A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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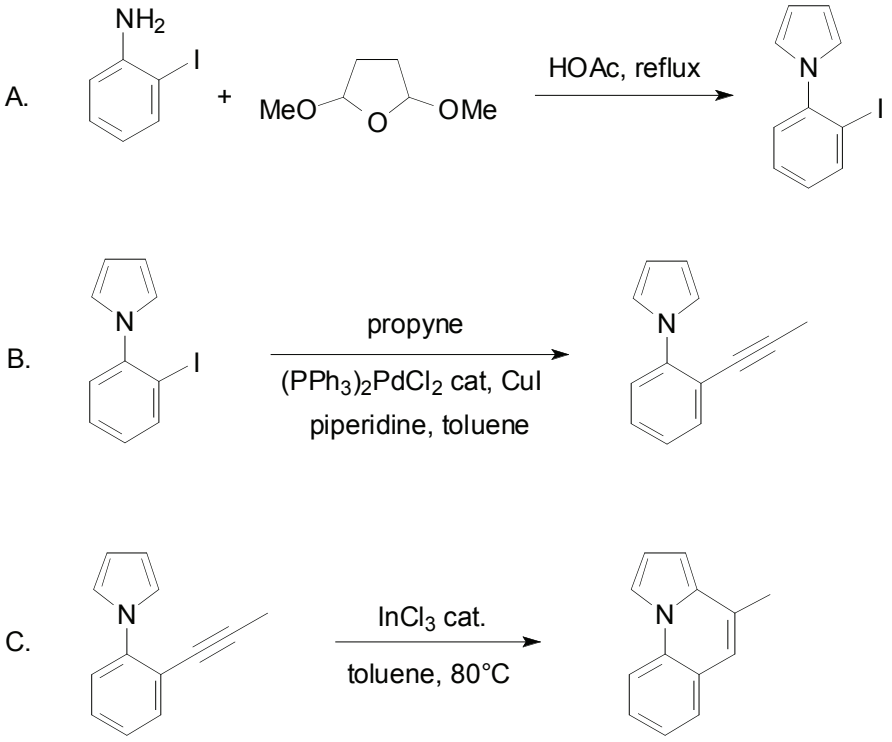
INDIUM-CATALYZED CYCLOISOMERIZATION: PREPARATION OF 4-METHYLPYRROLO[1,2-*a*]QUINOLINE



Submitted by Alois Fürstner, Victor Mamane, Günter Seidel, and Daniel Laurich.[1]
Checked by Scott E. Denmark and Jack Hung-Chang Liu.

1. Procedure

A. 1-(2-Iodophenyl)pyrrole. A 50-mL, three-necked, round-bottomed, flame-dried flask equipped with a Teflon-coated stirbar, a glass stopper, an addition funnel and a reflux condenser is charged with 2-iodoaniline (12.5 g, 57.1 mmol) (Notes 1, 2) and glacial acetic acid (12.5 mL, Note 1). The resulting solution is heated to reflux before 2,5-dimethoxytetrahydrofuran (7.8 mL, 59.0 mmol, Note 1) is added over a period of 10 min via the dropping funnel and reflux is continued for 5 min once the addition is complete. The addition funnel and reflux condenser are replaced by an inlet for a thermocouple and a distillation head with condenser, respectively, and the acetic acid is slowly removed by distillation under reduced pressure (15 mmHg, bath temperature about 50 °C) over a period of approximately 4 h. During this time, the internal temperature slowly reaches 50 °C. The

remaining brown residue is transferred to a 25-mL round-bottomed flask and is purified by short-path distillation in vacuum (0.04 mmHg, Note 3). The fraction distilling at 90−102 °C is collected to give 1-(2-iodophenyl)pyrrole as a brown liquid (9.78–10.12 g, 64–66%) (Notes 4, 5).

B. 1-(2-(1-Propynyl)phenyl)pyrrole. A flame-dried 250-mL, two-necked, round-bottomed flask equipped with a magnetic stirbar, a gas dispersion tube (5 mm OD, Note 6), and a bubbler-sealed gas outlet is charged sequentially with 1-(2-iodophenyl)pyrrole (8.98 g, 33.4 mmol), piperidine (10 mL, Note 1), $(PPh_3)_2PdCl_2$ (1.20 g, 1.67 mmol, Note 1), CuI (318 mg, 1.67 mmol, Note 1) and toluene (90 mL, Note 1) to give a homogeneous reddish solution. Propyne is bubbled through this solution for 90 min at 0.1 L/min via the dispersion tube until the reaction mixture turns black (Notes 7, 8). For workup, the flask is vented, the reaction mixture is filtered through a short pad of silica (approximately 30 g) which is carefully rinsed with *tert*-butyl methyl ether (350 mL). The combined filtrates are transferred into a 1-L separatory funnel and are successively washed with water (2 x 200-mL portions) and brine (350 mL) before being dried over Na_2SO_4 and evaporated. The remaining orange liquid is transferred to a 25-mL round-bottomed flask and is purified by short-path distillation in vacuum (Note 3) to give 1-(2-(1-propynyl)phenyl)pyrrole as a pale yellow liquid (bp 70−75 °C, 0.005 mm Hg) (4.56–4.72 g, 75–78%, Notes 5, 9).

