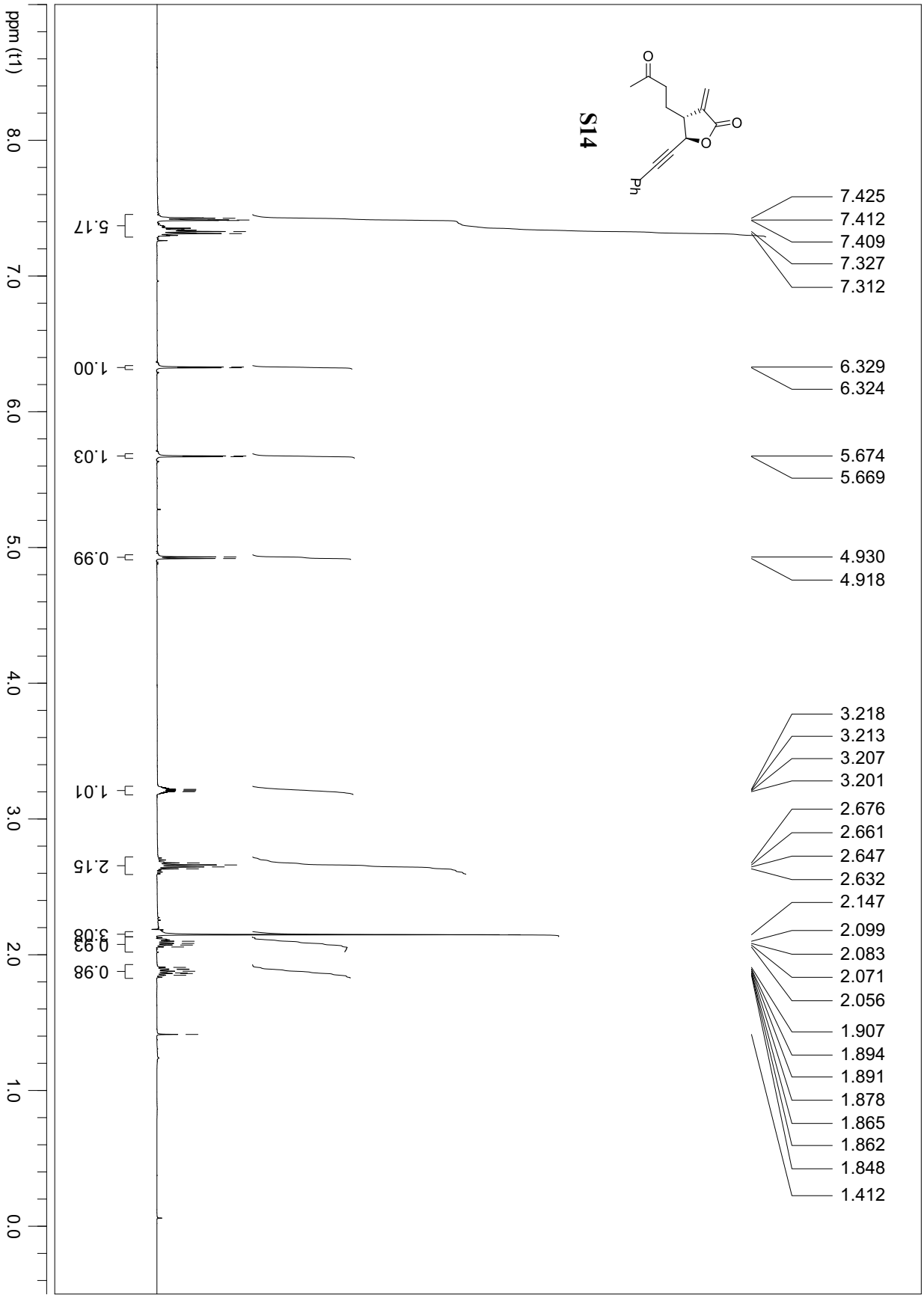
1.59
1.58
1.42
1.23
1.22
1.22
1.21


ppm (t1)
200
150
100
50
0
HO
O
O
Ph
169.30
137.43
131.62
129.00
128.24
122.99
121.30
87.69
84.97
77.25
77.00
76.75
72.34
67.31
46.80
35.30
29.25
23.53

