

# Supporting Information

## An Unprecedented Method for The Generation of *tert*-Butylperoxy Radical Using DIB/TBHP Protocol: Solvent Effect and Application on Allylic Oxidation

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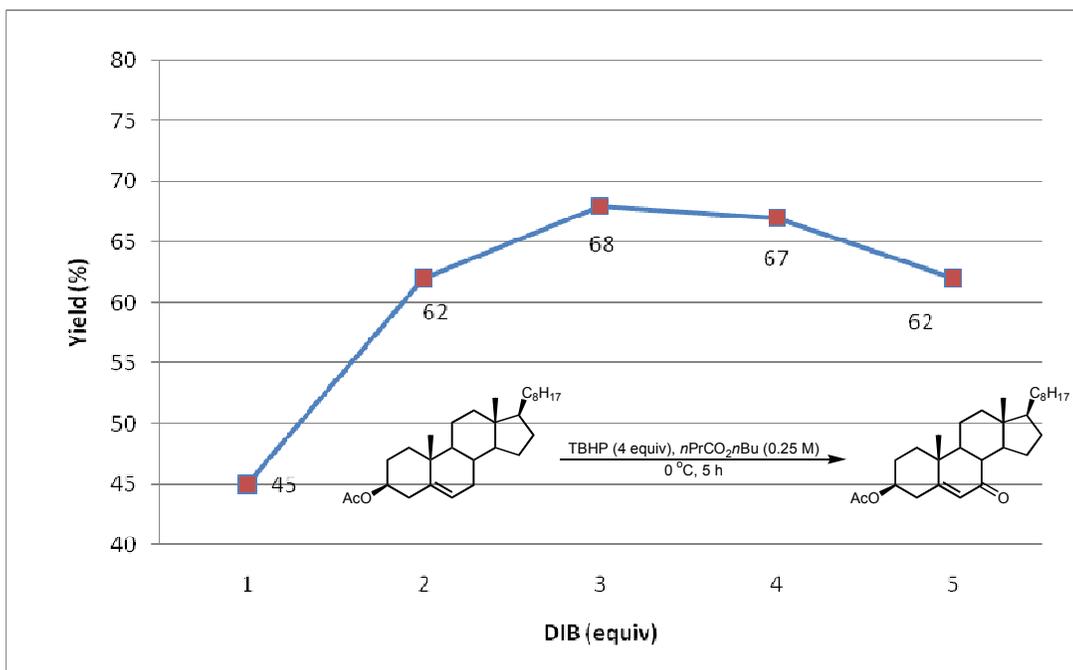
## Experimental procedures

**General.**  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were obtained in  $\text{CDCl}_3$  solutions with a Bruker DPX 500 spectrophotometer at 500 MHz and 125 MHz, respectively. NMR Spectra are referenced to residual chloroform ( $\delta$  7.26 ppm,  $^1\text{H}$ ;  $\delta$  77.0 ppm,  $^{13}\text{C}$ ). Chemical shifts are reported in ppm ( $\delta$ ); multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet) and br (broad). Coupling constants,  $J$ , are reported in Hertz. Thin-layer chromatography (TLC) was performed on Merck aluminum-precoated plates of silica gel 60 F<sub>254</sub>. Visualization was obtained by spraying with 5% (w/v) dodecamolybdophosphoric acid in ethanol 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. All chemicals are purchased from commercial suppliers and are used without further treatment. 3-[(1,1-dimethylethyl)dimethylsilyl]oxycholest-5-ene (**9**)<sup>[9]</sup> and 3-[(1,1-dimethylethyl)dimethylsilyl]-(3 $\beta$ ,25R)-spirost-5-ene (**14**)<sup>[10]</sup> were prepared accordingly to the literature procedures.

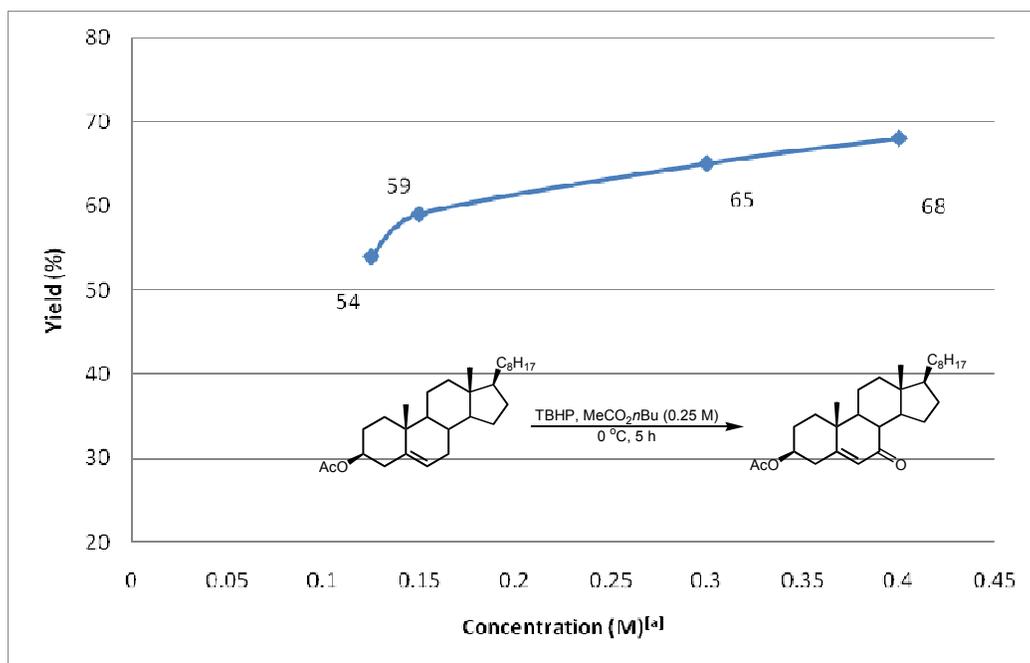
**Representative procedure for the allylic oxidation of  $\Delta^5$ -steroid.** To a solution of cholesterol acetate (**8**) (214 mg, 0.5 mmol) in *n*-butyl butanoate (1 mL) was added diacetoxyiodobenzene (**1**) (312 mg, 1.5 mmol) and Mg(OAc)<sub>2</sub>•4H<sub>2</sub>O (124 mg, 1.0 mmol) at 0 °C. The resultant suspension was vigorously stirred and a solution of *tert*-butylhydroperoxide (6.0M in decane, 333  $\mu$ L, 2.0 mmol) was added dropwise over 30 min. The solution was further stirred for 4.5 h at 0 °C (totally consumption of starting material as indicated by TLC) and filtered. The filtrate was chromatographed on silica gel eluted with *n*-hexanes/EtOAc (10:1) to yield 3 $\beta$ -acetoxycholest-5-ene-7-one (**8a**) as a white solid (181 mg, 82%).

**Representative procedure for the allylic oxidation of simple alkene.** To a solution of 1-phenylcyclohexene (**18**) (79 mg, 0.5 mmol) in *n*-butyl butanoate (1 mL) was added diacetoxyiodobenzene (**1**) (312 mg, 1.5 mmol) and K<sub>2</sub>CO<sub>3</sub> (35 mg, 0.25 mmol) at -20 °C. The resultant suspension was vigorously stirred and a solution of *tert*-butylhydroperoxide (6.0M in decane, 333  $\mu$ L, 2.0 mmol) was added dropwise over 30 min. The solution was further stirred for 6.5 h at -20 °C (totally consumption of starting material as indicated by TLC) and filtered. The filtrate was chromatographed on silica gel eluted with *n*-hexanes/diethyl ether (4:1) to yield 1-phenylcyclohex-1-en-3-one (**18a**) as a colourless oil (71 mg, 83%).

**Figure S1** Product yield as function of the equivalent of DIB for the allylic oxidation using 4 equivalents of TBHP

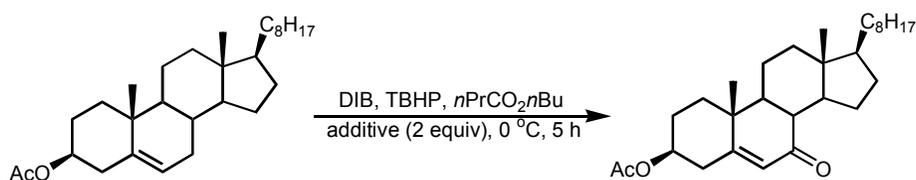


**Figure S2** Product yield as function of the concentration for the allylic oxidation using DIB and TBHP



[a] Substrate was highly insoluble when the concentration exceeded 0.4 M.

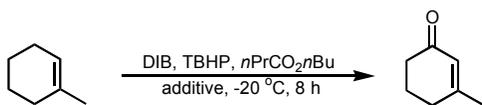
**Table S1** Allylic oxidation using different additives



Entry <sup>[a]</sup>	Additive	Yield (%) <sup>[b]</sup>
1	None	60
2	LiOAc·2H <sub>2</sub> O	53
3	NaOAc	72
4	KOAc	65
5	NH <sub>4</sub> OAc	62
6	Cu(OAc) <sub>2</sub>	60
7	Mg(OAc) <sub>2</sub> ·4H <sub>2</sub> O	79
8	K <sub>2</sub> CO <sub>3</sub> <sup>[a]</sup>	63

[a] 0.5 equivalent of K<sub>2</sub>CO<sub>3</sub> was used. [b] Isolated yield.

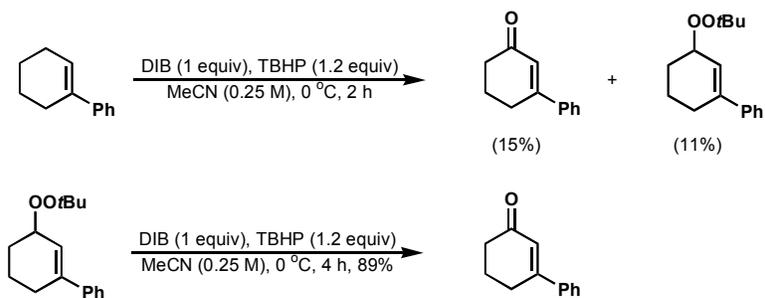
**Table S2** Allylic oxidation of 1-methylcyclohexene in the presence of additives

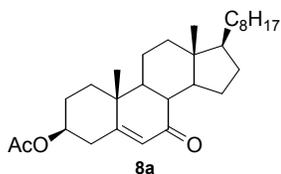


Entry <sup>[a]</sup>	Additive	Yield (%) <sup>[c]</sup>
1	None	46
2	$\text{Mg}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ <sup>[2]</sup>	45
3	$\text{K}_2\text{CO}_3$ <sup>[a]</sup>	73

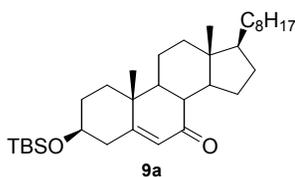
[a] 2 equivalents of  $\text{Mg}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$  were used. [b] 0.5 equivalent of  $\text{K}_2\text{CO}_3$  was used. [c] Isolated yield.

**Scheme S1** Attempt in the isolation of the allylic oxidation intermediate

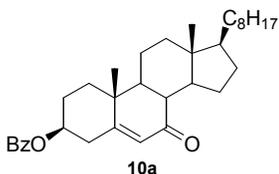




**3 $\beta$ -Acetoxycholest-5-ene-7-one (8a).**<sup>[1]</sup> Isolated as a white solid. <sup>1</sup>H NMR  $\delta$  5.70 (d,  $J$  = 1.5 Hz, 1H), 4.73 (m, 1H), 2.56-0.98 (m, 26H), 2.06 (s, 3H), 1.22 (s, 3H), 0.93 (d,  $J$  = 6.6 Hz, 3H), 0.87 (d,  $J$  = 6.6 Hz, 3H), 0.86 (d,  $J$  = 6.6 Hz, 3H), 0.68 (s, 3H). <sup>13</sup>C NMR  $\delta$  201.5, 169.9, 164.5, 127.6, 72.3, 55.1, 50.4, 50.2, 45.6, 40.1, 39.4, 37.9, 36.8, 36.4, 36.2, 29.1, 28.3, 27.9, 26.9, 24.3, 23.4, 23.1, 21.6, 19.3, 17.5, 12.5. (The data were in full accordance with the literature values)

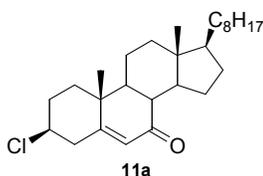


**3 $\beta$ -[(1,1-dimethylethyl)dimethylsilyl]oxycholest-5-ene-7-one (9a).**<sup>[1]</sup> Isolated as a white solid. <sup>1</sup>H NMR  $\delta$  5.66 (s, 1H), 3.59 (m, 1H), 2.44-0.98 (m, 26H), 1.18 (s, 3H), 0.92 (d,  $J$  = 6.5 Hz, 3H), 0.89 (s, 9H), 0.87 (dd,  $J$  = 6.5 Hz, 2 Hz, 6H), 0.68 (s, 3H), 0.06 (s, 6H). <sup>13</sup>C NMR  $\delta$  202.9, 166.4, 126.6, 72.1, 55.6, 50.9, 50.8, 46.2, 43.9, 43.3, 40.2, 39.6, 39.1, 37.2, 36.9, 36.4, 32.5, 29.3, 28.7, 27.1, 26.6, 24.6, 23.5, 23.3, 21.9, 19.6, 18.8, 18.1, 12.7, -3.9, -3.9; MS (ESI)  $m/z$  (relative intensive) 515.3.0 ([M+H]<sup>+</sup>, 100) (The data were in full accordance with the literature values)