C. 4-Methylpyrrolo[1,2-a]quinoline. A 250-mL, three-necked, round-bottomed flask equipped with a reflux condenser fitted with an argon inlet, a glass stopper, a stopcock gas inlet and a Teflon-coated magnetic stirbar is evacuated and flame-dried before it is flushed with argon and allowed to cool to ambient temperature. The flask is charged with 1-(2-(1-propynyl)phenyl)pyrrole (4.53 g, 25 mmol), toluene (100 mL, Note 1) and $InCl_3$ (277 mg, 1.25 mmol, Note 10) and the resulting mixture is stirred at 80 °C bath temperature for 4 h until GC analysis shows complete conversion of the substrate (Note 11). For workup, the dark mixture is filtered through a pad of silica gel (5 cm x 3 cm, approximately 30 g), which is then carefully rinsed with toluene (150 mL). The crude product was obtained as a yellow solid after removal of the solvent under reduced pressure. The product is dissolved in toluene (20 mL) and this solution is adsorbed on silica gel (approximately 10 g) and the resulting slurry is evaporated to dryness. The adsorbate is placed in a Soxhlet thimble (8.0 cm x 3.5 cm) and is extracted with pentane (80 mL, Note 1) for 18 h using a Soxhlet apparatus of ca. 50 mL inner volume (Note 12). Evaporation of the resulting pentane extract

provides 4-methylpyrrolo[1,2-a]quinoline as a pale yellow solid, which is pure enough for most applications (mp 63−65°C, ≥ 95% by GC, Note 11) (4.12 g, 91%). Analytically pure material is obtained by subsequent sublimation (60 °C bath temperature, 5 x 10^{-3} mmHg for 3.5 h, Note 13) as a white solid (3.05 g, 67%, Notes 14-16), mp 67−68 °C.

2. Notes

1. 2-Iodoaniline (>98%) was purchased from Aldrich Chemical Co. or from Lancaster. 2-Iodoaniline was recrystallized by dissolving the commercial material in benzene with stirring in a round-bottomed flask, in a 50 °C oil bath. For each gram of 2-iodoaniline, 1 mL of benzene was used. To the dark brown solution of 2-iodoaniline was then slowly added petroleum ether with stirring. Approximately four times the volume of benzene was added. This mixture was allowed to cool to room temperature and was then cooled in –20 °C for approximately 4 hours. The dark brown crystals were filtered off through a coarse fritted funnel, and were further purified by sublimation under high vacuum for approximately 2 to 2.5 h (oil bath temperature: 50–55 °C, cold finger of the sublimator cooled by a dry ice-acetone bath). The melting point of the purified material was 56–57 °C and was significantly less colored. Copper(I) iodide (CuI, 99.999%), $(PPh_3)_2PdCl_2$ (99.99%) (Aldrich Chemical Co.), glacial acetic acid (99%, Fluka), 2,5-dimethoxytetrahydrofuran (99%, *cis*/*trans* mixture, Acros Organics), and piperidine (99%, Aldrich Chemical Co.) were purchased from the suppliers indicated. 2,5-Dimethoxytetrahydrofuran was purified by short path distillation. Other chemicals were used as received. Toluene and pentane were dried by distillation over Na/benzophenone prior to use.

2. For Step A, the checkers were not able to reproduce the yield when the reaction was carried out on 114.0 mmol scale; considerably more unreacted 2-iodoaniline remained than when the reaction was carried out on 57.0 mmol scale. This was most likely due to the less efficient heat transfer into a 100-mL flask. Consequently, the distillate was a mixture of starting material and product.

3. The short path distillation was performed with a one-piece distillation apparatus consisting of a vacuum-jacketed distillation head and short (approximately 3 cm) condenser arm. This equipment enables adiabatic distillation and is commercially available from Ace Glass (catalog no. 9317-42).

4. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.35 (t, *J* = 2.1 Hz, 2 H), 6.82 (t, *J* = 2.1 Hz, 2 H), 7.11 (ddd, *J* = 9.0, 7.9, 1.7 Hz, 1 H), 7.31 (dd, *J* = 7.8, 1.6 Hz, 1 H), 7.42 (ddd, *J* = 8.8, 7.7, 1.4 Hz, 1 H), 7.95 (dd, *J* = 8.0, 1.4 Hz, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 96.0, 109.3, 122.3, 128.2, 129.1, 129.6, 140.1, 144.2; MS (EI) *m*/*z* (relative intensity): 269 (100%, [M^+]), 142 (35%), 115 (62%); HRMS (EI) *m/z*, calcd for $C_{10}H_8NI$: 268.9702; found: 268.9701; IR (film): 3101, 1582, 1494, 1438, 1072, 1012, 924, 760, 724 cm^{-1}. Anal. calcd for $C_{10}H_8IN$ (269.08): C, 44.64; H, 3.00; N, 5.21. Found: C, 44.65; H, 2.77; N, 5.02.

5. The product is stable for extended periods of time when kept in a refrigerator.

6. The gas dispersion tube was purchased from ACE Glass (catalog no. 9435-78).

7. Propyne purchased from Matheson Inc. was used without further purification.

8. A flow-meter (Matheson Tri-Gas: PM-1000, part no. MN11E101N201) showed that a total of approximately 9 L of propyne was passed through the solution. If a less vigorous stream of propyne is chosen, then the reaction time will increase accordingly. In all cases, however, the formation of a black precipitate indicated complete conversion.

9. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 2.03 (s, 3 H), 6.36 (dt, *J* = 2.5, 1.3 Hz, 2 H), 7.16 (dt, *J* = 2.2, 1.1 Hz, 2 H), 7.25 (dt, *J* = 7.5, 1.4 Hz, 1 H), 7.31 (dd, *J* = 8.0, 1.0 Hz, 1 H), 7.36 (dt, *J* = 8.1, 1.5 Hz, 1 H), 7.55 (d, *J* = 7.8 Hz, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 4.7, 76.8, 91.0, 109.2, 118.8, 121.8, 125.0, 126.3, 128.7, 134.2, 142.0; MS (EI) *m*/*z* (relative intensity): 181 (100%, [M^+]), 180 (95%), 166 (3%), 154 (18%), 152 (11%), 140 (2%), 127 (4%), 115 (3%), 89 (5%), 77 (7%); HRMS (EI) *m/z*, calcd for $C_{13}H_{11}N$: 181.0891, found: 181.0886; IR (film): 3102, 2914, 2849, 2226, 1599, 1501, 1478, 1332, 1102, 1070, 761, 725 cm^{-1}. Anal. calcd for $C_{13}H_{11}N$ (181.24): C, 86.15; H, 6.12; N, 7.73; Found: C, 86.02; H, 6.12; N, 8.04.