**S13**


S14
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
5.17
1.00
1.03
0.99
1.01
2.15
3.08
0.93
0.98
7.425
7.412
7.409
7.327
7.312
6.329
6.324
5.674
5.669
4.930
4.918
3.218
3.213
3.207
3.201
2.676
2.661
2.647
2.632
2.147
2.099
2.083
2.071
2.056
1.907
1.894
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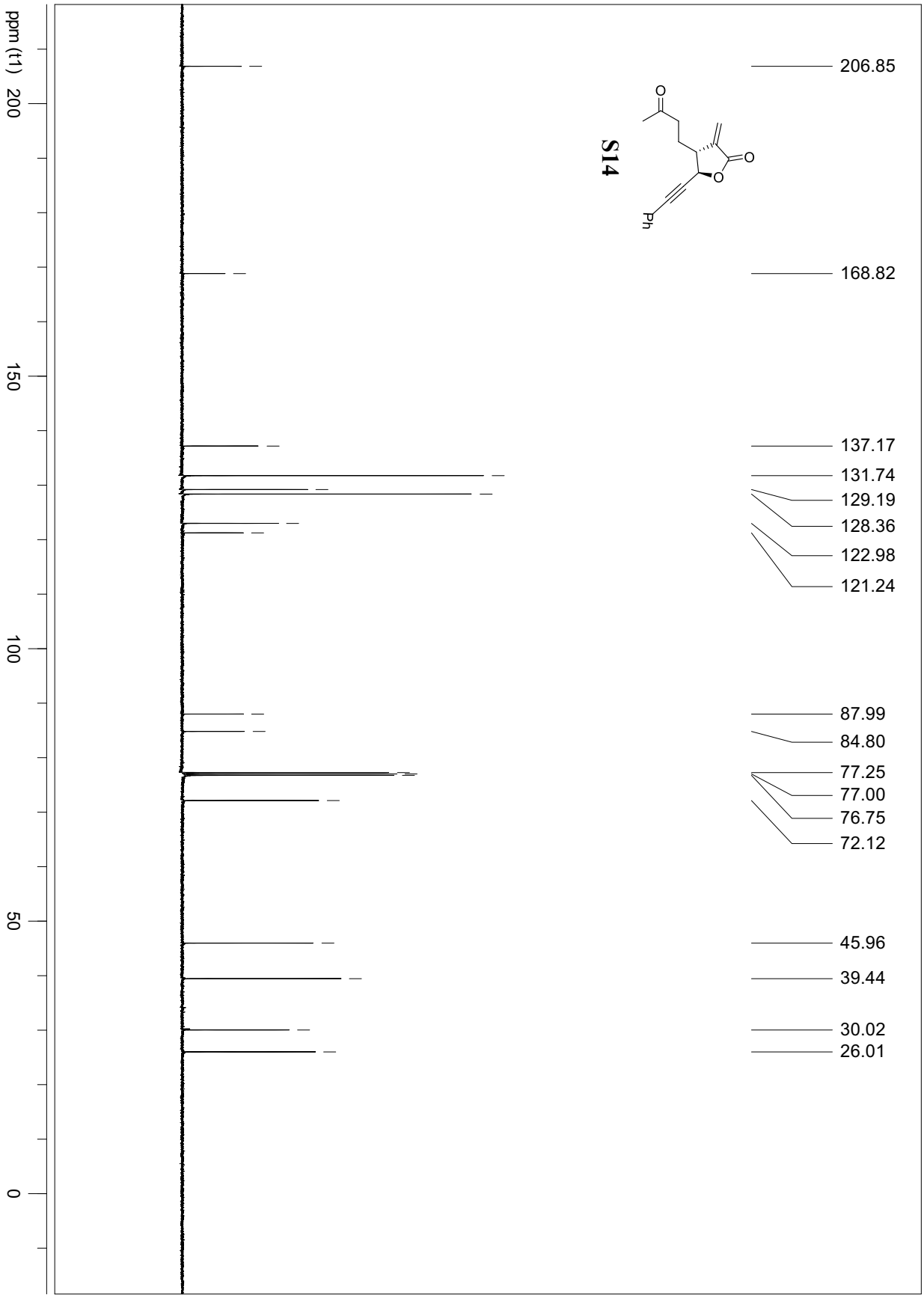
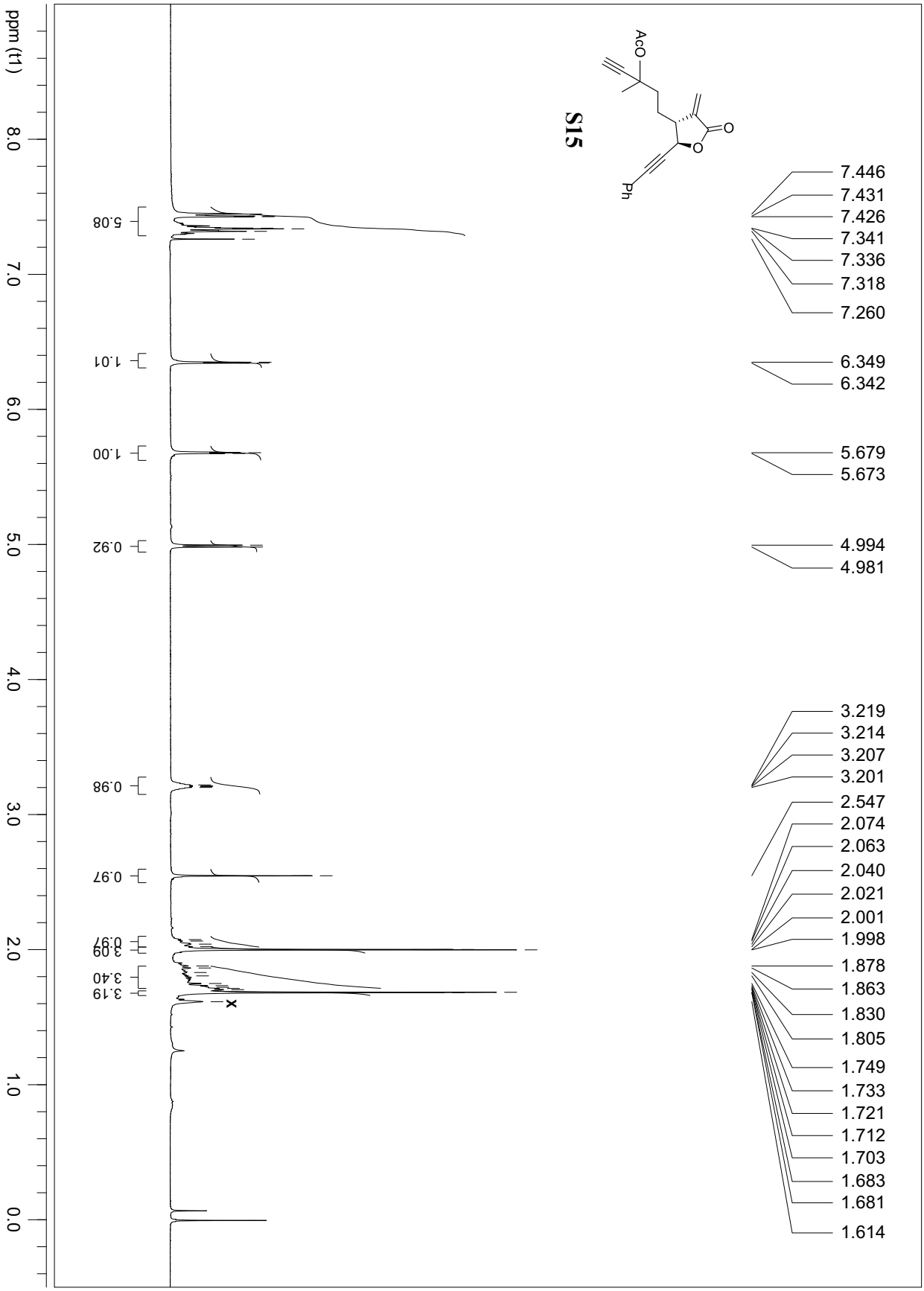
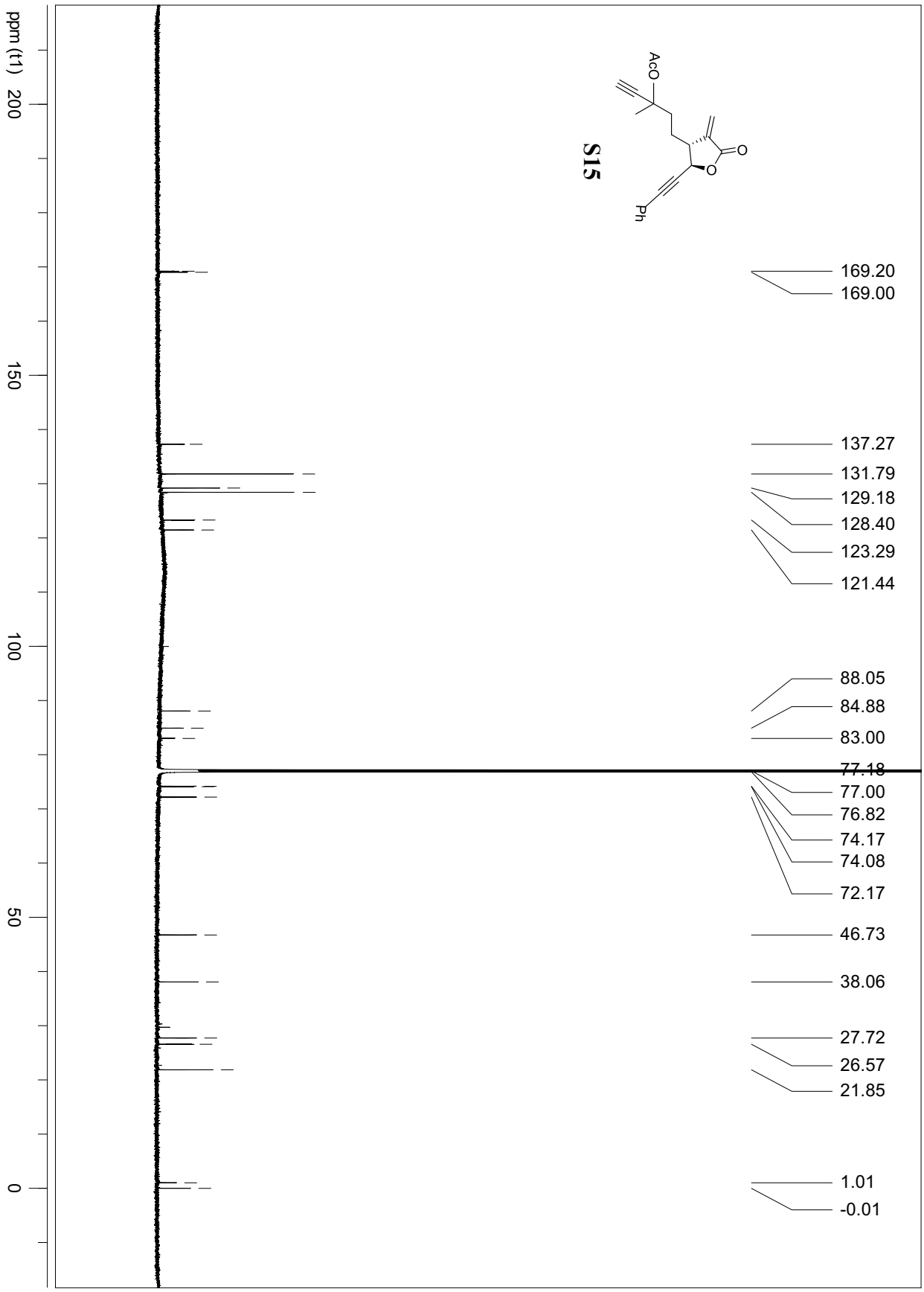
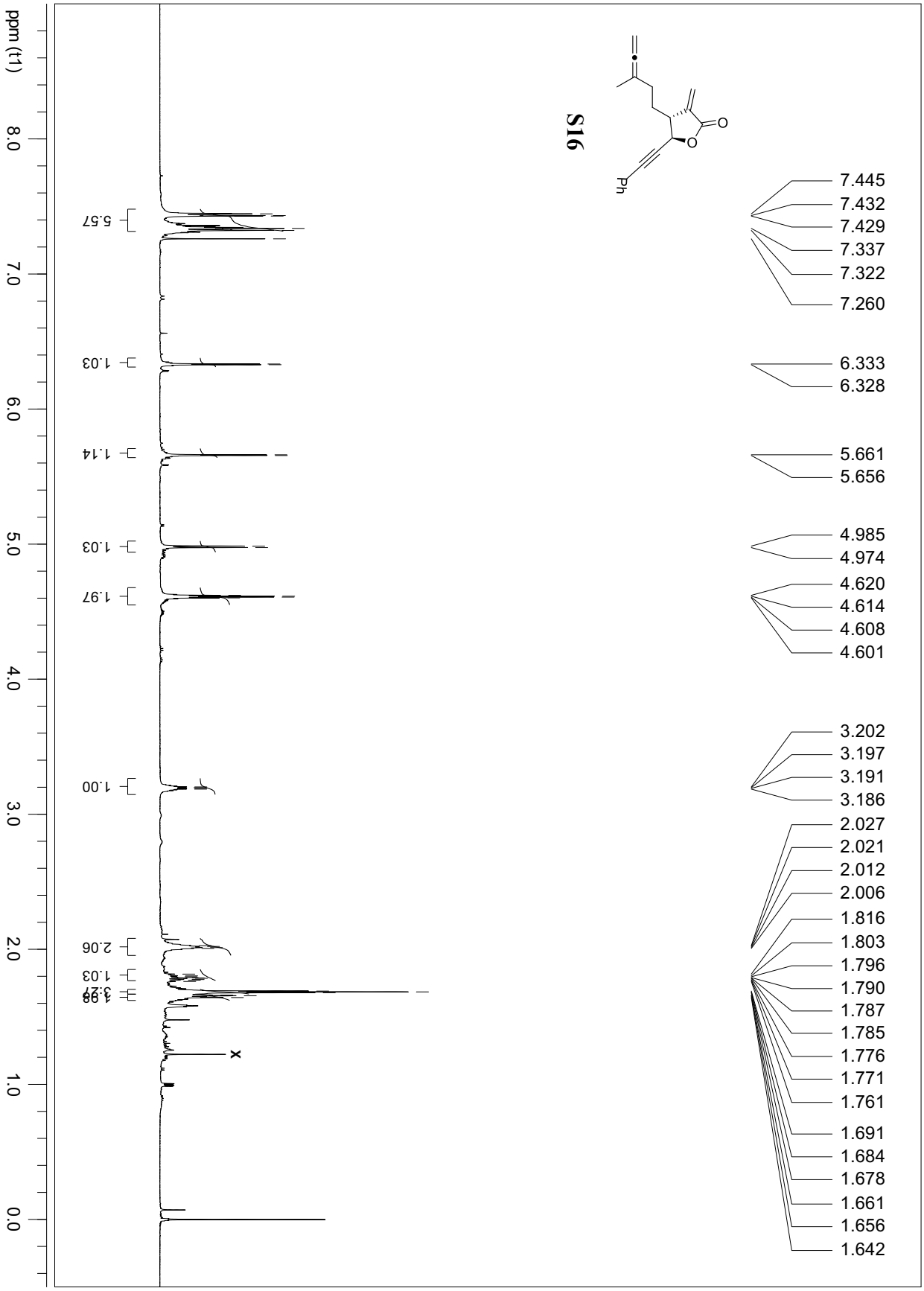
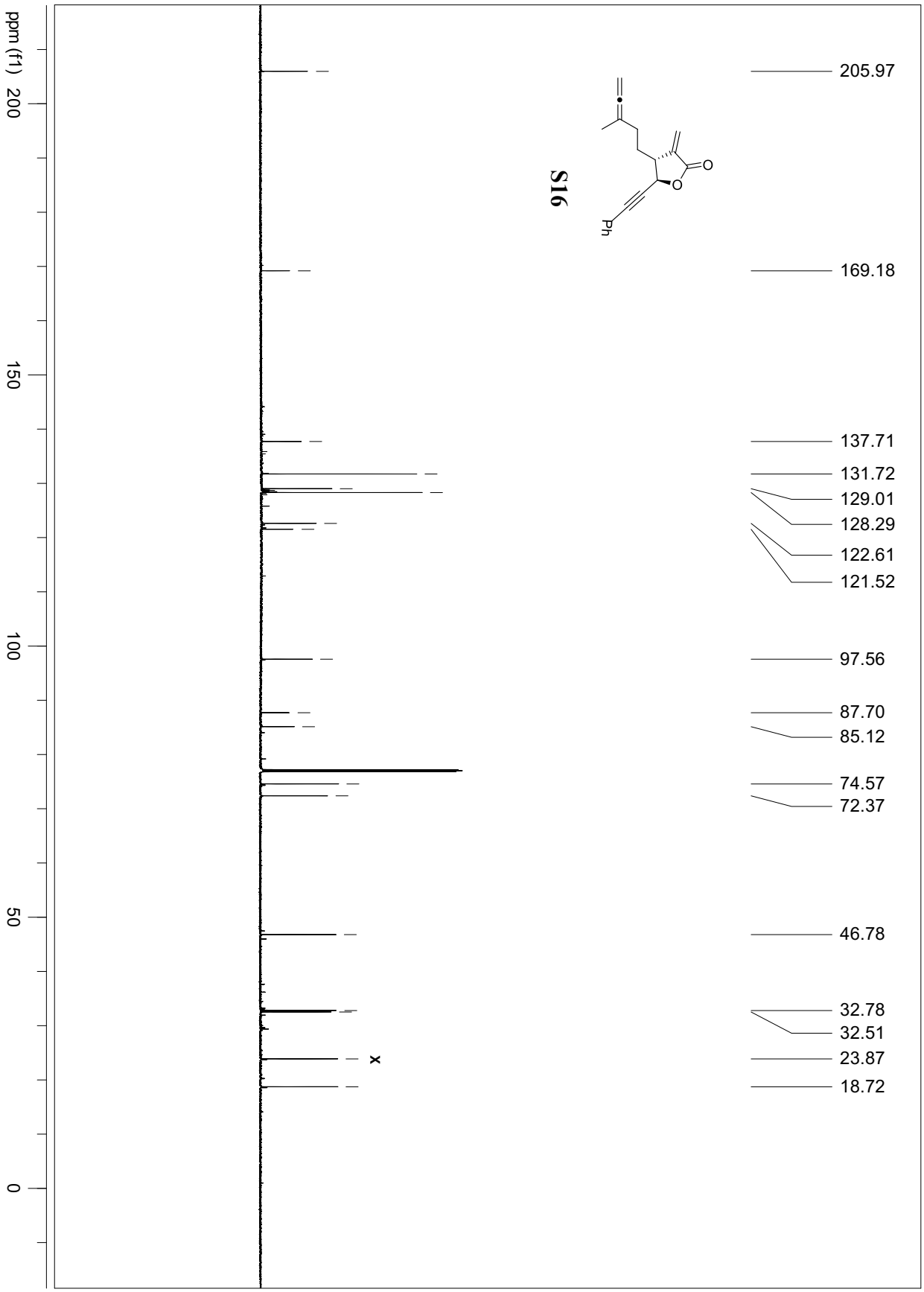
1.878
1.865
1.862
1.848
1.412

