

Supporting Information
for
**Palladium-catalyzed *ortho*-halogenations of
acetanilides with *N*-halosuccinimides via direct
sp² C–H bond activation in ball mills**

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Experimental, analytical data and NMR spectra of 2a–j, 3a and 4a

Table of Contents	Page
Experimental	S2
Analytical data of compounds 2a–j, 3a and 4a	S2–S4
References	S4
NMR spectra of compounds 2a–j, 3a and 4a	S5–S14

Experimental

General methods

Nuclear magnetic resonance spectra were acquired on a BRUKER 400 AV spectrometer (400 MHz and 100 MHz for ^1H NMR and ^{13}C NMR, respectively). All ^1H NMR spectra are reported in parts per million (ppm) relative to TMS. All ^{13}C NMR spectra were reported in ppm relative to residual CHCl_3 (77.16 ppm) and were obtained with ^1H -decoupling. Data for ^1H NMR are described as following: chemical shift (δ in ppm), multiplicity (s, singlet; d, doublet; t, triplet; brs, broad signal), coupling constant (Hz), integration. High resolution mass spectra were obtained on a Thermo Finnigan LCQ Advantage MAX.

Materials

Solvents, toluene and acetone were purchased from Sinopharm Chemical Reagent Co., Ltd. (China) and used as received. Chemicals purchased from Sinopharm Chemical Reagent Co., Ltd. (China), Energy Chemical, J&K, Alfa Aesar, Aladdin were used without further purification. Acetanilides were synthesized according to the literature procedure [1].

General procedure for synthesis of 2. A mixture of acetanilide **1** (0.4 mmol), NIS (90.0 mg, 0.4 mmol), $\text{Pd}(\text{OAc})_2$ (9.0 mg, 0.04 mmol), and PTSA (152.0 mg, 0.8 mmol) was added to a 3 mL stainless-steel jar with a stainless-steel ball of 5 mm diameter. The vessel was sealed and vibrated in a Spex SamplePrep 8000 Mixer Mill at a frequency of 875 cycles per minute at room temperature for 3 h. The same reaction was repeated again. Then, the reaction mixtures from two runs were washed with acetone and collected into a round-bottomed flask together with silica gel and concentrated under reduced pressure. The residue was purified by column chromatography over silica gel (ethyl acetate/petroleum ether = 1:3) to afford the corresponding product **2**.

Products **3a** and **4a** were synthesized by the general procedure for the synthesis of **2** except that NIS was replaced by NBS and NCS, respectively.

Analytical data of compounds 2a–j, 3a and 4a

N-(2-Iodo-4-methylphenyl)acetamide (2a)

^1H NMR (400 MHz, CDCl_3): δ 7.99 (d, J = 8.1 Hz, 1H), 7.60 (s, 1H), 7.35 (brs, 1H), 7.13 (d, J = 8.1 Hz, 1H), 2.27 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 168.3, 139.1, 136.2, 135.9, 130.0, 122.3, 90.4, 24.8, 20.4. The analytical data are in accordance with the literature [2].

***N*-(2-Iodo-5-methylphenyl)acetamide (2b)**

¹H NMR (400 MHz, CDCl₃): δ 8.03 (s, 1H), 7.62 (d, *J* = 7.9 Hz, 1H), 7.36 (brs, 1H), 6.68 (d, *J* = 7.9 Hz, 1H), 2.32 (s, 3H), 2.22 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 168.3, 139.8, 138.4, 138.1, 127.2, 123.0, 86.1, 24.9, 21.4. The analytical data are in accordance with the literature [2].

***N*-(2-Iodo-4,5-dimethylphenyl)acetamide (2c)**

¹H NMR (400 MHz, CDCl₃): δ 7.90 (s, 1H), 7.52 (s, 1H), 7.28 (brs, 1H), 2.22 (s, 3H), 2.21 (s, 3H), 2.17 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 168.3, 139.1, 138.2, 136.1, 135.2, 123.7, 86.8, 24.8, 19.8, 19.0; HR-MS (+EI) calcd for C₁₀H₁₂INO (M⁺) 288.9964, found 288.9970.

***N*-(2-Iodophenyl)acetamide (2d)**

¹H NMR (400 MHz, CDCl₃): δ 8.18 (d, *J* = 7.8 Hz, 1H), 7.77 (d, *J* = 7.5 Hz, 1H), 7.43 (brs, 1H), 7.33 (td, *J* = 7.8, 1.3 Hz, 1H), 6.84 (t, *J* = 7.5 Hz, 1H), 2.23 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 168.3, 138.9, 138.4, 129.4, 126.1, 122.3, 90.1, 24.9. The analytical data are in accordance with the literature [2].

***N*-(4-Fluoro-2-iodo-5-methylphenyl)acetamide (2e)**

¹H NMR (400 MHz, CDCl₃): δ 7.95 (d, *J* = 7.4 Hz, 1H), 7.40 (d, *J* = 8.5 Hz, 1H), 7.25 (brs, 1H), 2.24 (d, *J* = 1.8 Hz, 3H), 2.22 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 168.4, 157.7 (d, *J* = 246 Hz), 134.4, 126.2 (d, *J* = 18 Hz), 124.9 (d, *J* = 5 Hz), 124.7 (d, *J* = 27 Hz), 86.0 (d, *J* = 8 Hz), 24.7, 14.8 (d, *J* = 2.8 Hz); HR-MS (+EI) calcd for C₉H₉FINO (M⁺) 292.9713, found 292.9708.

***N*-(4-Chloro-2-iodo-5-methylphenyl)acetamide (2f)**

¹H NMR (400 MHz, CDCl₃): δ 8.10 (s, 1H), 7.72 (s, 1H), 7.32 (brs, 1H), 2.33 (s, 3H), 2.23 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 168.3, 137.9, 137.6, 137.0, 130.6, 123.7, 86.1, 24.9, 20.3; HR-MS (+EI) calcd for C₉H₉ClINO (M⁺) 308.9417, found 308.9418.

***N*-(5-Chloro-2-iodo-4-methylphenyl)acetamide (2g)**

¹H NMR (400 MHz, CDCl₃): δ 8.23 (s, 1H), 7.62 (s, 1H), 7.34 (brs, 1H), 2.29 (s, 3H), 2.22 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 168.3, 139.9, 137.1, 135.4, 134.0, 122.3, 87.0, 24.9, 19.3; HR-MS (+EI) calcd for C₉H₉ClINO (M⁺) 308.9417, found 308.9412.

***N*-(5-Chloro-2-iodophenyl)acetamide (2h)**

¹H NMR (400 MHz, CDCl₃): δ 8.31 (s, 1H), 7.66 (d, *J* = 8.5 Hz, 1H), 7.42 (brs, 1H), 6.85 (dd, *J* = 8.5, 2.4 Hz, 1H), 2.24 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 168.3, 147.8, 139.3, 135.5, 126.1, 121.8, 86.6, 25.0. The analytical data are in accordance with the literature [3].

***N*-(4-Bromo-2-iodo-5-methylphenyl)acetamide (2i)**

¹H NMR (400 MHz, CDCl₃): δ 8.13 (s, 1H), 7.89 (s, 1H), 7.32 (brs, 1H), 2.36 (s, 3H), 2.23 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 168.3, 140.8, 139.5, 137.6, 123.6, 120.3, 86.4, 25.0, 23.0; HR-MS (+EI) calcd for C₉H₉BrINO (M⁺) 352.8912, found 352.8915.

N-(4-Acetyl-2-iodophenyl)acetamide (2j)

¹H NMR (400 MHz, CDCl₃): δ 8.42 (d, *J* = 8.6 Hz, 1H), 8.39 (d, *J* = 1.9 Hz, 1H), 7.92 (dd, *J* = 8.6, 1.9 Hz, 3H), 7.65 (brs, 1H), 2.56 (s, 3H), 2.28 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 195.6, 168.5, 142.2, 139.2, 134.2, 130.0, 120.3, 89.1, 26.5, 25.2. The analytical data are in accordance with the literature [4].

N-(4-acetyl-2-bromophenyl)acetamide (3a)

¹H NMR (400 MHz, CDCl₃): δ 8.16 (d, *J* = 8.3 Hz, 1H), 7.51 (brs, 1H), 7.35 (s, 1H), 7.11 (d, *J* = 8.3 Hz, 1H), 2.30 (s, 3H), 2.22 (s, 3H). The analytical data are in accordance with the literature [5].