10. $InCl_3$ (99.999%, Strem Chemicals) was used as received. Due to its hygroscopic character, this compound was kept in a dry box.

11. Occasionally, the reaction time was found to be somewhat longer. The progress of the reaction and the purity of the product can be monitored by GC (Hewlett-Packard HP-5, 5% phenylmethylsiloxane column: 50 m, 0.25 mm), temperature program: 70 °C → 270 °C, 20 °C/min, then 270 °C for 6.0 min; retention time: 11.81 min (starting material), 14.24 min (product).

12. In a second run, the checkers found that a higher yield (4.20 g, 93%) could be obtained by exhaustive Soxhlet extraction for 48 h. Sublimation of this material (oil bath: 50−60 °C, 7 h), afforded 3.97 g (88%) of the product as a white solid.

13. The sublimation was performed by immersing a large side-armed test tube (42 mm x 175 mm) containing the crude product in an oil bath and subliming the product under reduced pressure onto the cold wall of a smaller test tube (25 mm x 150 mm) cooled with a dry-ice/acetone slush. The smaller test tube, which served as a cold finger, was secured by a large rubber stopper. During the sublimation, the bath temperature should not exceed 60 °C to avoid undue decomposition. The purity of the material can be checked by GC (Note 11).

14. The submitters found that analytically pure material (2.49 g, 55%) could also be obtained by recrystallization from hexanes (15 mL).

15. In solid form, the product can be stored in a refrigerator for extended periods of time. However, the product is sensitive in solution to traces of acid present in $CDCl_3$ that has not been rigorously purified.

16. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 2.45 (d, *J* = 1.3 Hz, 3 H), 6.54 (dd, *J* = 3.8, 1.4 Hz, 1 H), 6.81 (dd, *J* = 3.8, 3.7 Hz, 1 H), 6.83 (s, 1H), 7.30 (dt, *J* = 7.5, 1.1 Hz, 1 H), 7.46 (dt, *J* = 7.8, 1.5 Hz, 1 H), 7.60 (dd, *J* = 7.8, 1.4, 1 H), 7.87 (d, *J* = 1.3 Hz, 1 H), 7.87 (d, *J* = 7.7 Hz, 1 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 18.5, 101.3, 112.5, 112.6, 114.2, 117.7, 123.7, 124.6, 127.0, 128.0, 128.1, 129.4, 132.7; MS (EI) *m/z* (relative intensity): 181 (100%, [M+]), 180 (42%), 152 (8%), 91 (7%), 77 (7%); HRMS (EI) *m/z*, calcd for $C_{13}H_{11}N$: 181.0891; found: 181.0890; IR (film): 3142, 1608, 1541, 1486, 1458, 1419, 1366, 1192, 1088, 867, 840, 774, 753, 739, 703 cm^{-1}. Anal. calcd for $C_{13}H_{11}N$ (181.24): C, 86.15; H, 6.12; N, 7.73. Found: C, 86.03; H, 6.15; N, 7.85.

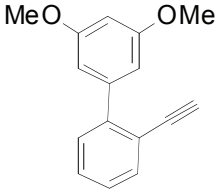
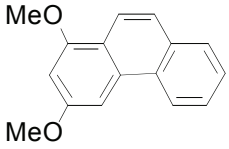
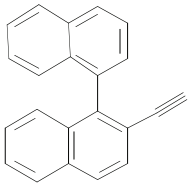
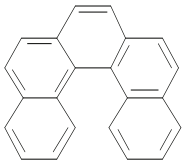
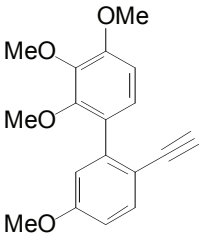
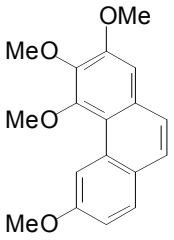
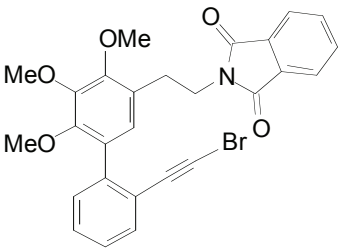
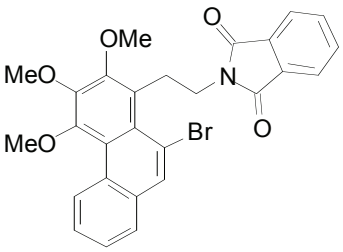
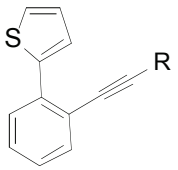
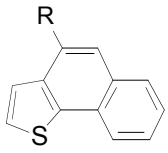
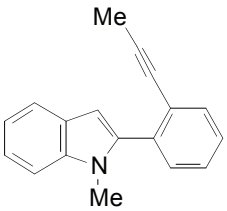
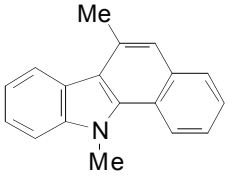
Safety and Waste Disposal Information

All hazardous materials should be handled and disposed of in accordance with “Prudent Practices in the Laboratory”; National Academy Press; Washington, DC, 1995.