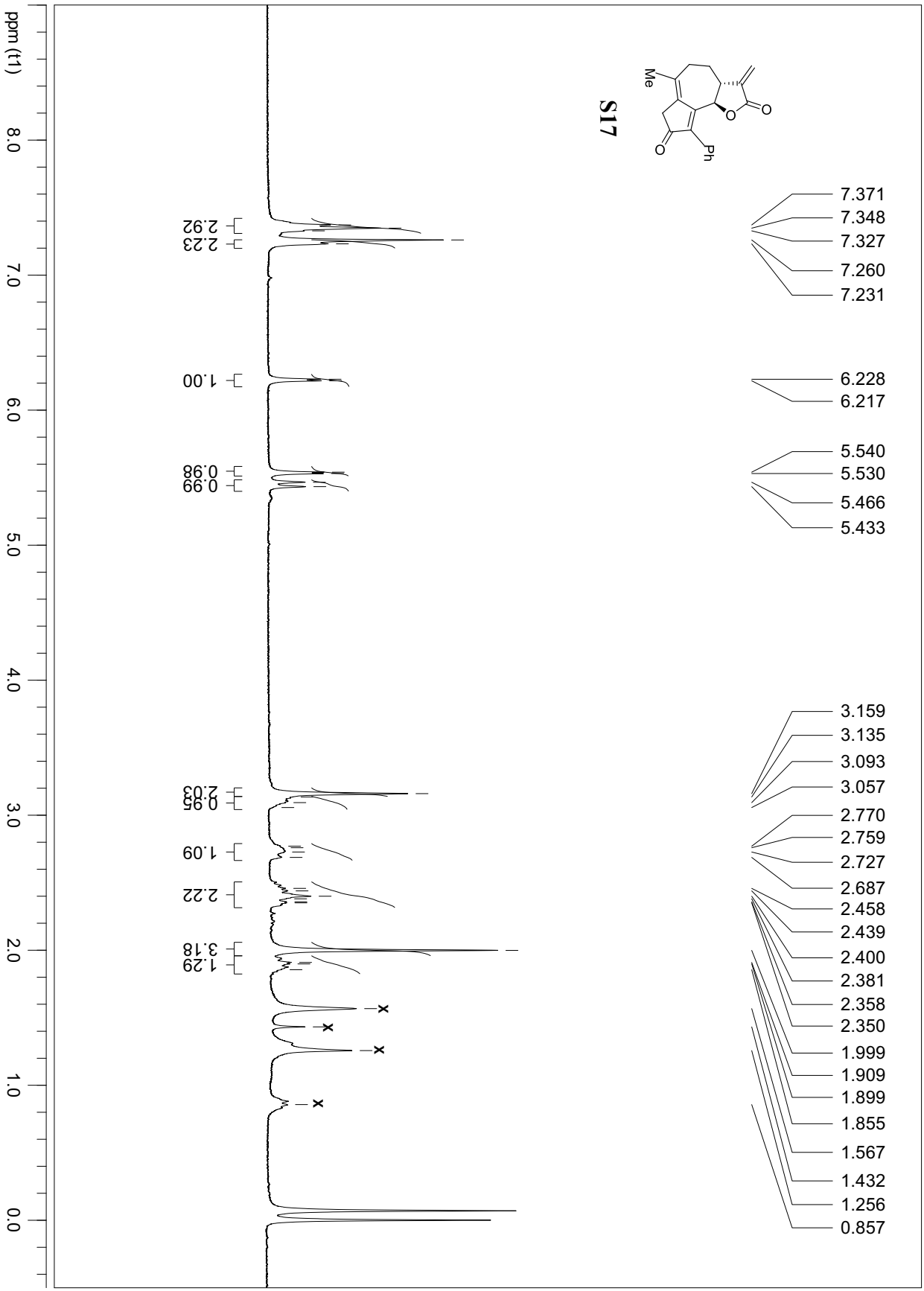

ppm (t1)
200
150
100
50
0
206.85
168.82
137.17
131.74
129.19
128.36
122.98
121.24
87.99
84.80
77.25
77.00
76.75
72.12
45.96
39.44
30.02
26.01
S14
O
O
O
Ph

