

Electronic Supplementary Information

Oxidative Transformation of Cyclic Ethers/Amines to Lactones/Lactams Using DIB/TBHP Protocol

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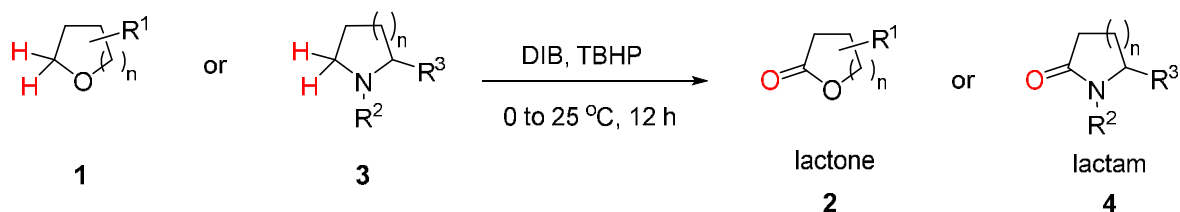
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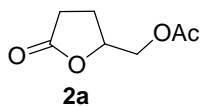
(A) Materials and Methods

Infrared spectra were recorded on a BIO-RAD FTS 165 FT-IR spectrophotometer and reported in wave numbers (cm^{-1}). ^1H NMR and ^{13}C NMR spectra were obtained in CDCl_3 solutions with a Bruker DPX 500 spectrophotometer at 500 MHz and 125 MHz, respectively. NMR spectra are referenced to residual chloroform (δ 7.26 ppm, 1H; δ 77.0 ppm, 13 C). Chemical shifts are reported in ppm (δ); multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), quin (quintet) and m (multiplet). Coupling constants, J, are reported in Hertz. Low resolution mass spectra were obtained on a Finnigan/MAT LCQ spectrometer in ESI mode. High resolution mass spectra were obtained on a Finnigan/MAT 95XL-T spectrometer. Enantiomeric excesses were determined by HPLC analysis on Shimadzu HPLC units, including the following instruments: pump, LC-20AD; detector, SPD-20A; column, Daicel Chiralpak IB. Thin-layer chromatography (TLC) was performed on Merck aluminum-precoated plates of silica gel 60 F₂₅₄. Visualization was obtained by spraying with basic potassium permanganate with subsequent heating. Column chromatography was performed with silica gel 60 (230-400) mesh. The *tert*-butyl hydroperoxide (TBHP) 5-6 M solution in decane was purchased from Aldrich Chemical Co. Commercially available reagents and solvents were used without further treatment.

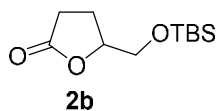
(B) DIB/TBHP oxidation of heterocycles



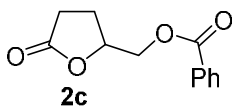
Representative procedure for the oxidation of heterocycle: To a solution of (tetrahydrofuran-2-yl)methyl acetate (**1a**) (72 mg, 0.50 mmol) in nitromethane (1 mL) was added diacetoxyiodobenzene (484 mg, 1.5 mmol) at 0 °C. The resultant suspension was vigorously stirred and a solution of *tert*-butylhydroperoxide (5.0 M in decane, 400 μL , 2.0 mmol) was added dropwise over 30 min. After 12 hours, the reaction was quenched with Na_2SO_3 (5 mL). The organic layer was separated and the aqueous layer was extracted with dichloromethane (4 x 5 mL). The combined extracts were dried (MgSO_4), filtered, and concentrated *in vacuo*. The residue was purified by flash column chromatography eluted with *n*-hexanes/EtOAc (2:1) to yield (5-oxotetrahydrofuran-2-yl)methyl acetate (**2a**) as a yellow oil (51 mg, 65%).



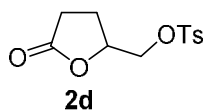
(5-oxotetrahydrofuran-2-yl)methyl acetate (2a).^[3] Isolated as a yellow oil. $^1\text{H NMR}$ d 4.76-4.71 (m, 1H), 4.32 (dd, $J = 10.5, 3.0$ Hz, 1H), 4.15 (dd, $J = 12.0, 5.6$ Hz, 1H), 2.64-2.52 (m, 2.0 H), 2.39-2.32 (m, 1H), 2.10 (s, 3H), 2.08-2.00 (m, 1H); $^{13}\text{C NMR}$ d 176.4, 170.6, 77.2, 65.3, 29.7, 28.2, 23.9, 20.7; MS (ESI+) m/z (relative intensive) 198.8 ($[\text{M}+\text{K}]^+$, 100). (The data were in full accordance with the literature values).



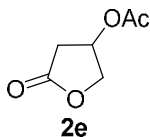
5-[[*tert*-butyldimethylsilyloxy]methyl]dihydrofuran-2(3H)-one (2b).^[4] Isolated as a colourless oil. ¹H NMR *d* 4.61-4.54 (m, 1H), 3.86 (dd, *J* = 11.4, 3.0 Hz, 1H), 3.68 (dd, *J* = 14.2, 3.2 Hz, 1H), 2.64-2.55 (m, 1.0 H), 2.51-2.42 (m, 1H), 2.30-2.15 (m, 2.0 H), 0.89 (s, 9H), 0.06 (s, 6H); ¹³C NMR *d* 177.6, 80.1, 64.9, 28.6, 25.8, 23.6, 18.3, -5.4, -5.5 ; MS (ESI+) *m/z* (relative intensive) 230.9 (M⁺). (The data were in full accordance with the literature values).



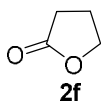
(5-oxotetrahydrofuran-2-yl)methyl benzoate (2c).^[5] Isolated as a yellow oil. ¹H NMR *d* 8.03 (dd, *J* = 8.4, 1.4 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.46(t, *J* = 7.2 Hz, 2H), 4.92-4.84 (m, 1H), 4.55 (dd, *J* = 12.3, 3.2 Hz, 1H), 4.45 (dd, *J* = 12.2, 5.4 Hz, 1H), 2.67-2.59 (m, 2H), 2.49-2.37 (m, 1H), 2.20-2.07 (m, 1H); ¹³C NMR *d* 176.5, 166.2, 133.5, 129.7, 129.3, 128.6, 77.4, 65.7, 28.2, 24.0; MS (ESI+) *m/z* (relative intensive) 243.0 ([M+Na]⁺, 100). (The data were in full accordance with the literature values).



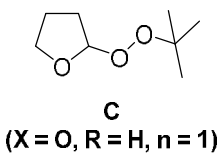
(5-oxotetrahydrofuran-2-yl)methyl 4-methylbenzenesulfonate (2d).^[6] Isolated as a yellow oil. ¹H NMR *d* 7.78 (d, *J* = 8.4 Hz, 2H), 7.36 (d, *J* = 7.8 Hz, 2H), 4.72-4.65 (m, 1H), 4.19 (dd, *J* = 11.0, 3.3 Hz, 1H), 4.13 (dd, *J* = 11.0, 4.2 Hz, 1H), 2.59-2.50 (m, 2H), 2.46 (s, 3H), 2.41-2.28 (m, 1H), 2.18-2.06 (m, 1H); ¹³C NMR *d* 175.9, 145.3, 132.2, 130.0, 127.9, 69.8, 29.6, 27.8, 23.5, 21.6; MS (ESI+) *m/z* (relative intensive) 563.7 (2[M+Na]⁺, 100). (The data were in full accordance with the literature values).



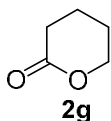
(5-oxotetrahydrofuran-3-yl) acetate (2e).^[9] Isolated as a yellow oil. ¹H NMR *d* 5.44-5.41 (m, 1H), 4.50 (dd, *J* = 11.0, 4.3 Hz, 1H), 4.37 (d, *J* = 10.8 Hz, 1H), 2.85 (dd, *J* = 18.5, 6.8 Hz, 1H), 2.62 (d, *J* = 18.3 Hz, 1H), 2.10 (s, 3H); ¹³C NMR *d* 174.4, 170.3, 73.0, 69.7, 34.5, 20.8 ; MS (ESI+) *m/z* (relative intensive) 166.7 ([M+Na]⁺, 100). (The data were in full accordance with the literature values).



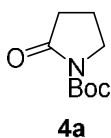
dihydrofuran-2(3H)-one (2f).^[1] Isolated as a clear oil. ¹H NMR *d* 4.33 (t, *J* = 7.1 Hz, 2H), 2.47 (t, *J* = 7.6 Hz, 2H), 2.24 (quin, *J* = 7.2 Hz, 2H); ¹³C NMR *d* 177.8, 68.5, 27.7, 22.1; MS (ESI+) *m/z* (relative intensive) 108.9 ([M+Na]⁺, 100). (The data were in full accordance with the authentic sample).



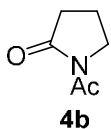
2-(tert-butylperoxy)tetrahydrofuran (C; X = O, R = H, n = 1).^[2] Isolated as a yellow oil. ¹H NMR *d* 5.56 (dd, *J* = 6.0, 1.8 Hz, 1H), 3.93 (t, *J* = 6.2 Hz, 2H), 2.05-1.92 (m, 2H), 1.89-1.73 (m, 2H), 1.25 (s, 9H); ¹³C NMR *d* 106.6, 80.5, 67.4, 29.3, 26.4, 24.0; MS (ESI+) *m/z* (relative intensive) 182.8 ([M+Na]⁺, 100). (The data were in full accordance with the literature values)



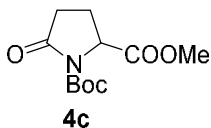
Tetrahydro-2H-pyran-2-one (2g).^[11] Isolated as a yellow oil. ¹H NMR *d* 4.34 (t, J = 6.7 Hz, 2H), 2.58 (t, J = 7.0 Hz, 2H), 1.95-1.85 (m, 4H); ¹³C NMR *d* 171.4, 69.4, 29.8, 22.3, 19.1 ; MS (ESI+) *m/z* (relative intensive) 123.0 ([M+Na]⁺, 100). (The data were in full accordance with the literature values).



tert-butyl 2-oxopyrrolidine-1-carboxylate (4a).^[11] Isolated as a yellow oil. ¹H NMR *d* 3.72 (t, J = 7.2 Hz, 2H), 2.49 (t, J = 7.9 Hz, 2H), 1.97 (quin, J = 8.1 Hz, 2H), 1.50 (s, 9H) ; ¹³C NMR *d* 174.3, 150, 82.7, 46.4, 32.9, 27.9, 17.3; MS (ESI+) *m/z* (relative intensive) 207.9 ([M+Na]⁺, 100). (The data were in full accordance with the literature values).

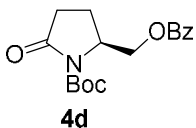


1-acetylpyrrolidin-2-one (4b).^[12] Isolated as a yellow oil. ¹H NMR *d* 3.80 (t, J = 7.4 Hz, 2H), 2.59 (t, J = 8.0 Hz, 2H), 2.49 (s, 3H), 2.03 (quin, J = 8.1 Hz, 2H); ¹³C NMR *d* 175.5, 171.3, 45.2, 33.5, 24.8, 17.1; MS (ESI+) *m/z* (relative intensive) 150.1 ([M+Na]⁺, 100). (The data were in full accordance with the literature values).

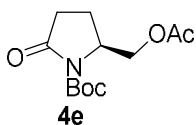


1-tert-butyl 2-methyl 5-oxopyrrolidine-1,2-dicarboxylate (4c).^[13] Isolated as a yellow oil. ¹H NMR *d* 4.61 (dd, J = 9.4, 2.9 Hz, 1H), 3.78 (s, 3H), 2.66-2.57 (m, 1H), 2.54-2.44 (m, 1H), 2.35-2.28 (m, 1H), 2.08-1.99 (m, 1H), 1.49 (s, 9H); ¹³C NMR *d* 173.3, 171.8, 149.2, 83.6, 58.8, 52.5, 31.2, 27.8, 21.4; MS

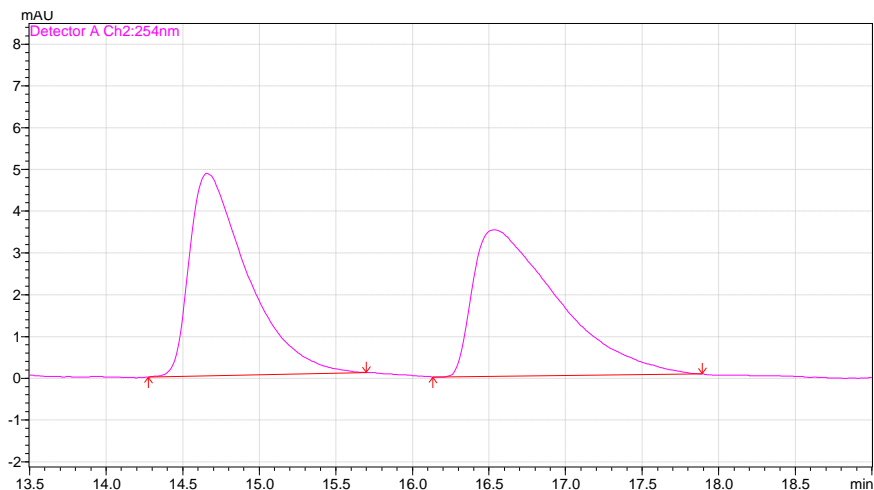
(ESI+) m/z (relative intensive) 265.9 ($[M+Na]^+$, 100). (The data were in full accordance with the literature values).



tert-butyl 2-((benzoyloxy)methyl)-5-oxopyrrolidine-1-carboxylate (4d). Isolated as a yellow oil. ^1H NMR d 8.00 (d, $J = 8.0$ Hz, 2H), 7.58 (t, $J = 7.5$ Hz, 1H), 7.45 (t, $J = 8.0$ Hz, 2H), 4.57-4.50 (m, 3H), 2.76-2.69 (m, 1H), 2.52-2.47 (m, 1H), 2.28-2.23 (m, 1H), 2.06-2.01 (m, 1H), 1.54 (s, 9H); ^{13}C NMR d 174.0, 166.3, 149.7, 133.4, 129.7, 129.4, 128.6, 83.5, 65.3, 56.3, 31.8, 28.0, 21.0; IR (solution): 3427, 2980, 1786, 1720, 1646, 1453, 1369, 1313, 1273, 1152, 713; HRMS (ESI+) calcd for $\text{C}_{17}\text{H}_{21}\text{NO}_5$ $[M+Na]^+$: 342.1321, found 342.1312.

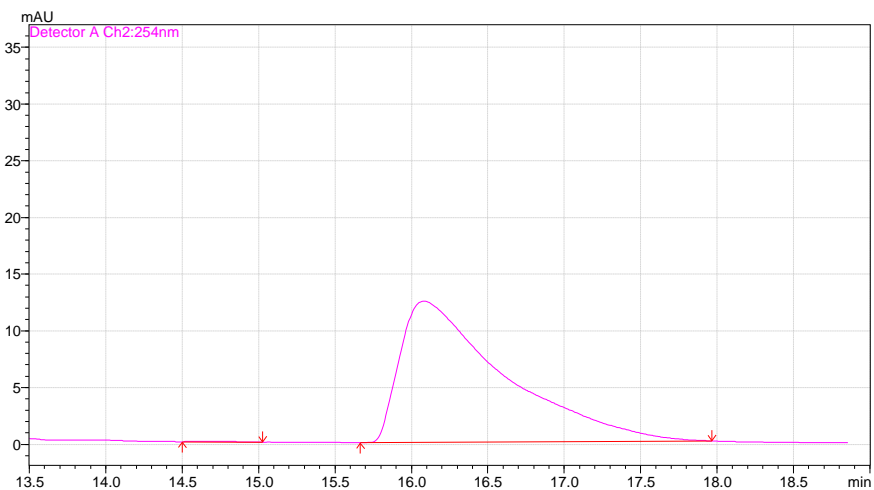


tert-butyl 2-(acetoxymethyl)-5-oxopyrrolidine-1-carboxylate (4e). Isolated as a colorless oil. ^1H NMR d 4.36 (dd, $J = 12.9, 4.5$ Hz, 2H), 4.15 (dd, $J = 13.3, 5.0$ Hz, 1H), 2.67-2.59 (m, 1H), 2.46-2.40 (m, 1H), 2.21-2.13 (m, 1H), 2.05 (s, 3H), 1.94-1.90 (m, 1H), 1.51 (s, 9H); ^{13}C NMR d 174.1, 170.6, 149.6, 83.4, 64.9, 56.1, 31.7, 28.0, 21.0, 20.8; IR (solution): 3427, 1784, 1744, 1645, 1457, 1369, 1310, 1255, 1153, 751; HRMS (ESI+) calcd for $\text{C}_{12}\text{H}_{19}\text{NO}_5$ $[M+Na]^+$: 280.1158, found 280.1155. HPLC (Daicel Chiralpak IB, *i*-PrOH/hexane = 25/75, 0.6 mL/min, 254 nm) $t_1 = 14.6$ min (minor), $t_2 = 16.0$ min (major).



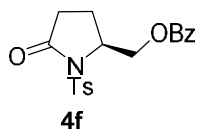
Detector A Ch2 254nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|--------------|-----------|---------------|-------------|----------------|----------------|
| 1 | 14.655 | 127745 | 4847 | 48.549 | 57.999 |
| 2 | 16.529 | 135382 | 3510 | 51.451 | 42.001 |
| Total | | 263127 | 8356 | 100.000 | 100.000 |

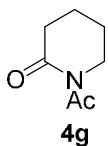


Detector A Ch2 254nm

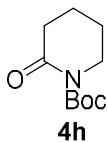
| Peak# | Ret. Time | Area | Height | Area % | Height % |
|--------------|-----------|---------------|--------------|----------------|----------------|
| 1 | 14.661 | 591 | 27 | 0.101 | 0.214 |
| 2 | 16.076 | 586121 | 12439 | 99.899 | 99.786 |
| Total | | 586712 | 12466 | 100.000 | 100.000 |



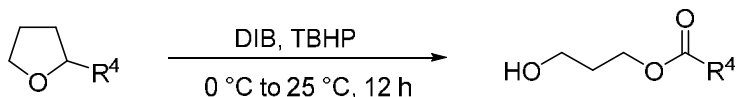
(5-oxo-1-tosylpyrrolidin-2-yl)methyl benzoate (4f). Isolated as a yellow oil. ^1H NMR δ 7.95 (d, $J = 8.0$ Hz, 2H), 7.68 (d, $J = 8.3$ Hz, 2H), 7.54 (t, $J = 7.5$ Hz, 1H), 7.36 (t, $J = 8.0$ Hz, 2H), 7.17 (d, $J = 8.3$ Hz, 2H), 4.78-4.75 (m, 1H), 4.71 (dd, $J = 11.9, 4.0$ Hz, 1H), 4.50 (dd, $J = 12.0, 1.75$ Hz, 1H), 2.63 (dt, $J = 17.8, 10.8$ Hz, 1H), 2.49-2.34 (m, 2H), 2.22 (s, 3H), 2.07-2.03 (m, 1H); ^{13}C NMR δ 173.6, 165.9, 145.2, 135.4, 133.3, 129.5, 129.4, 129.1, 128.4, 128.2, 65.8, 57.9, 31.2, 22.8, 21.5; IR (solution): 3447, 3023, 2926, 2855, 1725, 1601, 1453, 1362, 1272, 1216, 1171, 1119, 712; HRMS (ESI+) calcd for $\text{C}_{19}\text{H}_{19}\text{NO}_5$ $[\text{M}+\text{Na}]^+$: 396.0881, found 396.0876.



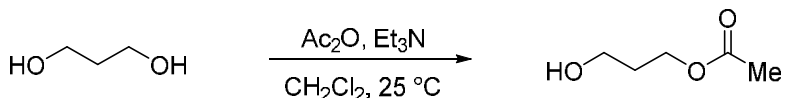
1-acetypiperidine-2-one (4g).^[14] Isolated as a yellow oil. ^1H NMR δ 3.74-3.69 (m, 2H), 2.55 (m, 2H), 2.52 (s, 3H), 1.84-1.81 (m, 4H); ^{13}C NMR δ 173.8, 173.4, 43.8, 34.8, 27.7, 22.4, 20.3; MS (ESI+) m/z (relative intensive) 163.8 ($[\text{M}+\text{Na}]^+$, 100). (The data were in full accordance with the literature values).



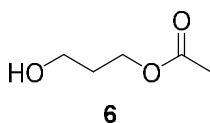
tert-butyl 2-oxopiperidine-1-carboxylate (4h).^[14] Isolated as a yellow oil. ^1H NMR δ 3.63-3.59 (m, 2H), 2.49-2.45 (m, 2H), 1.81-1.77 (m, 4H), 1.48 (s, 9H); ^{13}C NMR δ 171.4, 152.6, 82.8, 46.2, 34.7, 27.9, 22.7, 20.4; MS (ESI+) m/z (relative intensive) 221.9 ($[\text{M}+\text{Na}]^+$, 100). (The data were in full accordance with the literature values).