3. Discussion

The ability of "soft" metal salts such as $PtCl_2$, $AuCl_3$ or $InCl_3$ to render alkynes susceptible to attack by (tethered) nucleophiles such as alkenes, allyl ethers, or aromatic systems has been recognized only recently.[2-4] The ensuing skeletal rearrangements are inherently attractive for increasing molecular complexity. In this context, it has been shown that readily available biaryl derivatives containing an alkyne unit at one of their *ortho*-positions are converted into substituted phenanthrenes, or heterocyclic congeners thereof, on exposure to catalytic amounts of these salts in an inert solvent.[5-7] The most widely applied catalyst for this purpose is $PtCl_2$, although $GaCl_3$ and $InCl_3$ tend to give higher yields with heteroaromatic substrates as well as with biaryl derivatives bearing halo-alkynes. Such metal-catalyzed transformations accommodate substantial structural variations as can be seen from the selected examples compiled in the Table. In addition to a host of phenanthrene derivatives, substituted helicenes, benzoindoles, benzocarbazoles, napthothiophenes, or pyrrolo[1,2-*a*]quinolines can be obtained in good to excellent yields.[5-7] Since the latter class of heterocycles is endowed with promising biological activities but is difficult to make by more conventional methodology,[8] the procedure detailed above provides a good illustration for the advantages associated with this novel catalytic approach.

TABLE. Selected Examples of Phenanthrenes and Polycyclic Heteroarenes Formed by Metal-Catalyzed Cycloisomerization Reactions[5-7]

Substrate	Catalyst	Product	Yield
	$PtCl_2$		73%
	$PtCl_2$		56%
	$PtCl_2$		86%
	$PtCl_2$		87%
	$PtCl_2$ $InCl_3$		54% (R = H) 83% (R = Ph)
	$PtCl_2$		93%

1. Max-Planck-Institut für Kohlenforschung, Kaiser-Wilhelm-Platz 1, D-45470 Mülheim, Germany.

2. Reviews: (a) Méndez, M.; Mamane, V.; Fürstner, A. *Chemtracts – Org. Chem*. **2003**, *16*, 397. (b) Aubert, C.; Buisine, O.; Malacria, M. *Chem. Rev*. **2002**, *102*, 813. (c) Méndez, M.; Echavarren, A. M. *Eur. J. Org. Chem*. **2002**, 15. (d) Lloyd-Jones, G. C. *Org. Biomol. Chem.* **2003**, *1*, 215. (e) Fürstner, A.; Davies, P. W. Angew. *Chem. Int. Ed.* **2007**, *46*, 3410.

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Appendix
Chemical Abstracts Nomenclature; (Registry Number)

2-Iodoaniline: Benzenamine, 2-iodo-; (615-43-0)

2,5-Dimethoxytetrahydrofuran: Furan, tetrahydro-2,5-dimethoxy-; (696-59-3)

1-(2-Iodophenyl)pyrrole: 1*H*-Pyrrole, 1-(2-iodophenyl)-; (157017-41-9)

$(PPh_3)_2PdCl_2$: Palladium, dichlorobis(triphenylphosphine)-; (13965-03-2)

1-(2-(1-propynyl)phenyl)pyrrole: 1H-Pyrrole, 1-[2-(1-propynyl)phenyl]-; (796843-21-5)

4-Methylpyrrolo[1,2-a]quinoline; (796843-24-8)

$InCl_3$: Indium chloride; (10025-82-8

JHL-IX-83-5, 13C NMR

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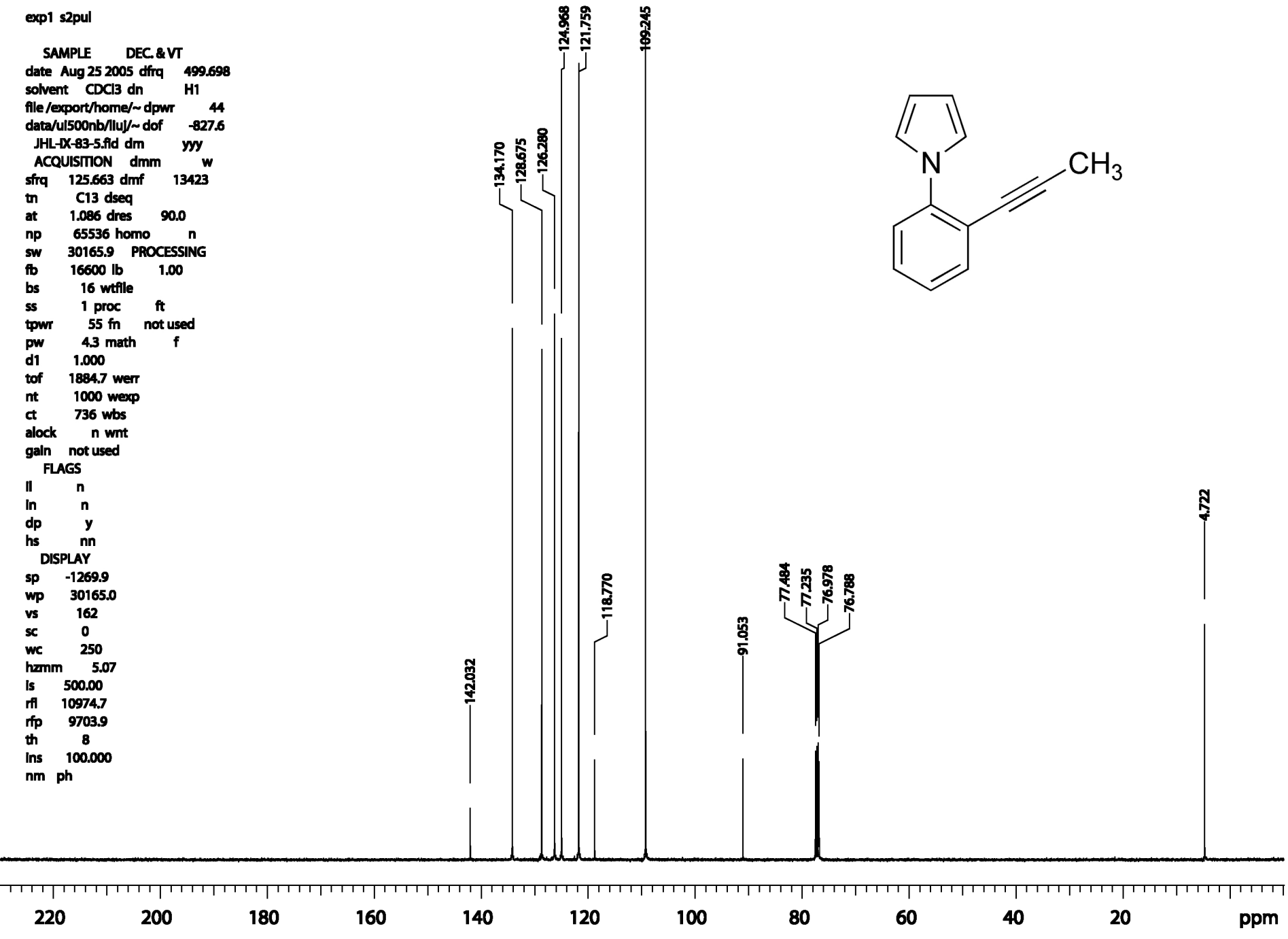