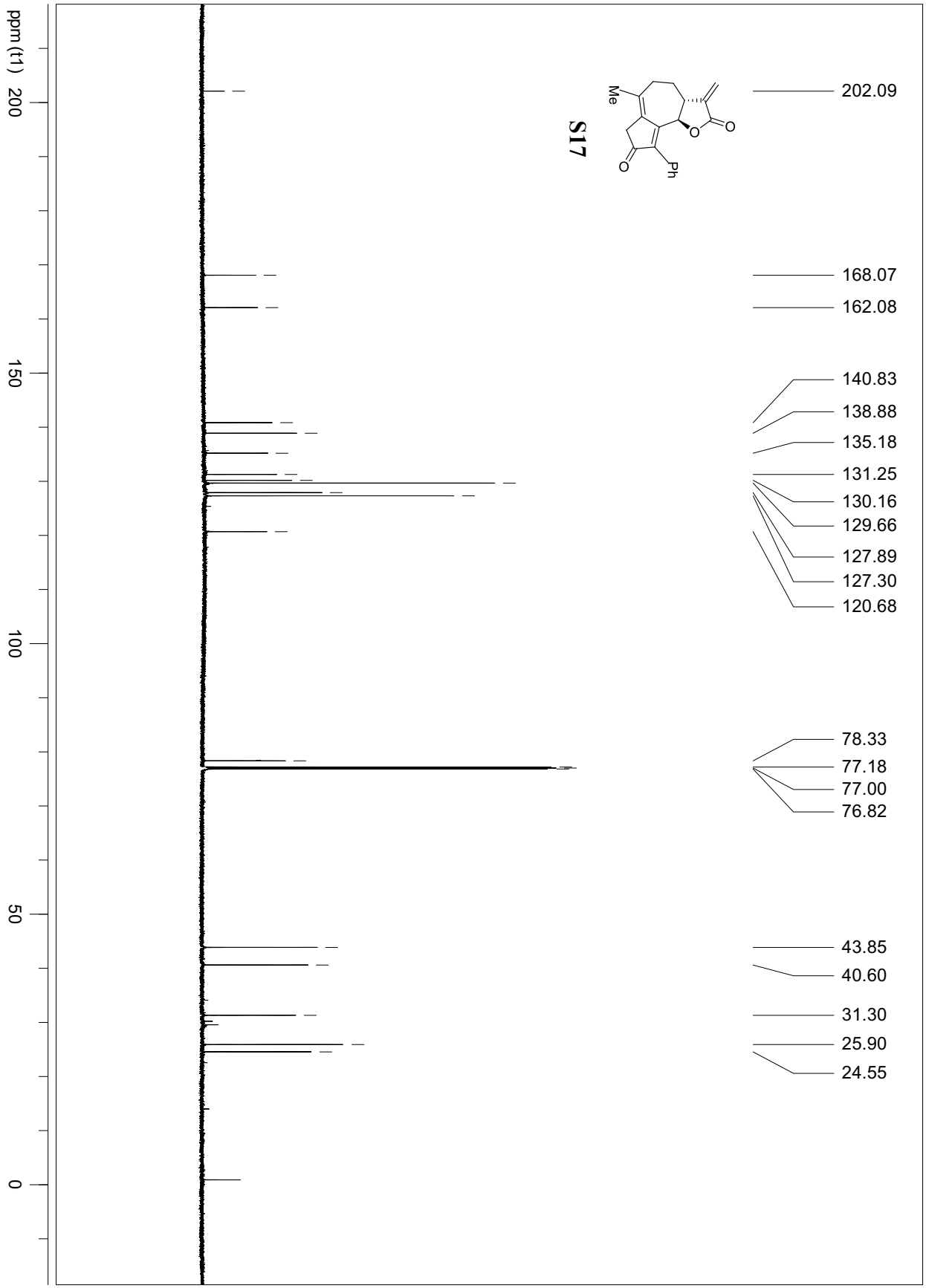

S15
AcO
Ph
O
O
O
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
5.08
1.01
1.00
0.92
0.98
0.97
3.09
0.97
3.40
3.19
7.446
7.431
7.426
7.341
7.336
7.318
7.260
6.349
6.342
5.679
5.673
4.994
4.981
3.219
3.214
3.207
3.201
2.547
2.074
2.063
2.040
2.021
2.001
1.998
1.878
1.863
1.830
1.805
1.749
1.733
1.721
1.712
1.703
1.683
1.681
1.614


ppm (t1)
200
150
100
50
0
AcO
O
O
Ph
S15
169.20
169.00
137.27
131.79
129.18
128.40
123.29
121.44
88.05
84.88
83.00
77.18
77.00
76.82
74.17
74.08
72.17
46.73
38.06
27.72
26.57
21.85
1.01
-0.01


S16
Ph
O
O
7.445
7.432
7.429
7.337
7.322
7.260
6.333
6.328
5.661
5.656
4.985
4.974
4.620
4.614
4.608
4.601
3.202
3.197
3.191
3.186
2.027
2.021
2.012
2.006
1.816
1.803
1.796
1.790
1.787
1.785
1.776
1.771
1.761
1.691
1.684
1.678
1.661
1.656
1.642
5.57
1.03
1.14
1.03
1.97
1.00
2.06
1.03
3.27
1.98
x
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0


ppm (f1)
200
150
100
50
0
S16
Ph
O
O
205.97
169.18
137.71
131.72
129.01
128.29
122.61
121.52
97.56
87.70
85.12
74.57
72.37
46.78
32.78
32.51
23.87
18.72
x


S17
Me
O
O
O
Ph
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
2.92
2.23
1.00
0.98
0.99
2.03
0.95
1.09
2.22
3.18
1.29
7.371
7.348
7.327
7.260
7.231
6.228
6.217
5.540
5.530
5.466
5.433
3.159
3.135
3.093
3.057
2.770
2.759
2.727
2.687
2.458
2.439
2.400
2.381
2.358
2.350
1.999
1.909
1.899
1.855
1.567
1.432
1.256
0.857


ppm (t1)
200
150
100
50
0
S17
Me
O
O
O
Ph
202.09
168.07
162.08
140.83
138.88
135.18
131.25
130.16
129.66
127.89
127.30
120.68
78.33
77.18
77.00
76.82
43.85
40.60
31.30
25.90
24.55