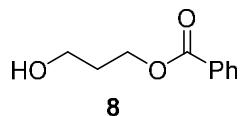
Representative procedure for the oxidation of simple alkyl and aryl substituted THF: To a solution of 2-methyltetrahydrofuran (**5**) (0.05 mL, 0.5 mmol) in *n*-butyl acetate (1 mL) was added diacetoxyiodobenzene (484 mg, 1.5 mmol) at 0 °C. The resultant suspension was vigorously stirred and a solution of *tert*-butylhydroperoxide (5.0 M in decane, 400 μ L, 2.0 mmol) was added dropwise over 30 min. After 12 hours, reaction was quenched with Na₂SO₃ (5 mL). The organic layer was separated and the aqueous layer was extracted with dichloromethane (4 x 5 mL). The combined extracts were dried (MgSO₄), filtered, and concentrated *in vacuo*. The residue was purified by flash column chromatography eluted with *n*-hexanes/EtOAc (3:1) to yield 3-hydroxypropyl acetate (**6**) as a yellow oil (43 mg, 72%).



A sample of **6** was prepared by the reaction between propane-1,3-diol and acetic anhydride. The resulting product has an identical ¹H NMR as compound **6** synthesized from the oxidation.



3-hydroxypropyl acetate (6).^[15] Isolated as a yellow oil. ¹H NMR *d* 4.23 (t, *J* = 6.3 Hz, 2H), 3.70 (t, *J* = 6.3 Hz, 2H), 2.06 (s, 3H), 1.87 (quin, *J* = 6.3 Hz, 2H); ¹³C NMR *d* 171.6, 61.5, 59.2, 31.7, 20.9; MS (ESI+) *m/z* (relative intensive) 118.8 (M⁺, 100). (The data were in full accordance with the literature values).



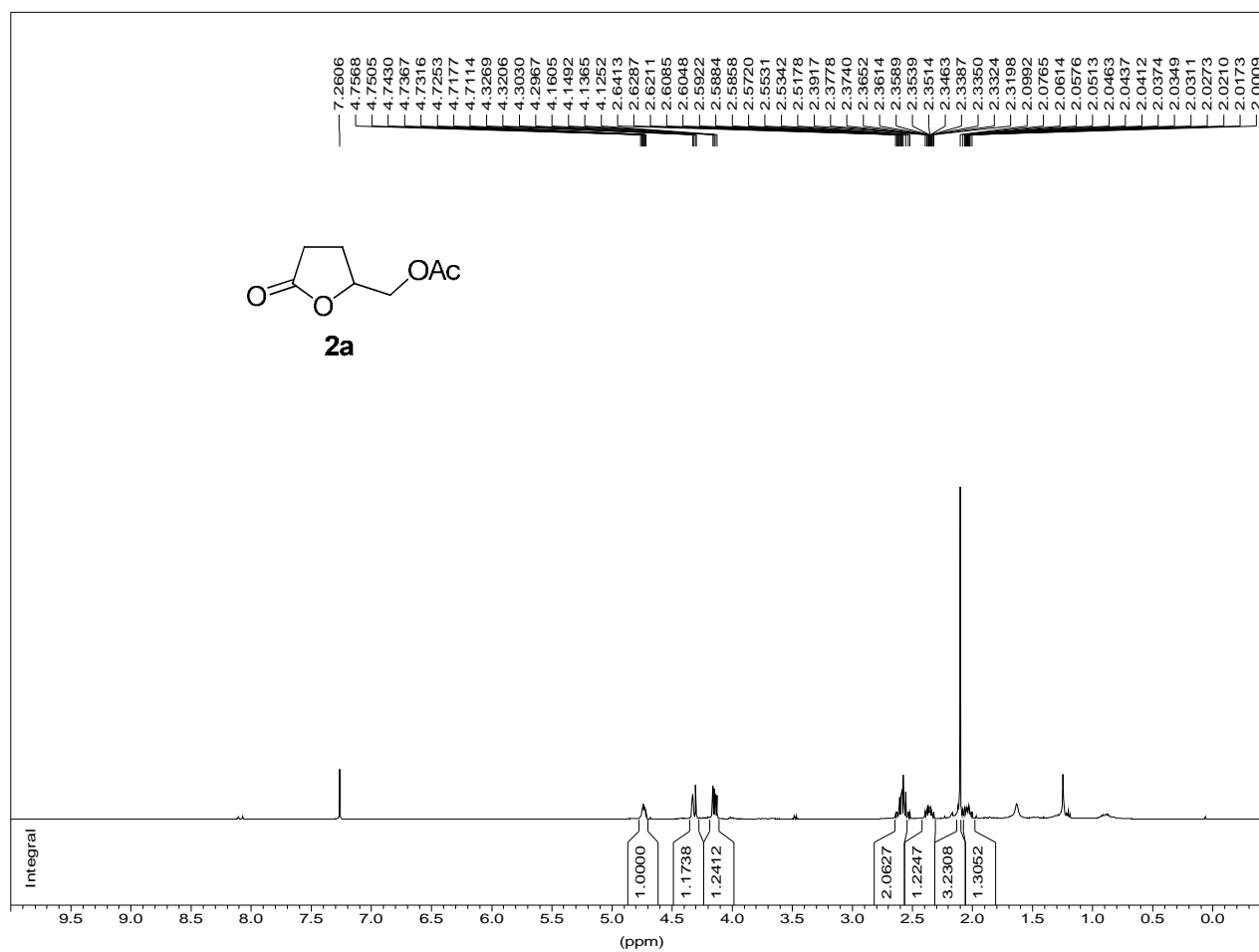
3-hydroxypropyl benzoate (8).^[16] Isolated as a yellow oil. ¹H NMR *d* 8.04 (dd, *J* = 8.4, 1.5 Hz, 2H), 7.57 (tt, *J* = 7.5, 1.5 Hz, 1H), 7.45 (t, *J* = 6.9 Hz, 2H), 4.50 (t, *J* = 6.0 Hz, 2H), 3.70 (t, *J* = 6.0 Hz, 2H), 2.02 (quin, *J* = 6.0 Hz, 2H); ¹³C NMR *d* 167.0, 133.0, 130.1, 129.6, 128.4, 61.8, 59.2, 31.9 ; MS (ESI +) *m/z* (relative intensive) 180.9 (*M*⁺, 100). (The data were in full accordance with the literature values).

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1H AMX500

jas-2-9-1



*** Current Data Parameters ***

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EXPNO : 1
PROCNO : 1

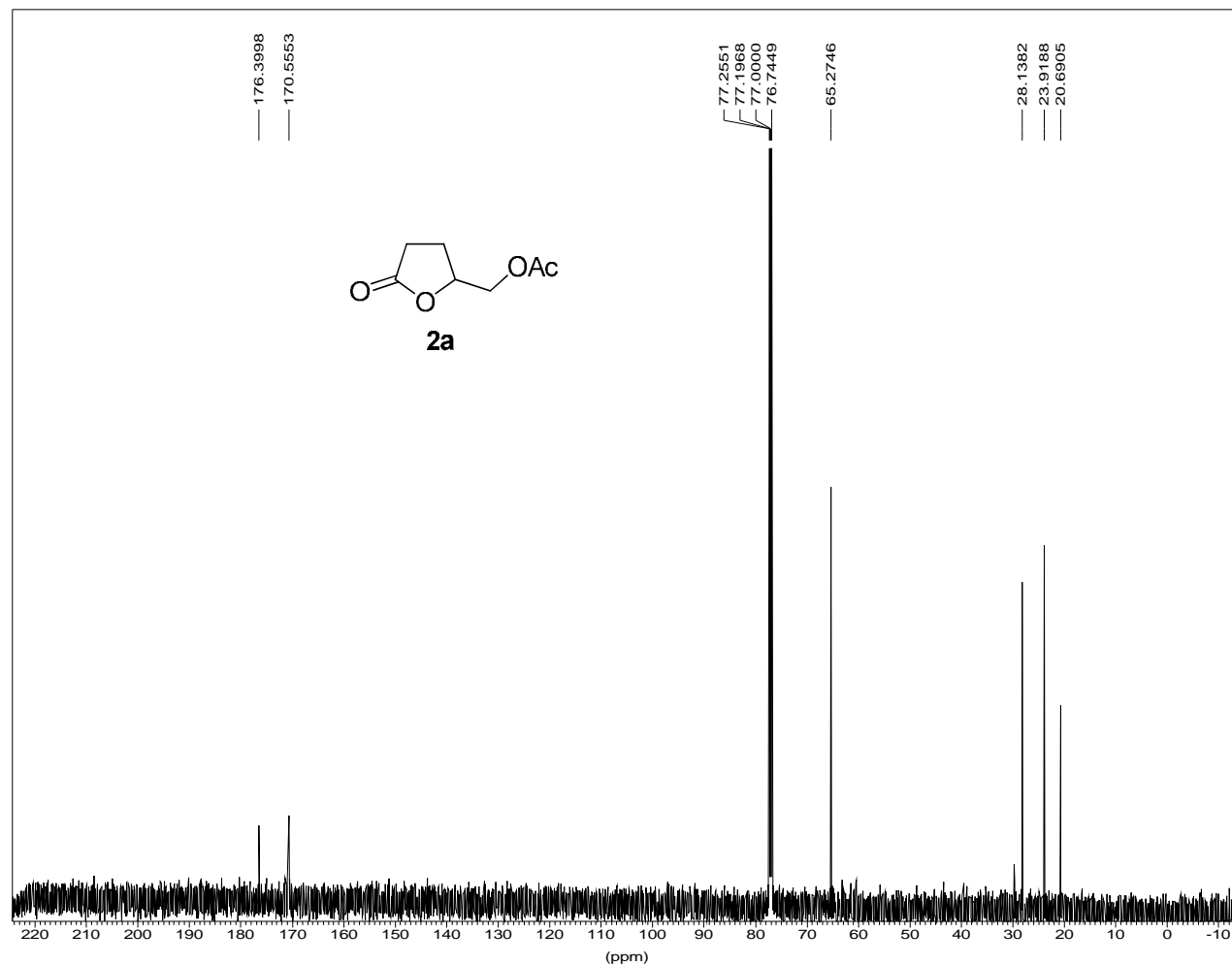
*** Acquisition Parameters ***

BF1 : 500.130000 MHz
LOCNUC : 2H
NS : 8
O1 : 3088.51 Hz
PULPROG : zg30
SFO1 : 500.1330885 MHz
SOLVENT : CDCl3
SW : 20.6557 ppm

*** Processing Parameters ***

LB : 0.30 Hz
PHC0 : 56.574 degree
PHC1 : -1.075 degree

13C AMX500
jas-2-9-1



*** Current Data Parameters ***

NAME : ja15jas1
EXPNO : 2
PROCNO : 1

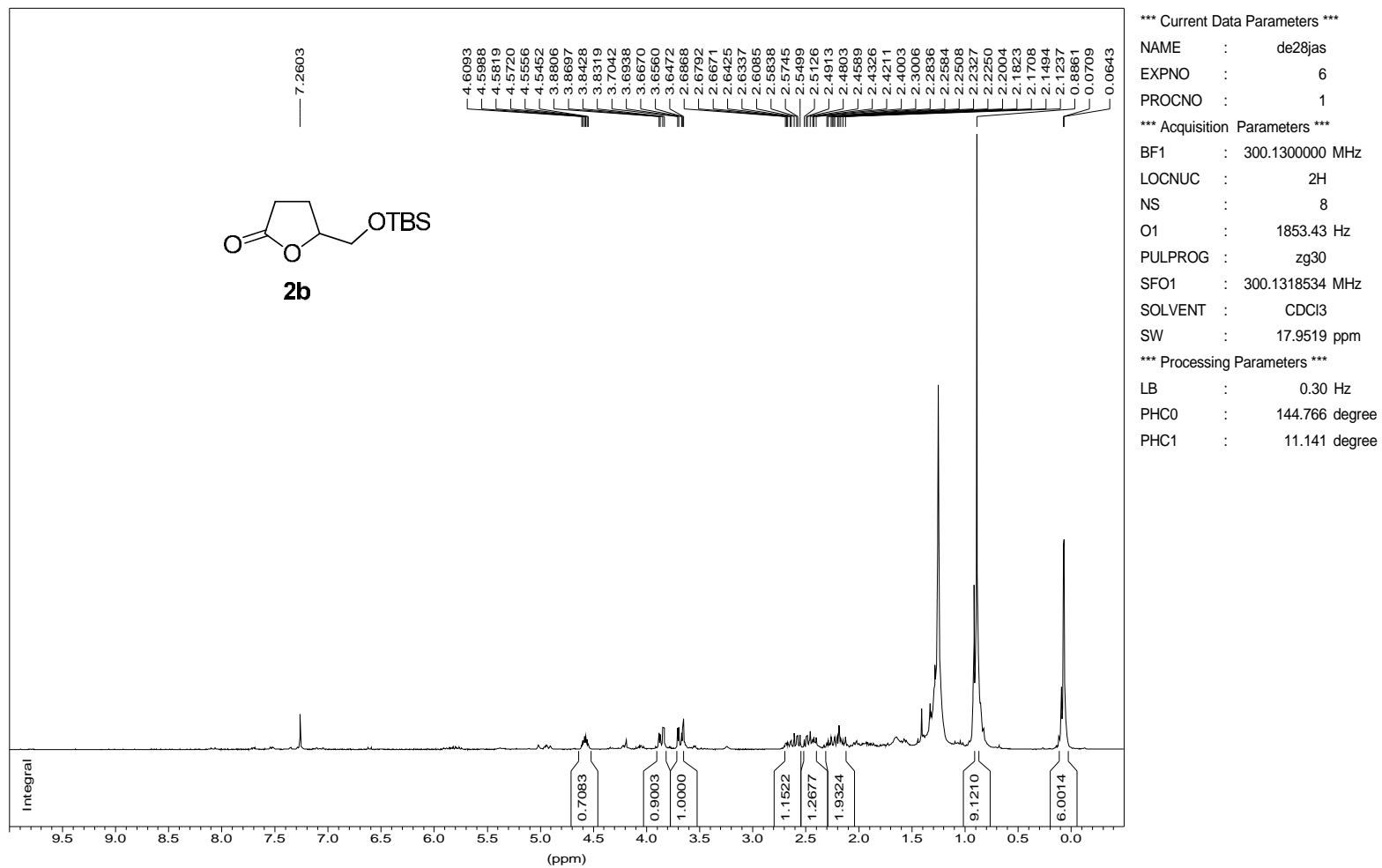
*** Acquisition Parameters ***

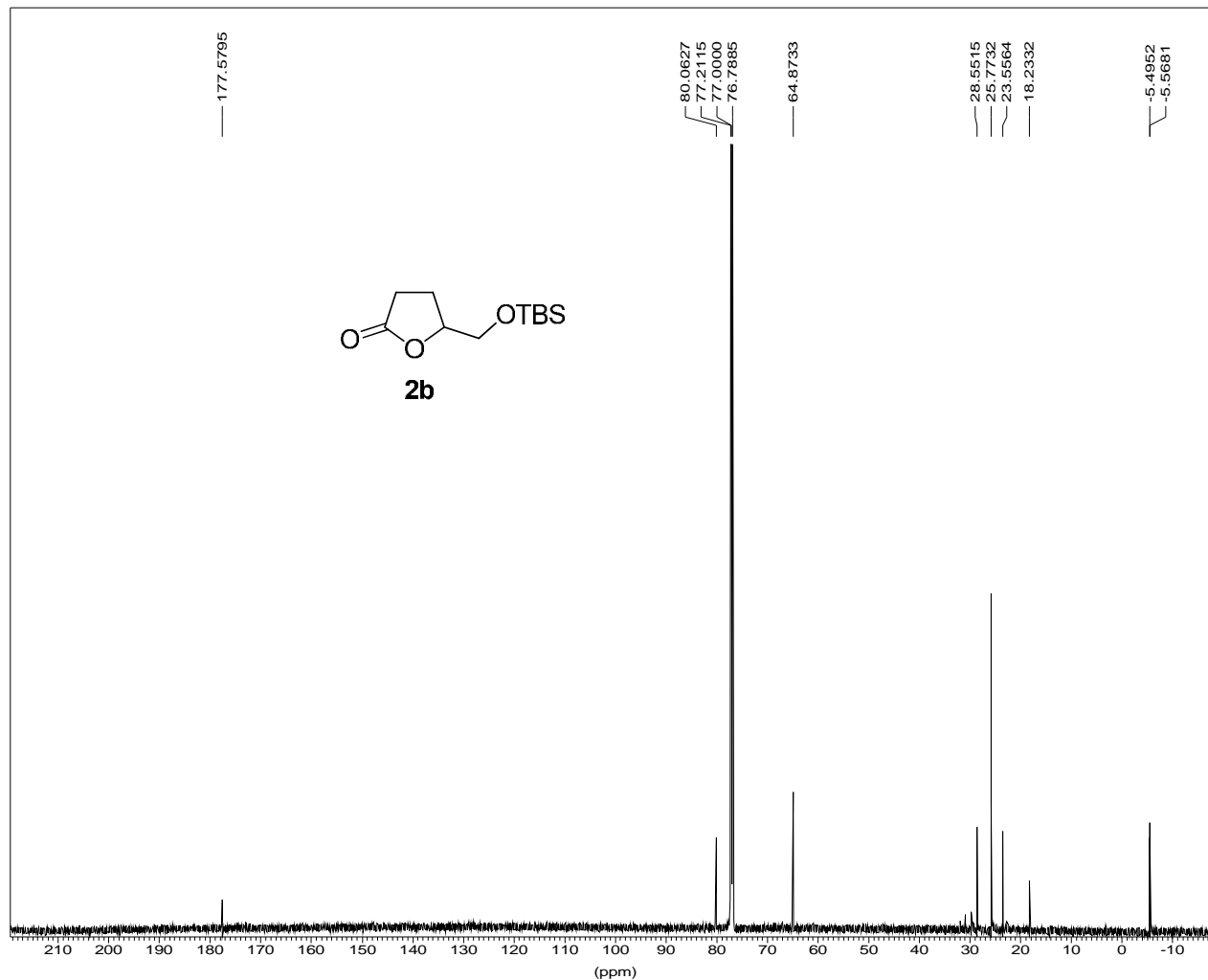
BF1 : 125.7577890 MHz
LOCNUC : 2H
NS : 236
O1 : 13204.57 Hz
PULPROG : zgpg30
SFO1 : 125.7709936 MHz
SOLVENT : CDCl3
SW : 238.7675 ppm

*** Processing Parameters ***

LB : 1.00 Hz
PHC0 : 184.629 degree
PHC1 : 32.388 degree

1H normal range AC300
jas-1-182-1





*** Current Data Parameters ***

NAME : 2013-j-2
EXPNO : 3
PROCNO : 1

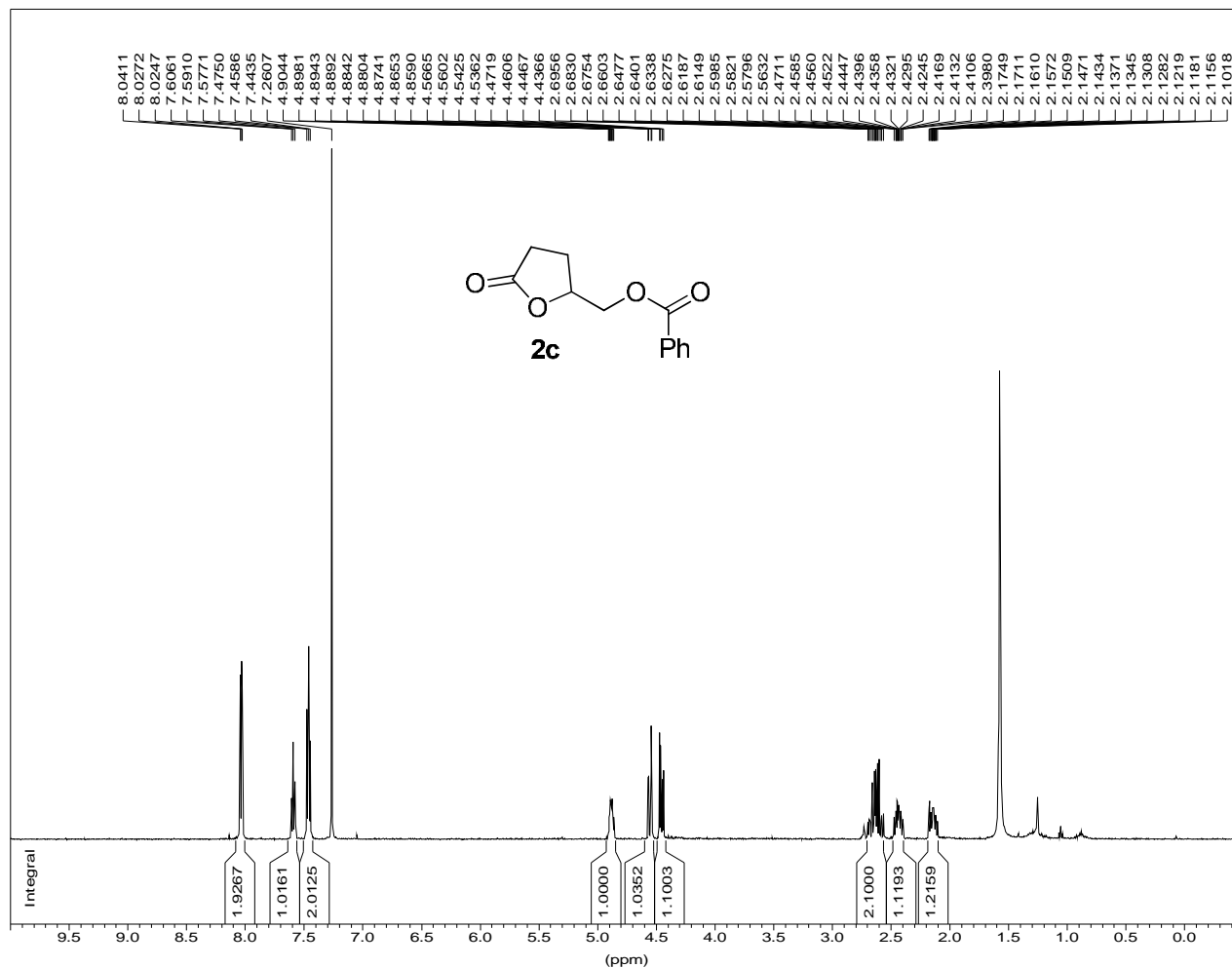
*** Acquisition Parameters ***

BF1 : 150.9028090 MHz
LOCNUC : 2H
NS : 206
O1 : 15089.81 Hz
PULPROG : zgpg30
SFO1 : 150.9178988 MHz
SOLVENT : CDCl3
SW : 238.9226 ppm