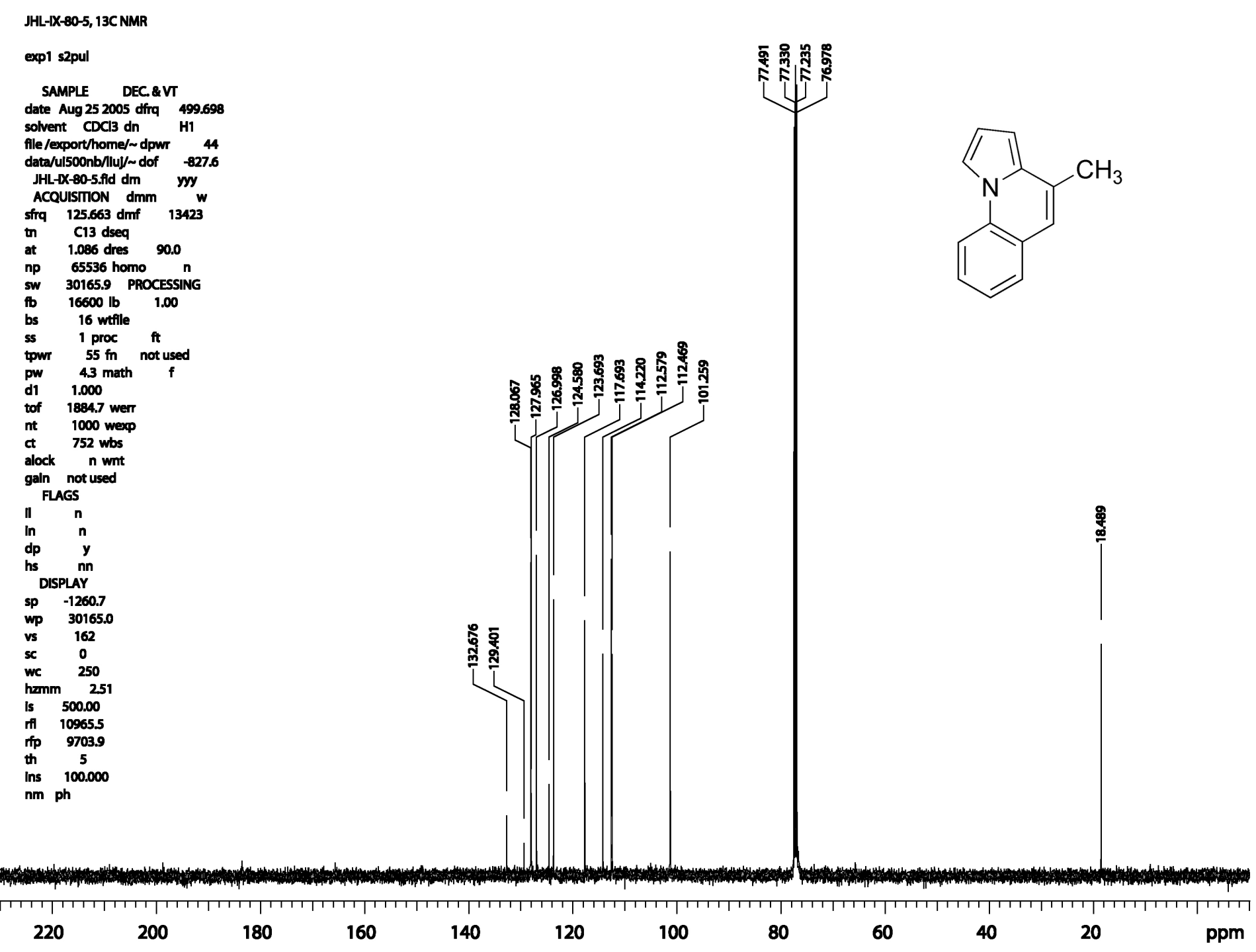
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nm ph
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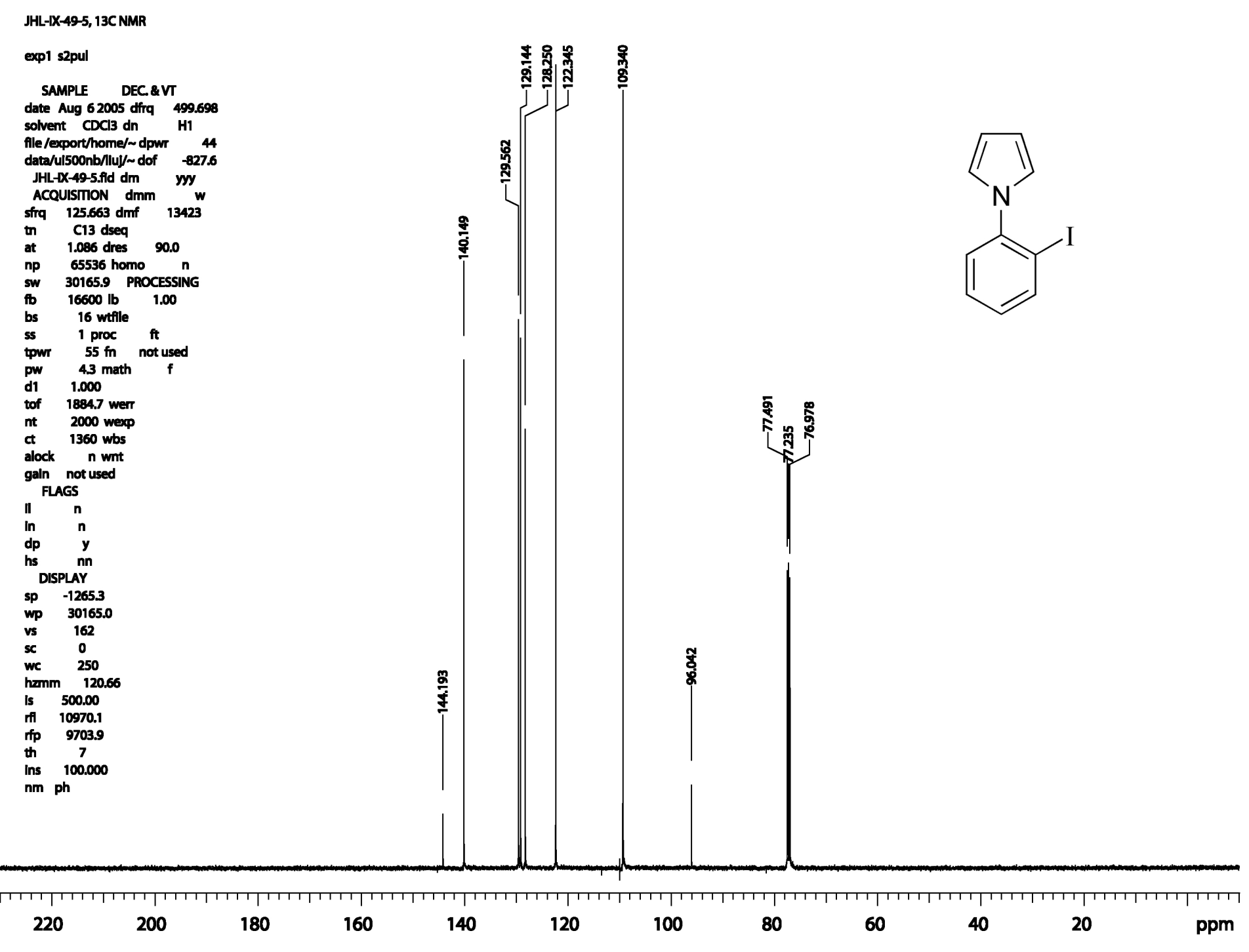
77.330
77.235
76.978
128.067
127.965
126.998
124.580
123.693
117.693
114.220
112.579
112.469
101.259
132.676
129.401
18.489
N
CH3
220
200
180
160
140
120
100
80
60
40
20
ppm