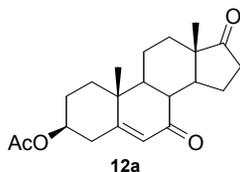


**3 $\beta$ -Benzoyloxycholest-5-en-7-one (10a).**<sup>[3]</sup> Isolated as a white solid. <sup>1</sup>H NMR  $\delta$  8.04 (dd,  $J$  = 8.5, 1.5 Hz, 2H), 7.57 (t,  $J$  = 7.5 Hz, 1H), 7.44 (t,  $J$  = 7.5 Hz, 2H), 5.75 (d,  $J$  =

1.5 Hz, 1H), 4.98 (m, 1H), 2.70 (m, 1H), 2.63 (m, 1H), 2.42 (m, 1H), 2.26 (t,  $J = 10.5$  Hz, 1H), 2.14 (m, 1H), 2.04 (m, 2H), 2.14 (m, 1H), 1.60-1.01 (m, 15H), 1.26 (s, 3H), 0.93 (d,  $J = 6.5$  Hz, 3H), 0.87 (dd,  $J = 6.5, 2.5$  Hz, 6H), 0.7 (s, 6H).  $^{13}\text{C}$  NMR  $\delta$  201.8, 165.7, 163.7, 133.1, 130.4, 129.6, 128.4, 126.9, 72.8, 54.9, 54.9, 50.1, 49.9, 45.5, 43.2, 39.5, 38.8, 38.4, 37.9, 36.2, 36.1, 35.7, 28.5, 28.0, 27.5, 26.3, 23.9, 22.8, 22.6, 21.2, 18.9, 17.3; MS (ESI)  $m/z$  (relative intensive) 383.4 ( $[\text{M}-\text{PhCOO}]^+$ , 100) (The data were in full accordance with the literature values)

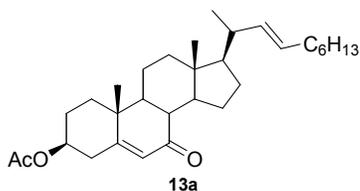


**3 $\beta$ -Chlorocholest-5-ene-7-one (11a).**<sup>[21]</sup> Isolated as a white solid.  $^1\text{H}$  NMR  $\delta$  5.68 (s, 1H), 3.87-3.80 (m, 1H), 2.70 (d,  $J = 8.5$  Hz, 2H), 2.42-2.36 (m, 1H), 2.23 (t,  $J = 12.0$  Hz, 1H), 2.17 (m, 1H), 2.05-1.87 (m, 4H), 1.59-1.00 (m, 20H), 1.22 (s, 3H), 0.92 (d,  $J = 6.5$  Hz, 3H), 0.86 (dd,  $J = 6.5, 2.0$  Hz, 6H), 0.68 (s, 3H).  $^{13}\text{C}$  NMR  $\delta$  201.9, 163.8, 126.2, 57.8, 54.8, 49.9, 49.8, 45.5, 43.1, 42.6, 39.5, 38.6, 38.1, 36.2, 35.7, 32.8, 28.5, 28.0, 26.3, 23.8, 22.8, 22.5, 21.1, 18.9, 17.2, 12.0. (The data were in full accordance with the literature values)

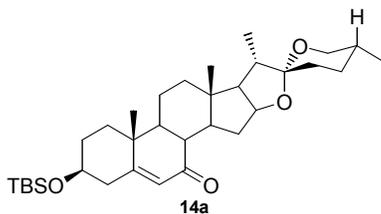


**3 $\beta$ -Acetoxyandrost-5-ene-7,17-dione (12a).**<sup>[11]</sup> Isolated as a white solid.  $^1\text{H}$  NMR  $\delta$  5.72 (d,  $J = 2.0$  Hz, 1H), 4.69 (m, 1H), 2.80-2.75 (m, 1H), 2.59-2.54 (m, 1H), 2.49-2.46 (m, 1H), 2.44-2.35 (m, 2H), 2.02 (s, 3H), 2.14-1.22 (m, 12H), 1.21 (s, 3H), 0.86 (s, 3H).  $^{13}\text{C}$  NMR  $\delta$  220.0, 200.6, 170.1, 164.7, 126.4, 71.9, 49.9, 47.8, 45.7, 44.3,

38.4, 37.7, 35.9, 35.5, 30.6, 27.2, 24.1, 21.1, 20.5, 17.3, 13.7; MS (ESI)  $m/z$  (relative intensive) 303.4 ( $[M-Ac]^+$ , 100) (The data were in full accordance with the literature values)

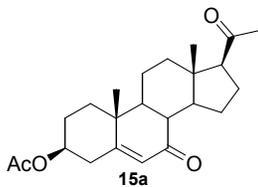


**3β-Acetoxy-5,22-diene-7-one (13a).**<sup>[8]</sup> Isolated as a white solid. <sup>1</sup>H NMR  $\delta$  5.69 (d,  $J = 1.2$  Hz, 1H), 5.17 (dd,  $J = 15.3, 8.7$  Hz, 1H), 5.02 (dd,  $J = 15.0, 8.4$  Hz, 1H), 4.71 (m, 1H), 2.58-1.13 (m, 23H), 2.05 (s, 3H), 1.21 (s, 3H), 1.03 (d,  $J = 6.6$  Hz, 3H), 0.85 (d,  $J = 6.3$  Hz, 3H), 0.80 (m, 6H), 0.70 (s, 3H), <sup>13</sup>C NMR  $\delta$  201.8, 170.3, 163.8, 138.1, 129.5, 126.7, 72.2, 54.7, 51.2, 50.0, 49.8, 45.4, 43.0, 40.2, 38.6, 38.3, 37.8, 36.0, 31.9, 29.0, 27.4, 26.4, 25.4, 21.4, 21.3, 21.2, 21.0, 19.0, 17.3, 12.2, 12.2. (The data were in full accordance with the literature values)

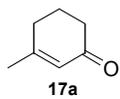


**3-[(1,1-dimethylethyl)dimethyl]-(3β,25R)-spirost-5-en-7-one (14a).**<sup>[1]</sup> Isolated as a white solid. <sup>1</sup>H NMR  $\delta$  5.64 (d,  $J = 1.5$  Hz, 1H), 4.45 (m, 1H), 3.54 (m, 1H), 3.44 (m, 1H), 3.37 (t,  $J = 11.0$  Hz, 1H), 2.85 (m, 1H), 2.42-2.40 (m, 1H), 2.37-2.35 (m, 2H), 1.90-1.06 (m, 12H), 1.18 (s, 3H), 0.95 (d,  $J = 7.0$  Hz, 3H), 0.86 (s, 9H), 0.76 (d,  $J = 6.0$  Hz, 6H), 0.04 (s, 6H). <sup>13</sup>C NMR  $\delta$  201.6, 166.0, 125.5, 109.1, 80.9, 71.2, 66.7, 61.1, 49.8, 49.5, 44.8, 42.5, 41.5, 40.9, 38.7, 38.4, 36.3, 33.7, 31.7, 31.4, 30.3, 28.8,

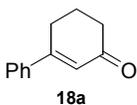
25.8, 20.9, 18.1, 17.3, 17.1, 16.4, 14.6, -4.7; MS (ESI)  $m/z$  (relative intensive) 515.3 ( $[M+H]^+$ , 100) (The data were in full accordance with the literature values)



**3  $\beta$ -Acetoxypregn-5-ene-7,20-dione (15a).**<sup>[1]</sup> Isolated as a white solid.  $^1\text{H}$  NMR  $\delta$  5.63 (d,  $J = 1.5$  Hz, 1H), 4.64 (m, 1H), 2.51-1.18 (m, 18H), 2.05 (s, 3H), 1.97 (s, 3H), 1.15 (s, 3H), 0.58 (s, 3H).  $^{13}\text{C}$  NMR  $\delta$  209.3, 200.8, 170.0, 164.0, 126.2, 71.9, 62.0, 49.8, 49.5, 45.0, 44.2, 38.2, 37.6, 37.4, 35.8, 31.4, 27.1, 26.3, 23.4, 21.0, 17.1, 13.0. (The data were in full accordance with the literature values)



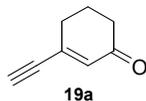
**3-methyl-2-cyclohexen-1-one (17a).** Isolated as a pale yellow oil.  $^1\text{H}$  NMR  $\delta$  5.88 (d,  $J = 1.5$  Hz, 1H), 2.34 (t,  $J = 6.5$  Hz, 2H), 2.29-2.24 (m, 2H), 2.01-1.97 (m, 2H), 1.95 (s, 3H);  $^{13}\text{C}$  NMR  $\delta$  199.7, 162.7, 126.7, 37.0, 31.0, 24.4, 22.6; MS (ESI)  $m/z$  (relative intensive) 110.0 ( $M^+$ , 100) (The data were in full accordance with the authentic sample)



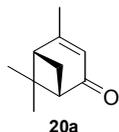
**3-Phenyl-2-cyclohexen-1-one (18a).**<sup>[1]</sup> Isolated as a pale yellow oil.  $^1\text{H}$  NMR  $\delta$  7.54-7.52 (m, 2H), 7.41-7.40 (m, 3H), 6.42 (m, 1H), 2.77 (dt,  $J = 6.5, 1.5$  Hz, 2H), 2.48 (t,  $J = 6.5$  Hz, 2H), 2.18-2.13 (m, 2H);  $^{13}\text{C}$  NMR  $\delta$  199.7, 159.7, 138.8, 129.9, 128.7,

126.0, 125.4, 37.2, 28.1, 22.8; MS (ESI)  $m/z$  (relative intensive) 195.1 ( $[M+Na]^+$ , 100)

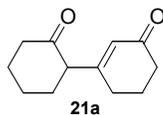
(The data were in full accordance with the literature values)



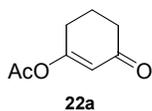
**3-ethynylcyclohex-2-enone (19a).**<sup>[8]</sup> Isolated as a colourless oil.  $^1\text{H}$  NMR  $\delta$  6.26 (s, 1H), 3.52 (s, 1H), 2.46 (dt,  $J = 6.0, 1.5$  Hz, 2H), 2.42 (t,  $J = 6.5$  Hz, 2H), 2.04 (t,  $J = 6.0$  Hz, 2H);  $^{13}\text{C}$  NMR  $\delta$  198.5, 142.1, 134.0, 87.1, 82.5, 37.3, 30.2, 22.5. (The data were in full accordance with the literature values)



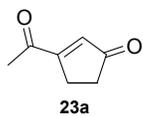
**(1S)-(-)-Verbinone (20a).**<sup>[1]</sup> Isolated as a pale yellow oil.  $^1\text{H}$  NMR  $\delta$  5.72 (m, 1H), 2.82-2.78 (m, 1H), 2.64 (dt,  $J = 6.5, 2.0$  Hz, 1H), 2.41 (dt,  $J = 6.0, 1.0$  Hz, 1H), 2.07 (d,  $J = 9.0$  Hz, 1H), 2.01 (d,  $J = 1.5$  Hz, 3H), 1.49 (s, 3H), 1.00 (s, 3H);  $^{13}\text{C}$  NMR  $\delta$  204.0, 170.2, 121.1, 57.6, 54.0, 49.7, 40.8, 26.5, 23.5, 22.0; MS (ESI)  $m/z$  (relative intensive) 151.0 ( $[M+H]^+$ , 100). (The data were in full accordance with the authentic sample)



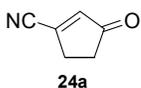
**3-(2-Oxo-cyclohexanyl)-2-cyclohexene-1-one (21a).**<sup>[1]</sup> Isolated as a colourless oil.  $^1\text{H}$  NMR  $\delta$  5.83 (s, 1H), 3.18 (dd,  $J = 12.5, 5.0$  Hz, 1H), 2.43-2.32 (m, 1H), 2.30 (t,  $J = 6.0$  Hz, 2H), 2.13-1.71(m, 8H);  $^{13}\text{C}$  NMR  $\delta$  208.7, 199.7, 162.9, 127.3, 58.9, 42.3, 37.6, 31.6, 28.9, 27.5, 22.7;; MS (ESI)  $m/z$  (relative intensive) 193.1 ( $[M+H]^+$ , 100).