*** Processing Parameters ***

LB : 1.00 Hz
PHC0 : 242.214 degree
PHC1 : -121.130 degree

1H AMX500
jas-1-172-1



*** Current Data Parameters ***

NAME : zfy1221
EXPNO : 9
PROCNO : 1

*** Acquisition Parameters ***

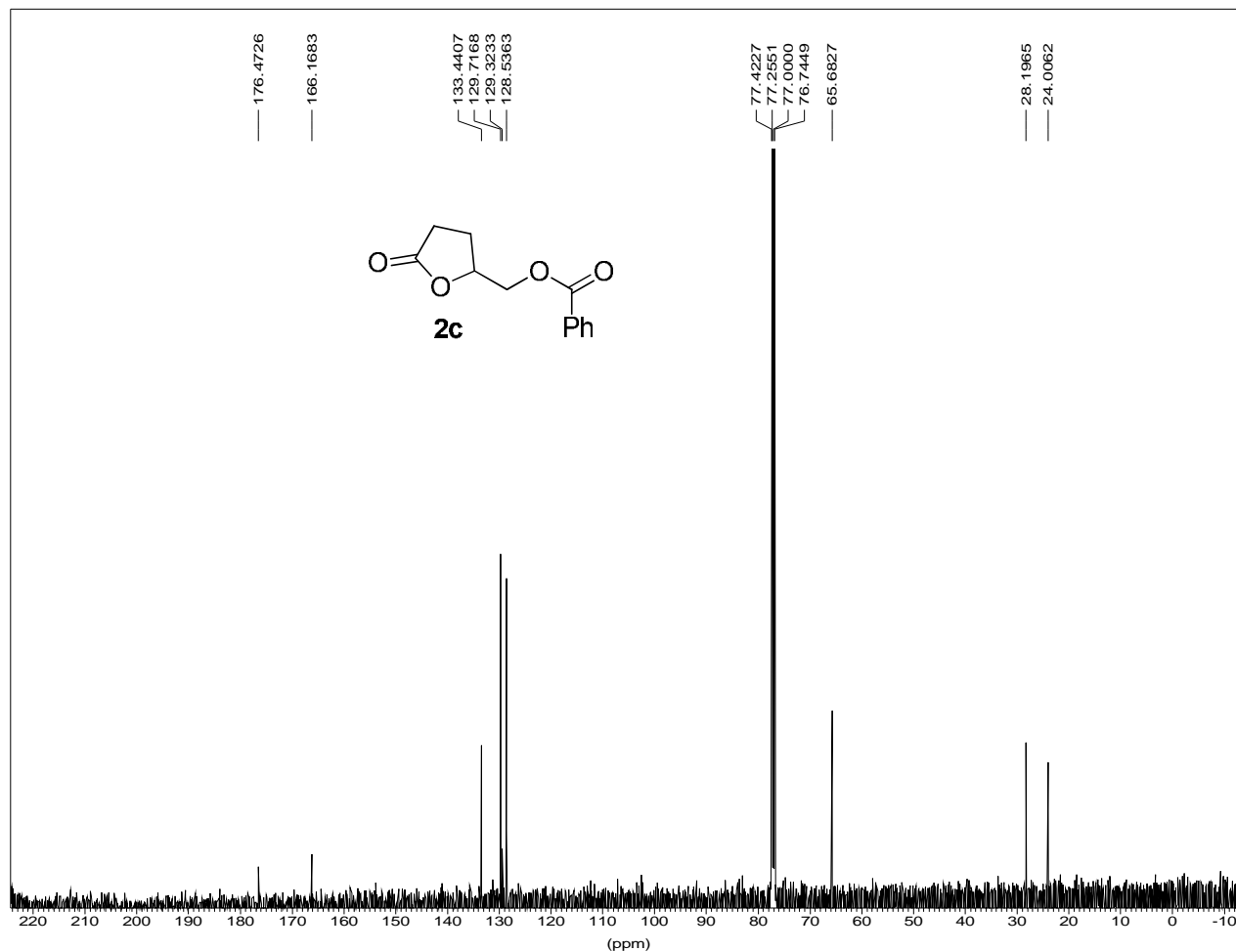
BF1 : 500.130000 MHz
LOCNUC : 2H
NS : 8
O1 : 3088.51 Hz
PULPROG : zg30
SFO1 : 500.1330885 MHz
SOLVENT : CDCl3
SW : 20.6557 ppm

*** Processing Parameters ***

LB : 0.30 Hz
PHC0 : 60.233 degree
PHC1 : 1.716 degree

13C AMX500

jas-1-172-1



*** Current Data Parameters ***

NAME : zfy0111

EXPNO : 1

PROCNO : 1

*** Acquisition Parameters ***

BF1 : 125.7577890 MHz

LOCNUC : 2H

NS : 615

O1 : 13204.57 Hz

PULPROG : zgpg30

SFO1 : 125.7709936 MHz

SOLVENT : CDCl3

SW : 238.7675 ppm

*** Processing Parameters ***

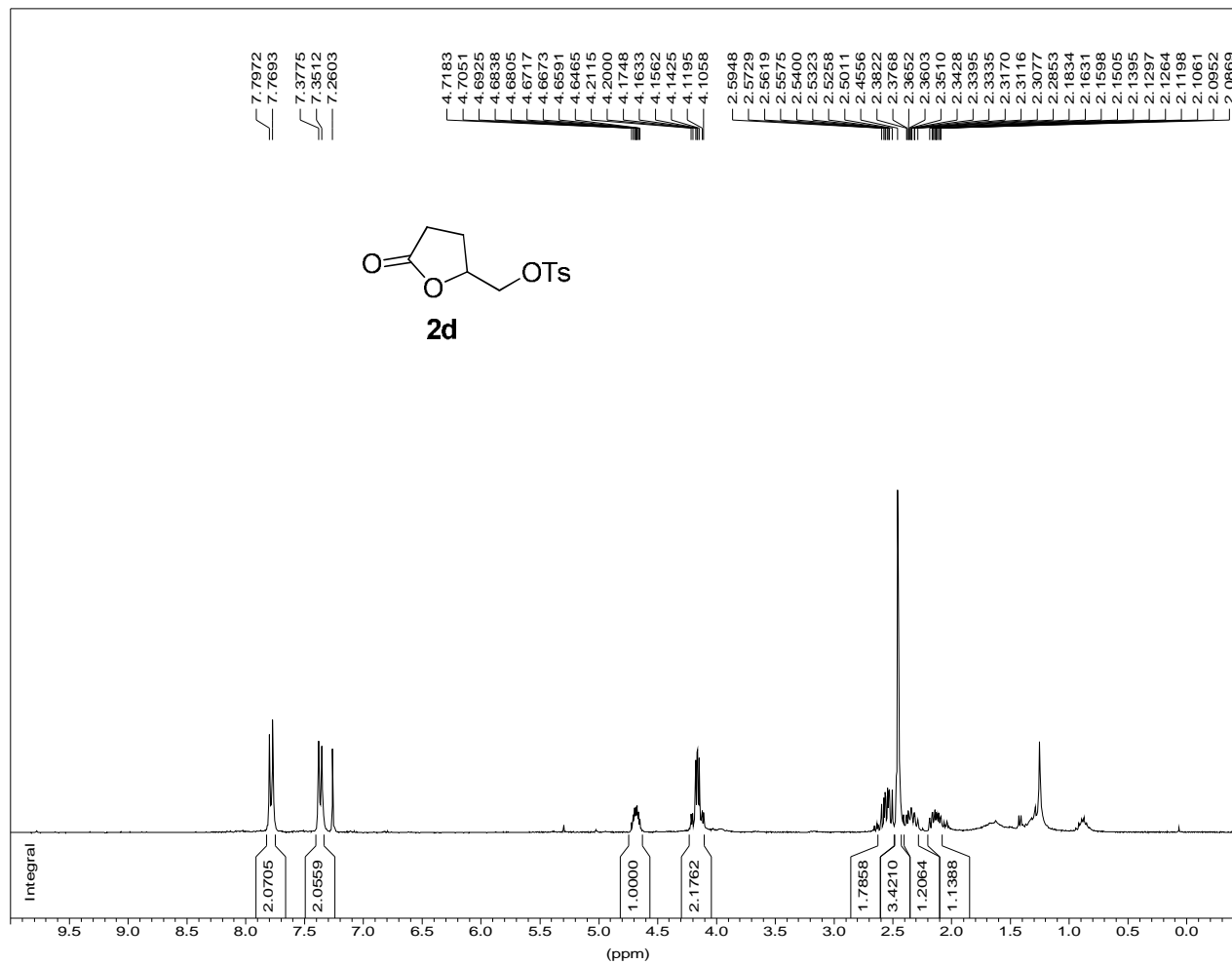
LB : 1.00 Hz

PHC0 : 167.251 degree

PHC1 : 41.795 degree

1H normal range AC300

jas-1-176-1



*** Current Data Parameters ***

NAME : dejas26
EXPNO : 2
PROCNO : 1

*** Acquisition Parameters ***

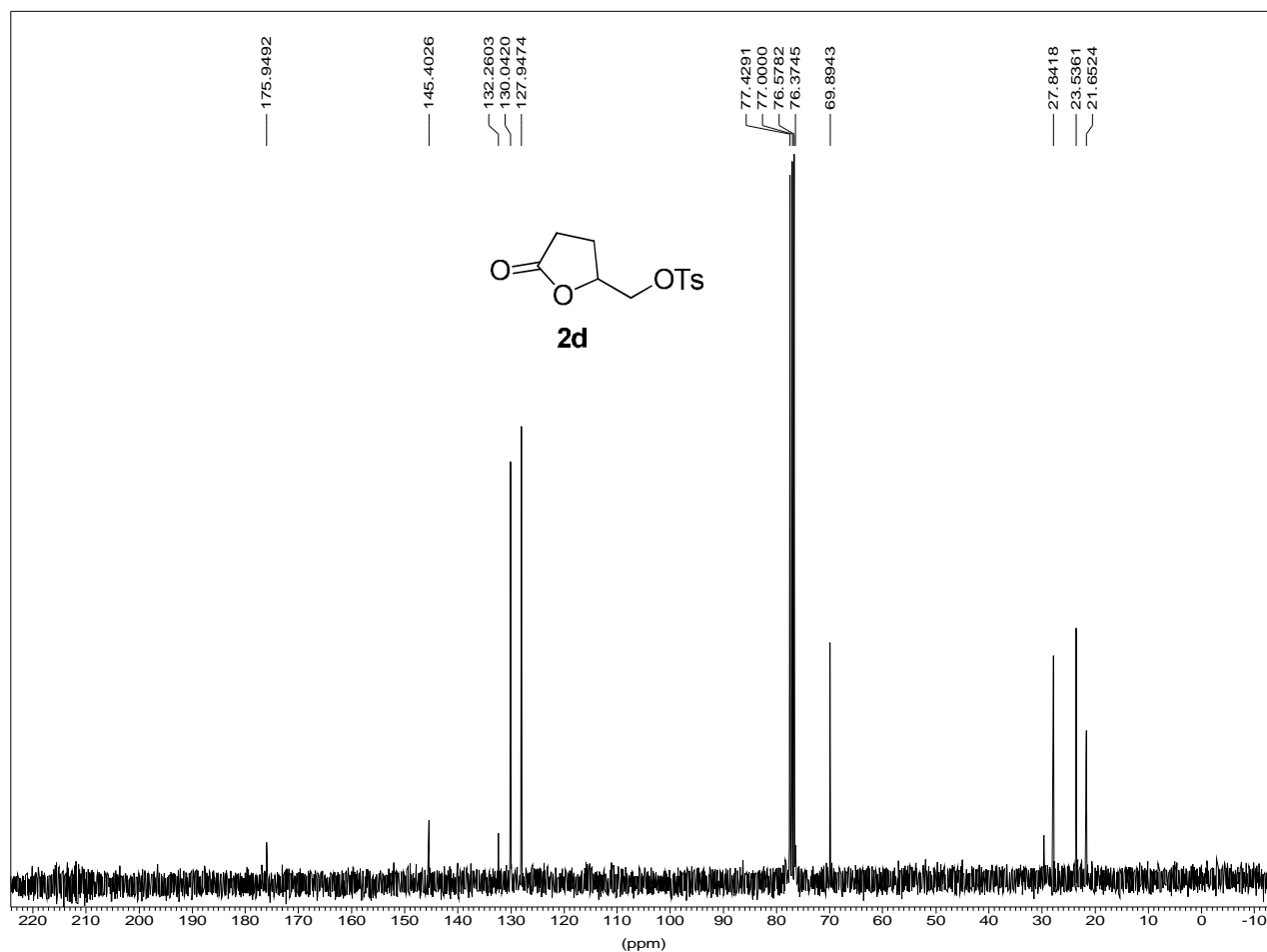
BF1 : 300.130000 MHz
LOCNUC : 2H
NS : 8
O1 : 1853.43 Hz
PULPROG : zg30
SFO1 : 300.1318534 MHz
SOLVENT : CDCl3
SW : 17.9519 ppm

*** Processing Parameters ***

LB : 0.30 Hz
PHC0 : 292.496 degree
PHC1 : 15.095 degree

13C Standard AC300

jas-1-176-1



*** Current Data Parameters ***

NAME : de27jas
EXPNO : 3
PROCNO : 1

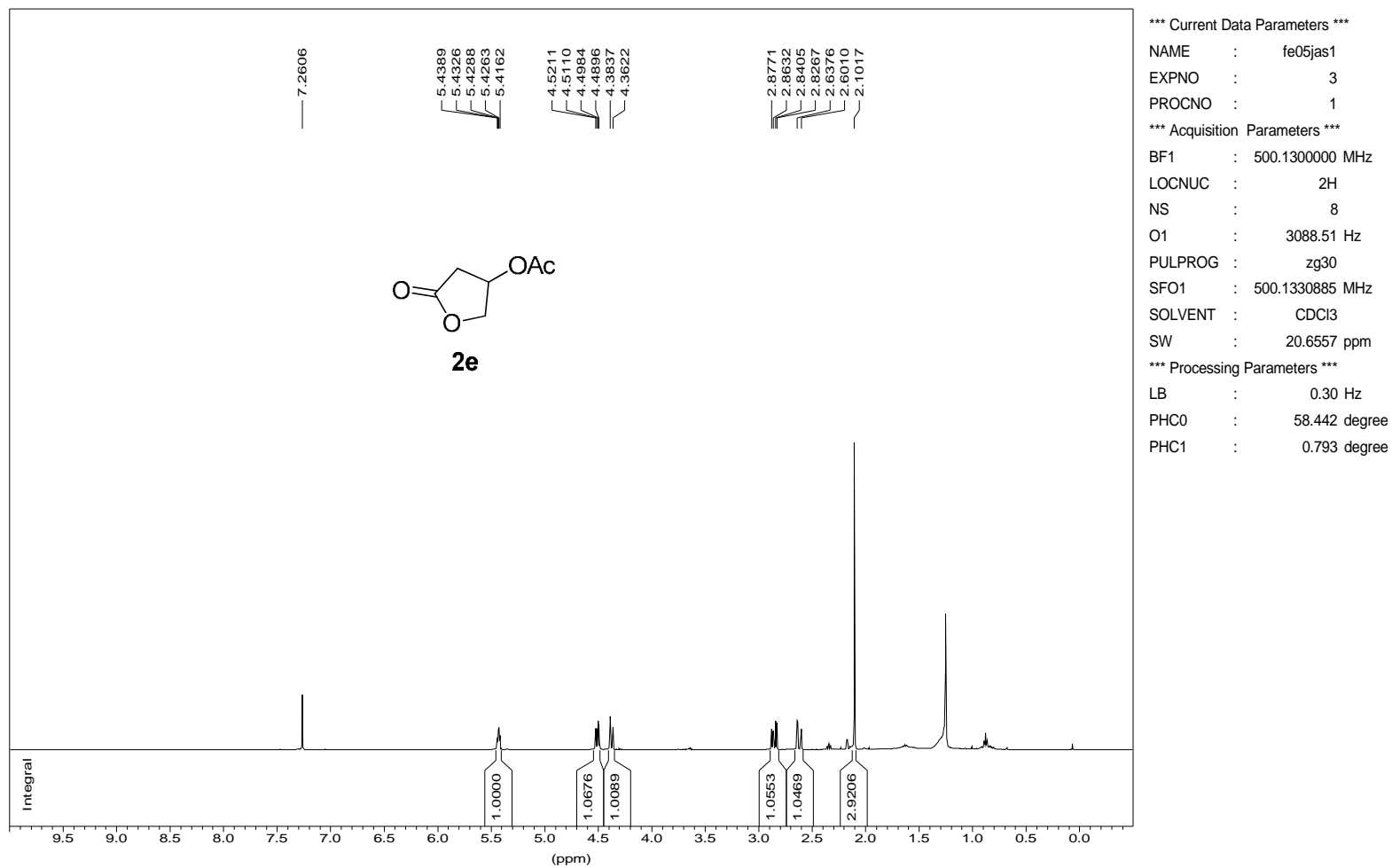
*** Acquisition Parameters ***

BF1 : 75.4677490 MHz
LOCNUC : 2H
NS : 1000
O1 : 7924.11 Hz
PULPROG : zgpg30
SFO1 : 75.4756731 MHz
SOLVENT : CDCl3
SW : 238.2968 ppm

*** Processing Parameters ***

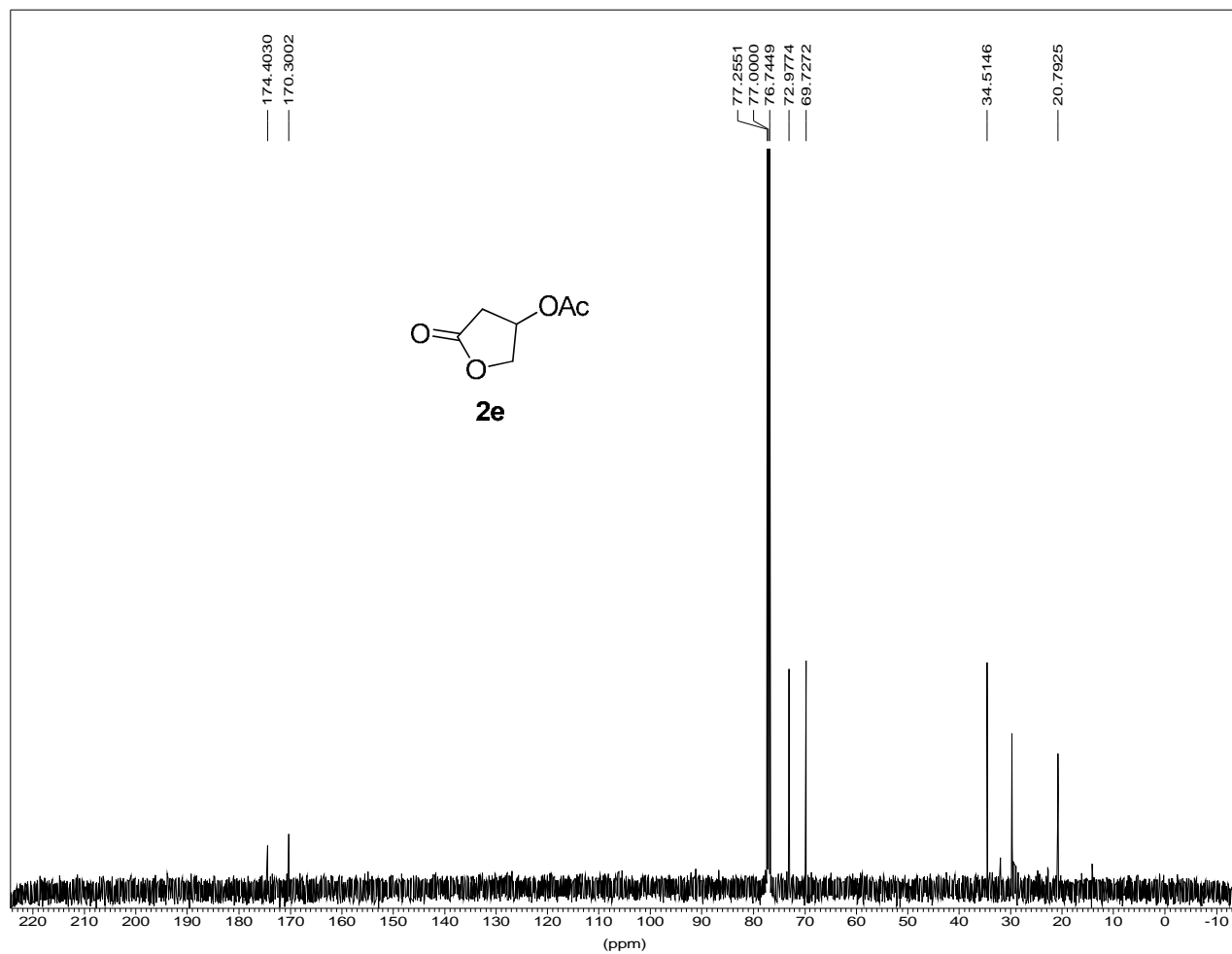
LB : 1.00 Hz
PHC0 : 10.834 degree
PHC1 : 20.848 degree