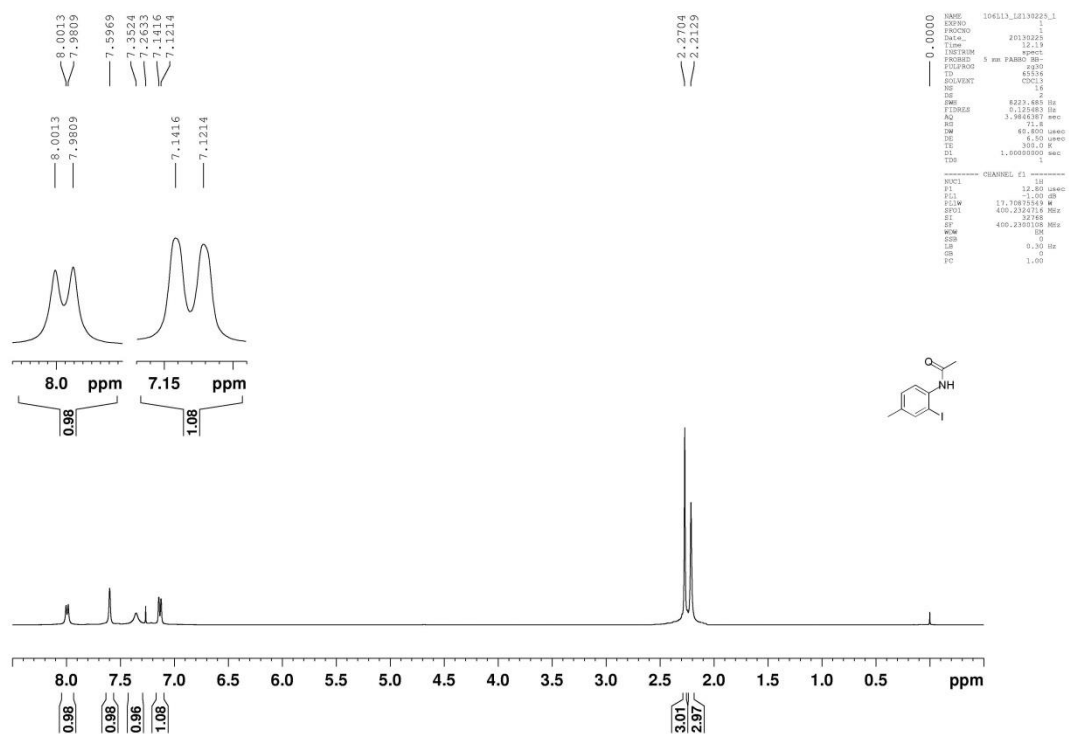
N-(2-chloro-4-methylphenyl)acetamide (4a)

¹H NMR (400 MHz, CDCl₃): δ 8.19 (d, *J* = 8.3 Hz, 1H), 7.53 (brs, 1H), 7.17 (s, 1H), 7.06 (d, *J* = 8.3 Hz, 1H), 2.29 (s, 3H), 2.22 (s, 3H). The analytical data are in accordance with the literature [5].

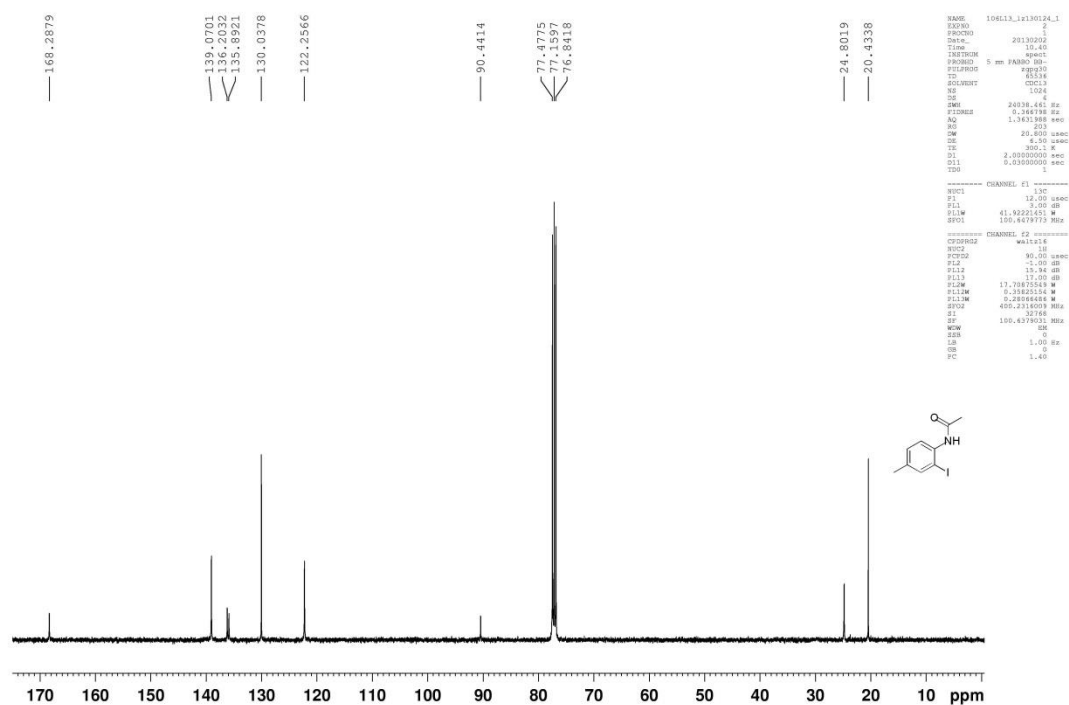
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5. Bedford, R. B.; Mitchell, C. J.; Webster, R. L. *Chem. Commun.* **2010**, 46, 3095-3097.

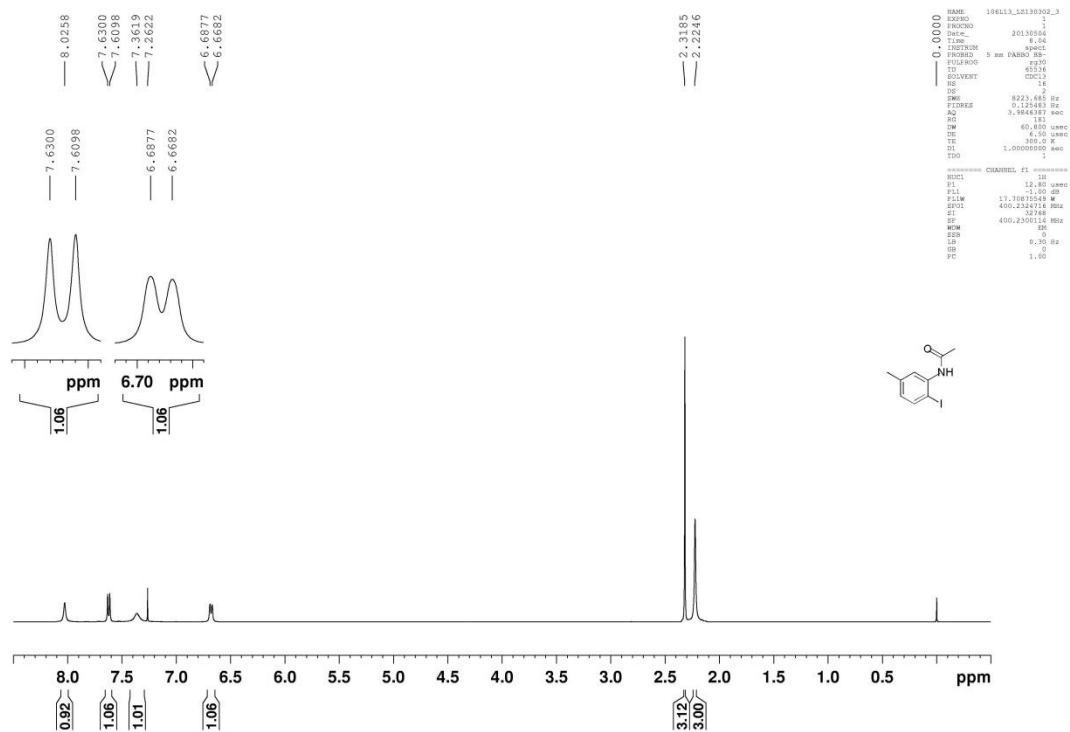
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¹³C NMR (100 MHz, CDCl₃) of compound 2a