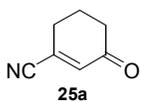
**3-Acetoxy-2-cyclohexene-1-one (22a).**<sup>[6]</sup> Isolated as a colourless oil. <sup>1</sup>H NMR  $\delta$  5.90 (s, 1H), 2.53 (t,  $J$  = 6.5 Hz, 2H), 2.40 (t,  $J$  = 6.5 Hz, 2H), 2.21 (s, 3H), 2.05 (m, 2H); <sup>13</sup>C NMR  $\delta$  199.5, 170.0, 167.3, 117.6, 36.7, 28.3, 21.2, 21.2; MS (ESI)  $m/z$  (relative intensive) 111.2 ([M-OAc]<sup>+</sup>, 100) (The data were in full accordance with the literature values)



**3-Acetyl-2-cyclopentenone (23a).**<sup>[7]</sup> Isolated as a pale yellow solid. <sup>1</sup>H NMR  $\delta$  6.63(t,  $J$  = 2.0 Hz, 1H), 2.81-2.79 (m, 2H), 2.53-2.51 (m, 2H), 2.49 (s, 3H); <sup>13</sup>C NMR  $\delta$  210.1, 197.1, 169.1, 137.0, 35.4, 27.7, 26.4. (The data were in full accordance with the literature values)

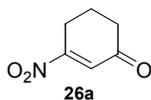


**3-Cyano-2-cyclopentenone (24a).**<sup>[11]</sup> Isolated as a pale yellow oil. <sup>1</sup>H NMR  $\delta$  6.76 (t,  $J$  = 2.0 Hz, 1H), 2.91 (m, 2H), 2.54 (m, 2H); <sup>13</sup>C NMR  $\delta$  206.1, 143.4, 142.9, 114.7, 34.1, 30.2; MS (ESI)  $m/z$  (relative intensive) 130.2 ([M+Na]<sup>+</sup>, 100) (The data were in full accordance with the literature values)

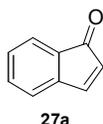


**3-Cyano-2-cyclohexen-1-one (25a).**<sup>[5]</sup> Isolated as a pale yellow oil. <sup>1</sup>H NMR  $\delta$  6.51 (s, 1H), 2.56 (dt,  $J$  = 6.5, 1.5 Hz, 2H), 2.50 (t,  $J$  = 6.5 Hz, 2H), 2.13 (m, 2H); <sup>13</sup>C

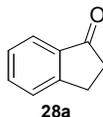
NMR  $\delta$  196.2, 138.7, 130.9, 117.0, 37.2, 27.7, 22.1. (The data were in full accordance with the literature values)



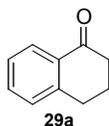
**3-Nitro-2-cyclohexen-1-one (26a).**<sup>[4]</sup> Isolated as a pale yellow oil. <sup>1</sup>H NMR  $\delta$  6.93 (m, 1H), 2.10 (dt,  $J = 6.5, 2.0$  Hz, 2H), 2.51 (t,  $J = 6.5$  Hz, 2H), 2.20-2.15 (m, 2H), <sup>13</sup>C NMR  $\delta$  198.1, 164.0, 125.7, 37.0, 24.4, 20.9; MS (ESI)  $m/z$  (relative intensive) 284.0 ( $2[M+H]^+$ , 100) (The data were in full accordance with the literature values)



**1H-inden-1-one (27a).**<sup>[12]</sup> Isolated as a pale yellow oil. <sup>1</sup>H NMR  $\delta$  7.57 (d,  $J = 6$  Hz, 1H), 7.43 (d,  $J = 7.0$  Hz, 1H), 7.35 (t,  $J = 7.0$  Hz, 1H), 7.24 (t,  $J = 7.5$  Hz, 1H), 7.07 (d,  $J = 7.5$  Hz, 1H), 5.90 (d,  $J = 6.0$  Hz, 1H); <sup>13</sup>C NMR  $\delta$  199.1, 150.5, 145.2, 134.2, 130.8, 129.2, 127.4, 123.3, 122.4. (The data were in full accordance with the literature values)



**2,3-Dihydro-1H-inden-1-one (28a).**<sup>[9]</sup> Isolated as a colourless oil. <sup>1</sup>H NMR  $\delta$  7.76 (d,  $J = 7.5$  Hz, 1H), 7.59 (dt,  $J = 7.5, 1.0$  Hz, 1H), 7.48 (d,  $J = 8.0$  Hz, 1H), 7.37 (t,  $J = 7.5$  Hz, 1H), 3.15 (t,  $J = 5.5$  Hz, 2H), 2.69 (m, 2H); <sup>13</sup>C NMR  $\delta$  207.0, 155.1, 137.1, 134.6, 127.2, 126.7, 123.7, 36.2, 25.8; MS (EI)  $m/z$  (relative intensive) 155.1 ( $[M+Na]^+$ , 100) (The data were in full accordance with the literature values)



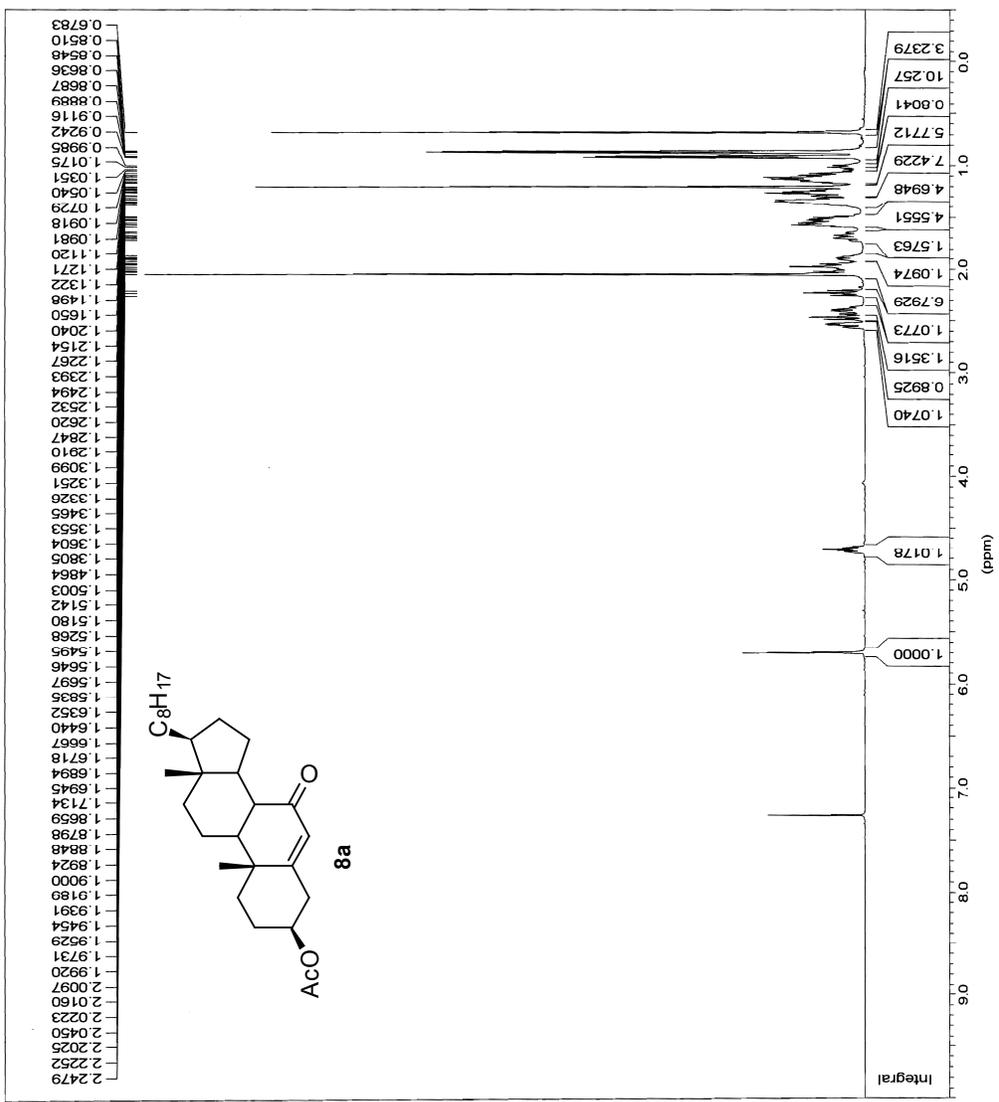
**3,4-Dihydronaphthalen-1(2H)-one (29a).**<sup>[9]</sup> Isolated as a pale yellow oil. <sup>1</sup>H NMR  $\delta$  8.02 (d,  $J = 7.5$  Hz, 1H), 7.44 (dt,  $J = 7.5, 1.5$  Hz, 1H), 7.28 (t,  $J = 7.5$  Hz, 1H), 7.23 (d,  $J = 7.5$  Hz, 1H), 2.95 (t,  $J = 6.0$  Hz, 2H), 2.64 (t,  $J = 6.0$  Hz, 2H), 2.12 (m, 2H); <sup>13</sup>C NMR  $\delta$  198.3, 144.4, 133.3, 132.6, 128.7, 127.1, 126.5, 39.1, 29.6, 23.2. (The data were in full accordance with the literature values)

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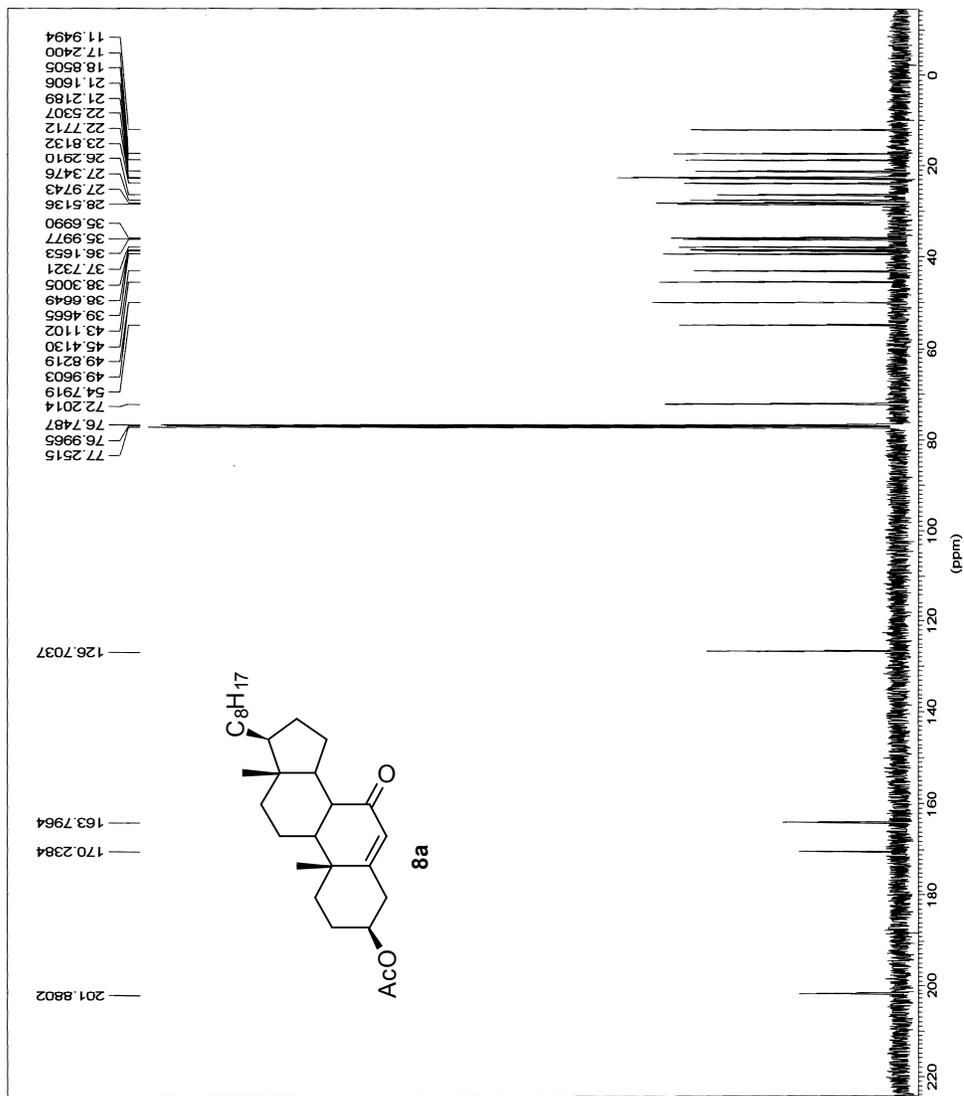
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1H AMX500  
cholesterol acetate



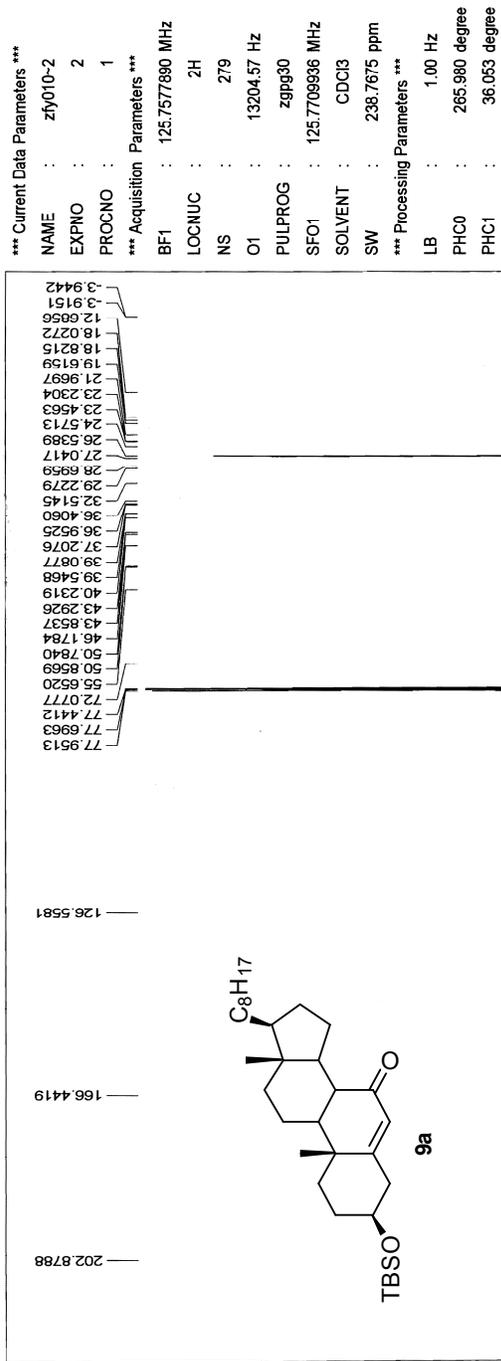
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 LOCNUG : 2H  
 NS : 8  
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 PULPROG : zg30  
 SFO1 : 500.1330885 MHz  
 SOLVENT : CDCl3  
 SW : 20.6557 ppm  
 \*\*\* Processing Parameters \*\*\*  
 LB : 0.30 Hz  
 PHC0 : 155.267 degree  
 PHC1 : 3.514 degree