1H AMX500
jas-2-38-2



13C AMX500

jas-2-38-2



*** Current Data Parameters ***

NAME : fe05jas1

EXPNO : 4

PROCNO : 1

*** Acquisition Parameters ***

BF1 : 125.7577890 MHz

LOCNUC : 2H

NS : 343

O1 : 13204.57 Hz

PULPROG : zgpg30

SFO1 : 125.7709936 MHz

SOLVENT : CDCl3

SW : 238.7675 ppm

*** Processing Parameters ***

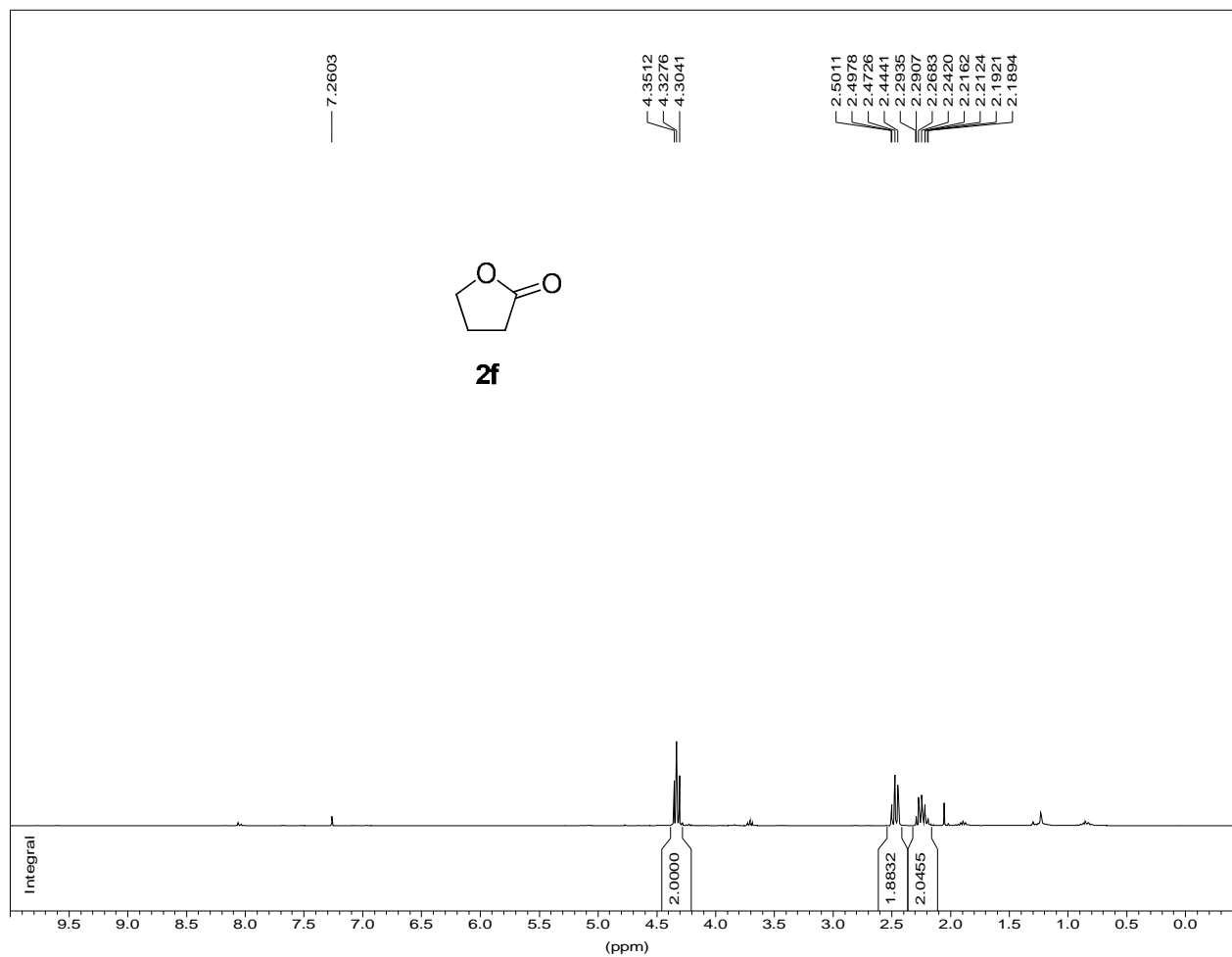
LB : 1.00 Hz

PHC0 : 188.455 degree

PHC1 : 29.919 degree

1H normal range AC300

139-9-11



*** Current Data Parameters ***

NAME : ma23hwk

EXPNO : 2

PROCNO : 1

*** Acquisition Parameters ***

BF1 : 300.1300000 MHz

LOCNUC : 2H

NS : 8

O1 : 1853.43 Hz

PULPROG : zg30

SFO1 : 300.1318534 MHz

SOLVENT : CDCl3

SW : 17.9519 ppm

*** Processing Parameters ***

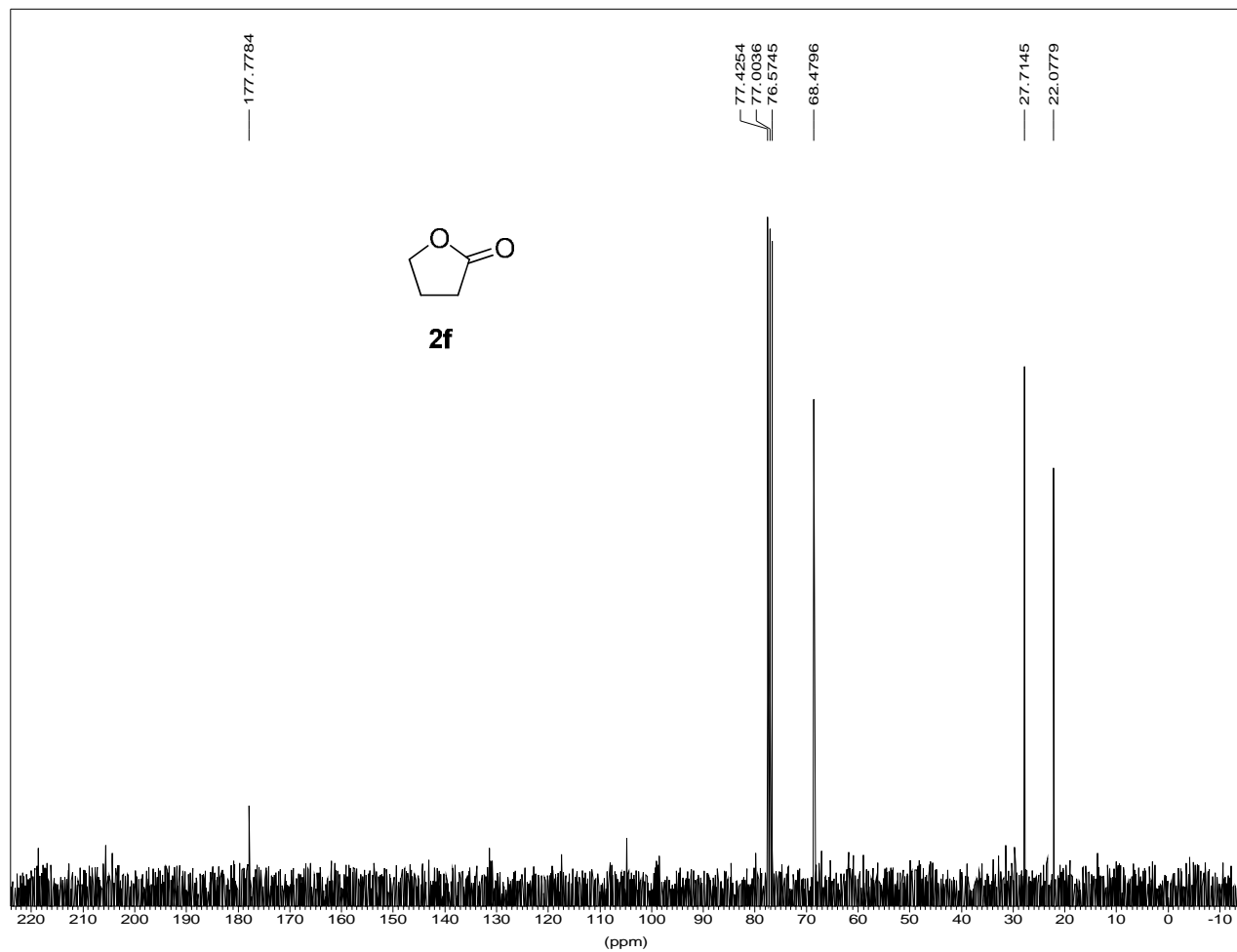
LB : 0.30 Hz

PHC0 : 190.734 degree

PHC1 : 50.514 degree

13C Standard AC300

139-9-11



*** Current Data Parameters ***

NAME : ma23hwk

EXPNO : 3

PROCNO : 1

*** Acquisition Parameters ***

BF1 : 75.4677490 MHz

LOCNUC : 2H

NS : 51

O1 : 7924.11 Hz

PULPROG : zgpg30

SFO1 : 75.4756731 MHz

SOLVENT : CDCl3

SW : 238.2968 ppm

*** Processing Parameters ***

LB : 1.00 Hz

PHC0 : 126.884 degree

PHC1 : 18.691 degree

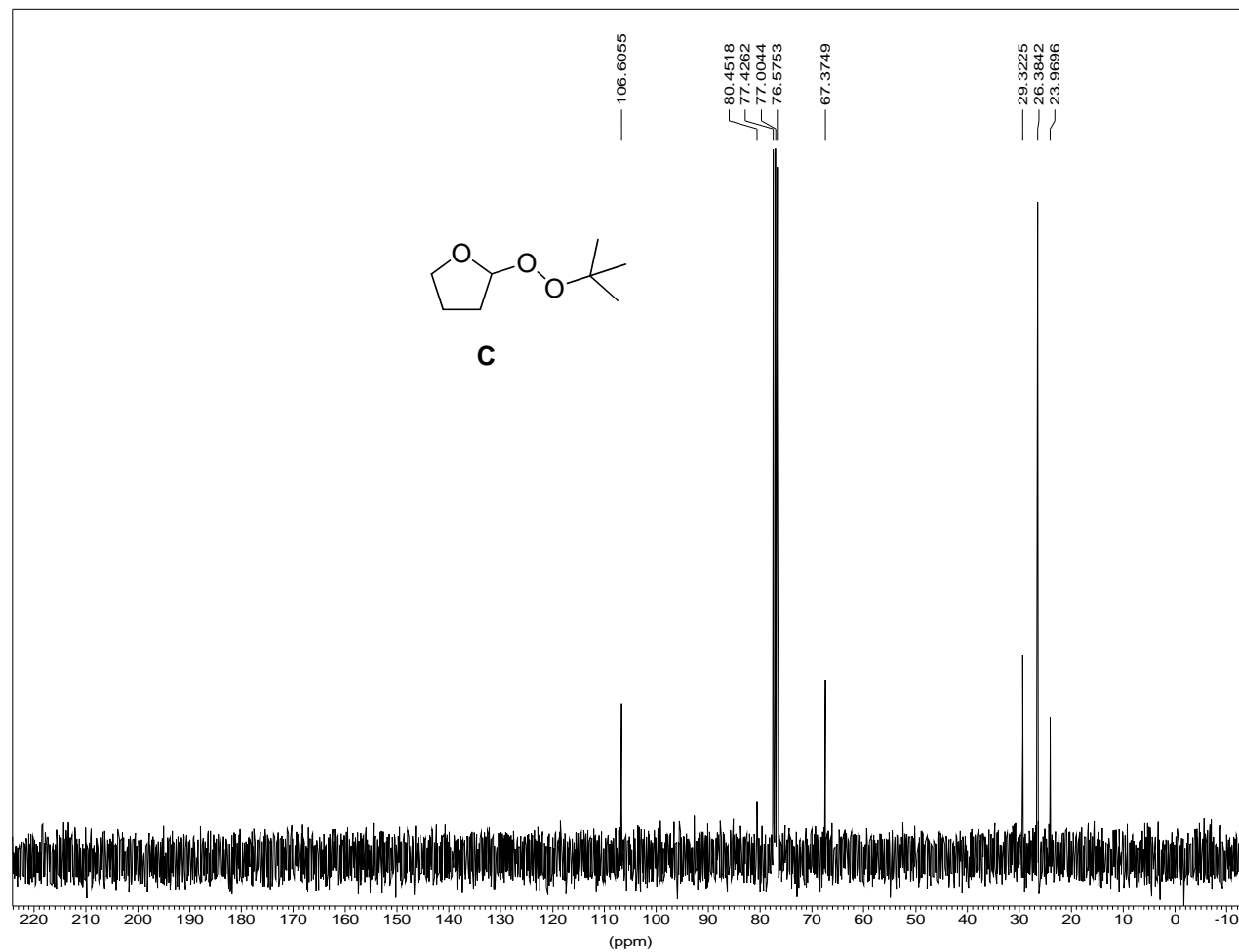
1H normal range AC300

133- 7-9



13C Standard AC300

133-THF



*** Current Data Parameters ***

NAME : fe10hwk
EXPNO : 3
PROCNO : 1

*** Acquisition Parameters ***

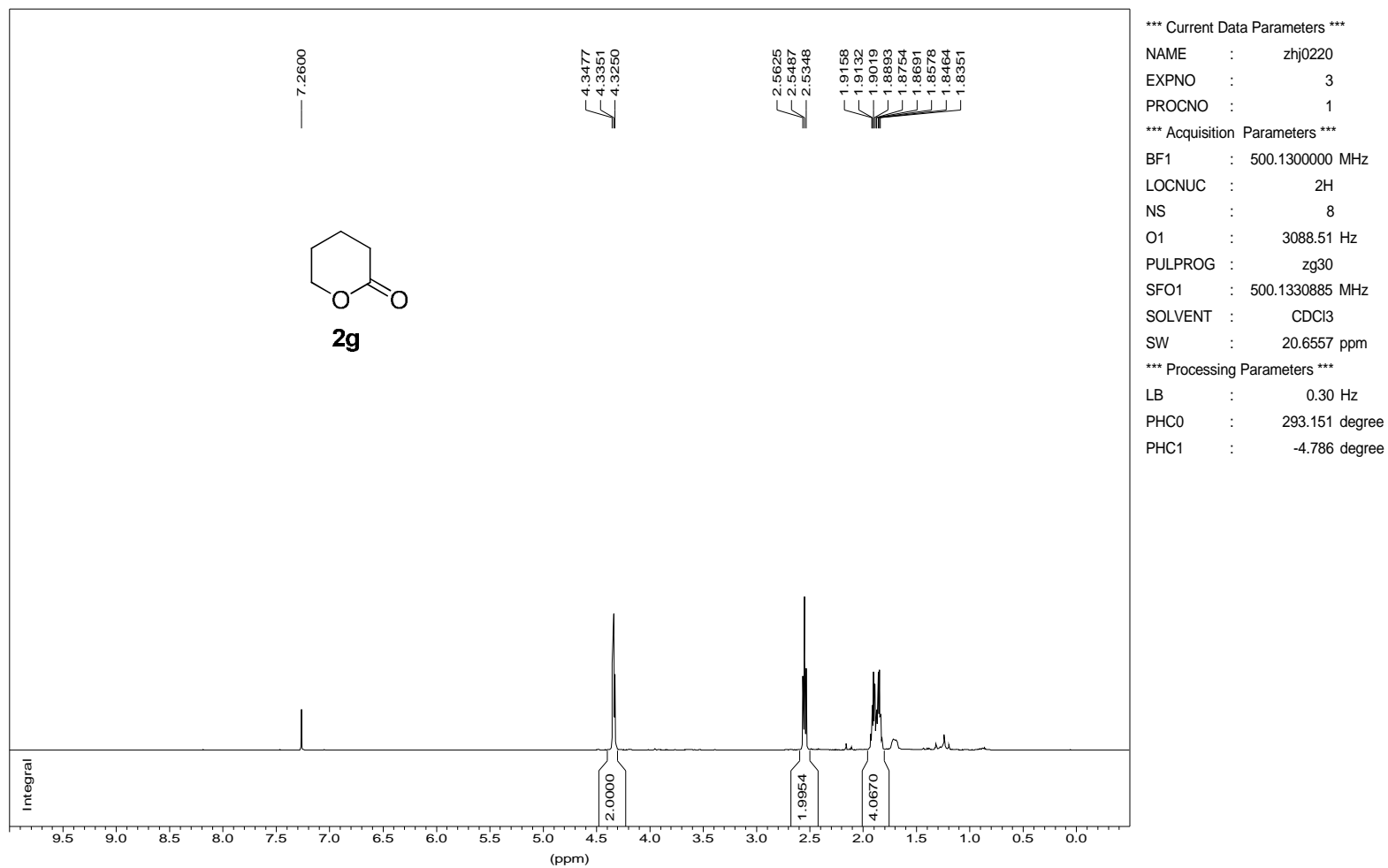
BF1 : 75.4677490 MHz
LOCNUC : 2H
NS : 206
O1 : 7924.11 Hz
PULPROG : zgpg30
SFO1 : 75.4756731 MHz
SOLVENT : CDCl3
SW : 238.2968 ppm