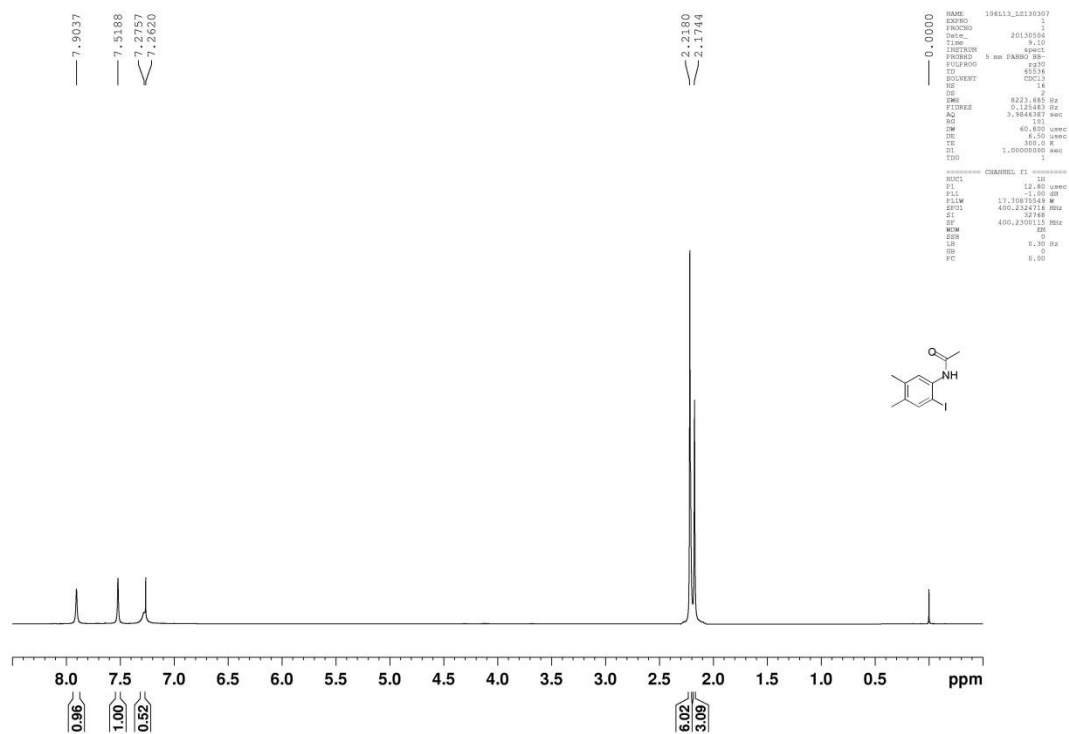
¹H NMR (400 MHz, CDCl₃) of compound 2b



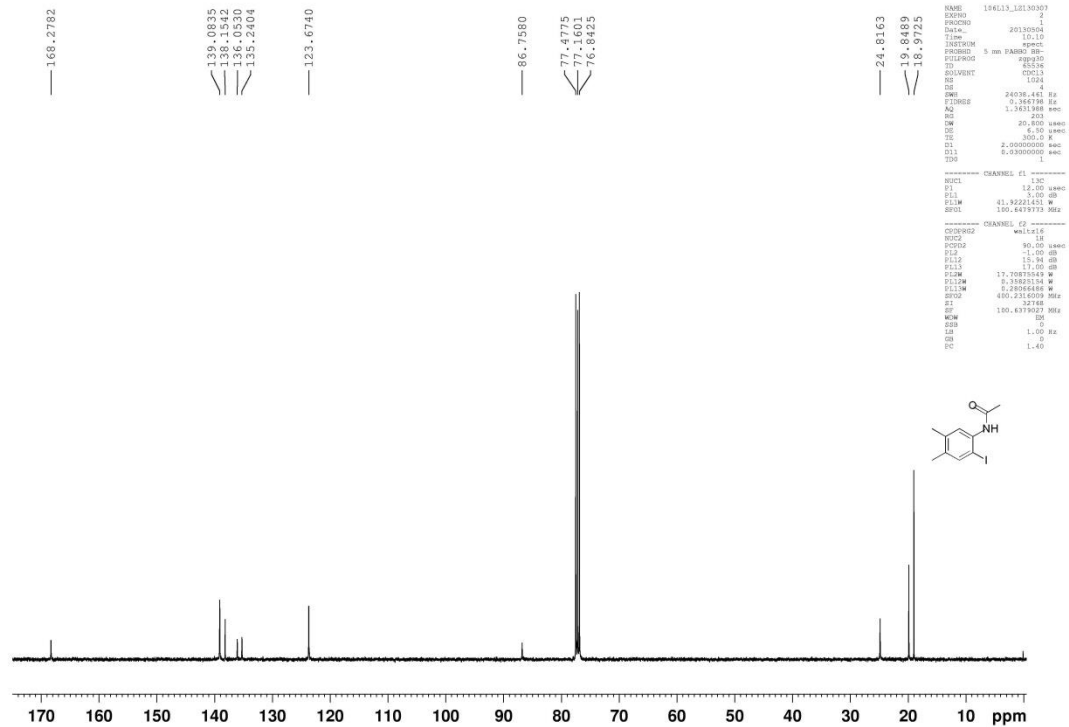
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SC         8271.885 Hz
FIDRES    0.125483 Hz
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SFO1       100.6261158 MHz
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PROCNO2    1
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Time2      8.04
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PULPROG2   zgpg30
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SOLVENT2    CDCl3
NS2         16
DS2         4
SC2         8271.885 Hz
FIDRES2    0.125483 Hz
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DE2         6.50 umsec
TE2         300.2 K
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PROCNO4    1
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NS4         16
DS4         4
SC4         8271.885 Hz
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NS6         16
DS6         4
SC6         8271.885 Hz
FIDRES6    0.125483 Hz
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RG6         181
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TE6         300.2 K
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PROCNO8    1
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Time8      8.04
INSTRUM8   spect
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PULPROG8   zgpg30
TD8         65536
SOLVENT8    CDCl3
NS8         16
DS8         4
SC8         8271.885 Hz
FIDRES8    0.125483 Hz
AQ8         9.964087 sec
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DE8         6.50 umsec
TE8         300.2 K
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TD08       1
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PL9        0.00 dB
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PROCNO10    1
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Time10     8.04
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PULPROG10   zgpg30
TD10        65536
SOLVENT10    CDCl3
NS10        16
DS10        4
SC10        8271.885 Hz
FIDRES10    0.125483 Hz
AQ10        9.964087 sec
RG10        181
RM10        60.620 umsec
DE10        6.50 umsec
TE10        300.2 K
D110        1.00000000 sec
TD010       1
===== CHANNEL f11 =====
NUC11      13C
P11         12.00 umsec
PL11        0.00 dB
PL112       17.7087049 Hz
PL113       0.2804688 Hz
SFO11       100.6261158 MHz
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EXPNO12    1
PROCNO12    1
F12 - F11    20110104
Time12     8.04
INSTRUM12   spect
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PULPROG12   zgpg30
TD12        65536
SOLVENT12    CDCl3
NS12        16
DS12        4
SC12        8271.885 Hz
FIDRES12    0.125483 Hz
AQ12        9.964087 sec
RG12        181
RM12        60.620 umsec
DE12        6.50 umsec
TE12        300.2 K
D112        1.00000000 sec
TD012       1
===== CHANNEL f13 =====
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P13         12.00 umsec
PL13        0.00 dB
PL132       17.7087049 Hz
PL133       0.2804688 Hz
SFO13       100.6261158 MHz
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PROCNO14    1
F14 - F13    20110104
Time14     8.04
INSTRUM14   spect
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PULPROG14   zgpg30
TD14        65536
SOLVENT14    CDCl3
NS14        16
DS14        4
SC14        8271.885 Hz
FIDRES14    0.125483 Hz
AQ14        9.964087 sec
RG14        181
RM14        60.620 umsec
DE14        6.50 umsec
TE14        300.2 K
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PL15        0.00 dB
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===== CHANNEL f16 =====
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PROCNO16    1
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Time16     8.04
INSTRUM16   spect
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TD16        65536
SOLVENT16    CDCl3
NS16        16
DS16        4
SC16        8271.885 Hz
FIDRES16    0.125483 Hz
AQ16        9.964087 sec
RG16        181
RM16        60.620 umsec
DE16        6.50 umsec
TE16        300.2 K
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NUC17      13C
P17         12.00 umsec
PL17        0.00 dB
PL172       17.7087049 Hz
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SFO17       100.6261158 MHz
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PROCNO18    1
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Time18     8.04
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PULPROG18   zgpg30
TD18        65536
SOLVENT18    CDCl3
NS18        16
DS18        4
SC18        8271.885 Hz
FIDRES18    0.125483 Hz
AQ18        9.964087 sec
RG18        181
RM18        60.620 umsec
DE18        6.50 umsec
TE18        300.2 K
D118        1.00000000 sec
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PL193       0.2804688 Hz
SFO19       100.6261158 MHz
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PROCNO20    1
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Time20     8.04
INSTRUM20   spect
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SOLVENT20    CDCl3
NS20        16
DS20        4
SC20        8271.885 Hz
FIDRES20    0.125483 Hz
AQ20        9.964087 sec
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TE20        300.2 K
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PL212       17.7087049 Hz
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PROCNO22    1
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INSTRUM22   spect
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DS22        4
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FIDRES22    0.125483 Hz
AQ22        9.964087 sec
RG22        181
RM22        60.620 umsec
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PROCNO24    1
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Time24     8.04
INSTRUM24   spect
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PULPROG24   zgpg30
TD24        65536
SOLVENT24    CDCl3
NS24        16
DS24        4
SC24        8271.885 Hz
FIDRES24    0.125483 Hz
AQ24        9.964087 sec
RG24        181
RM24        60.620 umsec
DE24        6.50 umsec
TE24        300.2 K
D124        1.00000000 sec
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PL253       0.2804688 Hz
SFO25       100.6261158 MHz
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PROCNO26    1
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Time26     8.04
INSTRUM26   spect