13C AMX500  
cholesterol acetate

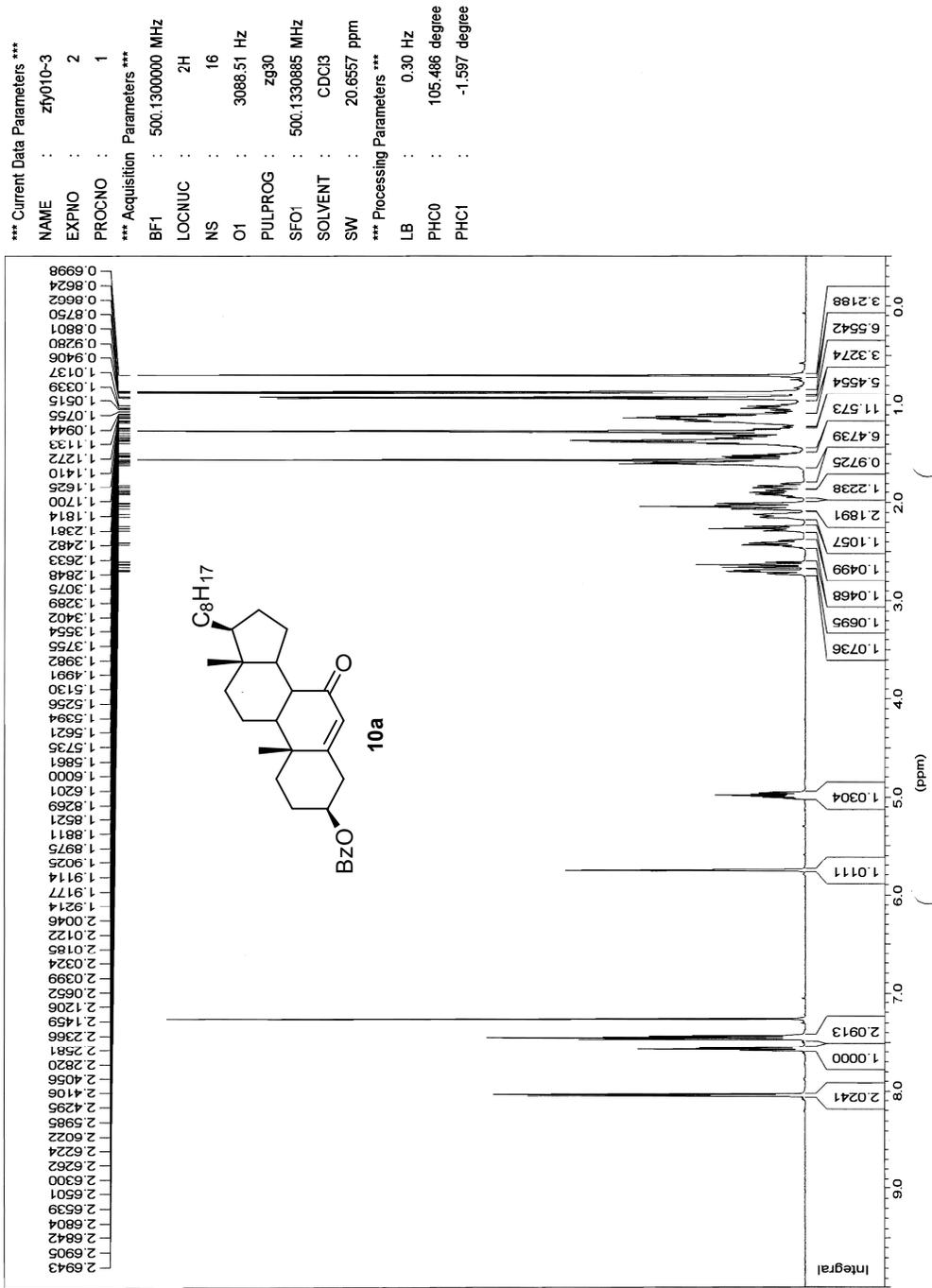




13C AMX500  
FY-2-35-1

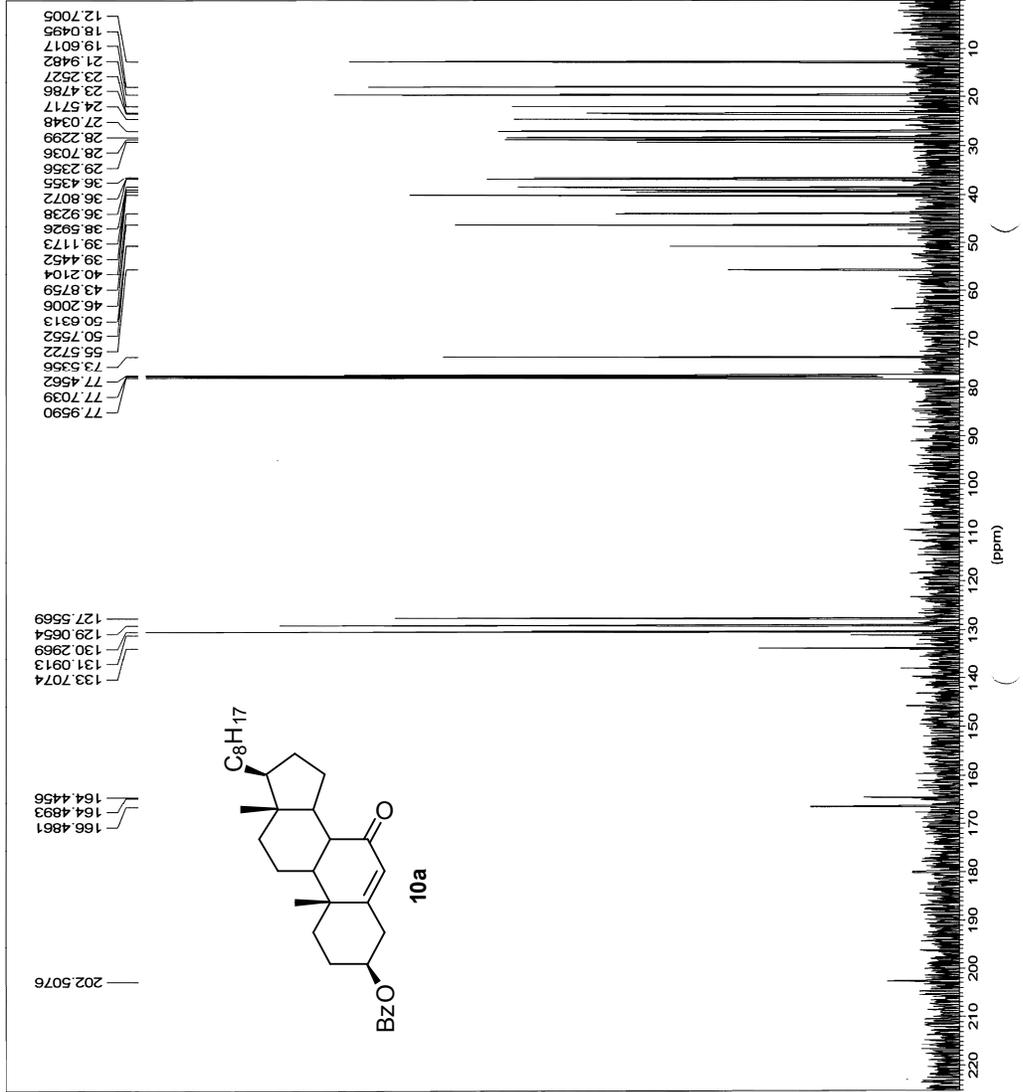


1H AMX500  
FY-2-39-1

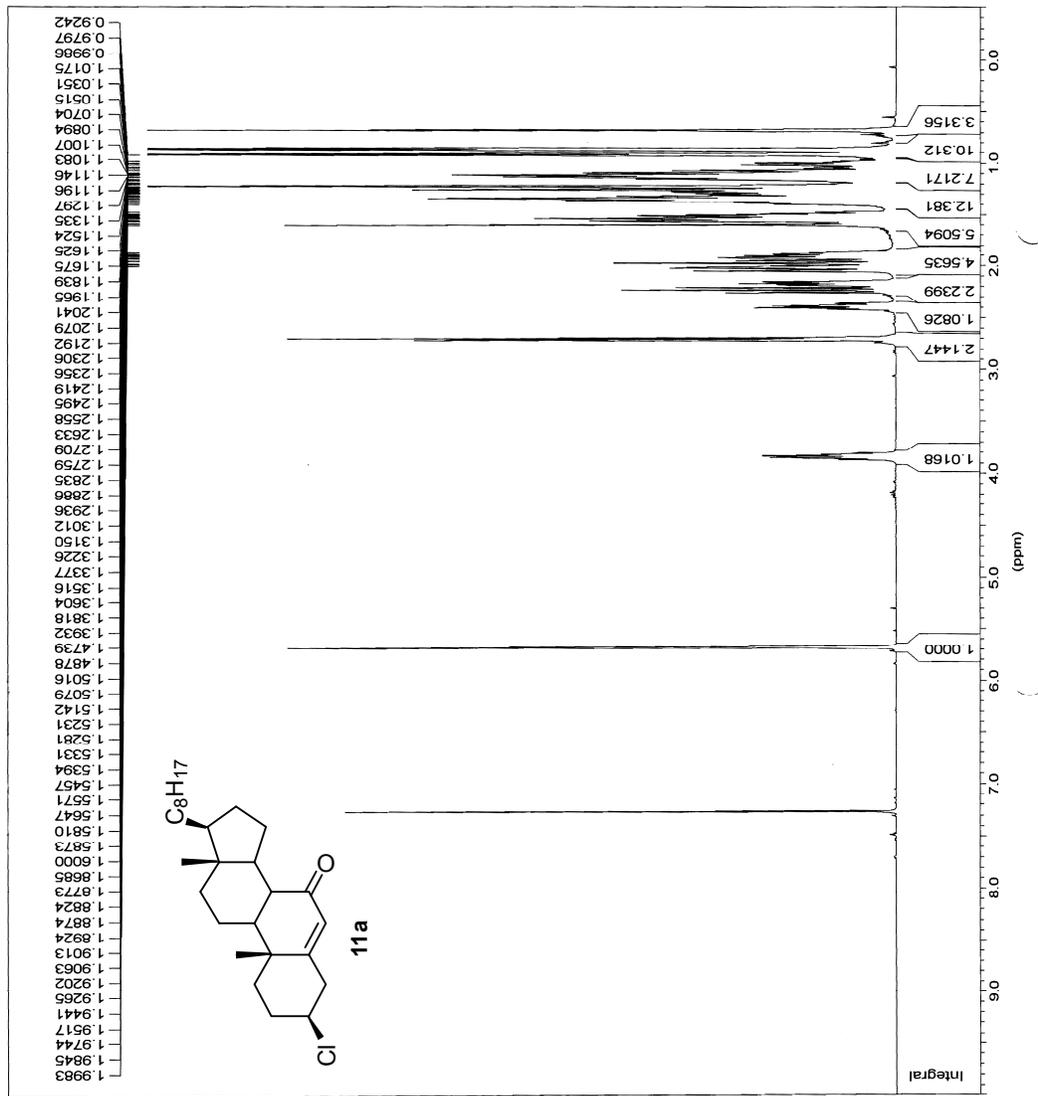


13C AMX500  
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PROCNO : 1  
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LOCNUC : 2H  
NS : 616  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709936 MHz  
SOLVENT : CDCl3  
SW : 238.7675 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
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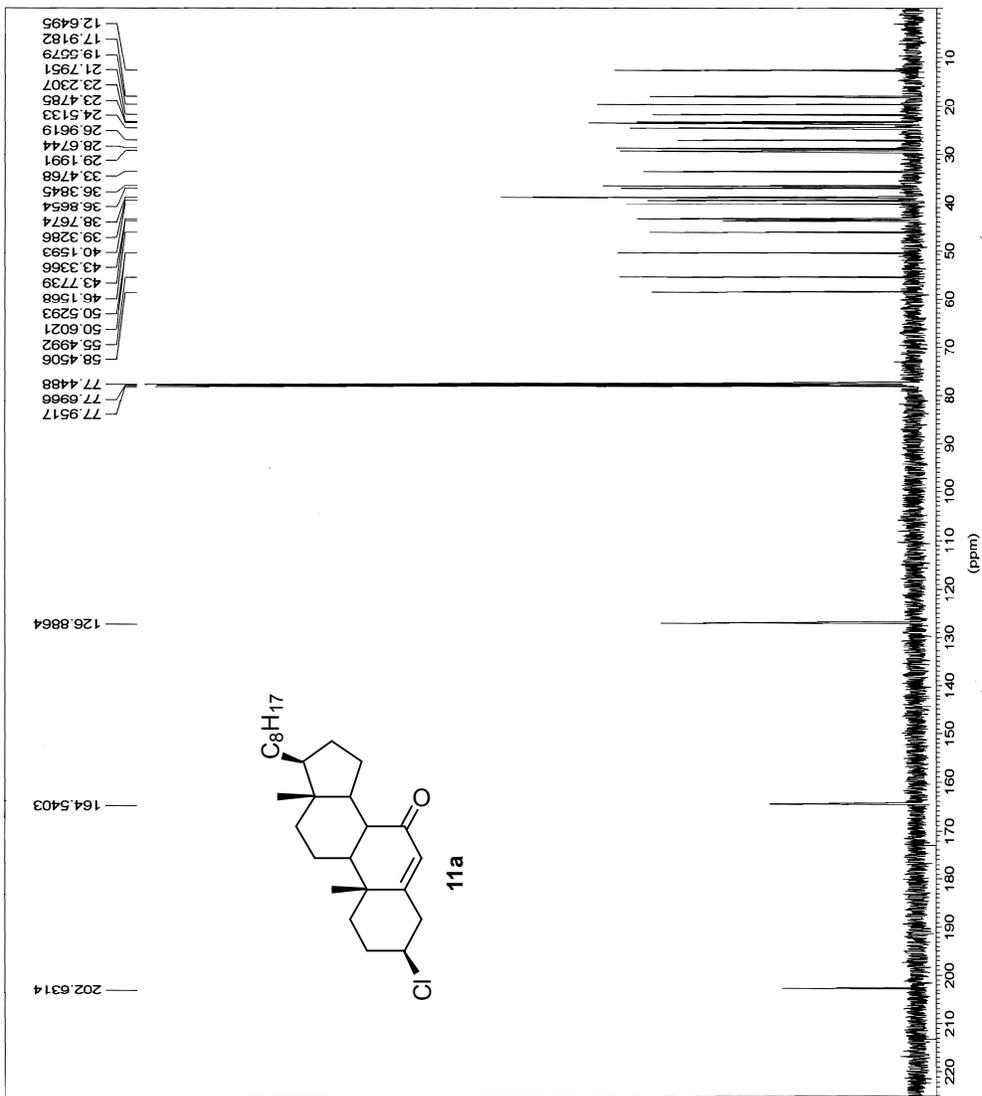
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FY-2-111-1