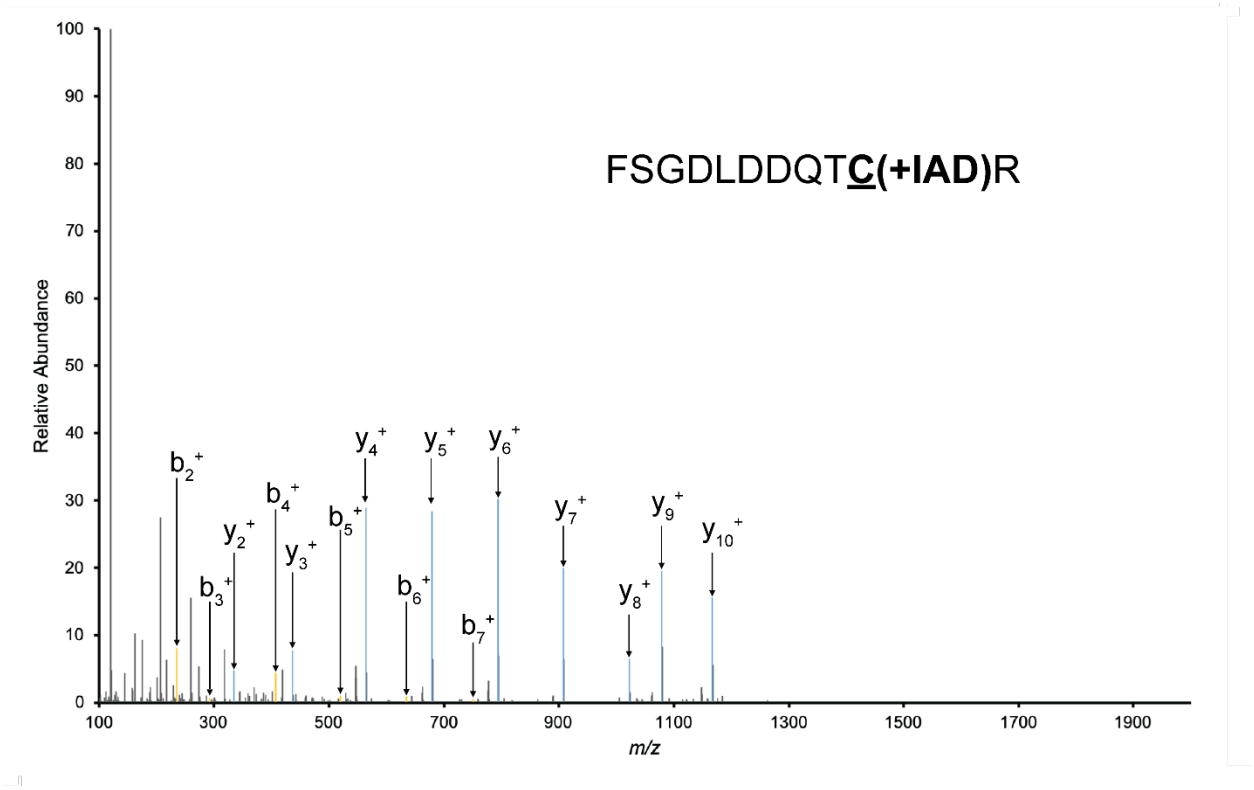



**Figure S27.** Example MS/MS spectrum from precursor ion *m/z* = 657.274 that is positively identified as a target peptide. The y- and b-ions of the carbamidomethylated FSGDLDDQT**C**$^{\mathbf{245}}$R are highlighted in blue and orange, respectively.

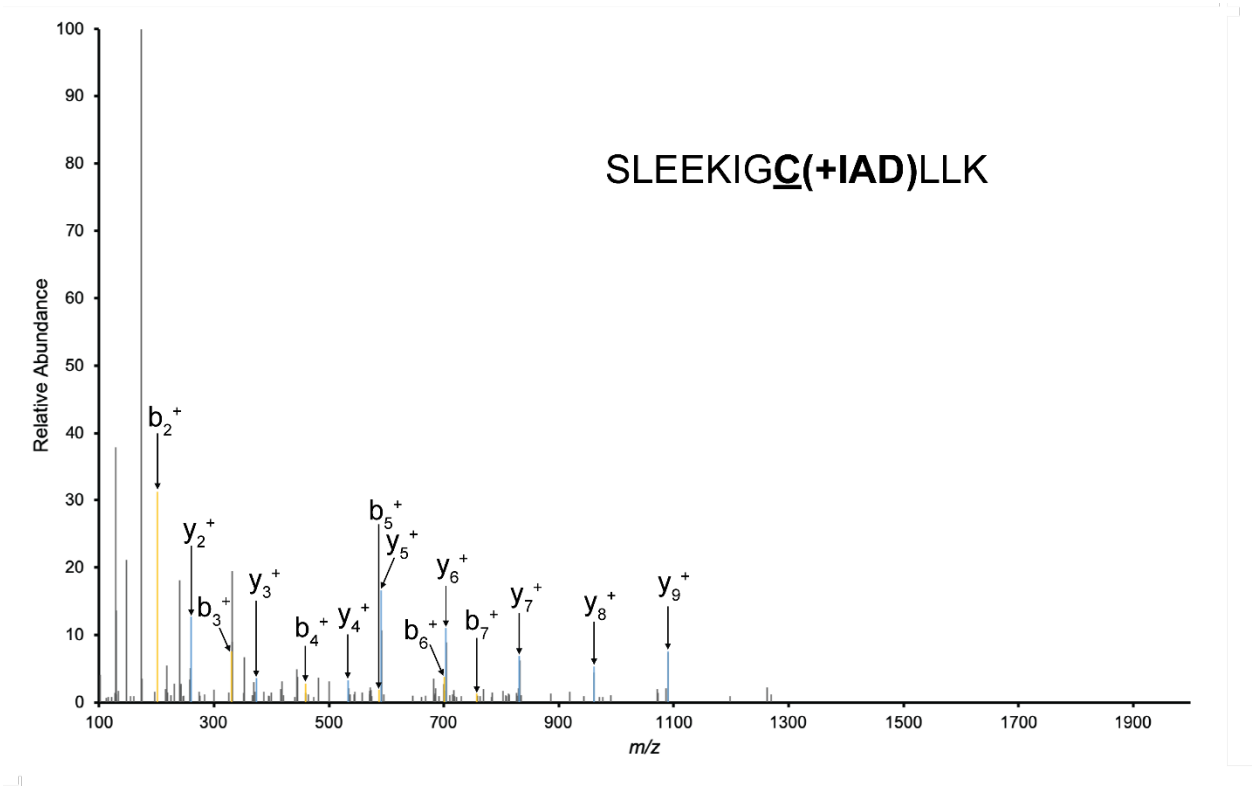



**Figure S28.** Example MS/MS spectrum from precursor ion *m/z* = 645.360 that is positively identified as a target peptide. The y- and b-ions of the carbamidomethylated SLEEKIG**C**$^{232}$LLK are highlighted in blue and orange, respectively.

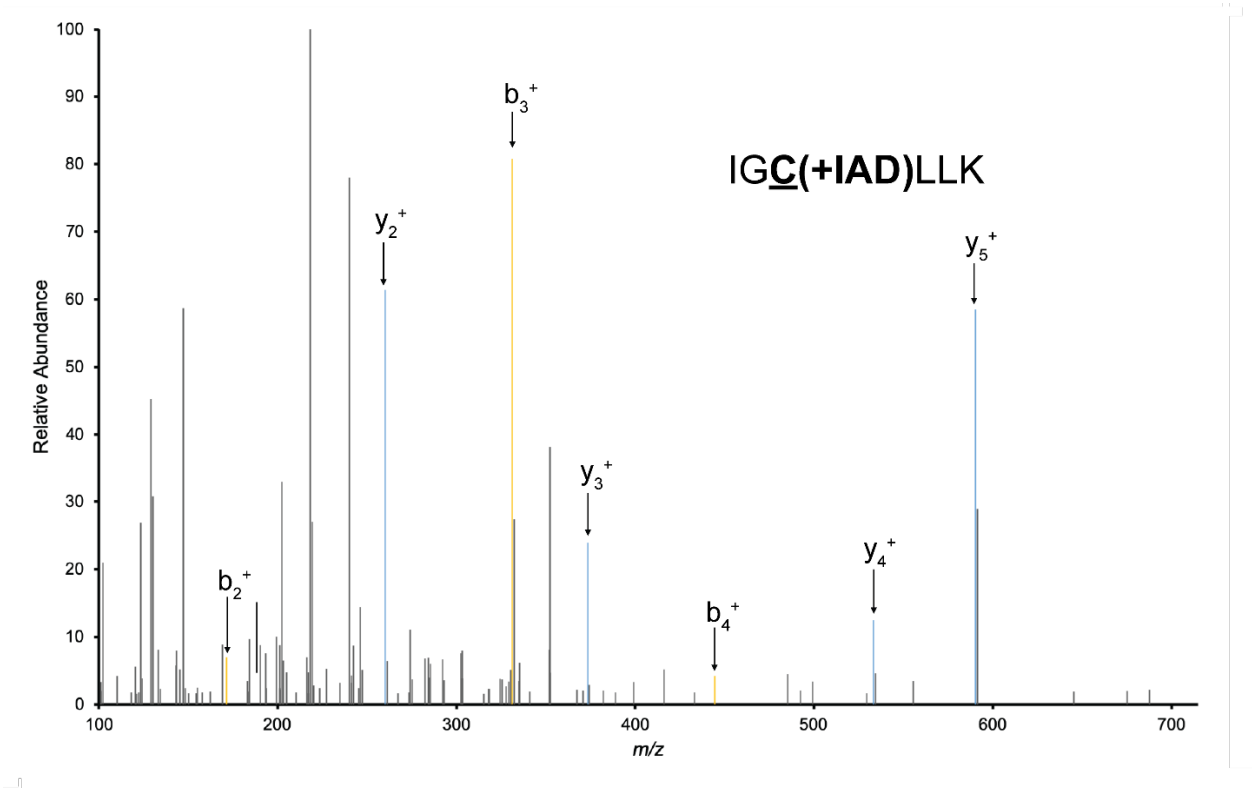



**Figure S29.** Example MS/MS spectrum from precursor ion *m/z* = 352.212 that is positively identified as a target peptide. The y- and b-ions of the carbamidomethylated IG**C**$^{232}$LLK are highlighted in blue and orange, respectively.