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PULPROG26   zgpg30
TD26        65536
SOLVENT26    CDCl3
NS26        16
DS26        4
SC26        8271.885 Hz
FIDRES26    0.125483 Hz
AQ26        9.964087 sec
RG26        181
RM26        60.620 umsec
DE26        6.50 umsec
TE26        300.2 K
D126        1.00000000 sec
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PL272       17.7087049 Hz
PL273       0.2804688 Hz
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PROCNO28    1
F28 - F27    20110104
Time28     8.04
INSTRUM28   spect
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PULPROG28   zgpg30
TD28        65536
SOLVENT28    CDCl3
NS28        16
DS28        4
SC28        8271.885 Hz
FIDRES28    0.125483 Hz
AQ28        9.964087 sec
RG28        181
RM28        60.620 umsec
DE28        6.50 umsec
TE28        300.2 K
D128        1.00000000 sec
TD028       1
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SFO29       100.6261158 MHz
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EXPNO30    1
PROCNO30    1
F30 - F29    20110104
Time30     8.04
INSTRUM30   spect
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PULPROG30   zgpg30
TD30        65536
SOLVENT30    CDCl3
NS30        16
DS30        4
SC30        8271.885 Hz
FIDRES30    0.125483 Hz
AQ30        9.964087 sec
RG30        181
RM30        60.620 umsec
DE30        6.50 umsec
TE30        300.2 K
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TD030       1
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PL312       17.7087049 Hz
PL313       0.2804688 Hz
SFO31       100.6261158 MHz
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PROCNO32    1
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Time32     8.04
INSTRUM32   spect
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TD32        65536
SOLVENT32    CDCl3
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DS32        4
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FIDRES32    0.125483 Hz
AQ32        9.964087 sec
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TE32        300.2 K
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PROCNO34    1
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Time34     8.04
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TD34        65536
SOLVENT34    CDCl3
NS34        16
DS34        4
SC34        8271.885 Hz
FIDRES34    0.125483 Hz
AQ34        9.964087 sec
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RM34        60.620 umsec
DE34        6.50 umsec
TE34        300.2 K
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P35         12.00 umsec
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PL353       0.2804688 Hz
SFO35       100.6261158 MHz
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EXPNO36    1
PROCNO36    1
F36 - F35    20110104
Time36     8.04
INSTRUM36   spect
PROBHD36    5 mm PABBO BB-
PULPROG36   zgpg30
TD36        65536
SOLVENT36    CDCl3
NS36        16
DS36        4
SC36        8271.885 Hz
FIDRES36    0.125483 Hz
AQ36        9.964087 sec
RG36        181
RM36        60.620 umsec
DE36        6.50 umsec
TE36        300.2 K
D136        1.00000000 sec
TD036       1
===== CHANNEL f37 =====
NUC37      13C
P37         12.00 umsec
PL37        0.00 dB
PL372       17.7087049 Hz
PL373       0.2804688 Hz
SFO37       100.6261158 MHz
===== CHANNEL f38 =====
EXPNO38    1
PROCNO38    1
F38 - F37    20110104
Time38     8.04
INSTRUM38   spect
PROBHD38    5 mm PABBO BB-
PULPROG38   zgpg30
TD38        65536
SOLVENT38    CDCl3
NS38        16
DS38        4
SC38        8271.885 Hz
FIDRES38    0.125483 Hz
AQ38        9.964087 sec
RG38        181
RM38        60.620 umsec
DE38        6.50 umsec
TE38        300.2 K
D138        1.00000000 sec
TD038       1
===== CHANNEL f39 =====
NUC39      13C
P39         12.00 umsec
PL39        0.00 dB
PL392       17.7087049 Hz
PL393       0.2804688 Hz
SFO39       100.6261158 MHz
===== CHANNEL f40 =====
EXPNO40    1
PROCNO40    1
F40 - F39    20110104
Time40     8.04
INSTRUM40   spect
PROBHD40    5 mm PABBO BB-
PULPROG40   zgpg30
TD40        65536
SOLVENT40    CDCl3
NS40        16
DS40        4
SC40        8271.885 Hz
FIDRES40    0.125483 Hz
AQ40        9.964087 sec
RG40        181
RM40        60.620 umsec
DE40        6.50 umsec
TE40        300.2 K
D140        1.00000000 sec
TD040       1
===== CHANNEL f41 =====
NUC41      13C
P41         12.00 umsec
PL41        0.00 dB
PL412       17.7087049 Hz
PL413       0.2804688 Hz
SFO41       100.6261158 MHz
===== CHANNEL f42 =====
EXPNO42    1
PROCNO42    1
F42 - F41    20110104
Time42     8.04
INSTRUM42   spect
PROBHD42    5 mm PABBO BB-
PULPROG42   zgpg30
TD42        65536
SOLVENT42    CDCl3
NS42        16
DS42        4
SC42        8271.885 Hz
FIDRES42    0.125483 Hz
AQ42        9.964087 sec
RG42        181
RM42        60.620 umsec
DE42        6.50 umsec
TE42        300.2 K
D142        1.00000000 sec
TD042       1
===== CHANNEL f43 =====
NUC43      13C
P43         12.00 umsec
PL43        0.00 dB
PL432       17.7087049 Hz
PL433       0.2804688 Hz
SFO43       100.6261158 MHz
===== CHANNEL f44 =====
EXPNO44    1
PROCNO44    1
F44 - F43    20110104
Time44     8.04
INSTRUM44   spect
PROBHD44    5 mm PABBO BB-
PULPROG44   zgpg30
TD44        65536
SOLVENT44    CDCl3
NS44        16
DS44        4
SC44        8271.885 Hz
FIDRES44    0.125483 Hz
AQ44        9.964087 sec
RG44        181
RM44        60.620 umsec
DE44        6.50 umsec
TE44        300.2 K
D144        1.00000000 sec
TD044       1
===== CHANNEL f45 =====
NUC45      13C
P45         12.00 umsec
PL45        0.00 dB
PL452       17.7087049 Hz
PL453       0.2804688 Hz
SFO45       100.6261158 MHz
===== CHANNEL f46 =====
EXPNO46    1
PROCNO46    1
F46 - F45    20110104
Time46     8.04
INSTRUM46   spect
PROBHD46    5 mm PABBO BB-
PULPROG46   zgpg30
TD46        65536
SOLVENT46    CDCl3
NS46        16
DS46        4
SC46        8271.885 Hz
FIDRES46    0.125483 Hz
AQ46        9.964087 sec
RG46        181
RM46        60.620 umsec
DE46        6.50 umsec
TE46        300.2 K
D146        1.00000000 sec
TD046       1
===== CHANNEL f47 =====
NUC47      13C
P47         12.00 umsec
PL47        0.00 dB
PL472       17.7087049 Hz
PL473       0.2804688 Hz
SFO47       100.6261158 MHz
===== CHANNEL f48 =====
EXPNO48    1
PROCNO48    1
F48 - F47    20110104
Time48     8.04
INSTRUM48   spect
PROBHD48    5 mm PABBO BB-
PULPROG48   zgpg30
TD48        65536
SOLVENT48    CDCl3
NS48        16
DS48        4
SC48        8271.885 Hz
FIDRES48    0.125483 Hz