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PROCNO : 1  
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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 : 347.748 degree  
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13C AMX500  
FY-2-11-1

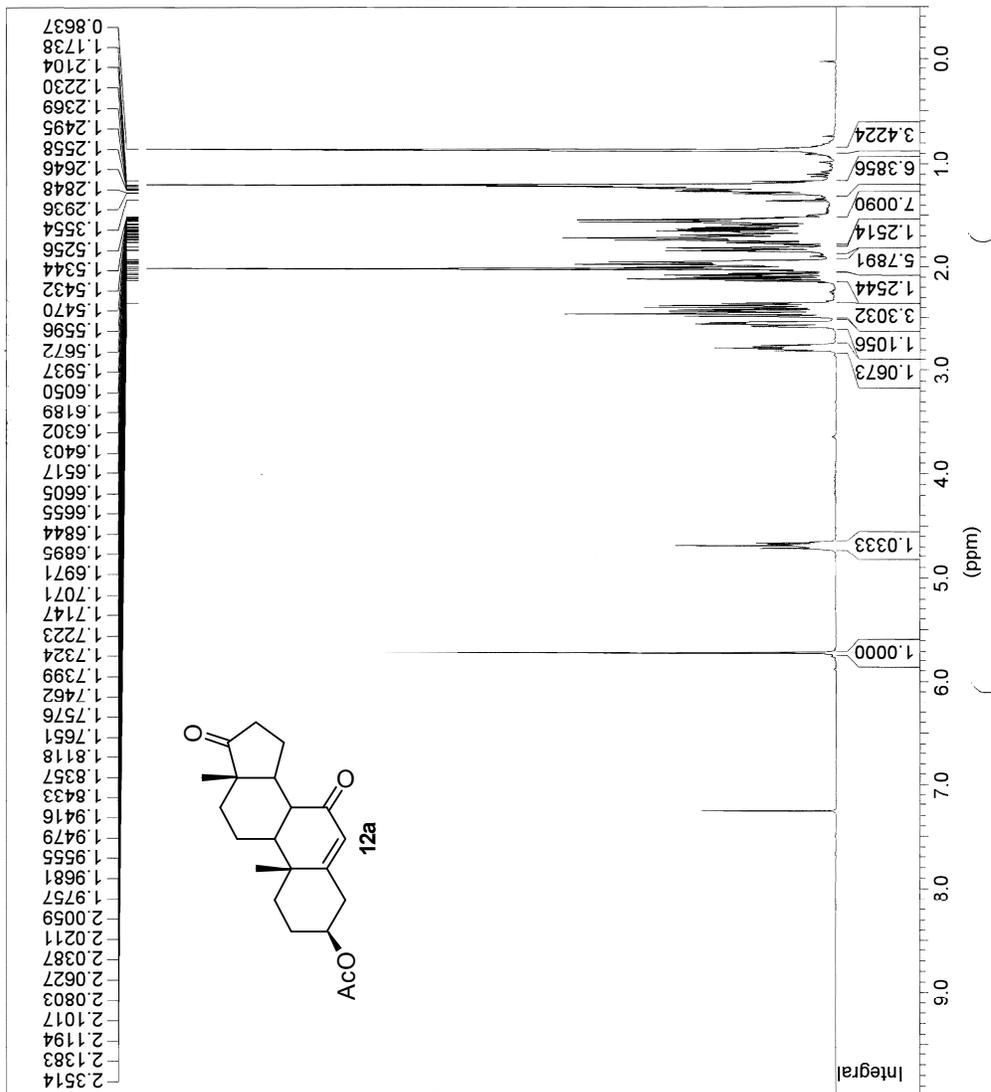
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LOCNUC : 2H  
NS : 206  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709986 MHz  
SOLVENT : CDC13  
SW : 238.7675 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
PHC0 : 273.345 degree  
PHC1 : 17.149 degree



1H AMX500  
FY-2-105-1



\*\*\* Current Data Parameters \*\*\*  
NAME : zfy0108  
EXPNO : 1  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
INSTRUM : av500  
LOCNUC : 2H  
NS : 8  
NUCLEUS : off  
O1 : 3088.51 Hz  
PULPROG : zg30  
SFO1 : 500.1330885 MHz  
SOLVENT : CDCl3  
SW : 20.6557 ppm  
TD : 32768  
\*\*\* Processing Parameters \*\*\*  
LB : 0.30 Hz  
\*\*\* 1D NMR Plot Parameters \*\*\*  
NUCLEUS : off

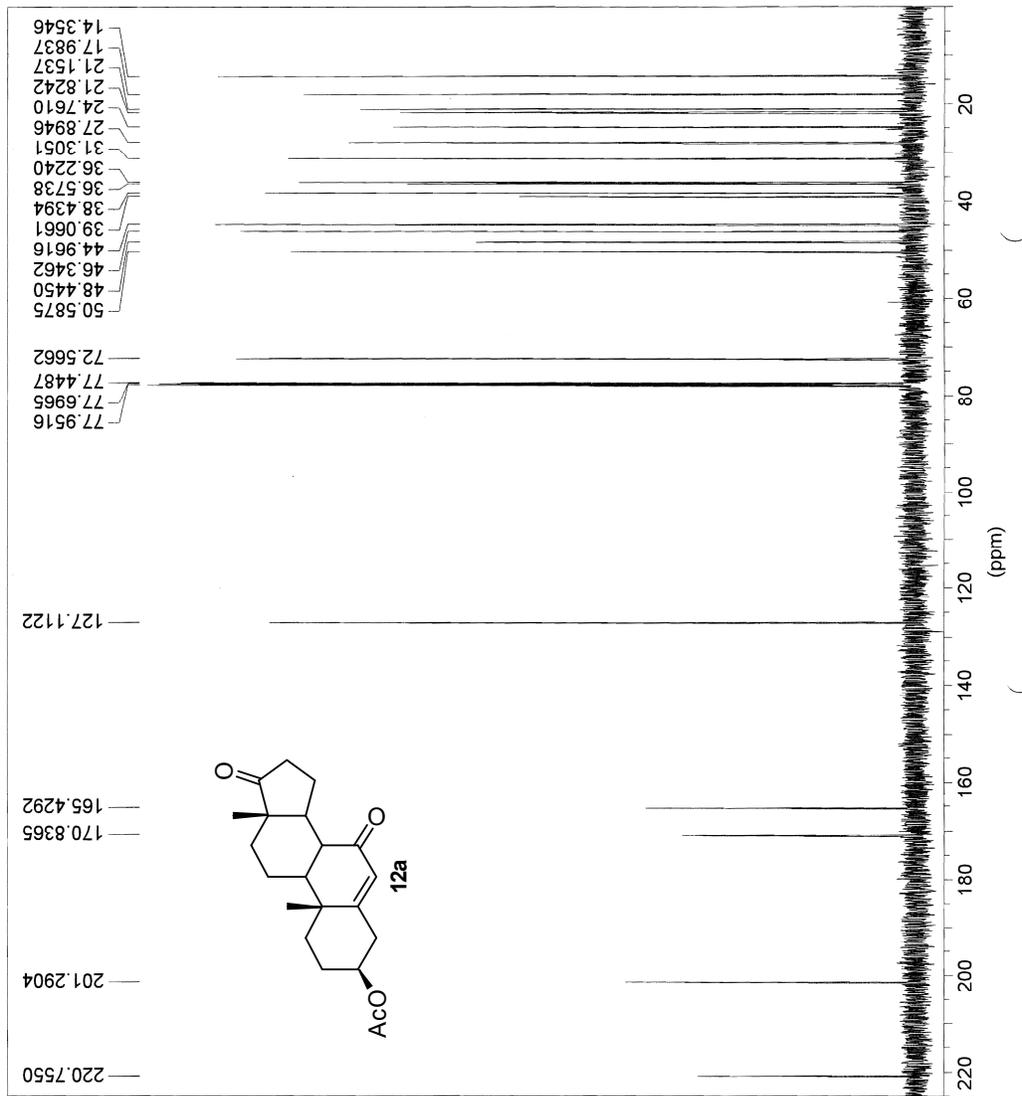


13C AMX500

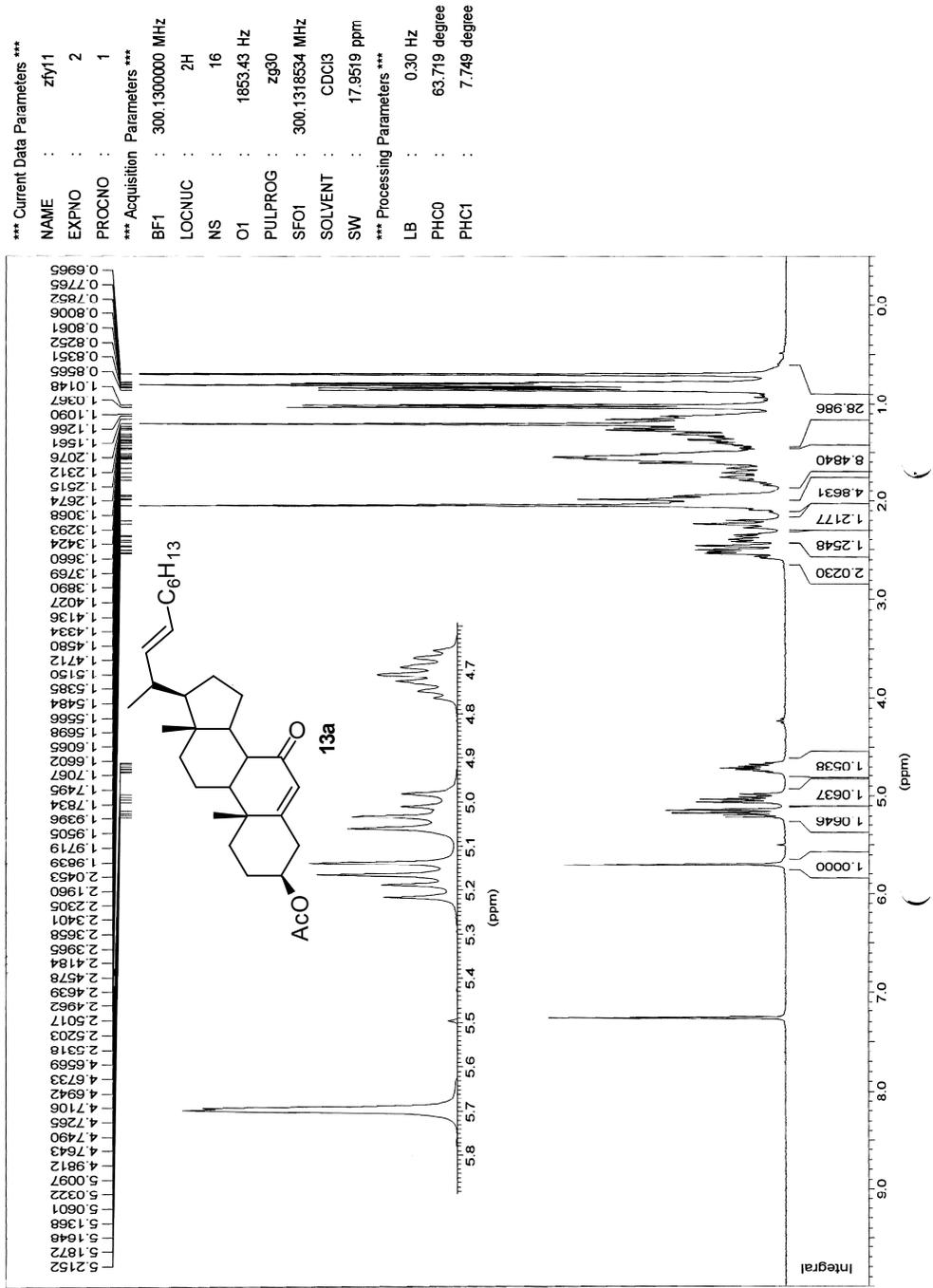
F1-X-1 (05-1)