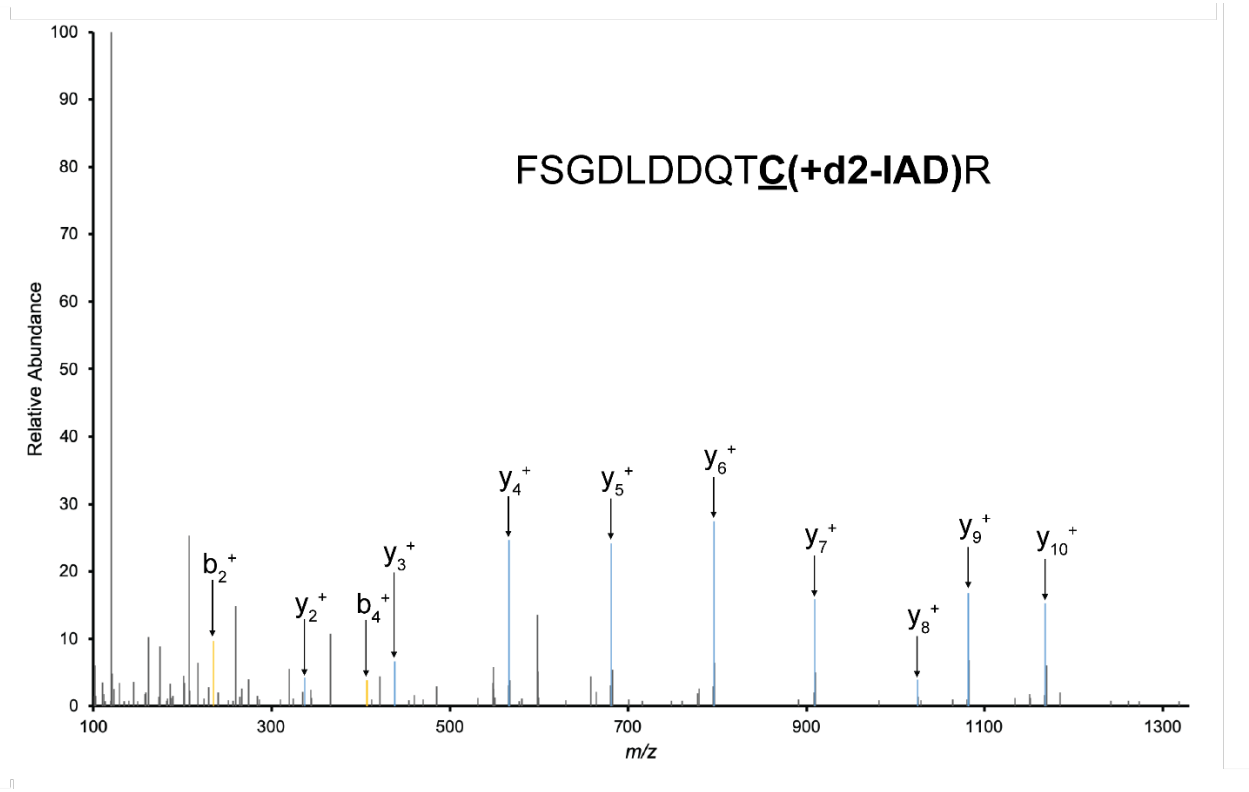



**Figure S30.** Example MS/MS spectrum from precursor ion *m/z* = 658.281 that is positively identified as a target peptide. The y- and b-ions of the $d_2$-carbamidomethylated FSGDLDDQT**C**$^{245}$R are highlighted in blue and orange, respectively.

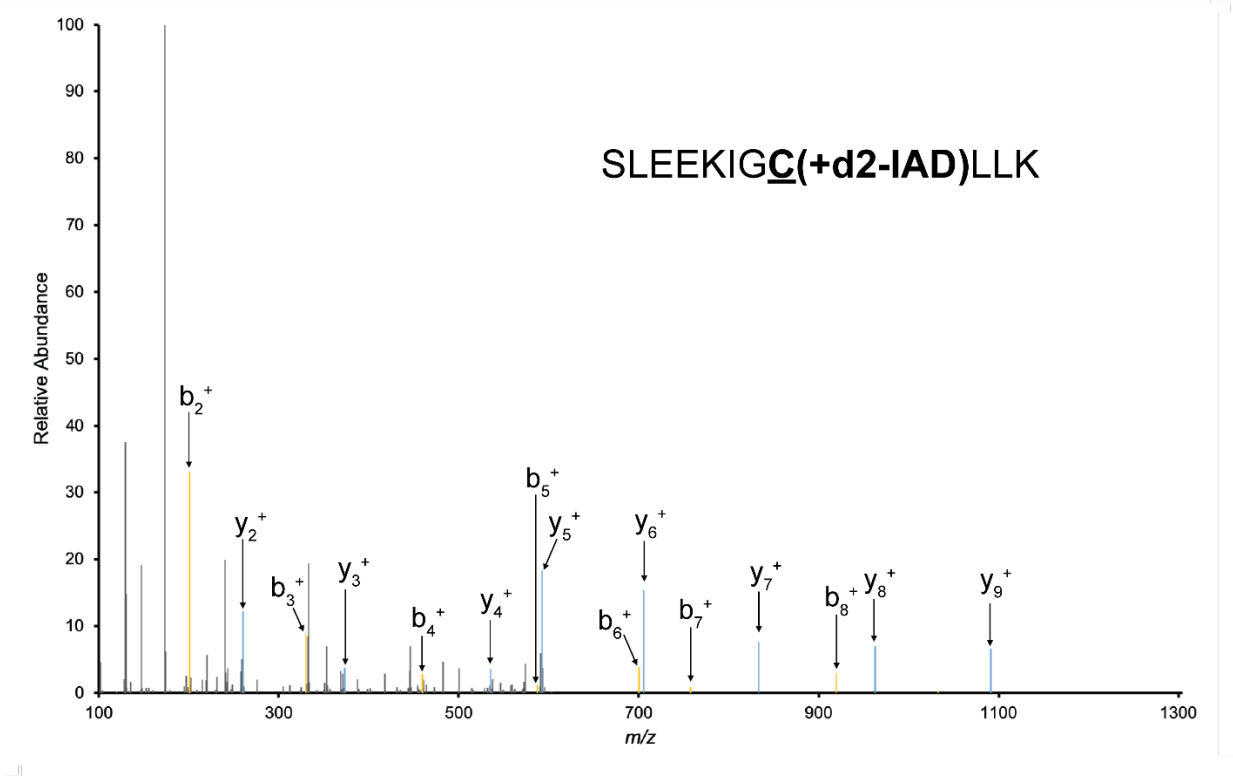



**Figure S31.** Example MS/MS spectrum from precursor ion *m/z* = 646.366 that is positively identified as a target peptide. The y- and b-ions of the $d_2$-carbamidomethylated SLEEKIG**C**$^{232}$LLK are highlighted in blue and orange, respectively.

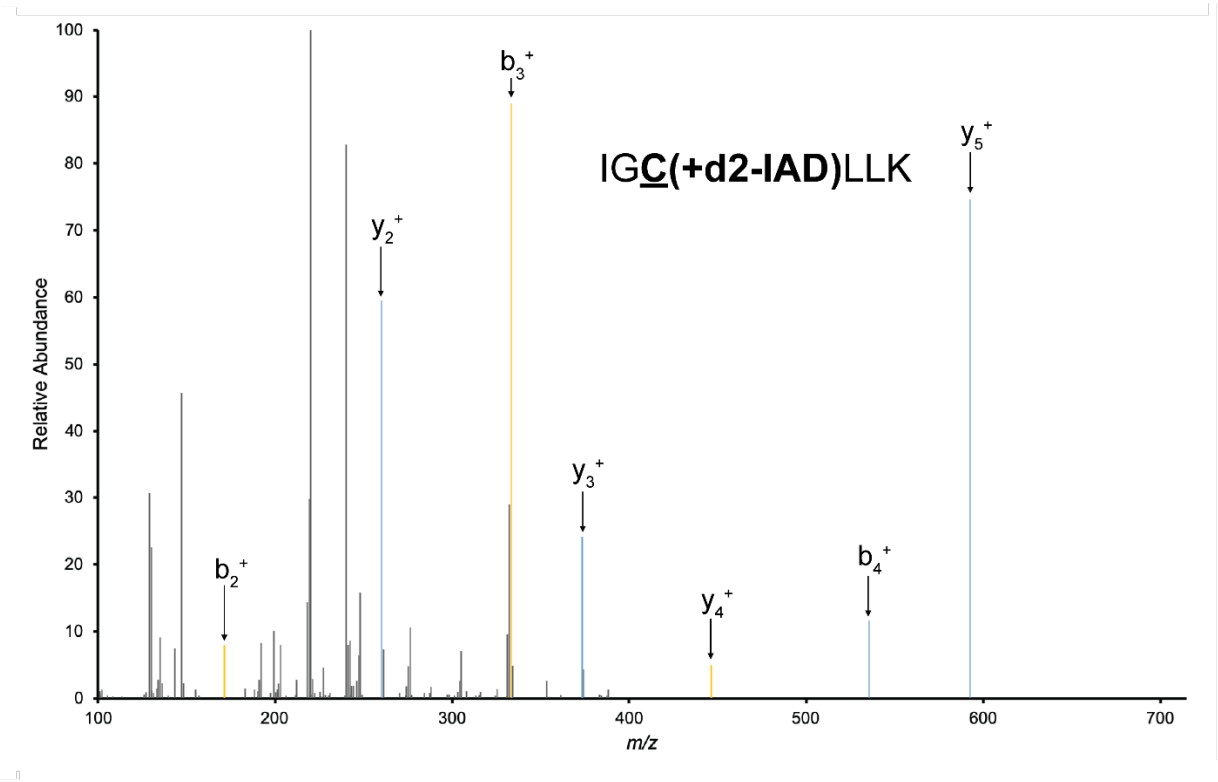



**Figure S32.** Example MS/MS spectrum from precursor ion *m/z* = 353.218 that is positively identified as a target peptide. The y- and b-ions of the $d_2$-carbamidomethylated IG**C**$^{232}$LLK are highlighted in blue and orange, respectively.