AQ48        9.964087 sec
RG48        181
RM48        60.620 umsec
DE48        6.50 umsec
TE48        300.2 K
D148        1.00000000 sec
TD048       1
===== CHANNEL f49 =====
NUC49      13C
P49         12.00 umsec
PL49        0.00 dB
PL492       17.7087049 Hz
PL493       0.2804688 Hz
SFO49       100.6261158 MHz
===== CHANNEL f50 =====
EXPNO50    1
PROCNO50    1
F50 - F49    20110104
Time50     8.04
INSTRUM50   spect
PROBHD50    5 mm PABBO BB-
PULPROG50   zgpg30
TD50        65536
SOLVENT50    CDCl3
NS50        16
DS50        4
SC50        8271.885 Hz
FIDRES50    0.125483 Hz
AQ50        9.964087 sec
RG50        181
RM50        60.620 umsec
DE50        6.50 umsec
TE50        300.2 K
D150        1.00000000 sec
TD050       1
===== CHANNEL f51 =====
NUC51      13C
P51         12.00 umsec
PL51        0.00 dB
PL512       17.7087049 Hz
PL513       0.2804688 Hz
SFO51       100.6261158 MHz
===== CHANNEL f52 =====
EXPNO52    1
PROCNO52    1
F52 - F51    20110104
Time52     8.04
INSTRUM52   spect
PROBHD52    5 mm PABBO BB-
PULPROG52   zgpg30
TD52        65536
SOLVENT52    CDCl3
NS52        16
DS52        4
SC52        8271.885 Hz
FIDRES52    0.125483 Hz
AQ52        9.964087 sec
RG52        181
RM52        60.620 umsec
DE52        6.50 umsec
TE52        300.2 K
D152        1.00000000 sec
TD052       1
===== CHANNEL f53 =====
NUC53      13C
P53         12.00 umsec
PL53        0.00 dB
PL532       17.7087049 Hz
PL533       0.2804688 Hz
SFO53       100.6261158 MHz
===== CHANNEL f54 =====
EXPNO54    1
PROCNO54    1
F54 - F53    20110104
Time54     8.04
INSTRUM54   spect
PROBHD54    5 mm PABBO BB-
PULPROG54   zgpg30
TD54        65536
SOLVENT54    CDCl3
NS54        16
DS54        4
SC54        8271.885 Hz
FIDRES54    0.125483 Hz
AQ54        9.964087 sec
RG54        181
RM54        60.620 umsec
DE54        6.50 umsec
TE54        300.2 K
D154        1.00000000 sec
TD054       1
===== CHANNEL f55 =====
NUC55      13C
P55         12.00 umsec
PL55        0.00 dB
PL552       17.7087049 Hz
PL553       0.2804688 Hz
SFO55       100.6261158 MHz
===== CHANNEL f56 =====
EXPNO56    1
PROCNO56    1
F56 - F55    20110104
Time56     8.04
INSTRUM56   spect
PROBHD56    5 mm PABBO BB-
PULPROG56   zgpg30
TD56        65536
SOLVENT56    CDCl3
NS56        16
DS56        4
SC56        8271.885 Hz
FIDRES56    0.125483 Hz
AQ56        9.964087 sec
RG56        181
RM56        60.620 umsec
DE56        6.50 umsec
TE56        300.2 K
D156        1.00000000 sec
TD056       1
===== CHANNEL f57 =====
NUC57      13C
P57         12.00 umsec
PL57        0.00 dB
PL572       17.7087049 Hz
PL573       0.2804688 Hz
SFO57       100.6261158 MHz
===== CHANNEL f58 =====
EXPNO58    1
PROCNO58    1
F58 - F57    20110104
Time58     8.04
INSTRUM58   spect
PROBHD58    5 mm PABBO BB-
PULPROG58   zgpg30
TD58        65536
SOLVENT58    CDCl3
NS58        16
DS58        4
SC58        8271.885 Hz
FIDRES58    0.125483 Hz
AQ58        9.964087 sec
RG58        181
RM58        60.620 umsec
DE58        6.50 umsec
TE58        300.2 K
D158        1.00000000 sec
TD058       1
===== CHANNEL f59 =====
NUC59      13C
P59         12.00 umsec
PL59        0.00 dB
PL592       17.7087049 Hz
PL593       0.2804688 Hz
SFO59       100.6261158 MHz
===== CHANNEL f60 =====
EXPNO60    1
PROCNO60    1
F60 - F59    20110104
Time60     8.04
INSTRUM60   spect
PROBHD60    5 mm PABBO BB-
PULPROG60   zgpg30
TD60        65536
SOLVENT60    CDCl3
NS60        16
DS60        4
SC60        8271.885 Hz
FIDRES60    0.125483 Hz
AQ60        9.964087 sec
RG60        181
RM60        60.620 umsec
DE60        6.50 umsec
TE60        300.2 K
D160        1.00000000 sec
TD060       1
===== CHANNEL f61 =====
NUC61      13C
P61         12.00 umsec
PL61        0.00 dB
PL612       17.7087049 Hz
PL613       0.2804688 Hz
SFO61       100.6261158 MHz
===== CHANNEL f62 =====
EXPNO62    1
PROCNO62    1
F62 - F61    20110104
Time62     8.04
INSTRUM62   spect
PROBHD62    5 mm PABBO BB-
PULPROG62   zgpg30
TD62        65536
SOLVENT62    CDCl3
NS62        16
DS62        4
SC62        8271.885 Hz
FIDRES62    0.125483 Hz
AQ62        9.964087 sec
RG62        181
RM62        60.620 umsec
DE62        6.50 umsec
TE62        300.2 K
D162        1.00000000 sec
TD062       1
===== CHANNEL f63 =====
NUC63      13C
P63         12.00 umsec
PL63        0.00 dB
PL632       17.7087049 Hz
PL633       0.2804688 Hz
SFO63       100.6261158 MHz
===== CHANNEL f64 =====
EXPNO64    1
PROCNO64    1
F64 - F63    20110104
Time64     8.04
INSTRUM64   spect
PROBHD64    5 mm PABBO BB-
PULPROG64   zgpg30
TD64        65536
SOLVENT64    CDCl3
NS64        16
DS64        4
SC64        8271.885 Hz
FIDRES64    0.125483 Hz
AQ64        9.964087 sec
RG64        181
RM64        60.620 umsec
DE64        6.50 umsec
TE64        300.2 K
D164        1.00000000 sec
TD064       1
===== CHANNEL f65 =====
NUC65      13C
P65         12.00 umsec
PL65        0.00 dB
PL652       17.7087049 Hz
PL653       0.2804688 Hz
SFO65       100.6261158 MHz
===== CHANNEL f66 =====
EXPNO66    1
PROCNO66    1
F66 - F65    20110104
Time66     8.04
INSTRUM66   spect
PROBHD66    5 mm PABBO BB-
PULPROG66   zgpg30
TD66        65536
SOLVENT66    CDCl3
NS66        16
DS66        4
SC66        8271.885 Hz
FIDRES66    0.125483 Hz
AQ66        9.964087 sec
RG66        181
RM66        60.620 umsec
DE66        6.50 umsec
TE66        300.2 K
D166        1.00000000 sec
TD066       1
===== CHANNEL f67 =====
NUC67      13C
P67         12.00 umsec
PL67        0.00 dB
PL672       17.7087049 Hz
PL673       0.2804688 Hz
SFO67       100.6261158 MHz
===== CHANNEL f68 =====
EXPNO68    1
PROCNO68    1
F68 - F67    20110104
Time68     8.04
INSTRUM68   spect
PROBHD68    5 mm PABBO BB-
PULPROG68   zgpg30
TD68        65536
SOLVENT68    CDCl3
NS68        16
DS68        4
SC68        8271.885 Hz
FIDRES68    0.125483 Hz
AQ68        9.964087 sec
RG68        181
RM68        60.620 umsec
DE68        6.50 umsec
TE68        300.2 K
D168        1.00000000 sec
TD068       1
===== CHANNEL f69 =====
NUC69      13C
P69         12.00 umsec
PL69        0.00 dB
PL692       17.7087049 Hz
PL693       0.2804688 Hz
SFO69       100.6261158 MHz
===== CHANNEL f70 =====
EXPNO70    1
PROCNO70    1
F70 - F69    20110104
Time70     8.04
INSTRUM70   spect
PROBHD70    5 mm PABBO BB-
PULPROG70   zgpg30
TD70        65536
SOLVENT70    CDCl3
NS70        16
DS70        4
SC70        8271.8
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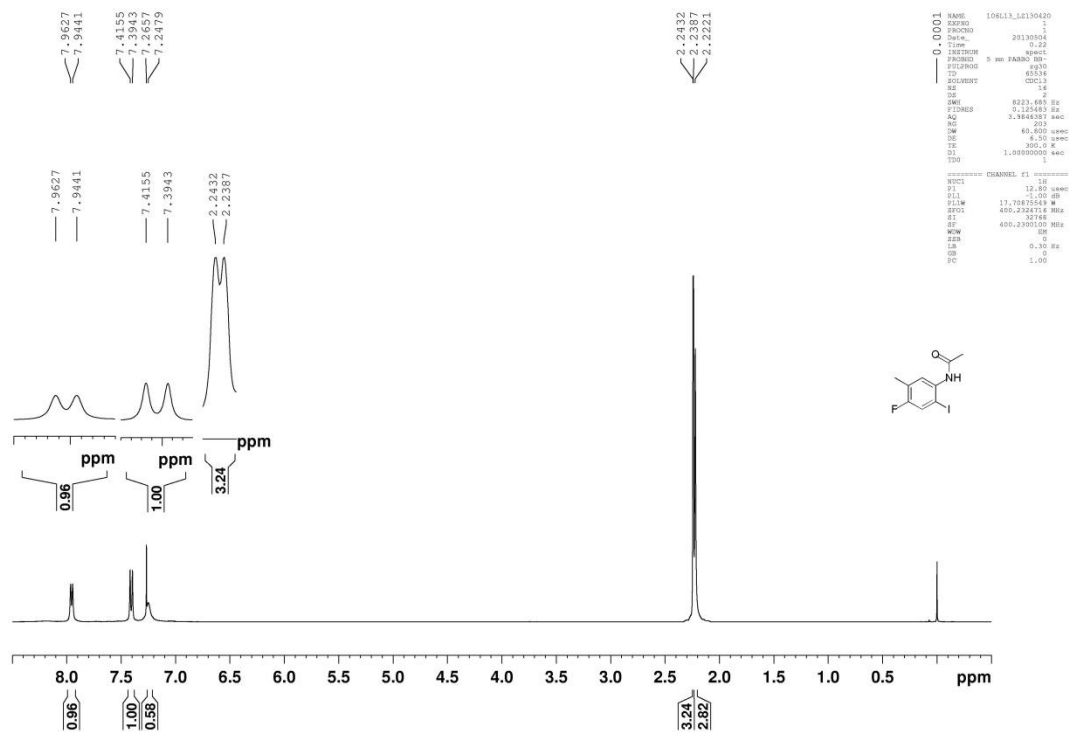
¹H NMR (400 MHz, CDCl₃) of compound 2c