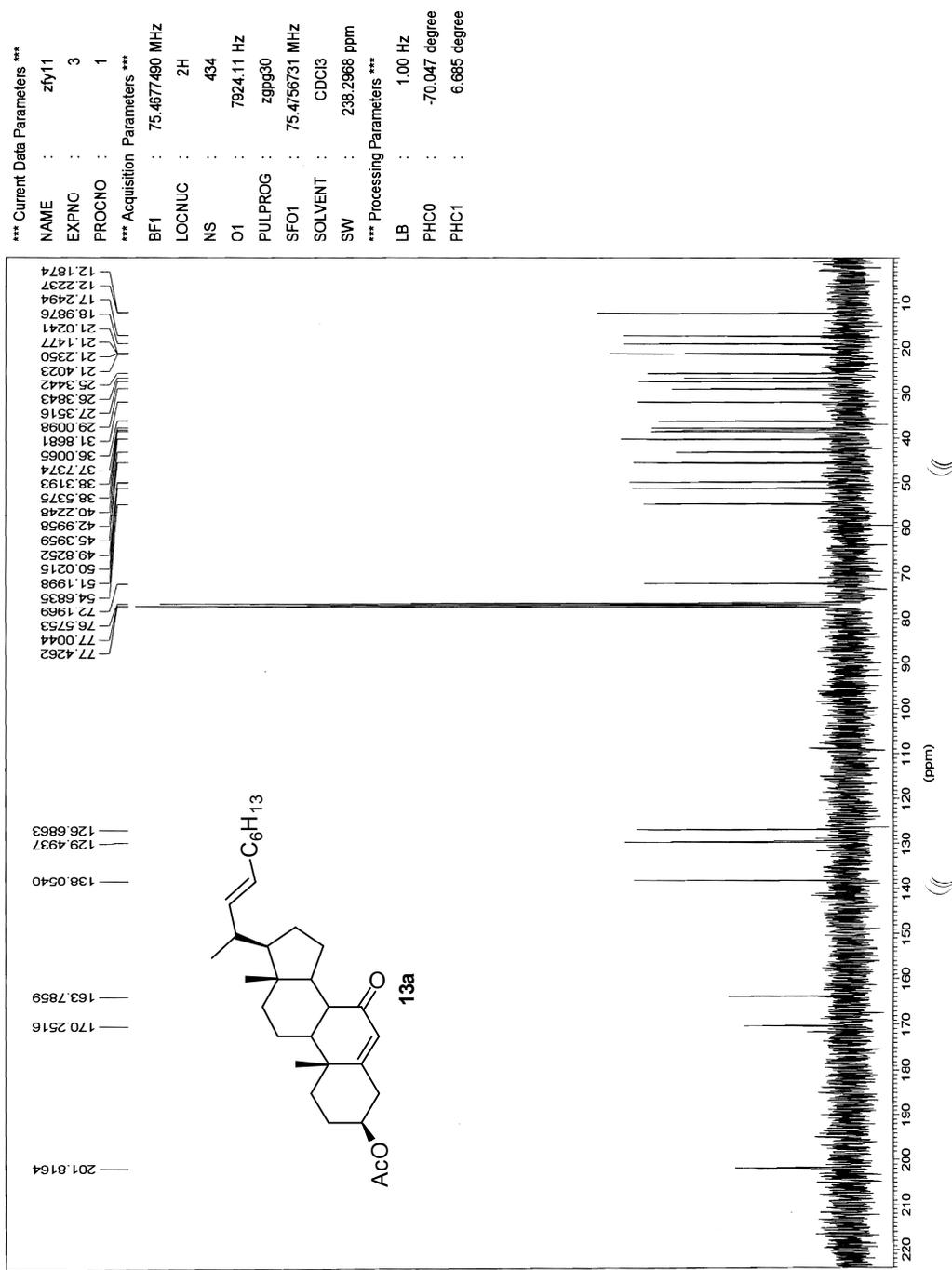
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\*\*\* Acquisition Parameters \*\*\*  
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LOCNUC : 2H  
NS : 101  
NUCLEUS : off  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709936 MHz  
SOLVENT : CDCl3  
SW : 238.7675 ppm  
TD : 65536  
\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
\*\*\* 1D NMR Plot Parameters \*\*\*  
NUCLEUS : off



<sup>1</sup>H normal range AC300  
FY-2-75-1



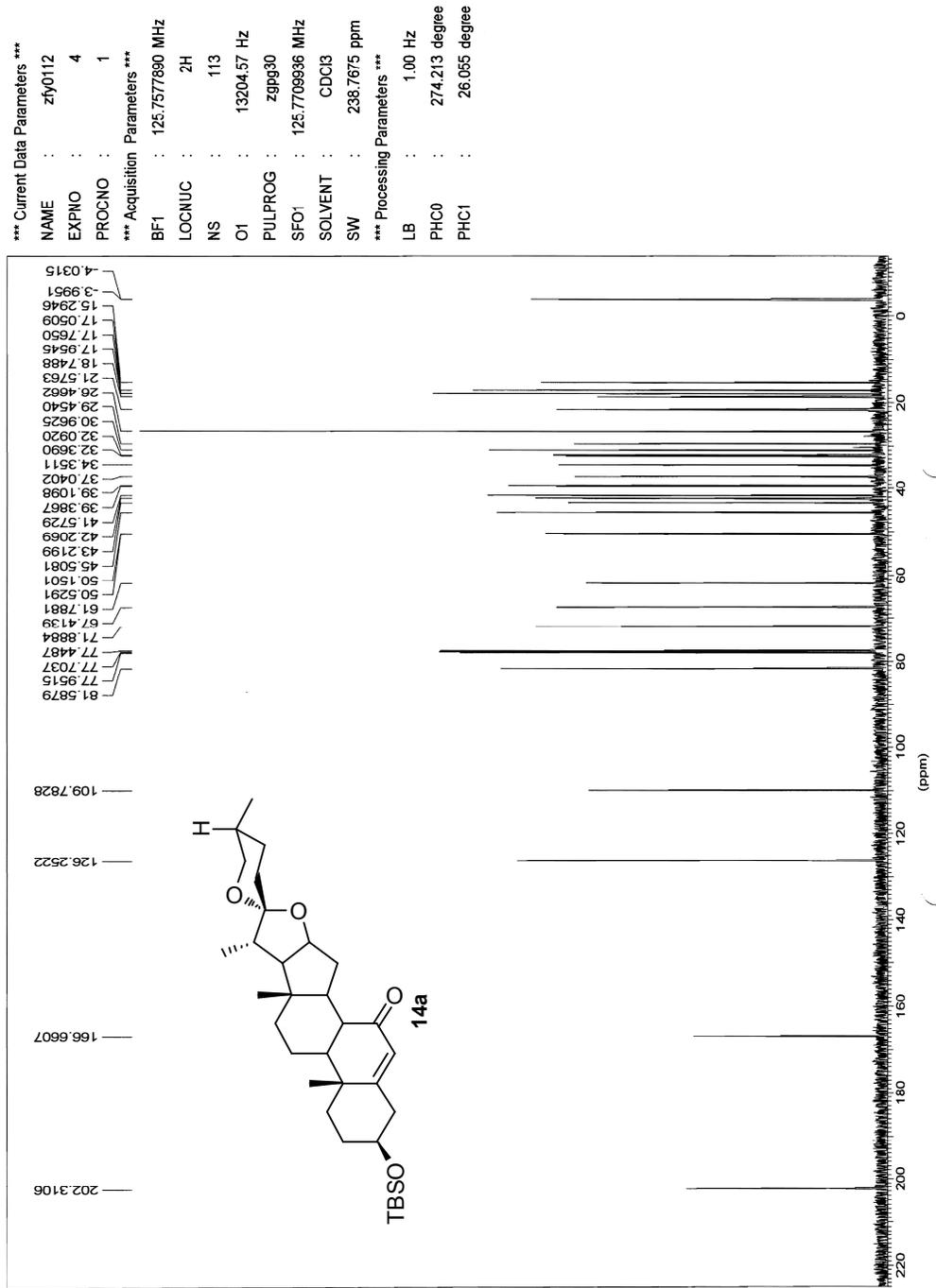
13C Standard AC300





13C AMX500

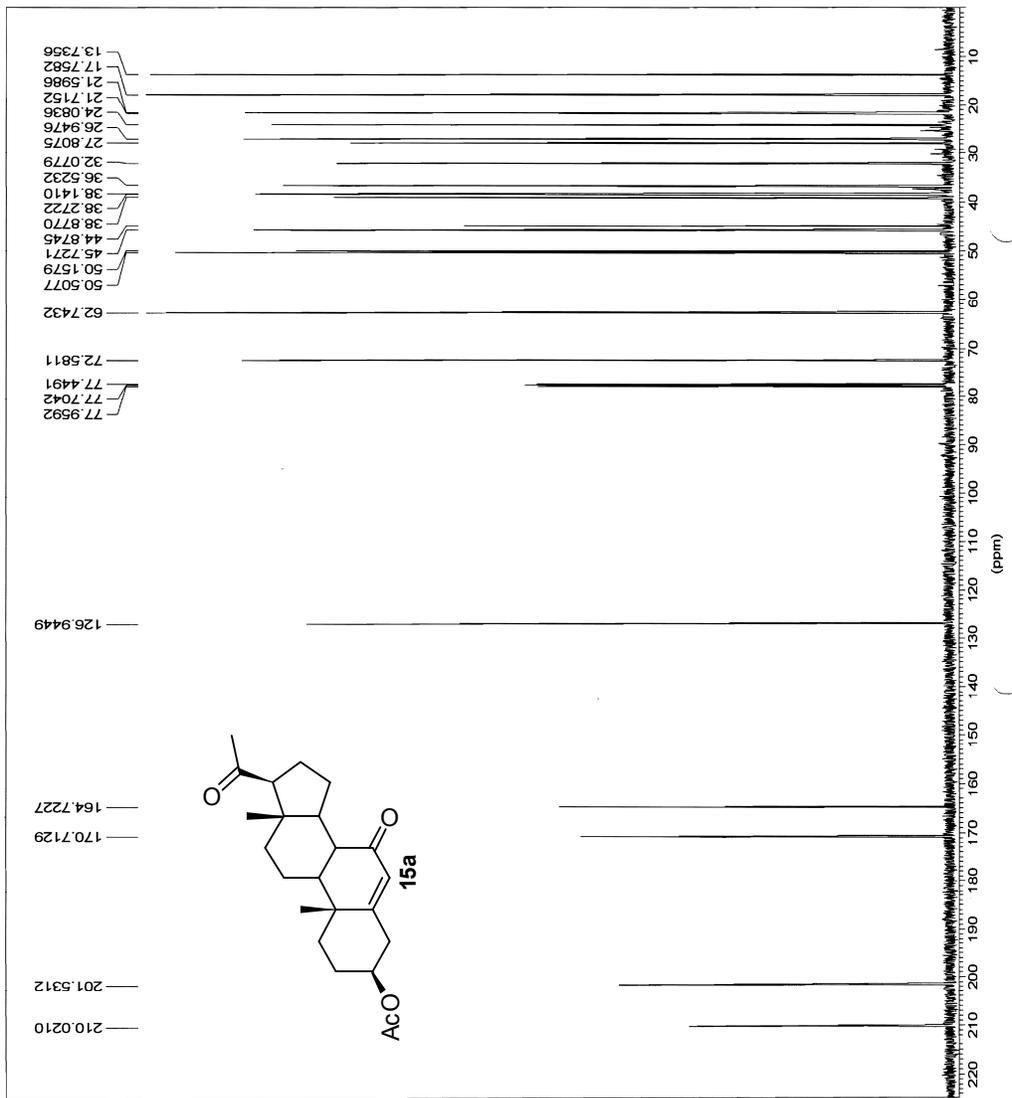
FY-2-119-1



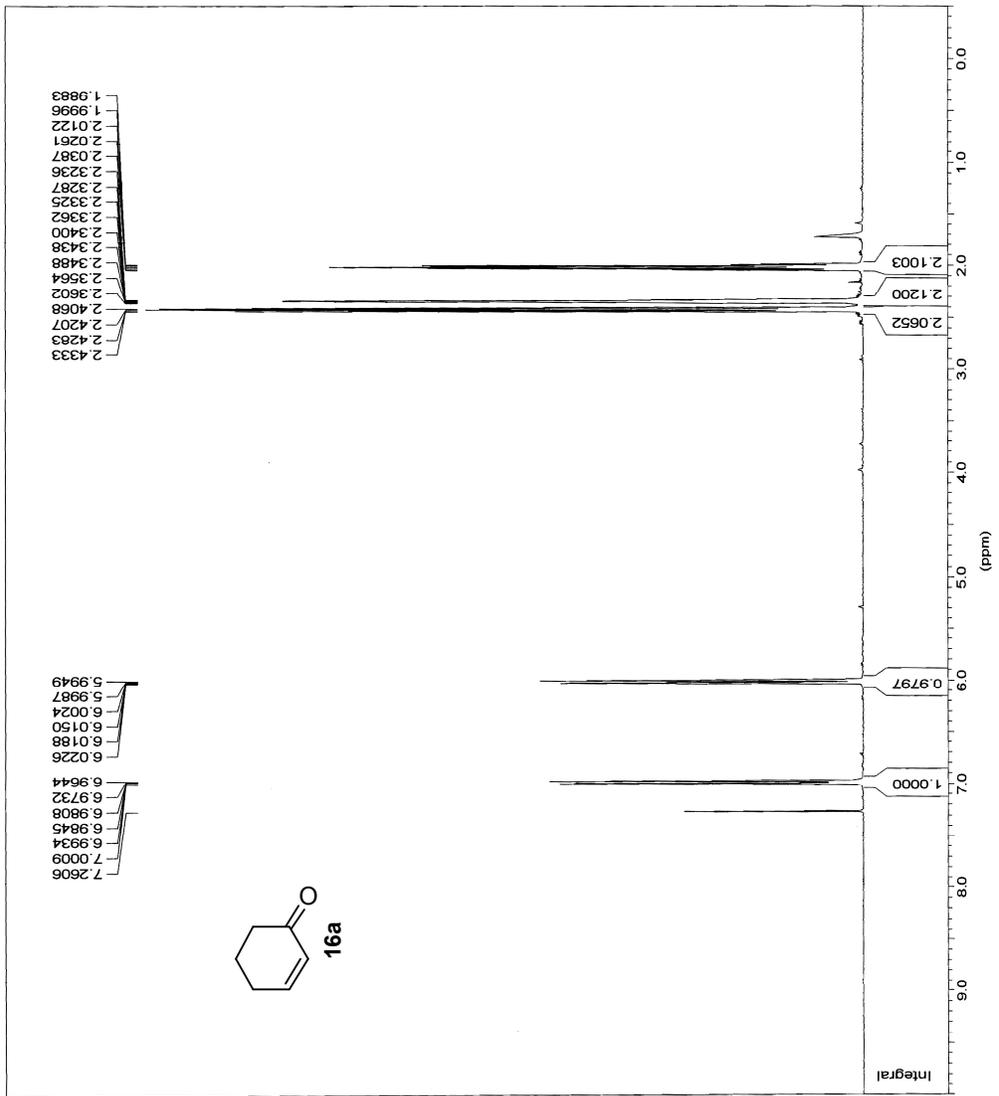


13C AMX500  
FY-2-123-1

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NAME : zfy0113  
EXPNO : 3  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 125.7577890 MHz  
LOCNUC : 2H  
NS : 159  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709936 MHz  
SOLVENT : CDCl3  
SW : 238.7675 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
PHC0 : 259.611 degree  
PHC1 : 32.872 degree