*** Processing Parameters ***

LB : 1.00 Hz
PHC0 : 91.206 degree
PHC1 : -78.407 degree

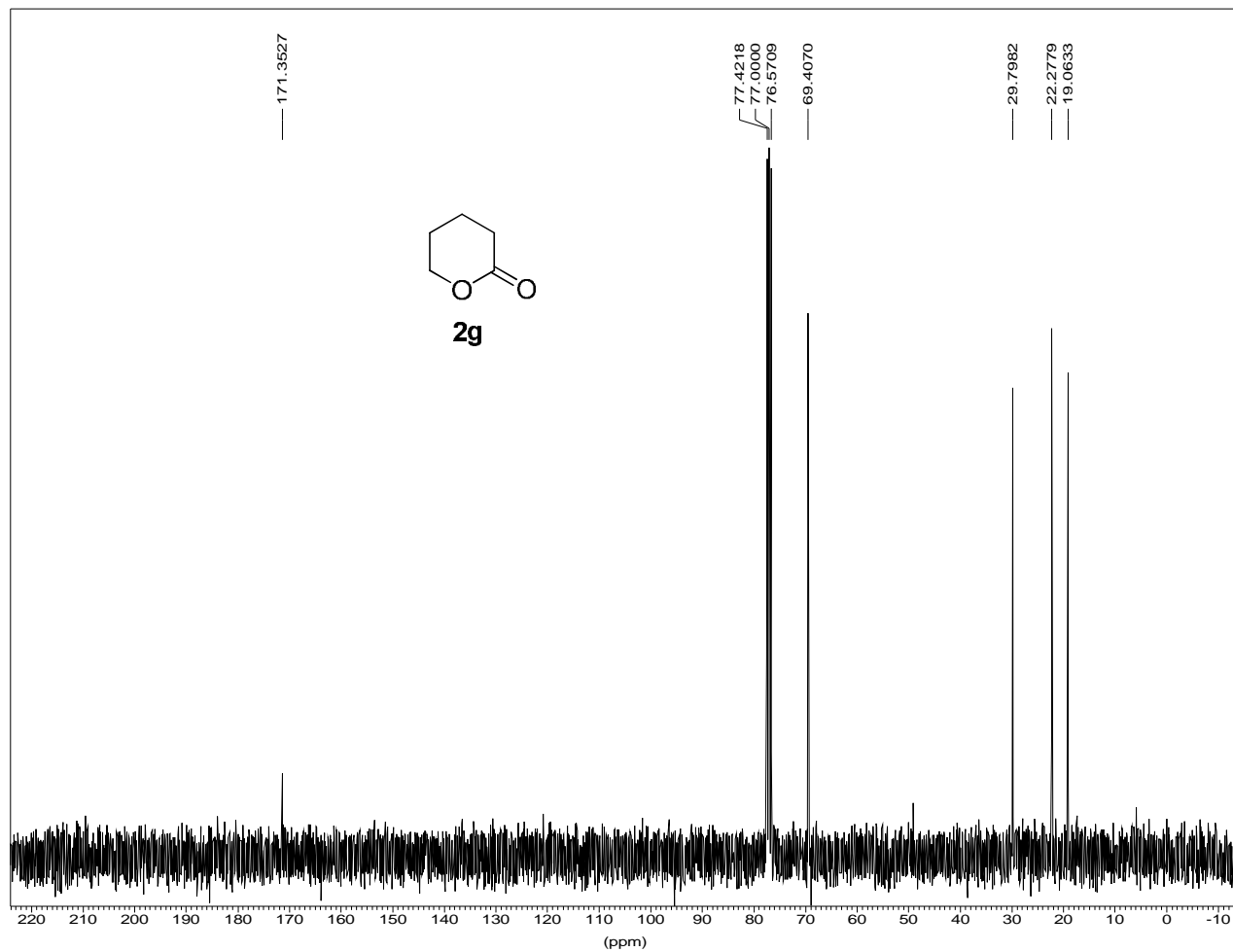
1H AMX500

Jas-2-44-1



13C Standard AC300

jas-2-44-1



*** Current Data Parameters ***

NAME : ma11jas
EXPNO : 2
PROCNO : 1

*** Acquisition Parameters ***

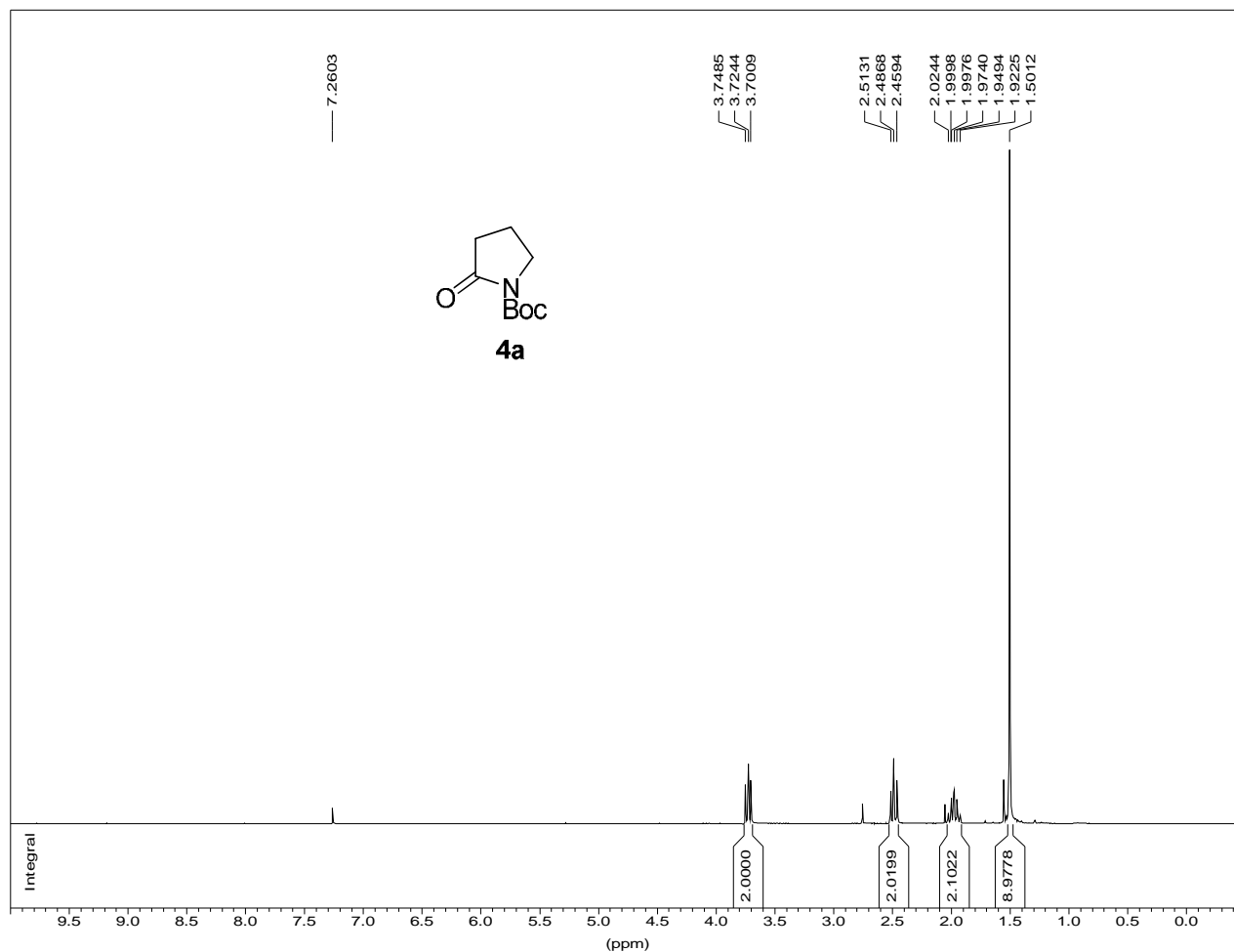
BF1 : 75.4677490 MHz
LOCNUC : 2H
NS : 416
O1 : 7924.11 Hz
PULPROG : zgpg30
SFO1 : 75.4756731 MHz
SOLVENT : CDCl3
SW : 238.2968 ppm

*** Processing Parameters ***

LB : 1.00 Hz
PHC0 : 202.782 degree
PHC1 : 9.367 degree

1H normal range AC300

68



*** Current Data Parameters ***

NAME : oc22hwk
EXPNO : 1
PROCNO : 1

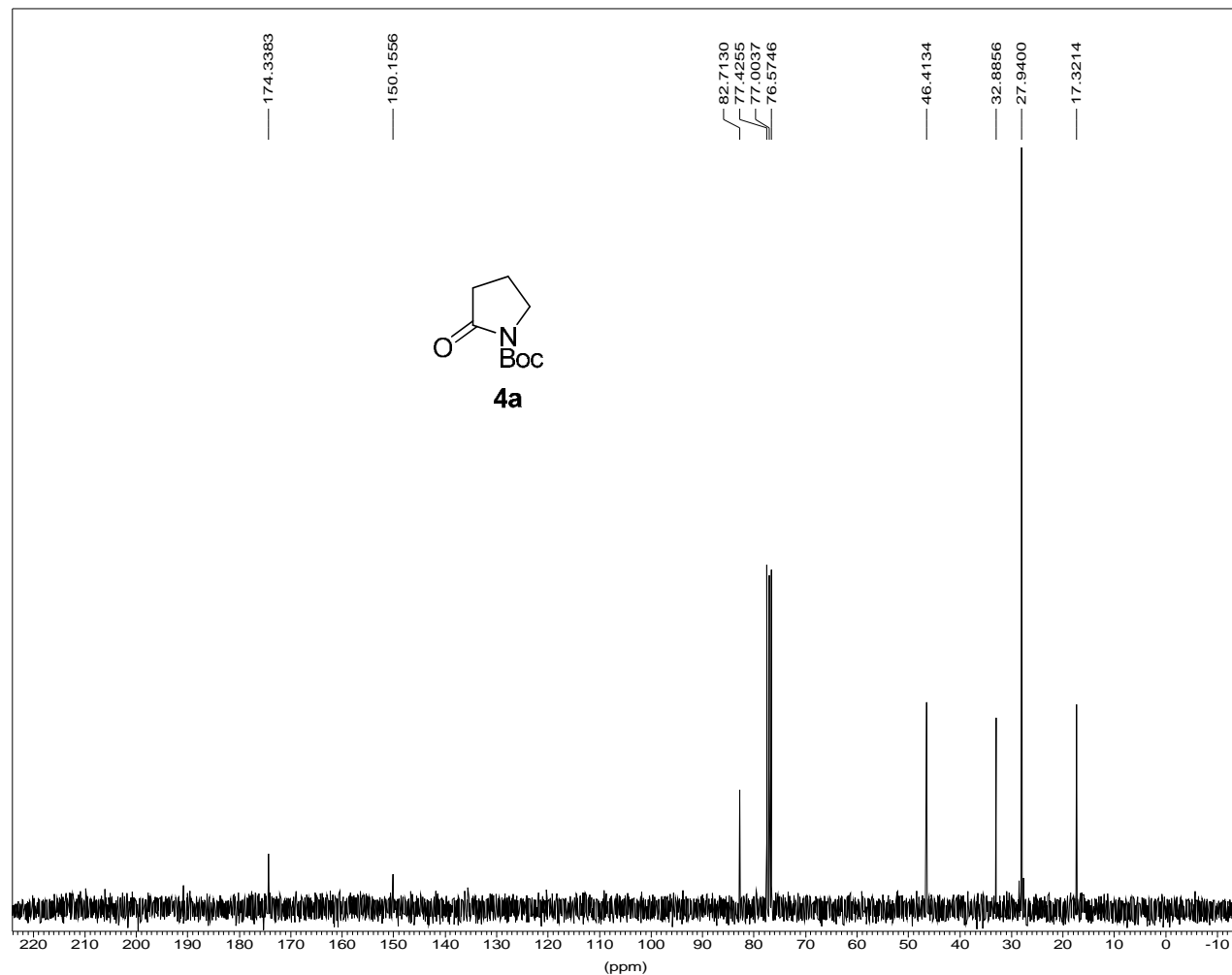
*** Acquisition Parameters ***

BF1 : 300.130000 MHz
LOCNUC : 2H
NS : 8
O1 : 1853.43 Hz
PULPROG : zg30
SFO1 : 300.1318534 MHz
SOLVENT : CDCl3
SW : 17.9519 ppm

*** Processing Parameters ***

LB : 0.30 Hz
PHC0 : 69.492 degree
PHC1 : -32.321 degree

13C Standard AC300



*** Current Data Parameters ***

NAME : oc22hwk
EXPNO : 2
PROCNO : 1

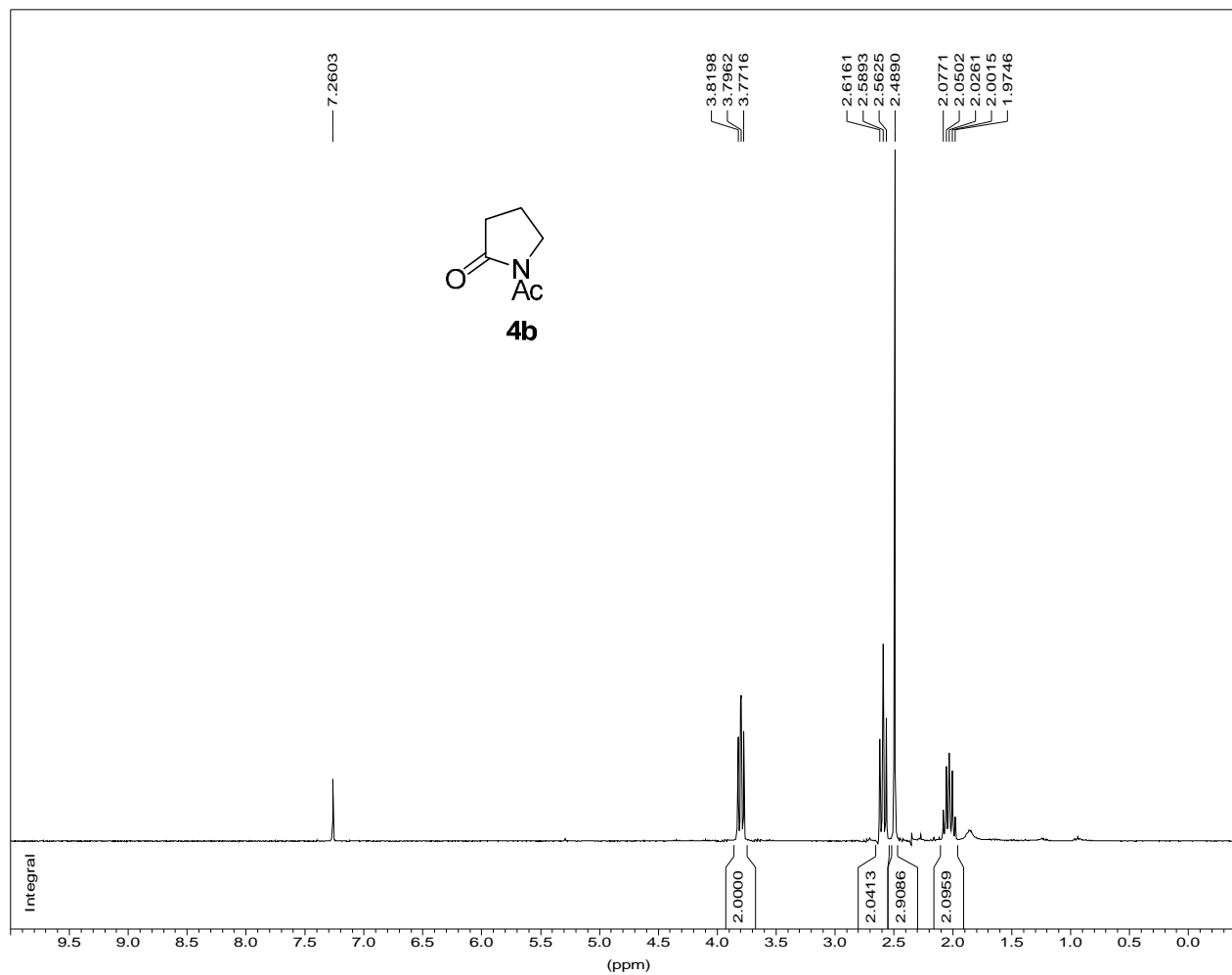
*** Acquisition Parameters ***

BF1 : 75.4677490 MHz
LOCNUC : 2H
NS : 80
O1 : 7924.11 Hz
PULPROG : zgpg30
SFO1 : 75.4756731 MHz
SOLVENT : CDCl3
SW : 238.2968 ppm

*** Processing Parameters ***

LB : 1.00 Hz
PHC0 : 231.564 degree
PHC1 : -13.674 degree

1H normal range AC300



*** Current Data Parameters ***

NAME : oc26hwk
EXPNO : 1
PROCNO : 1

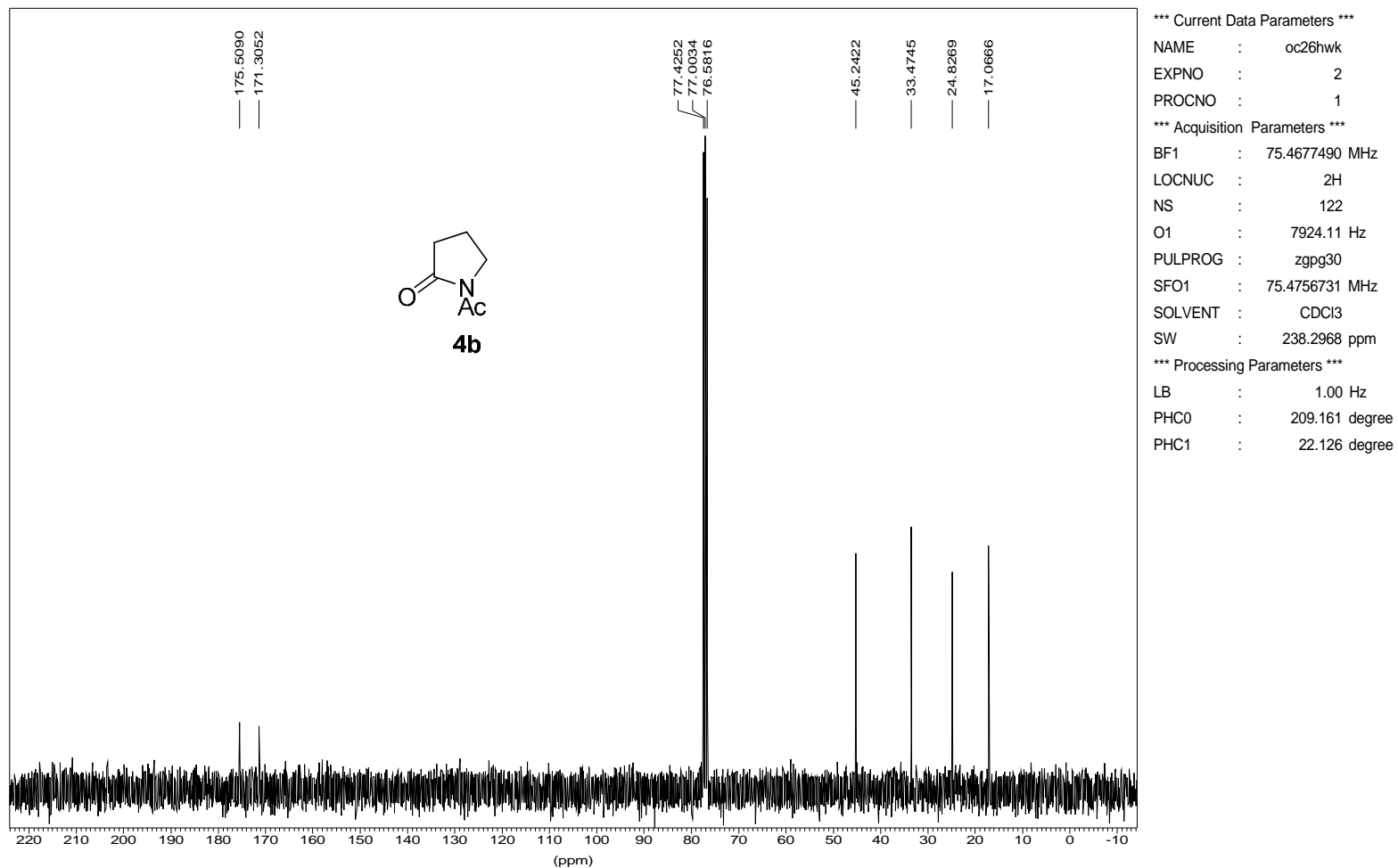
*** Acquisition Parameters ***

BF1 : 300.130000 MHz
LOCNUC : 2H
NS : 8
O1 : 1853.43 Hz
PULPROG : zg30
SFO1 : 300.1318534 MHz
SOLVENT : CDCl3
SW : 17.9519 ppm

*** Processing Parameters ***

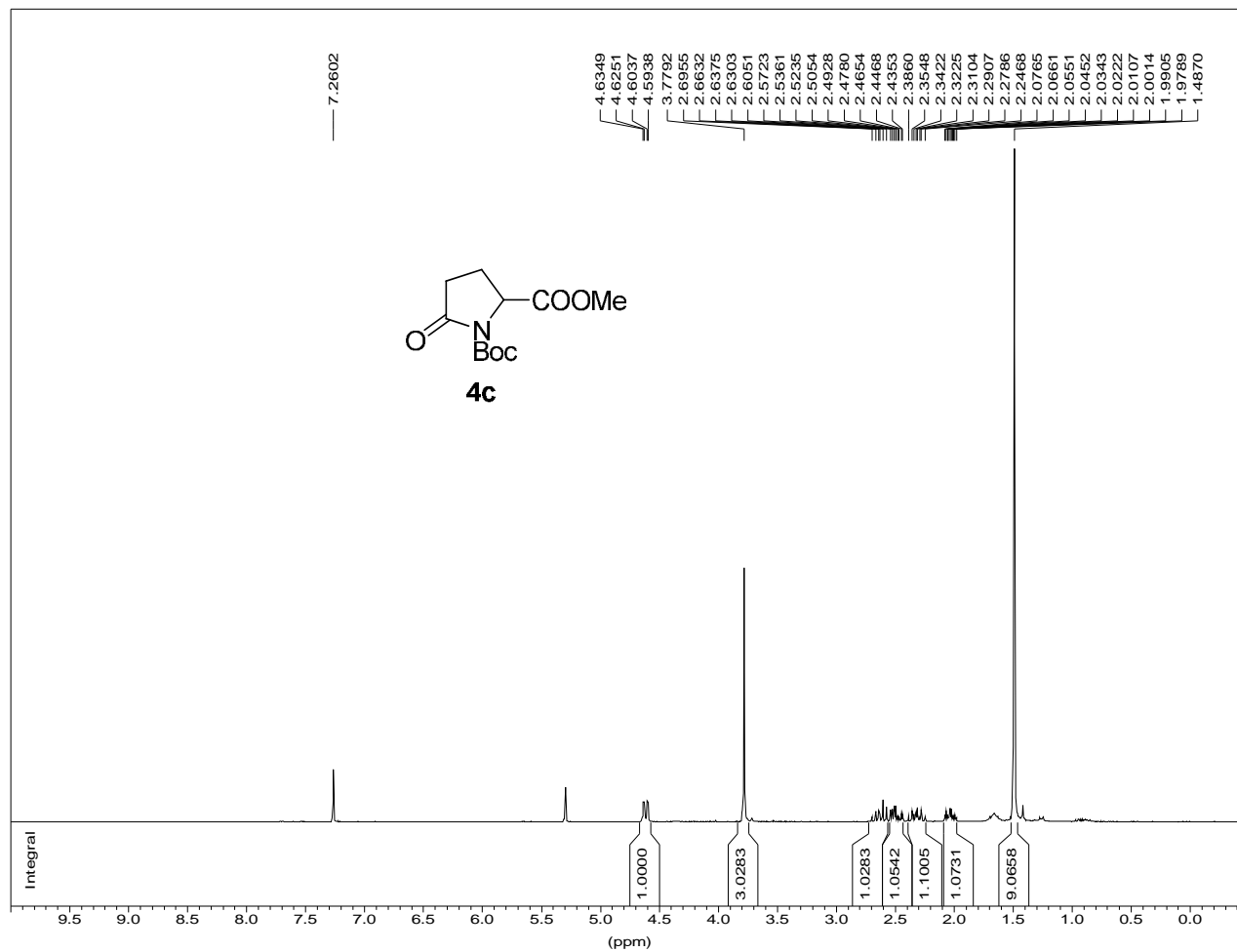
LB : 0.30 Hz
PHC0 : 310.673 degree
PHC1 : -4.202 degree