JHL-IX-49-5, 13C NMR

exp1 s2pul

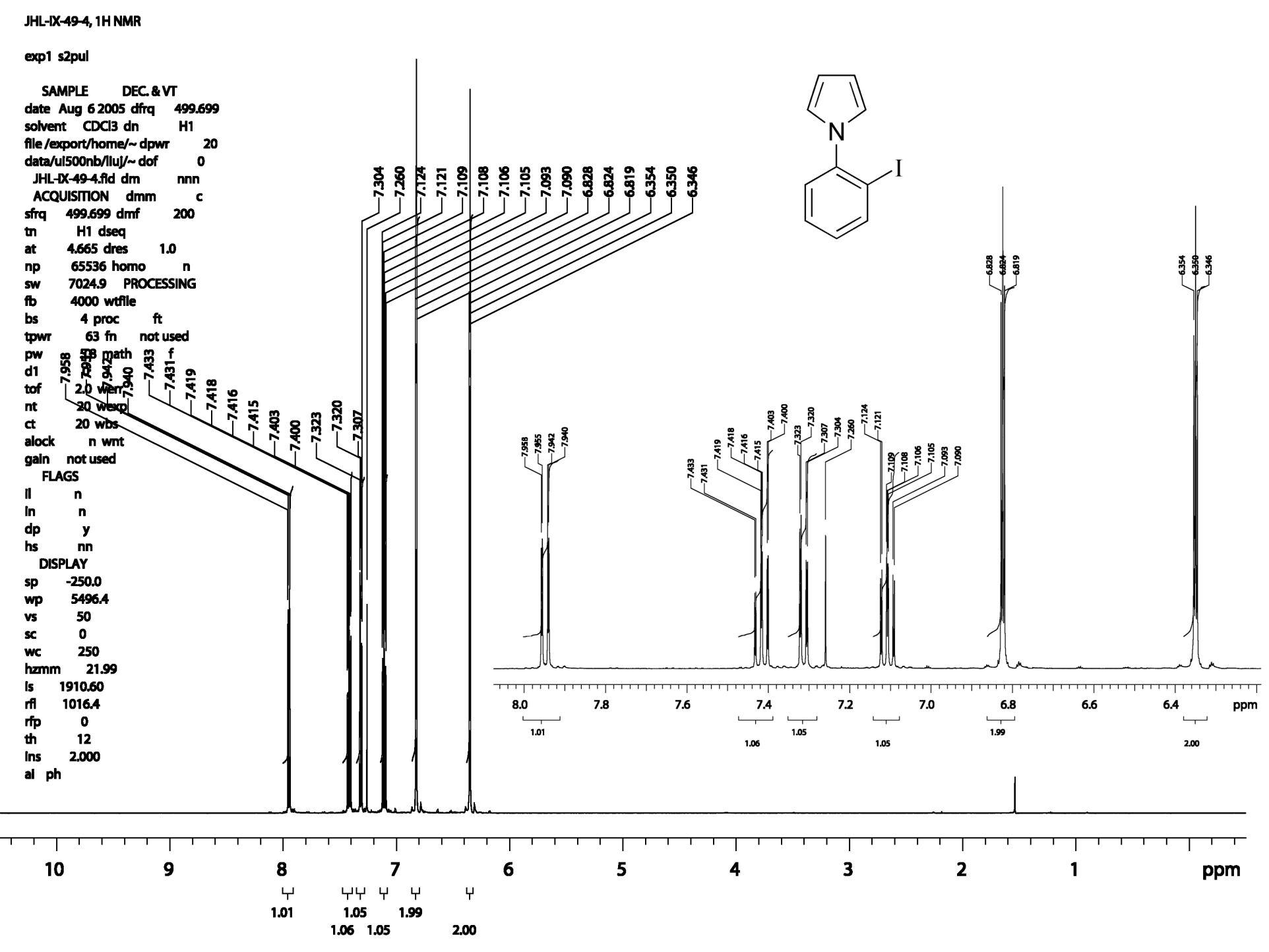
SAMPLE DEC. & VT
date Aug 6 2005 dfrq 499.698
solvent CDCl3 dn H1
file /export/home/~ dpwr 44
data/ul500nb/lluj/~ dof -827.6
JHL-IX-49-5.fid dm yyy
ACQUISITION dmm w
sfrq 125.663 dmf 13423
tn C13 dseq
at 1.086 dres 90.0
np 65536 homo n
sw 30165.9 PROCESSING
fb 16600 lb 1.00
bs 16 wtfile
ss 1 proc ft
tpwr 55 fn not used
pw 4.3 math f
d1 1.000
tof 1884.7 werr
nt 2000 wexp
ct 1360 wbs
alock n wnt
gain not used
FLAGS
ll n
ln n
dp y
hs nn
DISPLAY
sp -1265.3
wp 30165.0
vs 162
sc 0
wc 250
hzmm 120.66
ls 500.00
rfl 10970.1
rfp 9703.9
th 7
lns 100.000
nm ph



JHL-IX-49-4, 1H NMR

exp1 s2pul

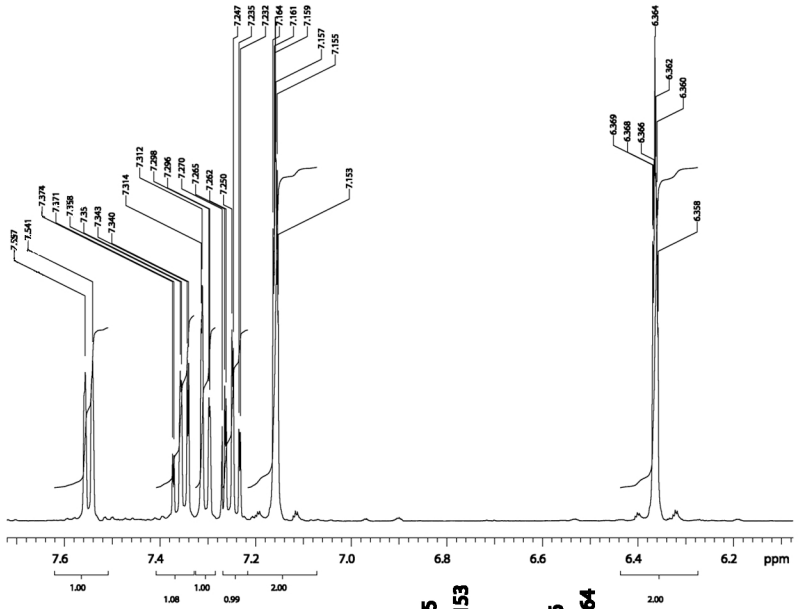
SAMPLE DEC. & VT
date Aug 6 2005 dfrq 499.699
solvent CDCl3 dn H1
file /export/home/~ dpwr 20
data/ul500nb/lluj/~ dof 0
JHL-IX-49-4.fid dm nnn
ACQUISITION dmm c
sfrq 499.699 dmf 200
tn H1 dseq
at 4.665 dres 1.0
np 65536 homo n
sw 7024.9 PROCESSING
fb 4000 wtfile
bs 4 proc ft
tpwr 63 fn not used
pw 8 math
d1 f
tof 2.0 wen
nt 20 wexp
ct 20 wbs
alock n wnt
gain not used
FLAGS
ll n
in n
dp y
hs nn
DISPLAY
sp -250.0
wp 5496.4
vs 50
sc 0
wc 250
hzmm 21.99
ls 1910.60
rfl 1016.4
rfp 0
th 12
lns 2.000
al ph