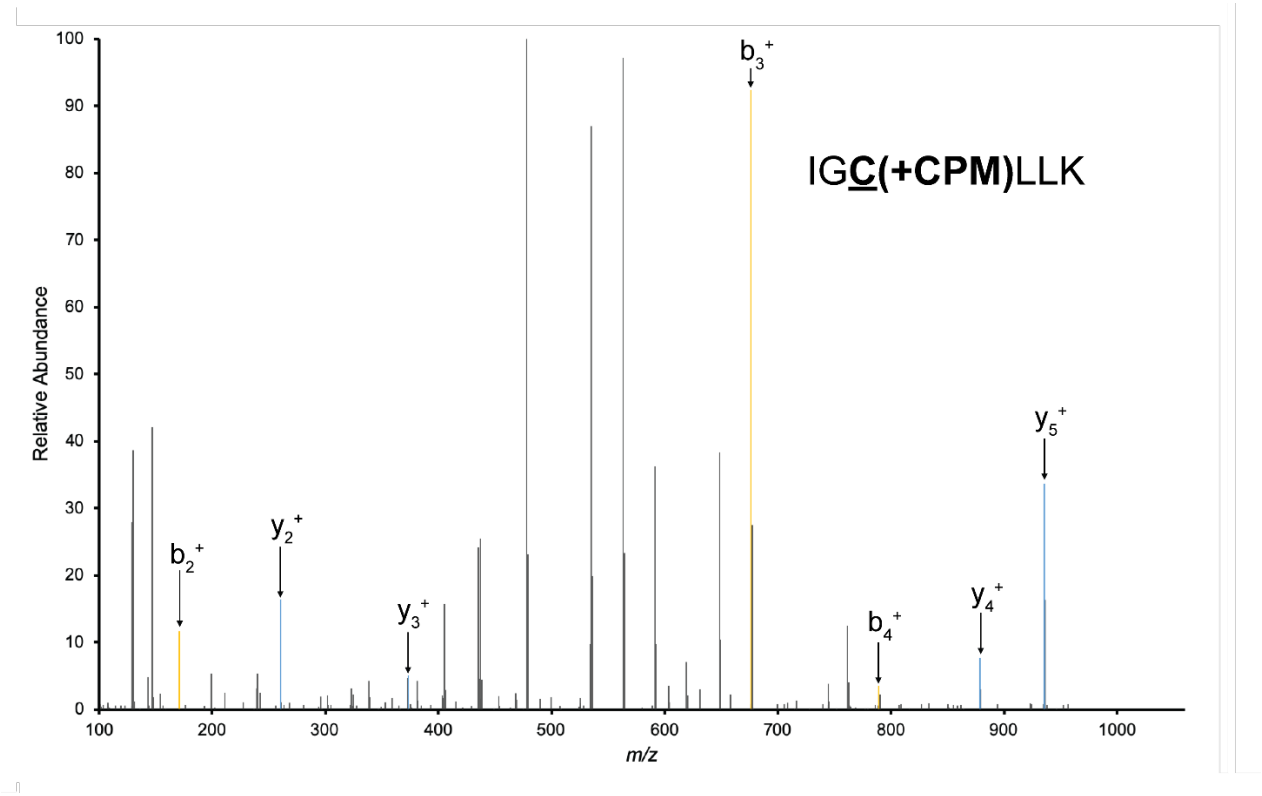



**Figure S33.** Example MS/MS spectrum from precursor ion *m/z* = 524.780 that is positively identified as a target peptide. The y- and b-ions of the CPM-adducted IG**C**$^{232}$LLK are highlighted in blue and orange, respectively.

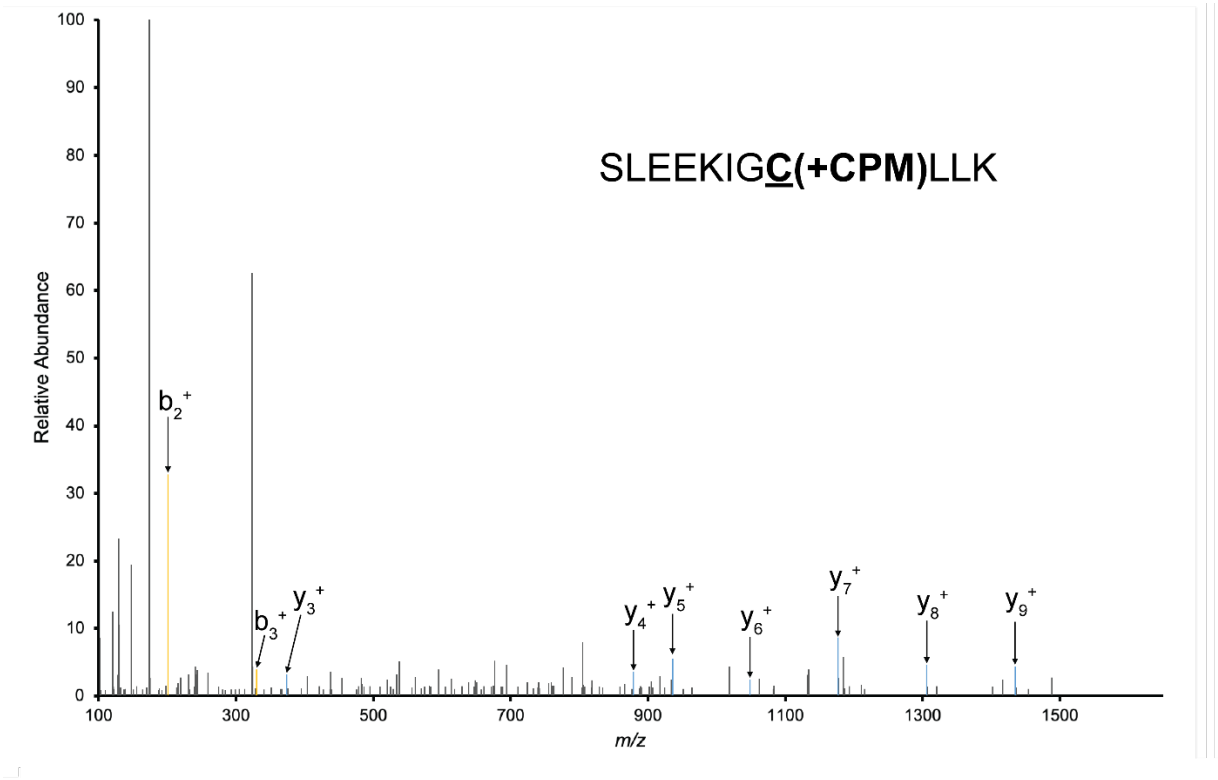



**Figure S34.** Example MS/MS spectrum from precursor ion *m/z* = 817.928 that is positively identified as a target peptide. The y- and b-ions of the CPM-adducted SLEEKIG**C**$^{232}$LLK are highlighted in blue and orange, respectively.