13C Standard AC300



1H normal range AC300

77



*** Current Data Parameters ***

NAME : nv08hwk
EXPNO : 2
PROCNO : 1

*** Acquisition Parameters ***

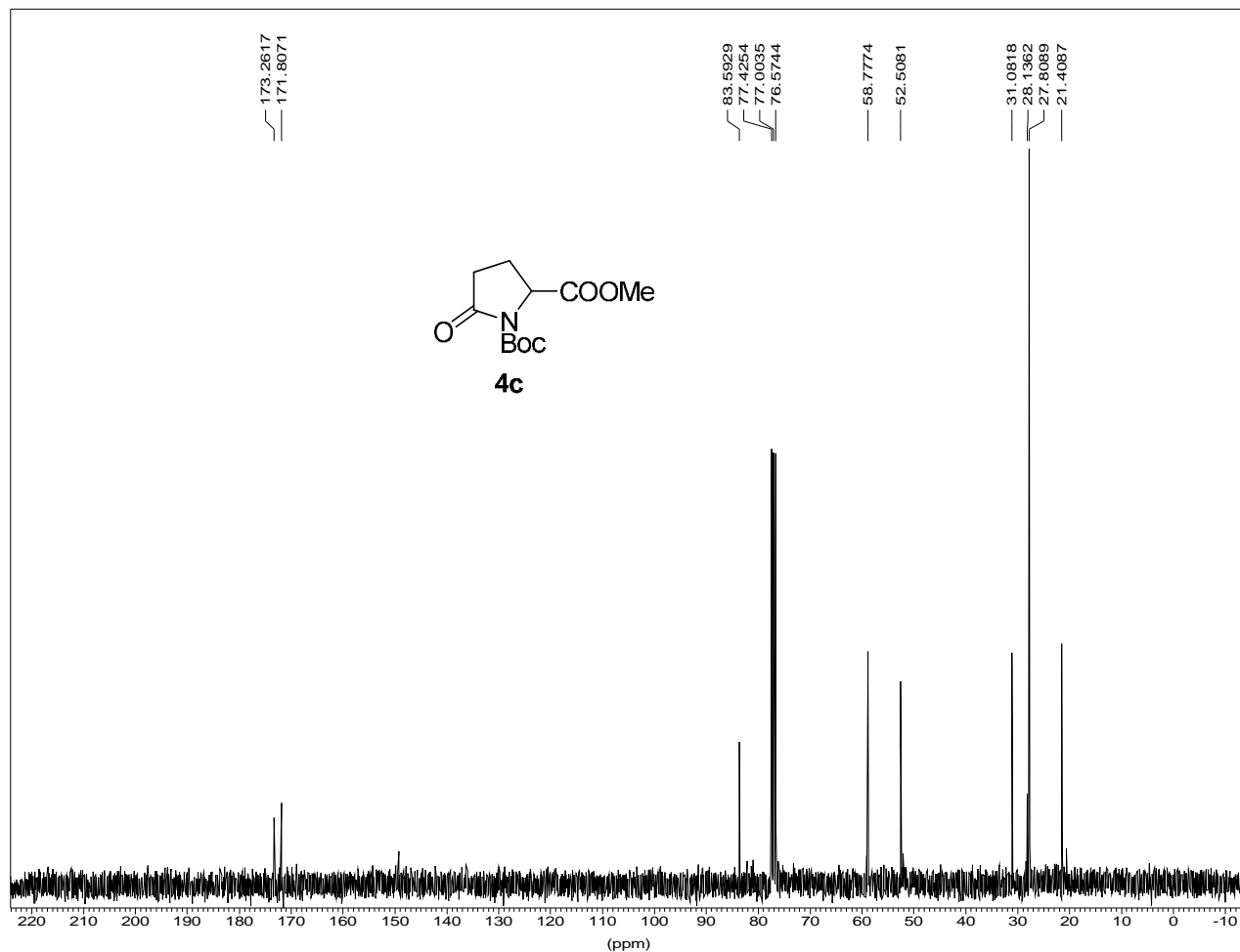
BF1 : 300.1300000 MHz
LOCNUC : 2H
NS : 8
O1 : 1853.43 Hz
PULPROG : zg30
SFO1 : 300.1318534 MHz
SOLVENT : CDCl3
SW : 17.9519 ppm

*** Processing Parameters ***

LB : 0.30 Hz
PHC0 : 304.447 degree
PHC1 : -3.649 degree

13C Standard AC300

7714,15



*** Current Data Parameters ***

NAME : nv04hwk
EXPNO : 4
PROCNO : 1

*** Acquisition Parameters ***

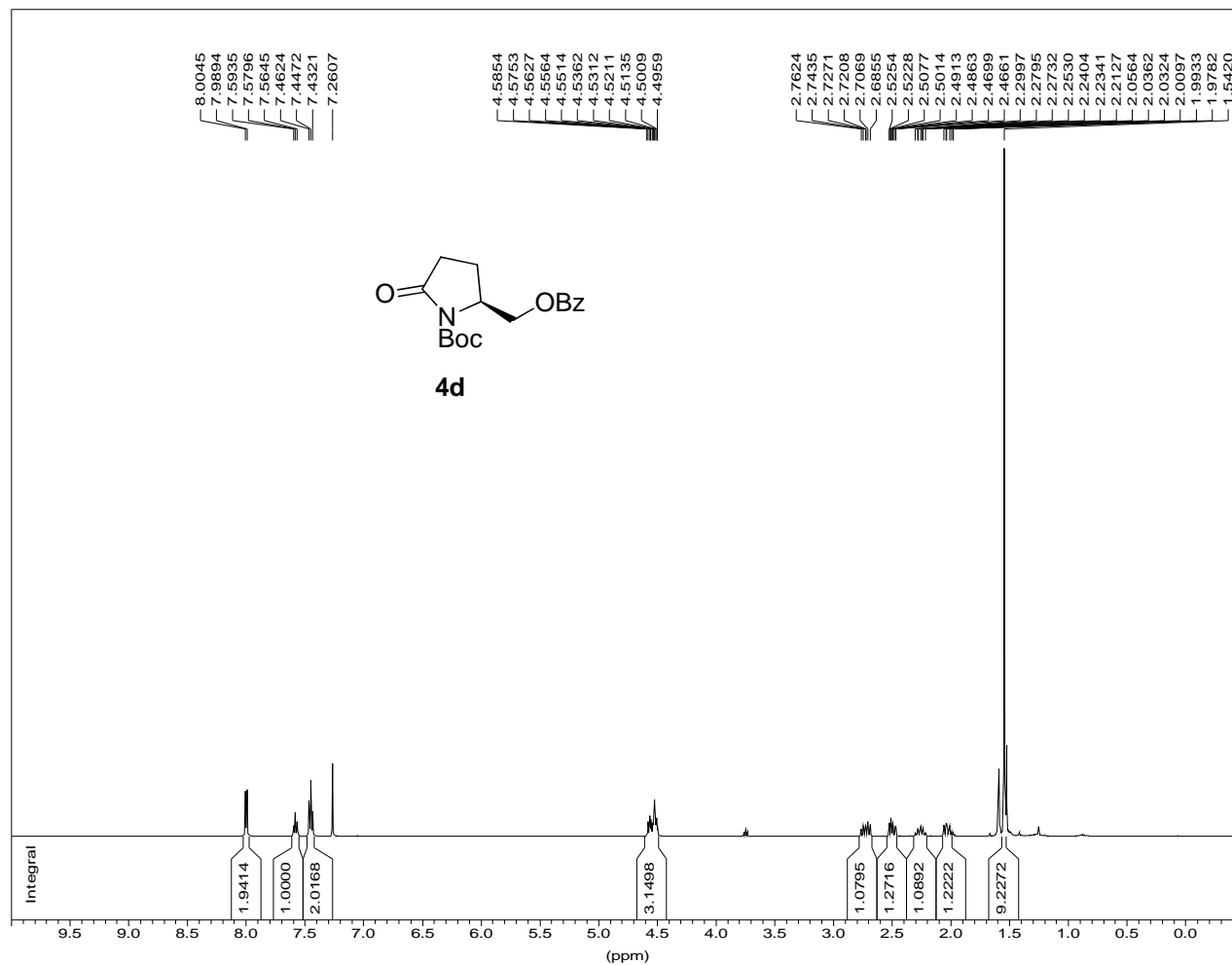
BF1 : 75.4677490 MHz
LOCNUC : 2H
NS : 223
O1 : 7924.11 Hz
PULPROG : zgpg30
SFO1 : 75.4756731 MHz
SOLVENT : CDCl3
SW : 238.2968 ppm

*** Processing Parameters ***

LB : 1.00 Hz
PHC0 : 187.926 degree
PHC1 : 65.569 degree

1H AMX500

jas-1-171-1



*** Current Data Parameters ***

NAME : zfy1221
EXPNO : 2
PROCNO : 1

*** Acquisition Parameters ***

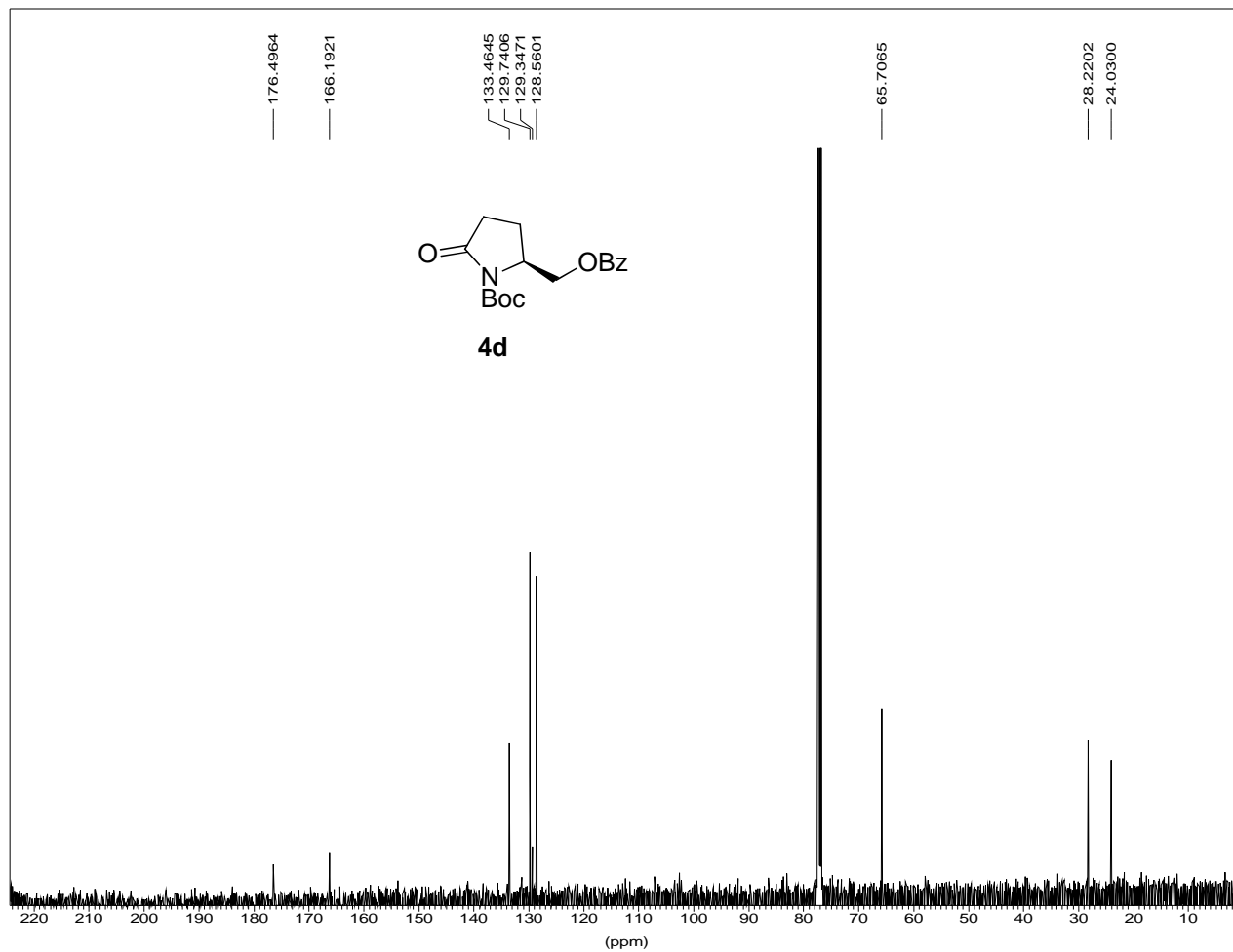
BF1 : 500.130000 MHz
LOCNUC : 2H
NS : 8
O1 : 3088.51 Hz
PULPROG : zg30
SFO1 : 500.133085 MHz
SOLVENT : CDCl3
SW : 20.6557 ppm