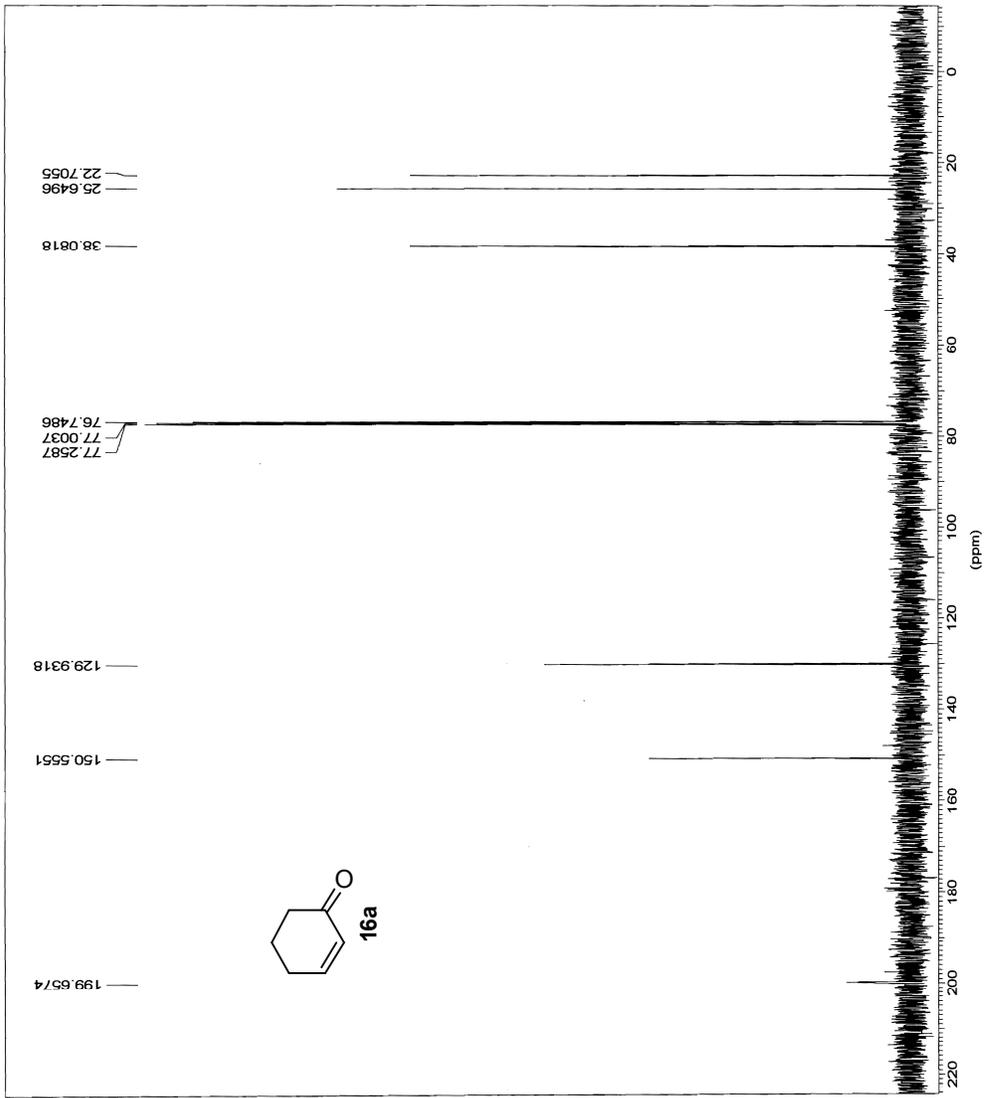
1H AMX500  
cyclo



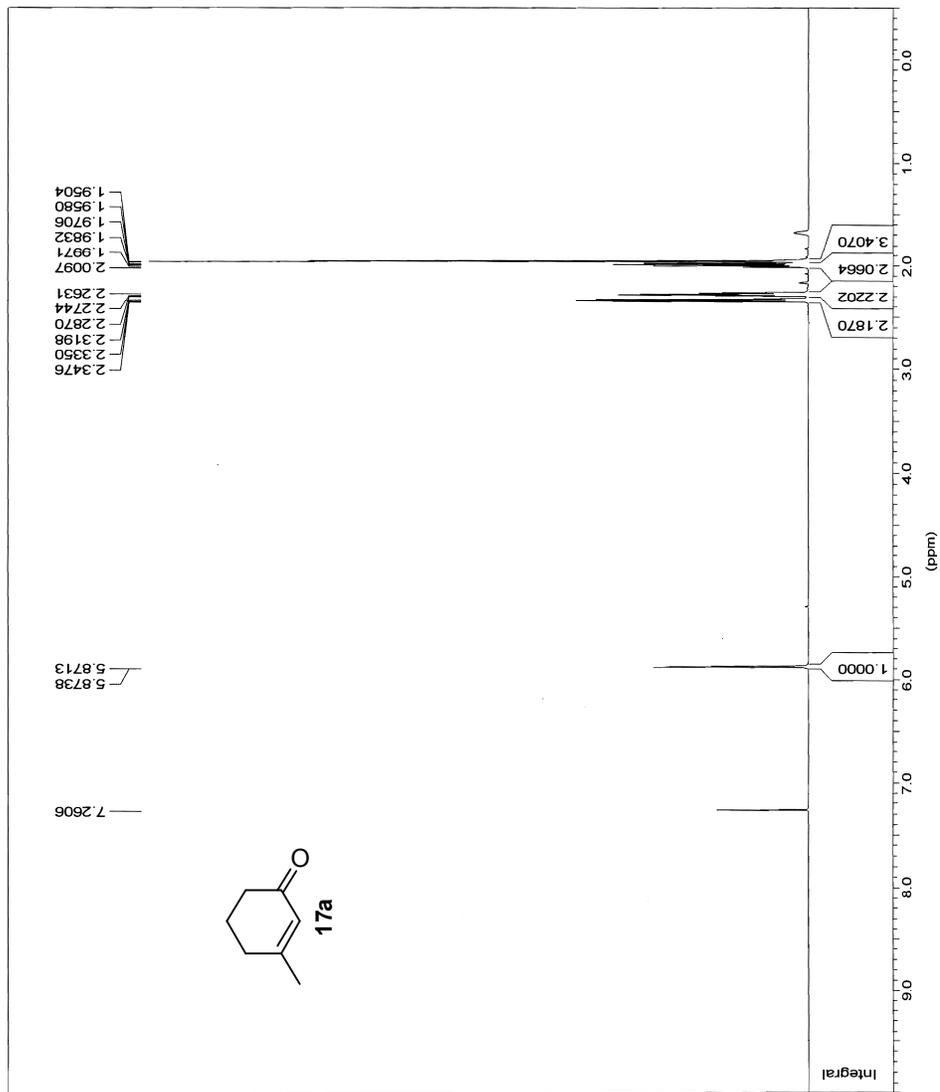
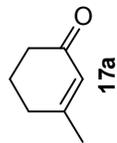
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EXPNO : 4  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
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LOCNUC : 2H  
NS : 8  
O1 : 3088.51 Hz  
PULPROG : zg30  
SFO1 : 500.1330885 MHz  
SOLVENT : CDCl3  
SW : 20.6557 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 0.30 Hz  
PHC0 : 277.597 degree  
PHC1 : -0.918 degree

13C AMX500  
cyclo

\*\*\* Current Data Parameters \*\*\*  
NAME : zfy031-3  
EXPNO : 5  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 125.7577890 MHz  
LOCNUC : 2H  
NS : 54  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709936 MHz  
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\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
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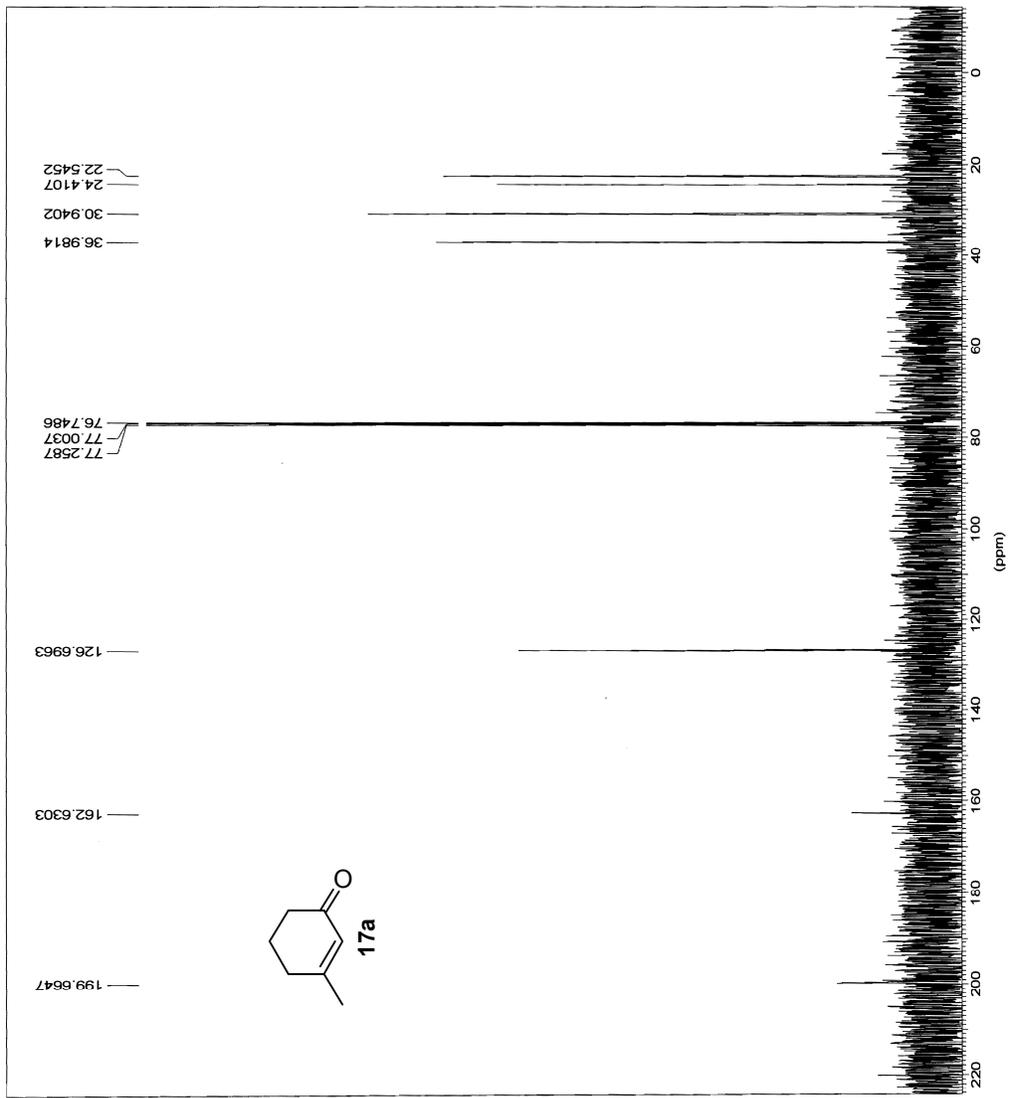


1H AMX500  
methyl



\*\*\* Current Data Parameters \*\*\*  
NAME : zfy031-3  
EXPNO : 2  
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\*\*\* Acquisition Parameters \*\*\*  
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NS : 8  
O1 : 3088.51 Hz  
PULPROG : zg30  
SFO1 : 500.1330885 MHz  
SOLVENT : CDCl3  
SW : 20.6557 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 0.30 Hz  
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PHC1 : -3.827 degree