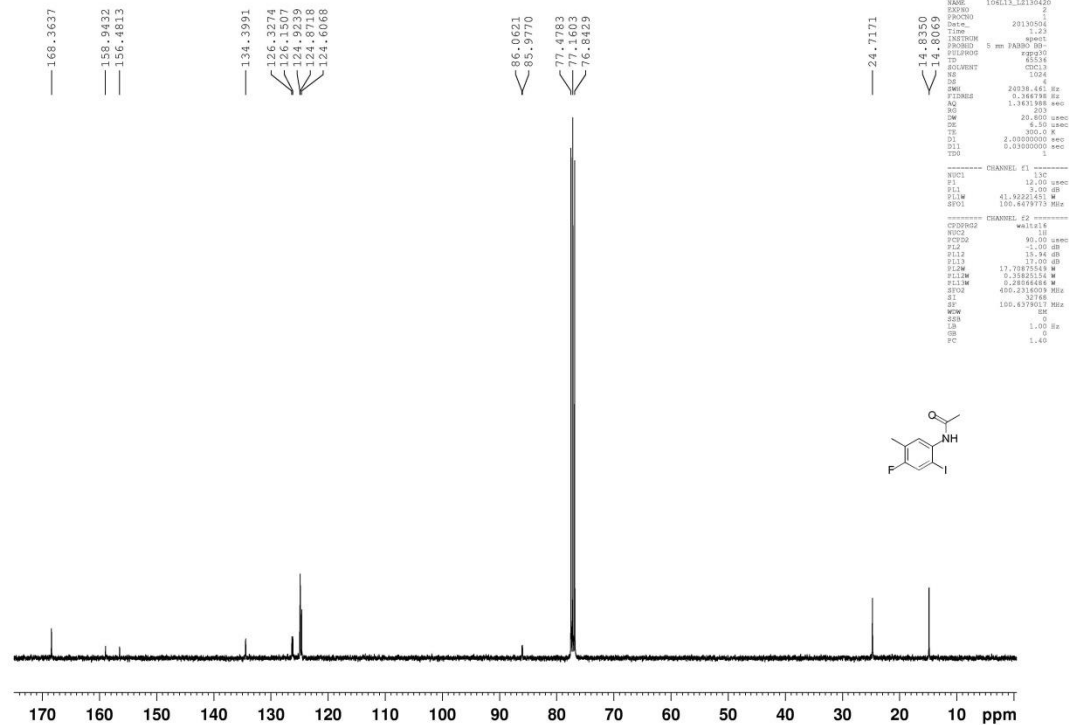
¹³C NMR (100 MHz, CDCl₃) of compound 2c



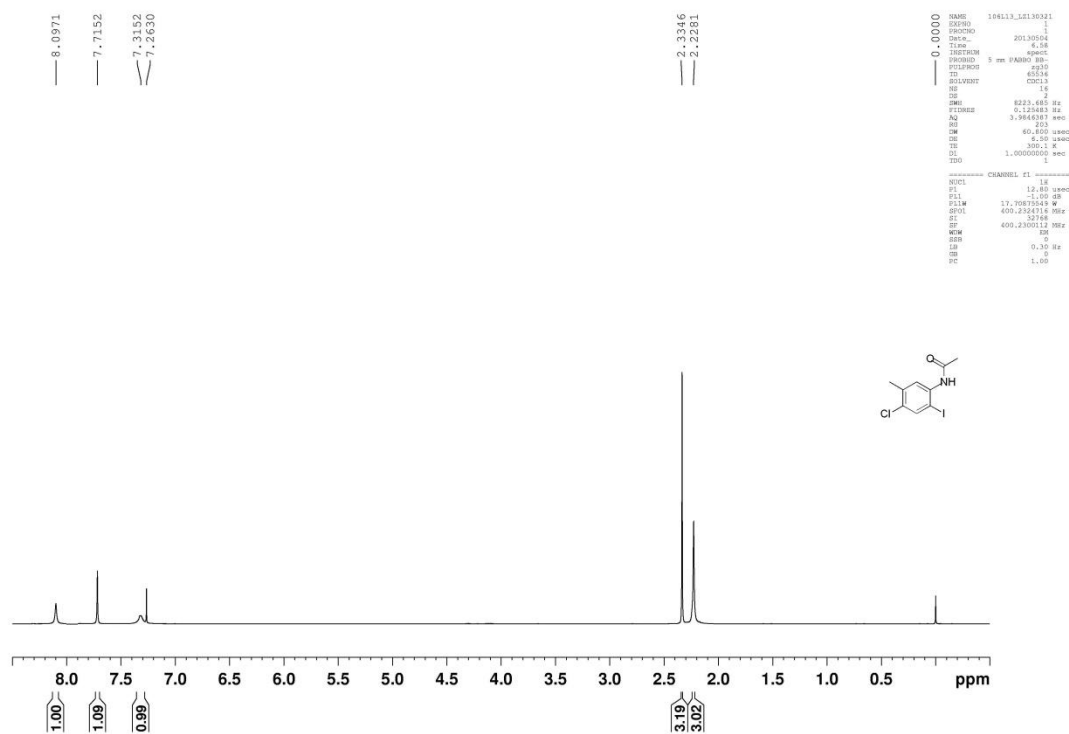
¹H NMR (400 MHz, CDCl₃) of compound 2e



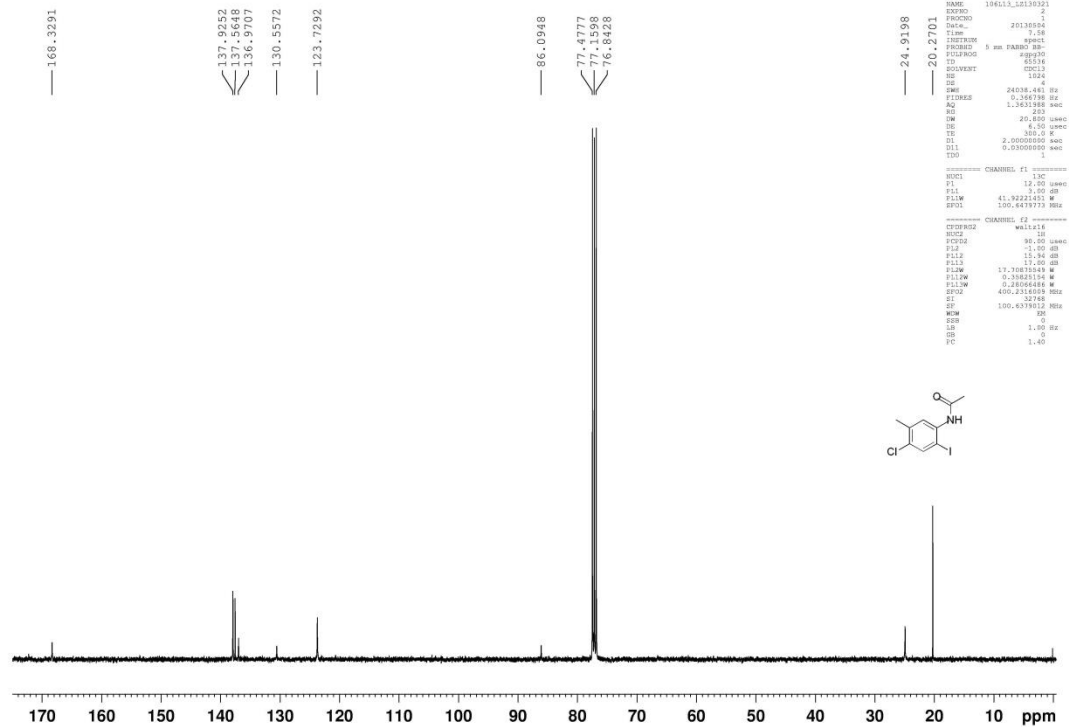
¹³C NMR (100 MHz, CDCl₃) of compound 2e



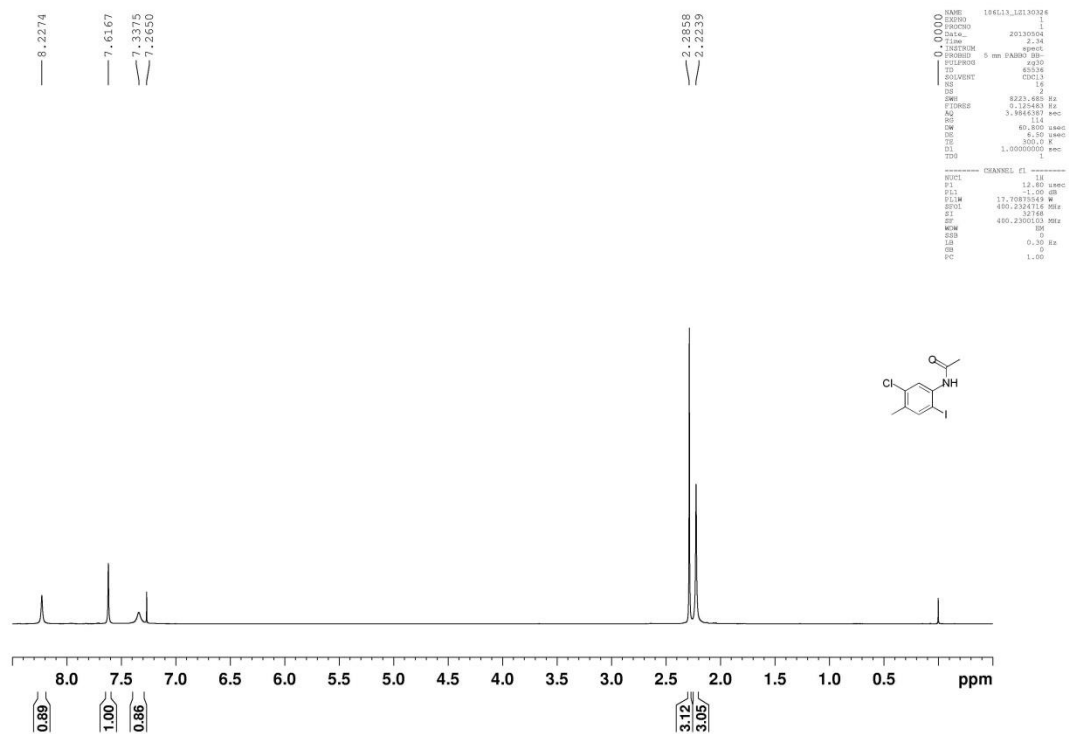
¹H NMR (400 MHz, CDCl₃) of compound 2f



¹³C NMR (100 MHz, CDCl₃) of compound 2f



¹H NMR (400 MHz, CDCl₃) of compound 2g

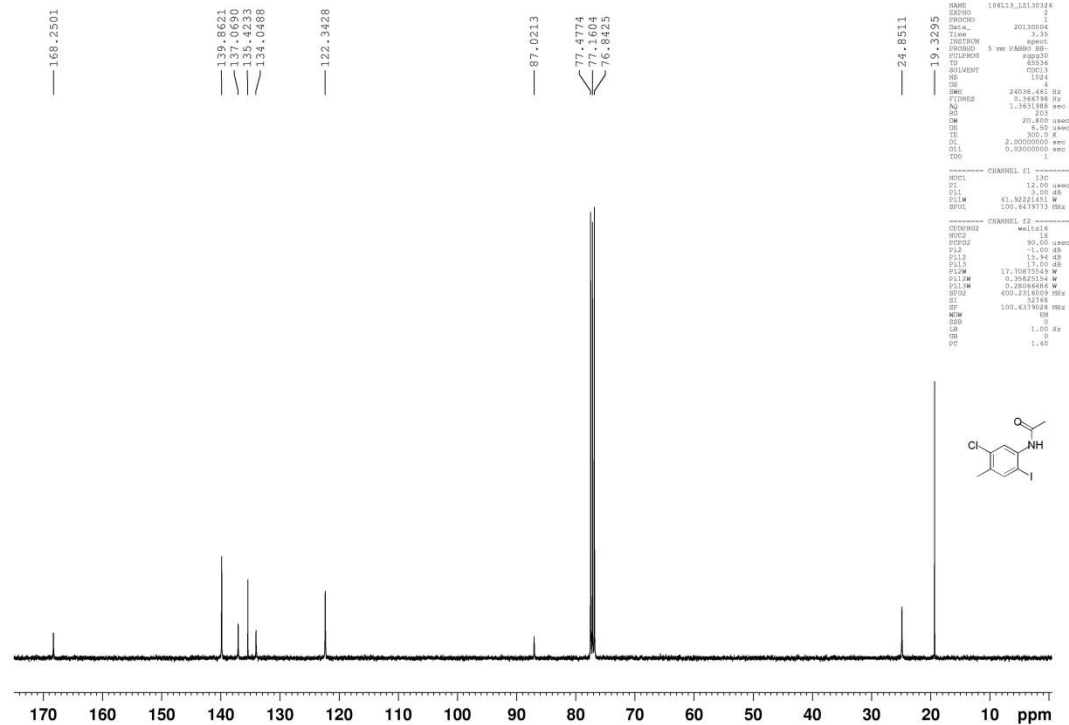


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PROCNO    1
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F3        2.34
F4        2.34
F5        2.34
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F7        2.34
F8        2.34
F9        2.34
F10       2.34
F11       2.34
F12       2.34
F13       2.34
F14       2.34
F15       2.34
F16       2.34
F17       2.34
F18       2.34
F19       2.34
F20       2.34
F21       2.34
F22       2.34
F23       2.34
F24       2.34
F25       2.34
F26       2.34
F27       2.34
F28       2.34
F29       2.34
F30       2.34
F31       2.34
F32       2.34
F33       2.34
F34       2.34
F35       2.34
F36       2.34
F37       2.34
F38       2.34
F39       2.34
F40       2.34
F41       2.34
F42       2.34
F43       2.34
F44       2.34
F45       2.34
F46       2.34
F47       2.34
F48       2.34
F49       2.34
F50       2.34
F51       2.34
F52       2.34
F53       2.34
F54       2.34
F55       2.34
F56       2.34
F57       2.34
F58       2.34
F59       2.34
F60       2.34
F61       2.34
F62       2.34
F63       2.34
F64       2.34
F65       2.34
F66       2.34
F67       2.34
F68       2.34
F69       2.34
F70       2.34
F71       2.34
F72       2.34
F73       2.34
F74       2.34
F75       2.34
F76       2.34
F77       2.34
F78       2.34
F79       2.34
F80       2.34
F81       2.34
F82       2.34
F83       2.34
F84       2.34
F85       2.34
F86       2.34
F87       2.34
F88       2.34
F89       2.34
F90       2.34
F91       2.34
F92       2.34
F93       2.34
F94       2.34
F95       2.34
F96       2.34
F97       2.34
F98       2.34
F99       2.34
F100      2.34

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¹³C NMR (100 MHz, CDCl₃) of compound 2g