*** Processing Parameters ***

LB : 0.30 Hz
PHC0 : 60.233 degree
PHC1 : 1.716 degree

13C AMX500

jas-1-172-1



*** Current Data Parameters ***

NAME : zfy0111

EXPNO : 1

PROCNO : 1

*** Acquisition Parameters ***

BF1 : 125.7577890 MHz

LOCNUC : 2H

NS : 615

O1 : 13204.57 Hz

PULPROG : zgpg30

SFO1 : 125.7709936 MHz

SOLVENT : CDCl3

SW : 238.7675 ppm

*** Processing Parameters ***

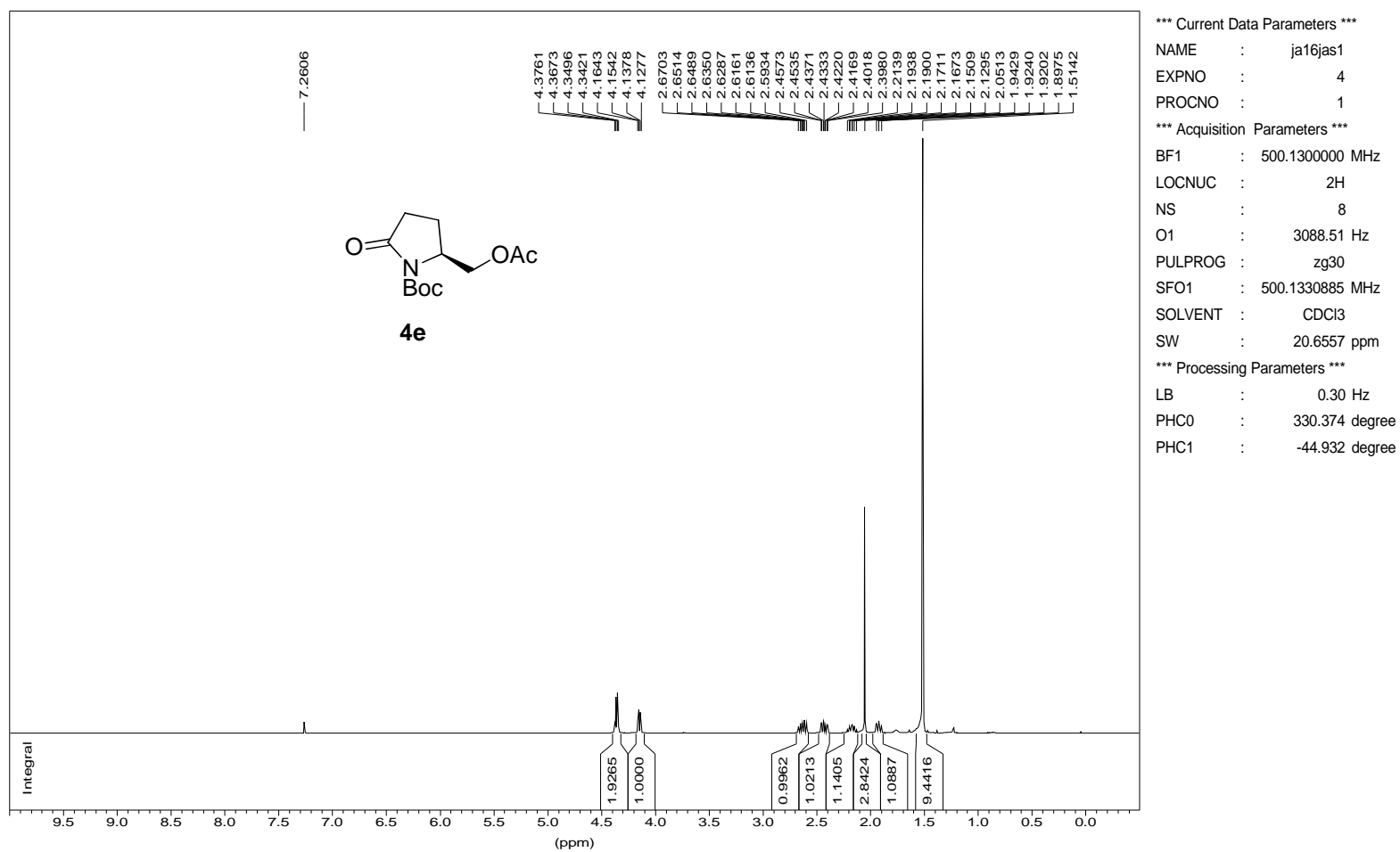
LB : 1.00 Hz

PHC0 : 167.251 degree

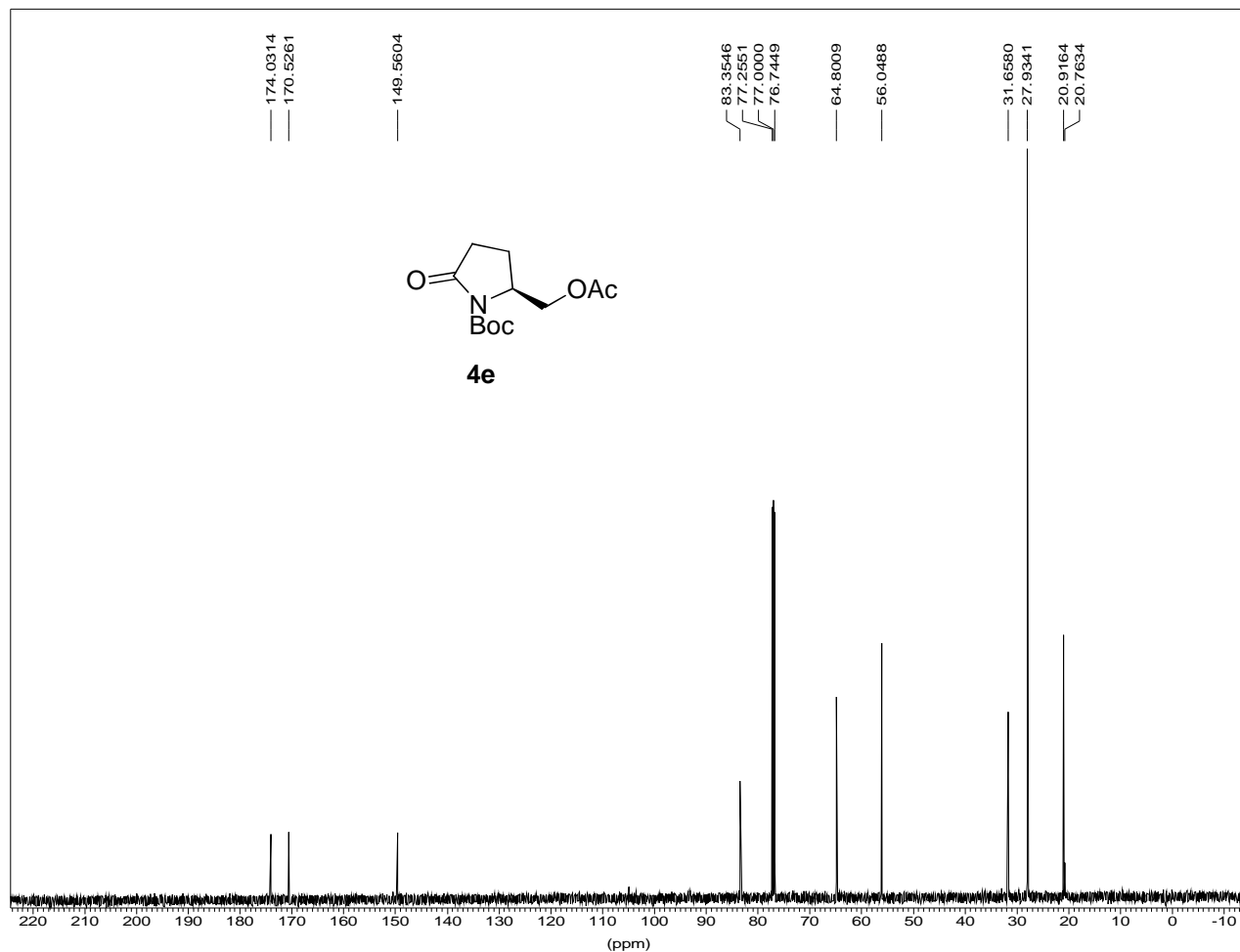
PHC1 : 41.795 degree

1H AMX500

jas-2-12-1



13C AMX500



*** Current Data Parameters ***

NAME : ja16jas1
EXPNO : 5
PROCNO : 1

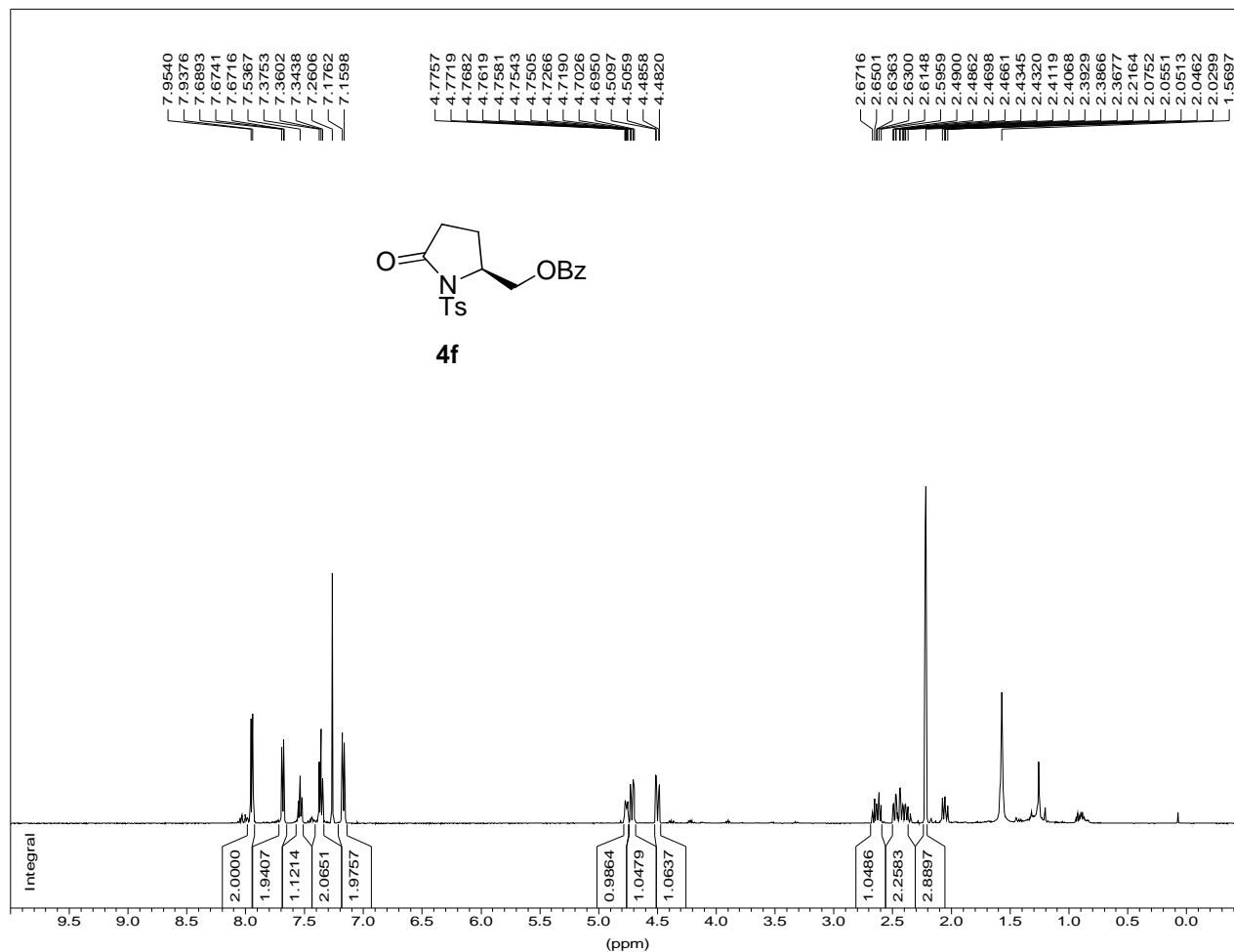
*** Acquisition Parameters ***

BF1 : 125.7577890 MHz
LOCNUC : 2H
NS : 121
O1 : 13204.57 Hz
PULPROG : zgpg30
SFO1 : 125.7709936 MHz
SOLVENT : CDCl3
SW : 238.7675 ppm

*** Processing Parameters ***

LB : 1.00 Hz
PHC0 : 168.214 degree
PHC1 : 38.846 degree

1H AMX500
jas-2-15-1



*** Current Data Parameters ***

NAME : ja24jas
EXPNO : 10
PROCNO : 1

*** Acquisition Parameters ***

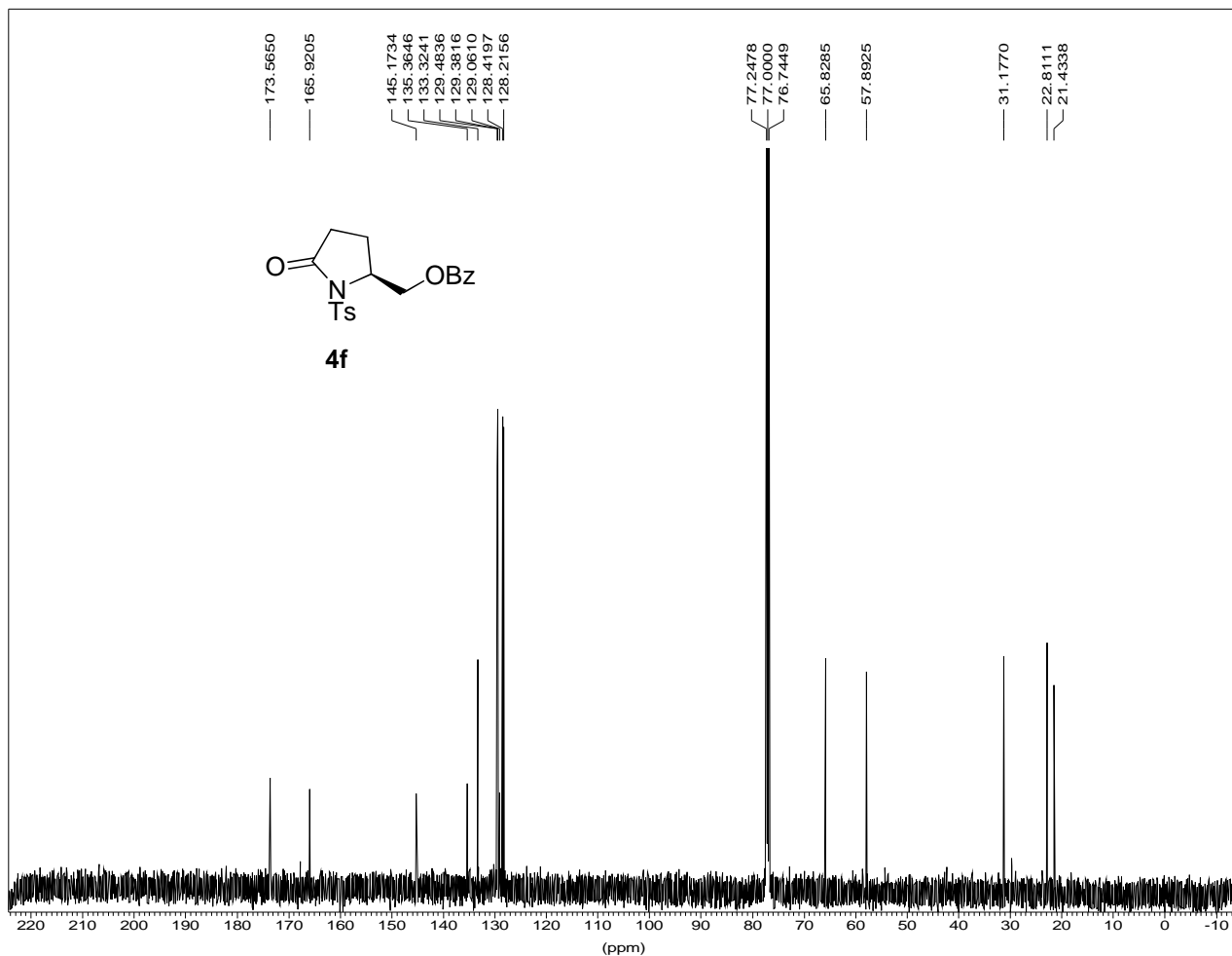
BF1 : 500.130000 MHz
LOCNUC : 2H
NS : 8
O1 : 3088.51 Hz
PULPROG : zg30
SFO1 : 500.1330885 MHz
SOLVENT : CDCl3
SW : 20.6557 ppm

*** Processing Parameters ***

LB : 0.30 Hz
PHC0 : 56.776 degree
PHC1 : 5.475 degree

13C AMX500

jas-2-15-1



*** Current Data Parameters ***

NAME : zfy012-1
EXPNO : 2
PROCNO : 1

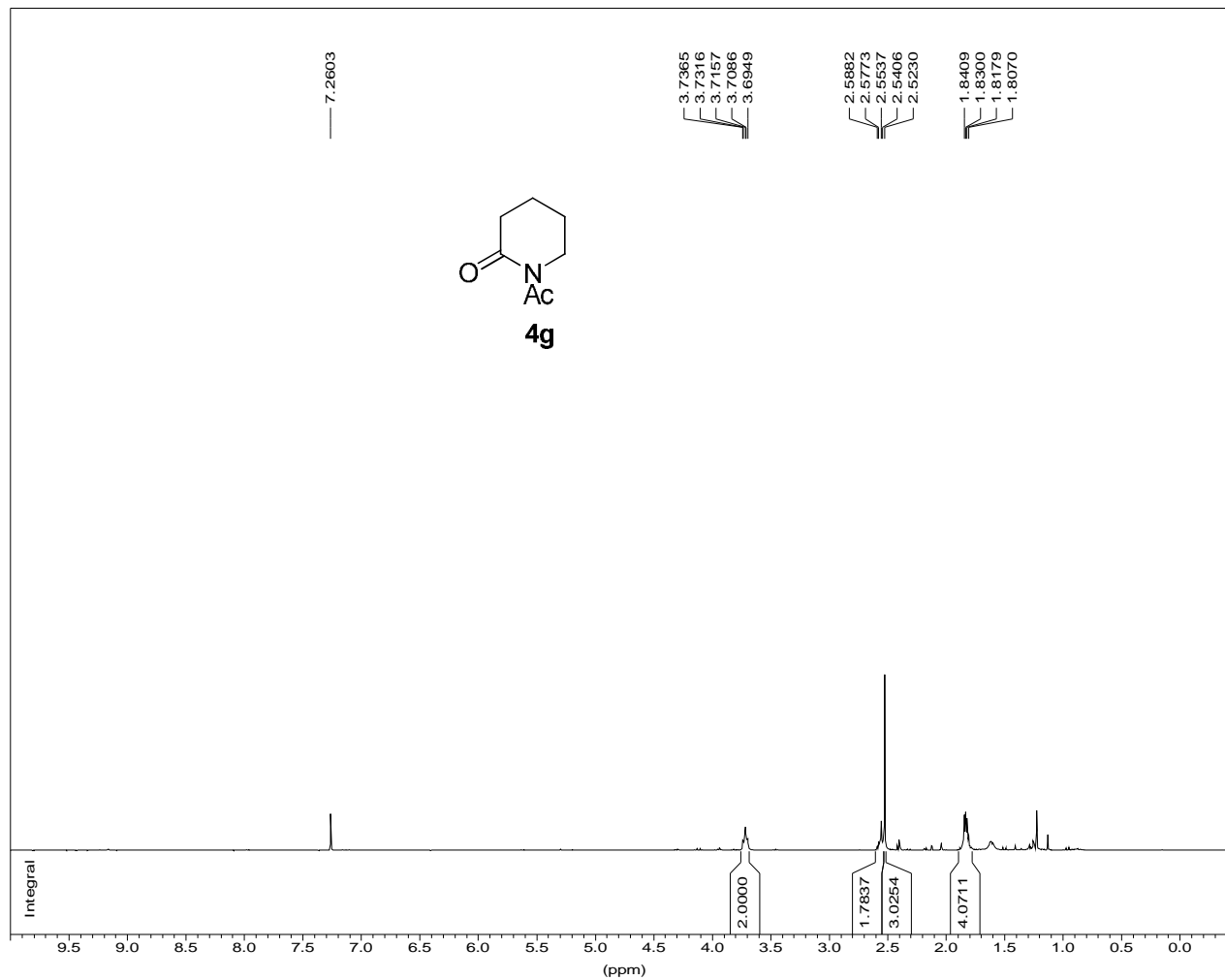
*** Acquisition Parameters ***

BF1 : 125.7577890 MHz
LOCNUC : 2H
NS : 985
O1 : 13204.57 Hz
PULPROG : zgpg30
SFO1 : 125.7709936 MHz
SOLVENT : CDCl3
SW : 238.7675 ppm

*** Processing Parameters ***

LB : 1.00 Hz
PHC0 : 159.245 degree
PHC1 : 62.600 degree

1H normal range AC300
105-16-18



*** Current Data Parameters ***

NAME : ja05hwk
EXPNO : 4
PROCNO : 1

*** Acquisition Parameters ***

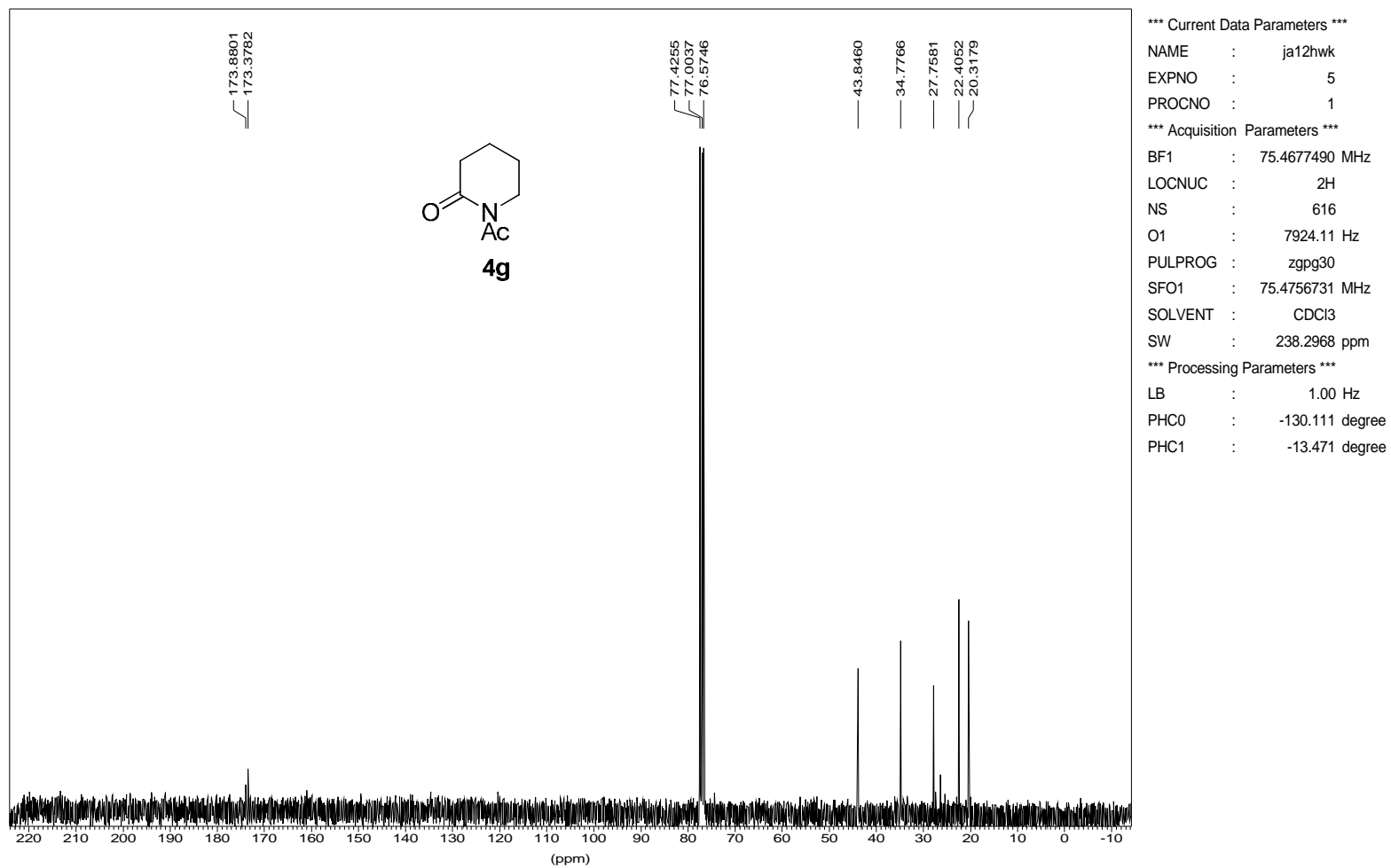
BF1 : 300.1300000 MHz
LOCNUC : 2H
NS : 8
O1 : 1853.43 Hz
PULPROG : zg30
SFO1 : 300.1318534 MHz
SOLVENT : CDCl3
SW : 17.9519 ppm