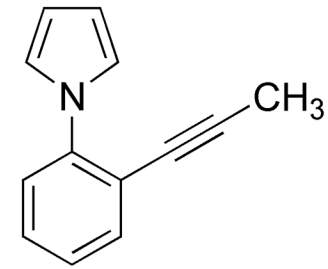


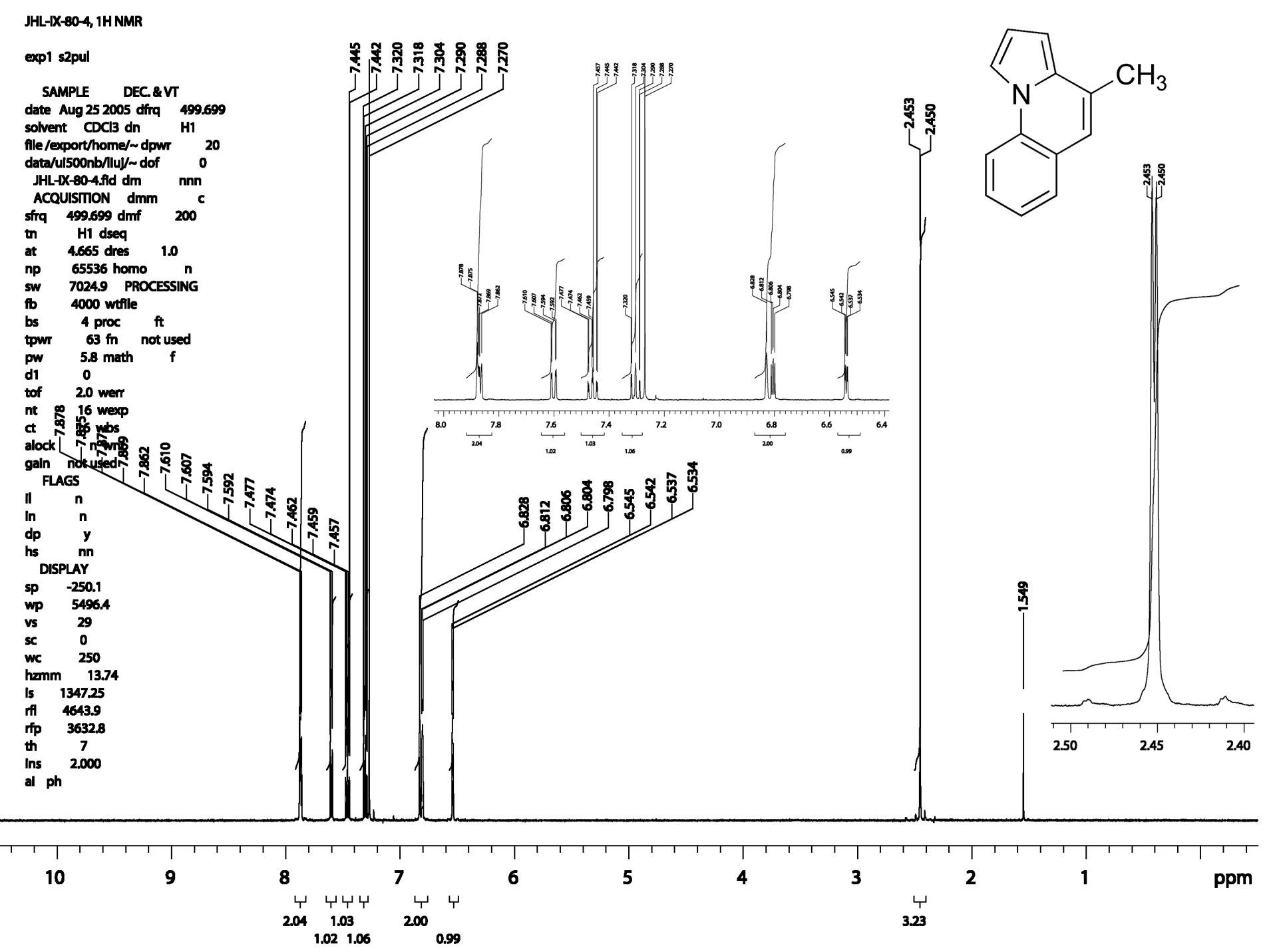
JHL-IX-83-4, 1H NMR

exp1 s2pul

SAMPLE DEC. & VT
date Aug 25 2005 dfrq 499.699
solvent CDCl3 dn H1
file /export/home/~ dpwr 20
data/ul500nb/lluj/~ dof 0
JHL-IX-83-4.fid dm nnn
ACQUISITION dmm c
sfrq 499.699 dmf 200
tn H1 dseq
at 4.665 dres 1.0
np 65536 homo n
sw 7024.9 PROCESSING
fb 4000 wtfile
bs 4 proc ft
tpwr 63 fn not used
pw 5.8 math f
d1 0
tof 2.0 werr
nt 8 wexp
ct 8 wbs
alock n wnt
gain not used
FLAGS
ll n
in n
dp y
hs nn
DISPLAY
sp -250.1
wp 5496.6
vs 25
sc 0
wc 250
hzmm 21.99
ls 1119.82
rfl 4643.5
rfp 3632.8
th 7
lns 2.000
al ph






JHL-IX-80-4, 1H NMR
exp1 s2pul
SAMPLE DEC. & VT
date Aug 25 2005 dfrq 499.699
solvent CDCl3 dn H1
file /export/home/~ dpwr 20
data/ul500nb/lluj/~ dof 0
JHL-IX-80-4.fid dm nnn
ACQUISITION dmm c
sfrq 499.699 dmf 200
tn H1 dseq
at 4.665 dres 1.0
np 65536 homo n
sw 7024.9 PROCESSING
fb 4000 wtfile
bs 4 proc ft
tpwr 63 fn not used
pw 5.8 math f
d1 0
tof 2.0 werr
nt 16 wexp
ct 8 wbs
alock n
gain not used
FLAGS
ll n
in n
dp y
hs nn
DISPLAY
sp -250.1
wp 5496.4
vs 29
sc 0
wc 250
hzmm 13.74
ls 1347.25
rfl 4643.9
rfp 3632.8
th 7
lns 2.000
al ph
7.878
7.875
7.869
7.862
7.610
7.607
7.594
7.592
7.477
7.474
7.462
7.459
7.457
6.828
6.812
6.806
6.804
6.798
6.545
6.542
6.537
6.534
2.453
2.450
1.549
CH3
N
10
9
8
7
6
5
4
3
2
1
ppm
2.04
1.03
2.00
1.02
1.06
0.99
3.23