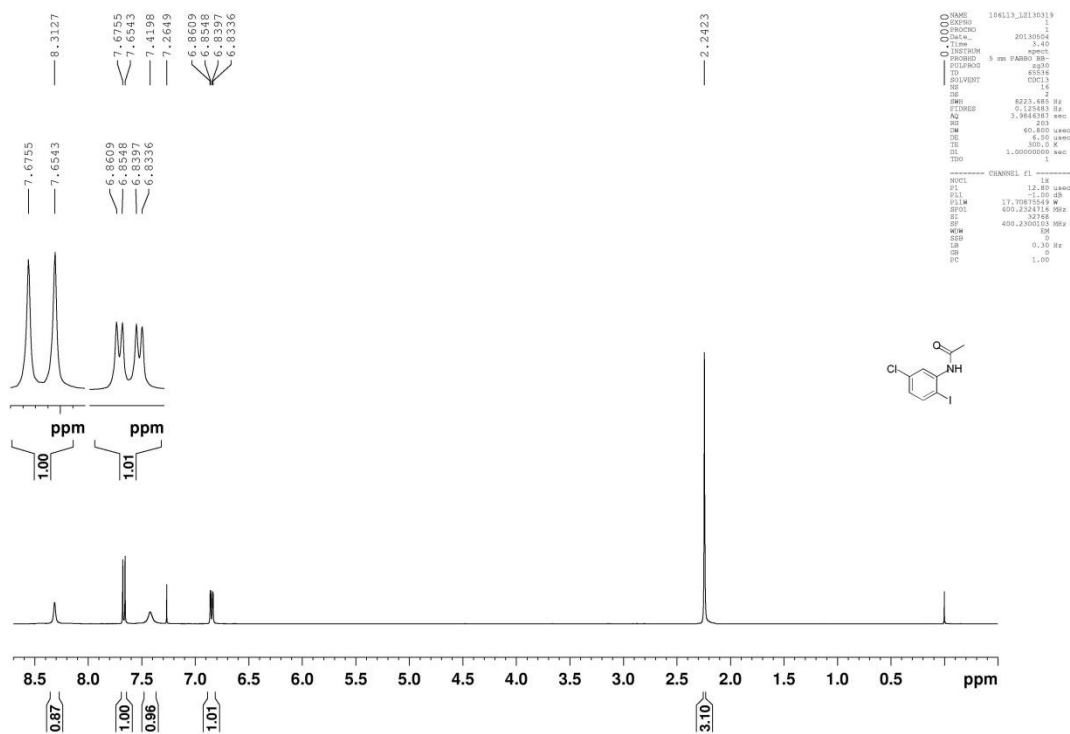


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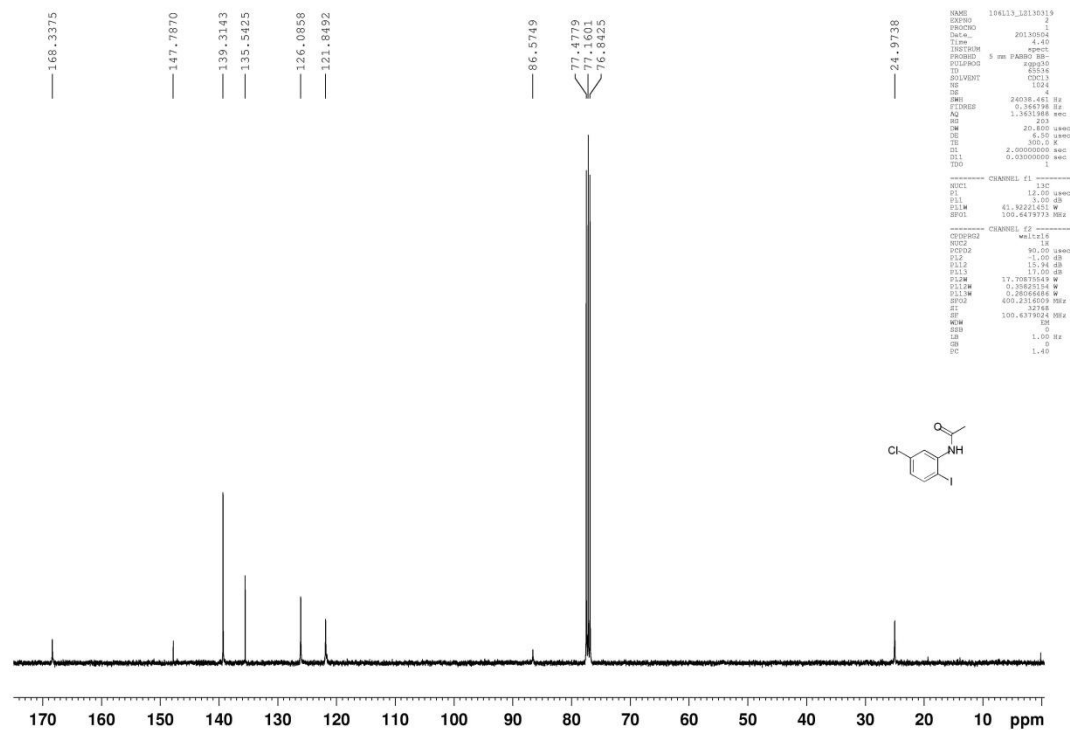
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PROCNO    1
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F3        2.34
F4        2.34
F5        2.34
F6        2.34
F7        2.34
F8        2.34
F9        2.34
F10       2.34
F11       2.34
F12       2.34
F13       2.34
F14       2.34
F15       2.34
F16       2.34
F17       2.34
F18       2.34
F19       2.34
F20       2.34
F21       2.34
F22       2.34
F23       2.34
F24       2.34
F25       2.34
F26       2.34
F27       2.34
F28       2.34
F29       2.34
F30       2.34
F31       2.34
F32       2.34
F33       2.34
F34       2.34
F35       2.34
F36       2.34
F37       2.34
F38       2.34
F39       2.34
F40       2.34
F41       2.34
F42       2.34
F43       2.34
F44       2.34
F45       2.34
F46       2.34
F47       2.34
F48       2.34
F49       2.34
F50       2.34
F51       2.34
F52       2.34
F53       2.34
F54       2.34
F55       2.34
F56       2.34
F57       2.34
F58       2.34
F59       2.34
F60       2.34
F61       2.34
F62       2.34
F63       2.34
F64       2.34
F65       2.34
F66       2.34
F67       2.34
F68       2.34
F69       2.34
F70       2.34
F71       2.34
F72       2.34
F73       2.34
F74       2.34
F75       2.34
F76       2.34
F77       2.34
F78       2.34
F79       2.34
F80       2.34
F81       2.34
F82       2.34
F83       2.34
F84       2.34
F85       2.34
F86       2.34
F87       2.34
F88       2.34
F89       2.34
F90       2.34
F91       2.34
F92       2.34
F93       2.34
F94       2.34
F95       2.34
F96       2.34
F97       2.34
F98       2.34
F99       2.34
F100      2.34

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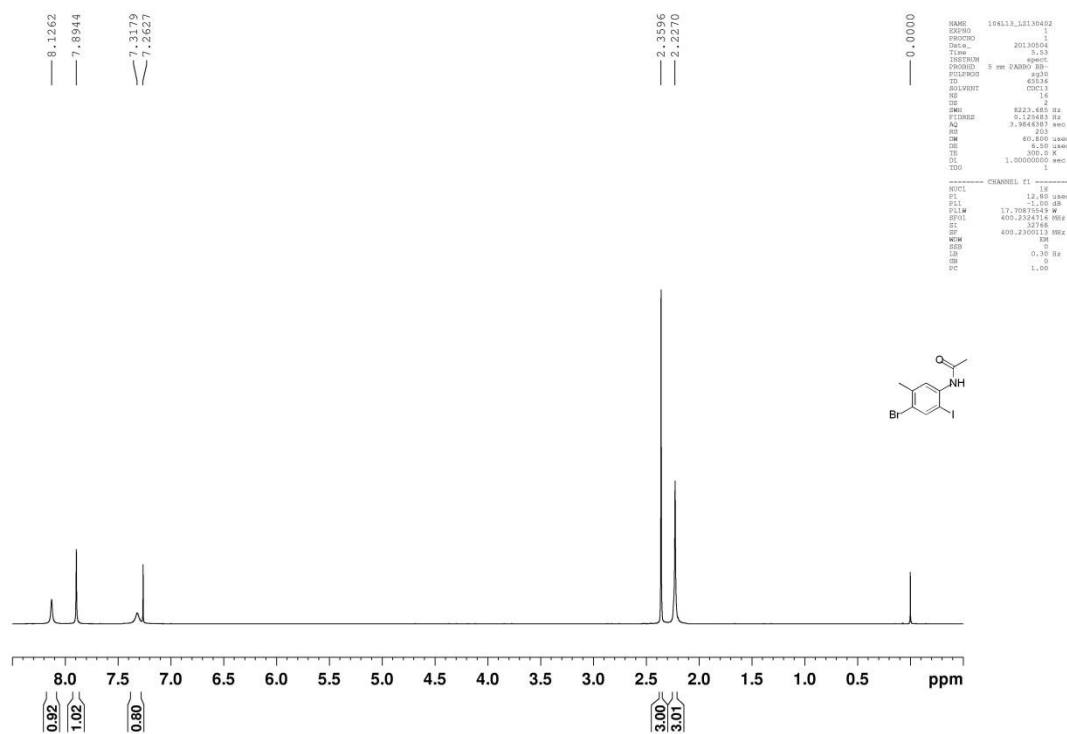
¹H NMR (400 MHz, CDCl₃) of compound 2h



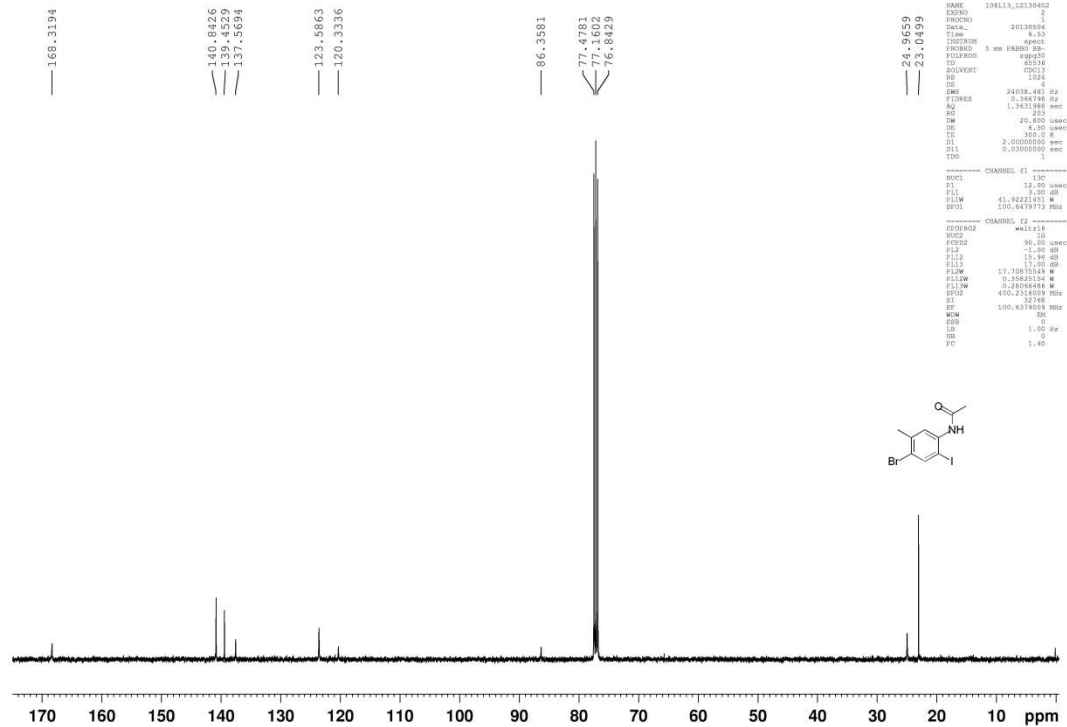
¹³C NMR (100 MHz, CDCl₃) of compound 2h



¹H NMR (400 MHz, CDCl₃) of compound 2i



¹³C NMR (100 MHz, CDCl₃) of compound 2i



¹H NMR (400 MHz, CDCl₃) of compound 2j