*** Processing Parameters ***

LB : 0.30 Hz
PHC0 : 311.493 degree
PHC1 : -0.157 degree

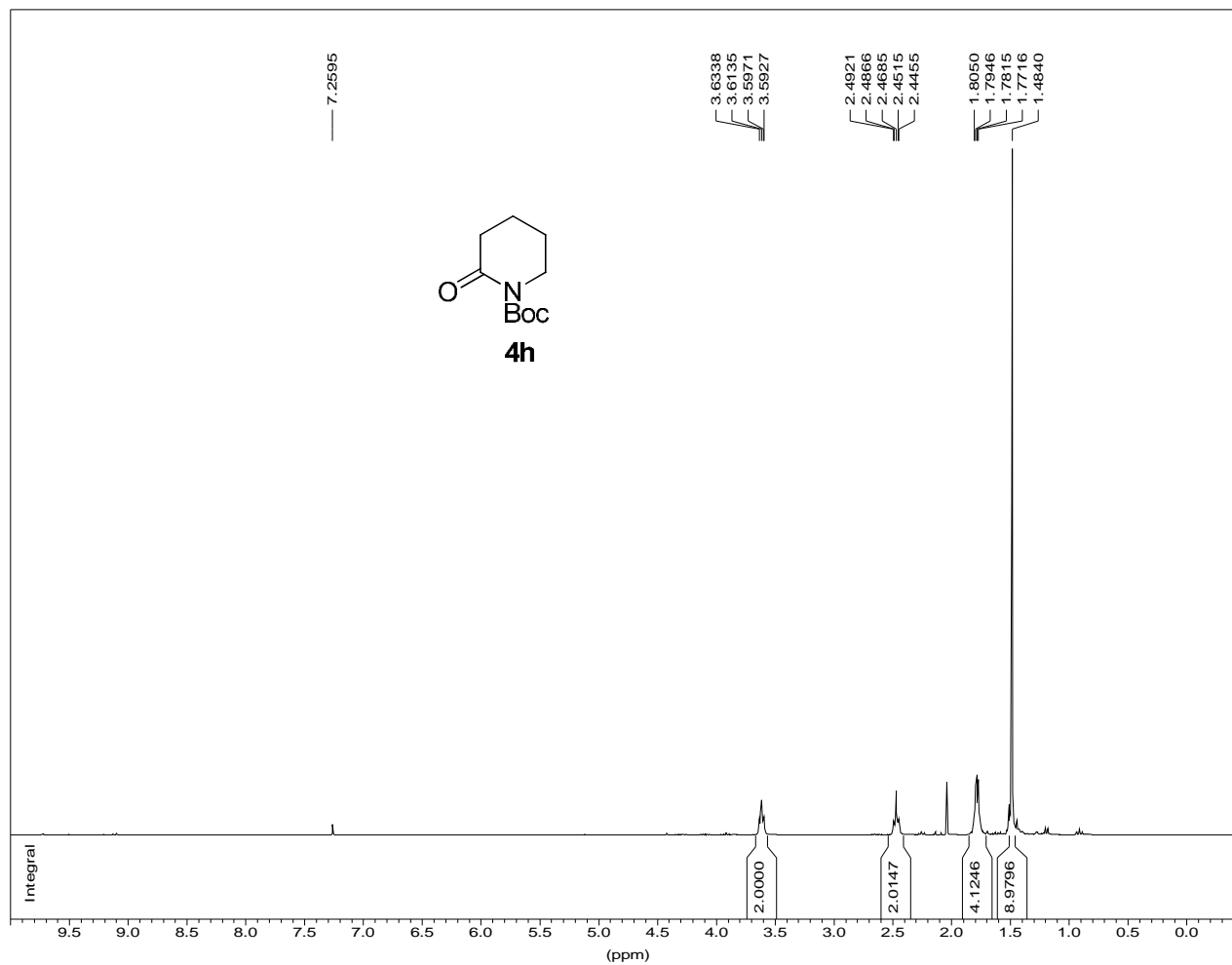
13C Standard AC300

105-16-18



1H normal range AC300

75-12,13



*** Current Data Parameters ***

NAME : oc20hwk
EXPNO : 1
PROCNO : 1

*** Acquisition Parameters ***

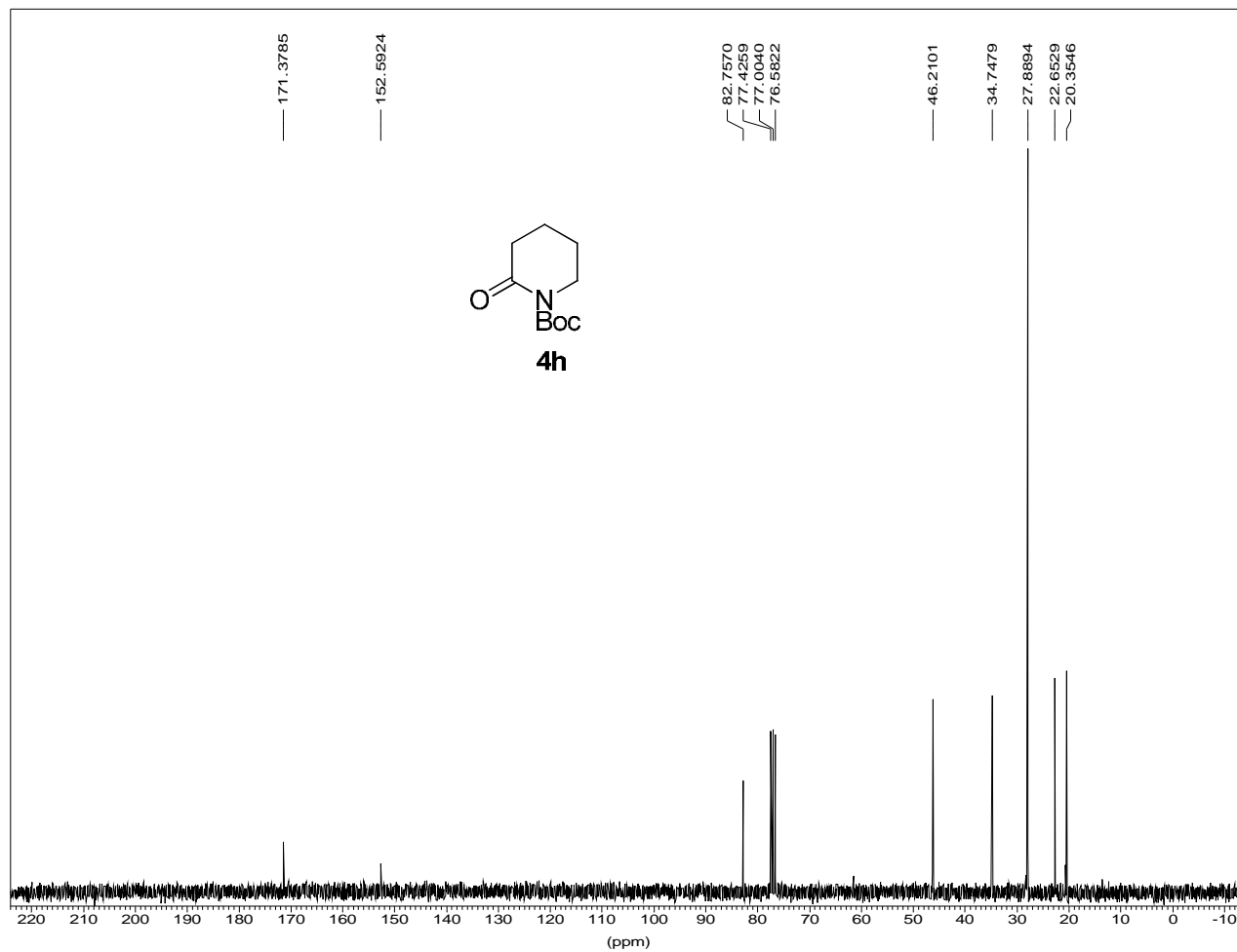
BF1 : 300.1300000 MHz
LOCNUC : 2H
NS : 8
O1 : 1853.43 Hz
PULPROG : zg30
SFO1 : 300.1318534 MHz
SOLVENT : CDCl3
SW : 17.9519 ppm

*** Processing Parameters ***

LB : 0.30 Hz
PHC0 : 167.157 degree
PHC1 : -8.047 degree

13C Standard AC300

75-12,13



*** Current Data Parameters ***

NAME : oc21hwk
EXPNO : 4
PROCNO : 1

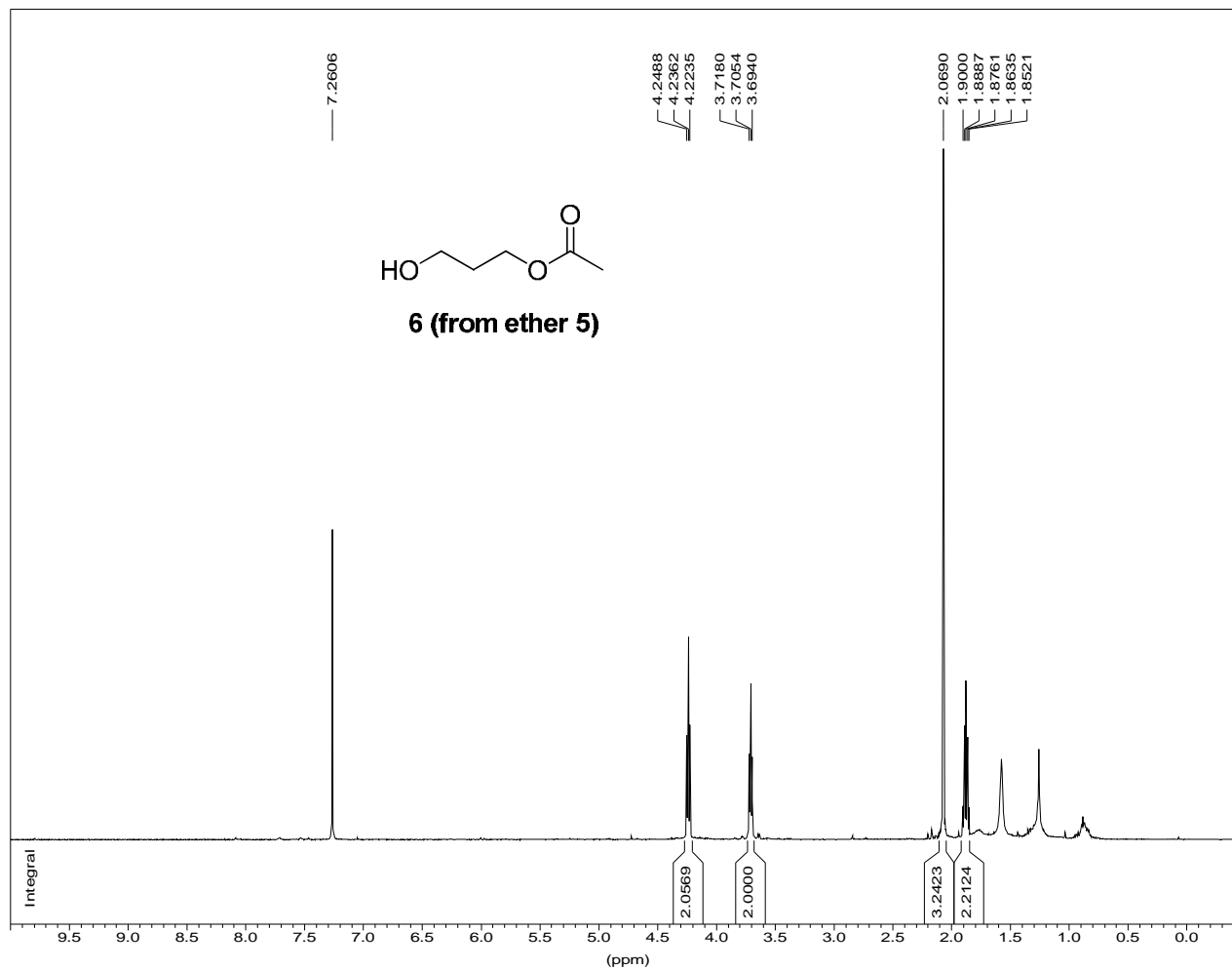
*** Acquisition Parameters ***

BF1 : 75.4677490 MHz
LOCNUC : 2H
NS : 101
O1 : 7924.11 Hz
PULPROG : zgpg30
SFO1 : 75.4756731 MHz
SOLVENT : CDCl3
SW : 238.2968 ppm

*** Processing Parameters ***

LB : 1.00 Hz
PHC0 : 244.579 degree
PHC1 : -34.382 degree

1H AMX500
jas-1-17-2



*** Current Data Parameters ***

NAME : zfy0730
EXPNO : 2
PROCNO : 1

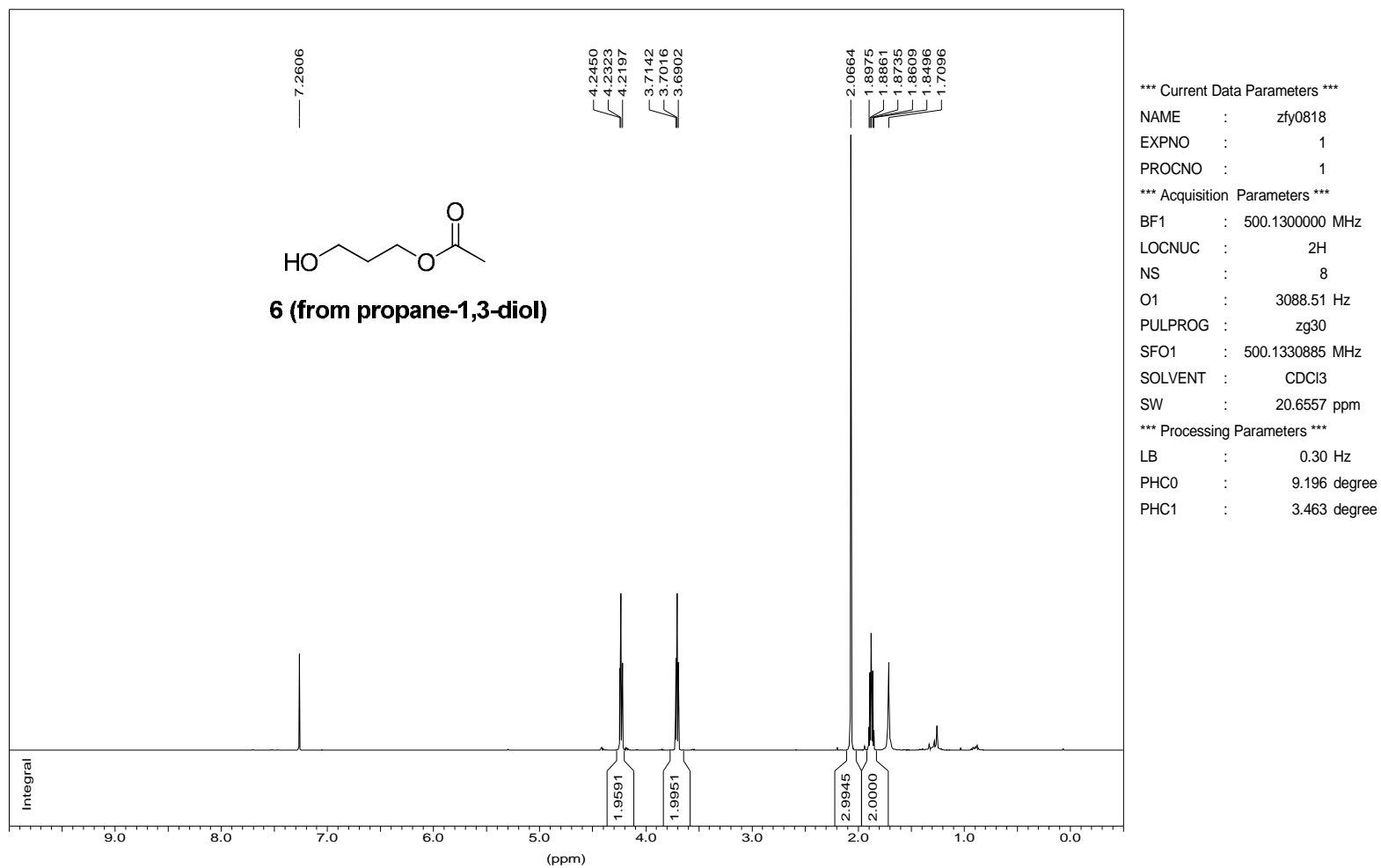
*** Acquisition Parameters ***

BF1 : 500.130000 MHz
LOCNUC : 2H
NS : 8
O1 : 3088.51 Hz
PULPROG : zg30
SFO1 : 500.1330885 MHz
SOLVENT : CDCl3
SW : 20.6557 ppm

*** Processing Parameters ***

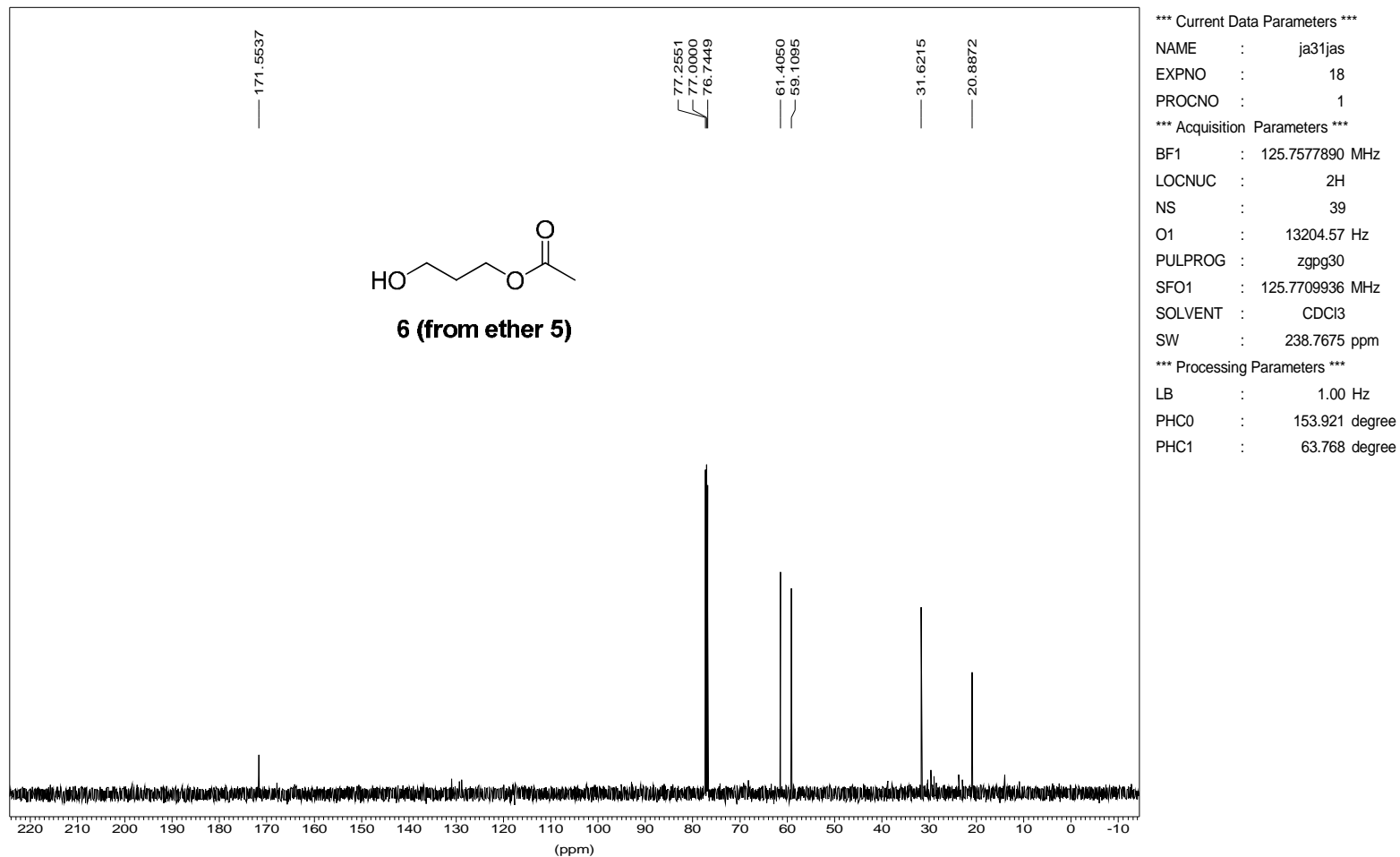
LB : 0.30 Hz
PHC0 : 23.993 degree
PHC1 : 3.174 degree

1H AMX500
jas-1-28-1

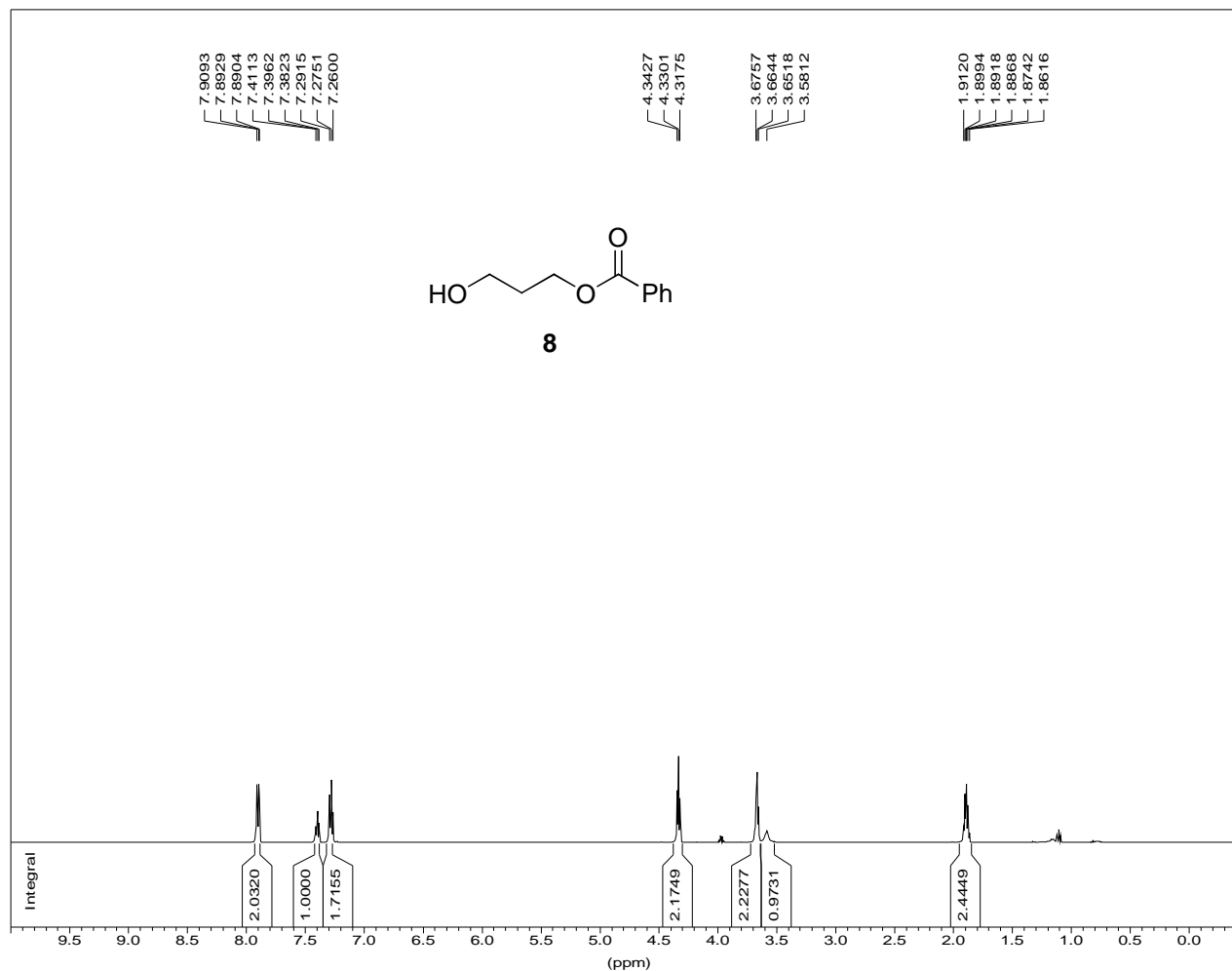


13C AMX500

jas-2-29-1



1H AMX500
jas-2-33-1



*** Current Data Parameters ***

NAME : ja31jas
EXPNO : 16
PROCNO : 1

*** Acquisition Parameters ***

BF1 : 500.130000 MHz
LOCNUC : 2H
NS : 8
O1 : 3088.51 Hz
PULPROG : zg30
SFO1 : 500.1330885 MHz
SOLVENT : CDCl3
SW : 20.6557 ppm

*** Processing Parameters ***

LB : 0.30 Hz
PHC0 : 43.097 degree
PHC1 : -5.521 degree

13C Standard AC300

jas-2-33-1