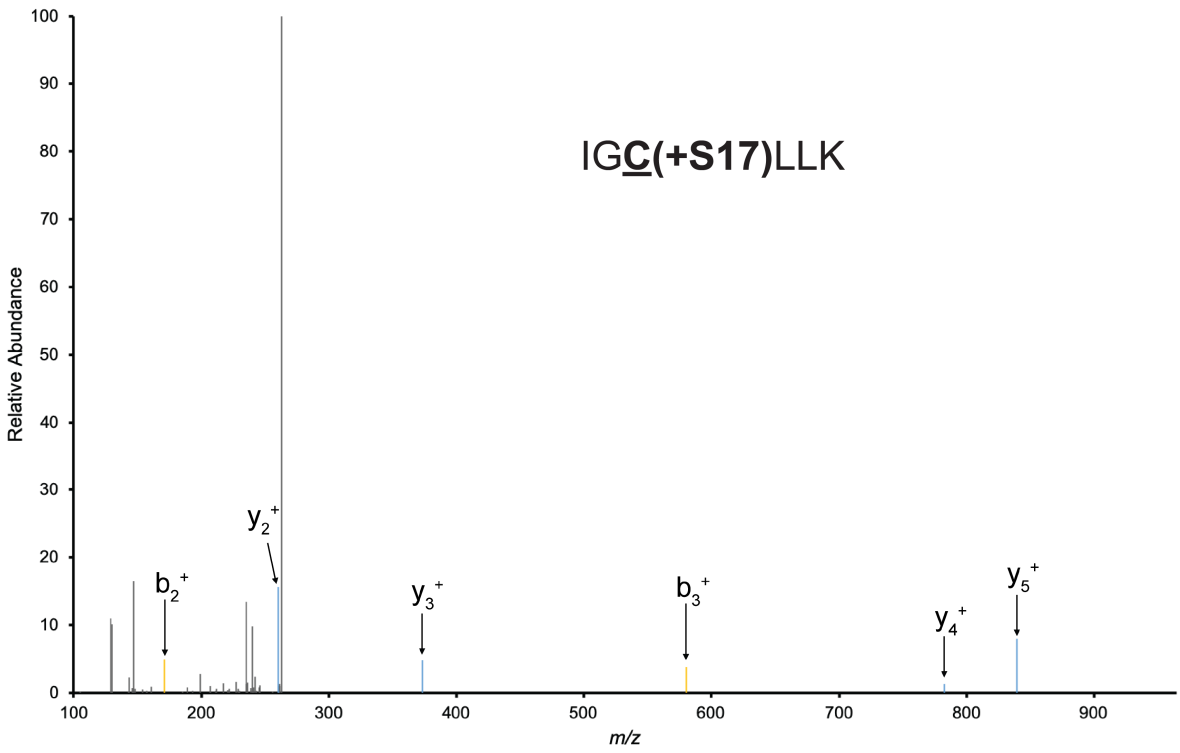



**Figure S35.** Example MS/MS spectrum from precursor ion *m/z* = 476.764 that is positively identified as a target peptide. The y- and b-ions of the **S17**-adducted IG**C**$^{232}$LLK are highlighted in blue and orange, respectively.

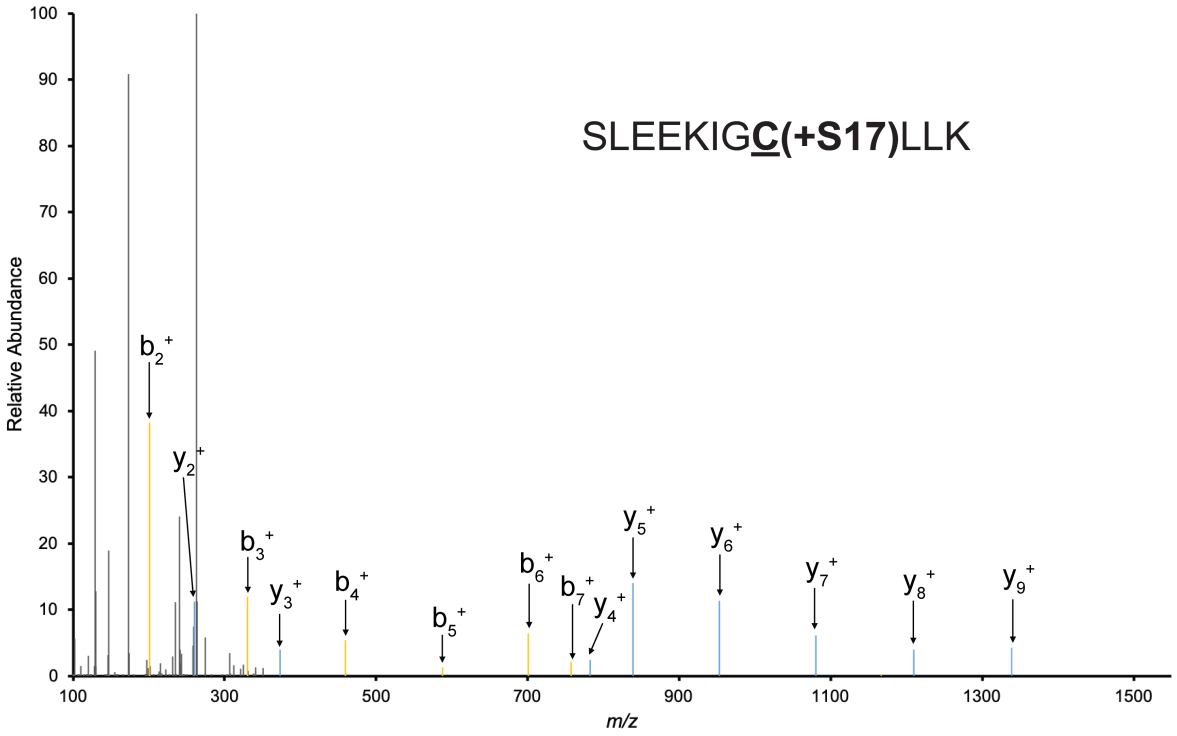



**Figure S36.** Example MS/MS spectrum from precursor ion *m/z* = 817.928 that is positively identified as a target peptide. The y- and b-ions of the **S17**-adducted SLEEKIG**C**$^{232}$LLK are highlighted in blue and orange, respectively.

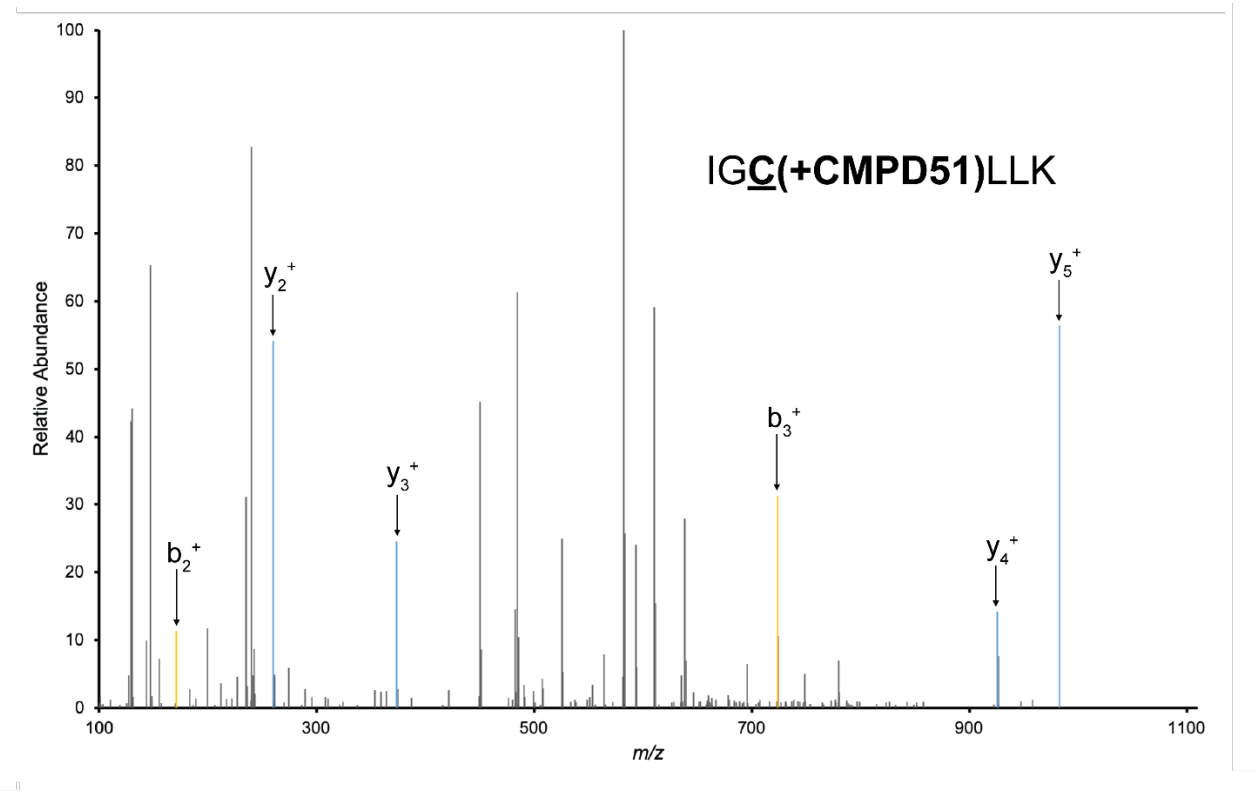



**Figure S37.** Example MS/MS spectrum from precursor ion *m/z* = 548.281 that is positively identified as a target peptide. The y- and b-ions of **51**-adducted IG**C**$^{232}$LLK are highlighted in blue and orange, respectively.

**HSQC of α-Methylene-γ-Lactam 52**