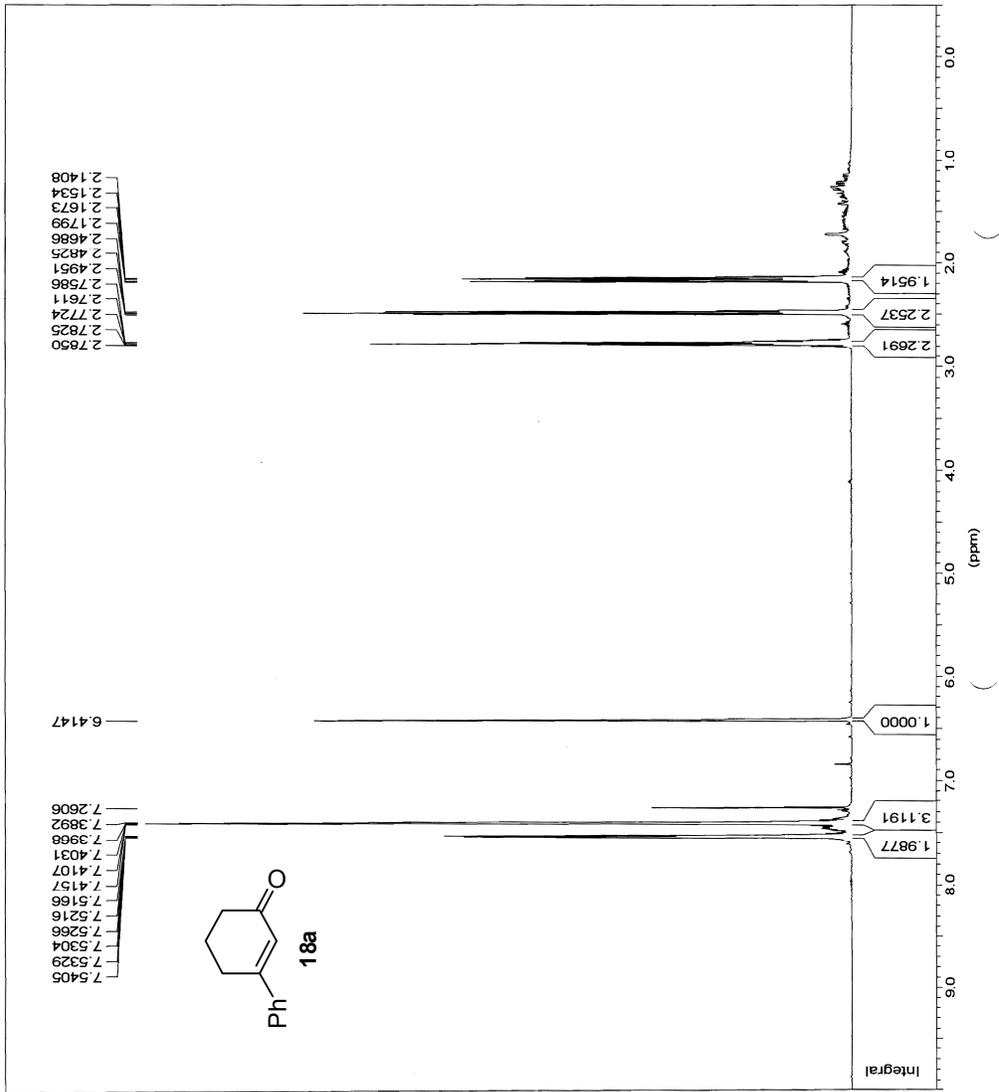
13C AMX500  
methyl



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EXPNO : 3  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 125.7577890 MHz  
LOCHUC : 2H  
NS : 29  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709936 MHz  
SOLVENT : CDCl3  
SW : 238.7675 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
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PHC1 : 26.930 degree

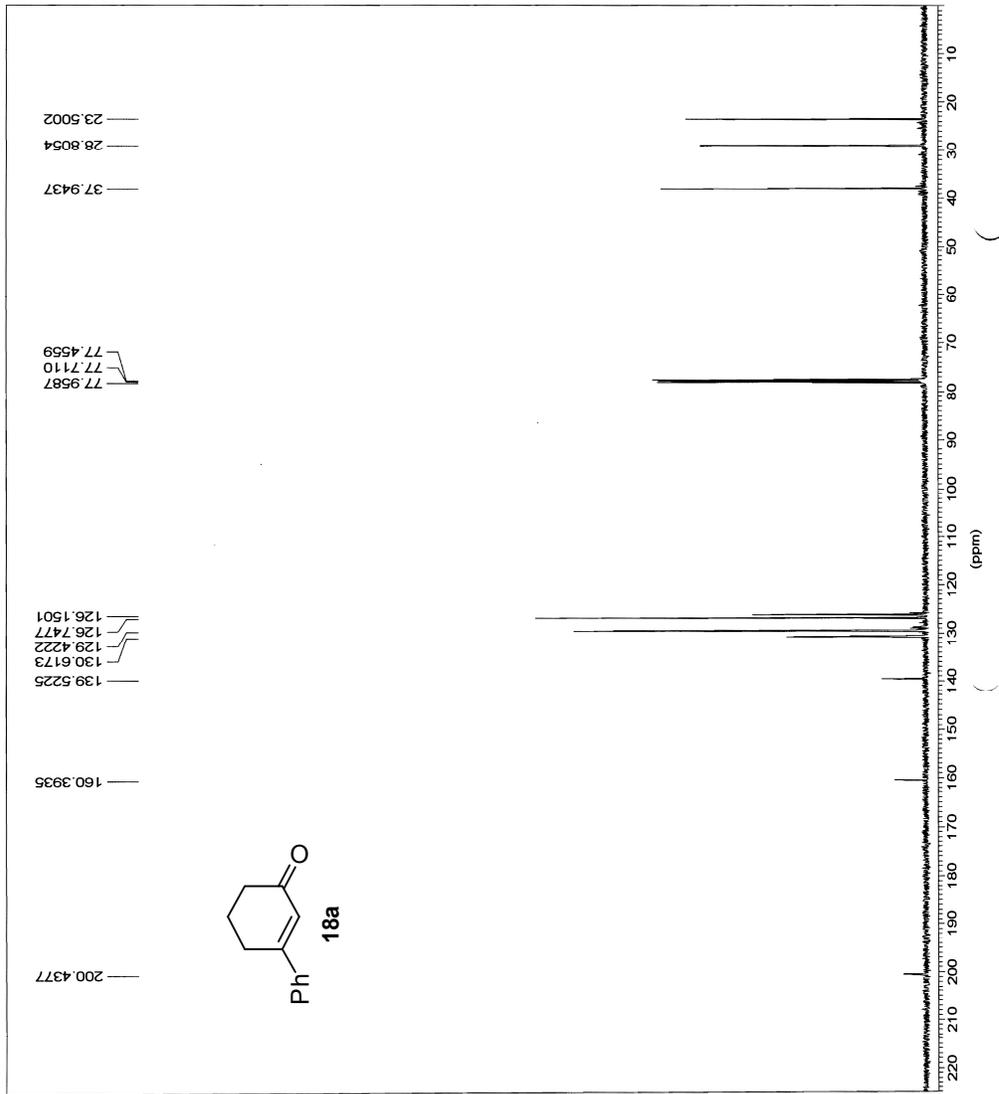
1H AMX500  
FY-2-95-1

\*\*\* Current Data Parameters \*\*\*  
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PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
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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  
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PHC1 : 2.378 degree



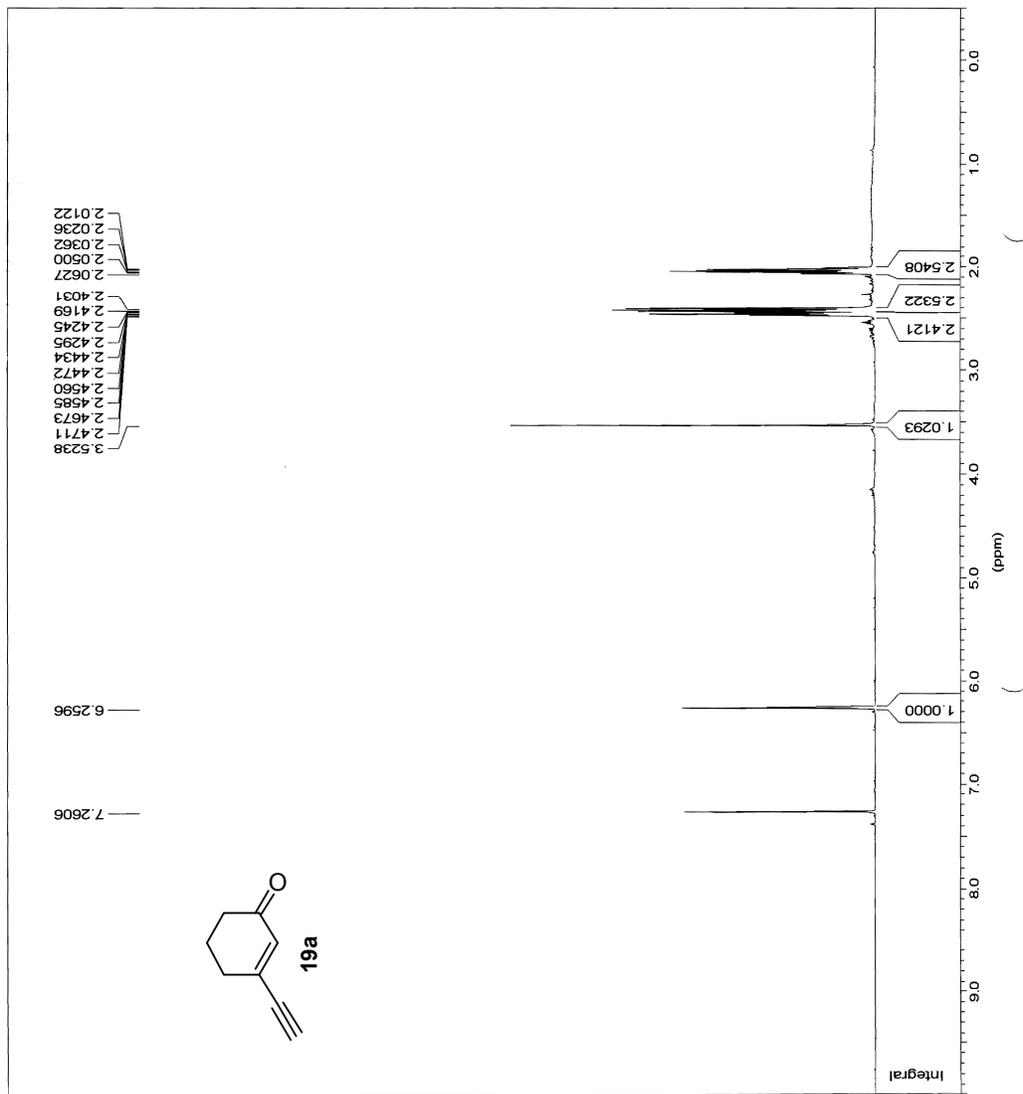
13C AMX500

FY-2-95-1



\*\*\* Current Data Parameters \*\*\*  
NAME : zfy010-1  
EXPNO : 2  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 125.777690 MHz  
LOCNUC : 2H  
NS : 401  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709836 MHz  
SOLVENT : CDCl3  
SW : 238.7675 ppm  
\*\*\* Processing Parameters \*\*\*  
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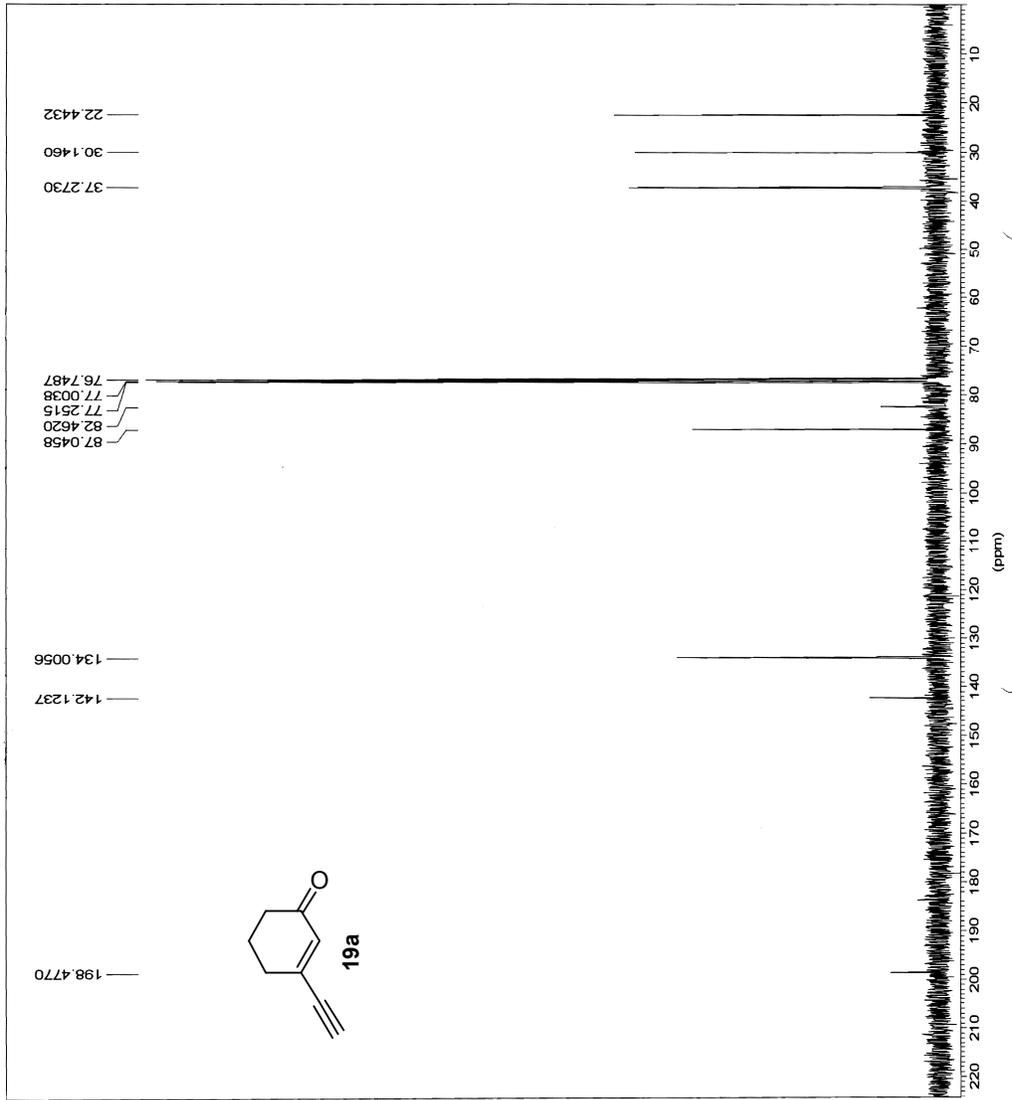
1H AMX500



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EXPNO : 1  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
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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  
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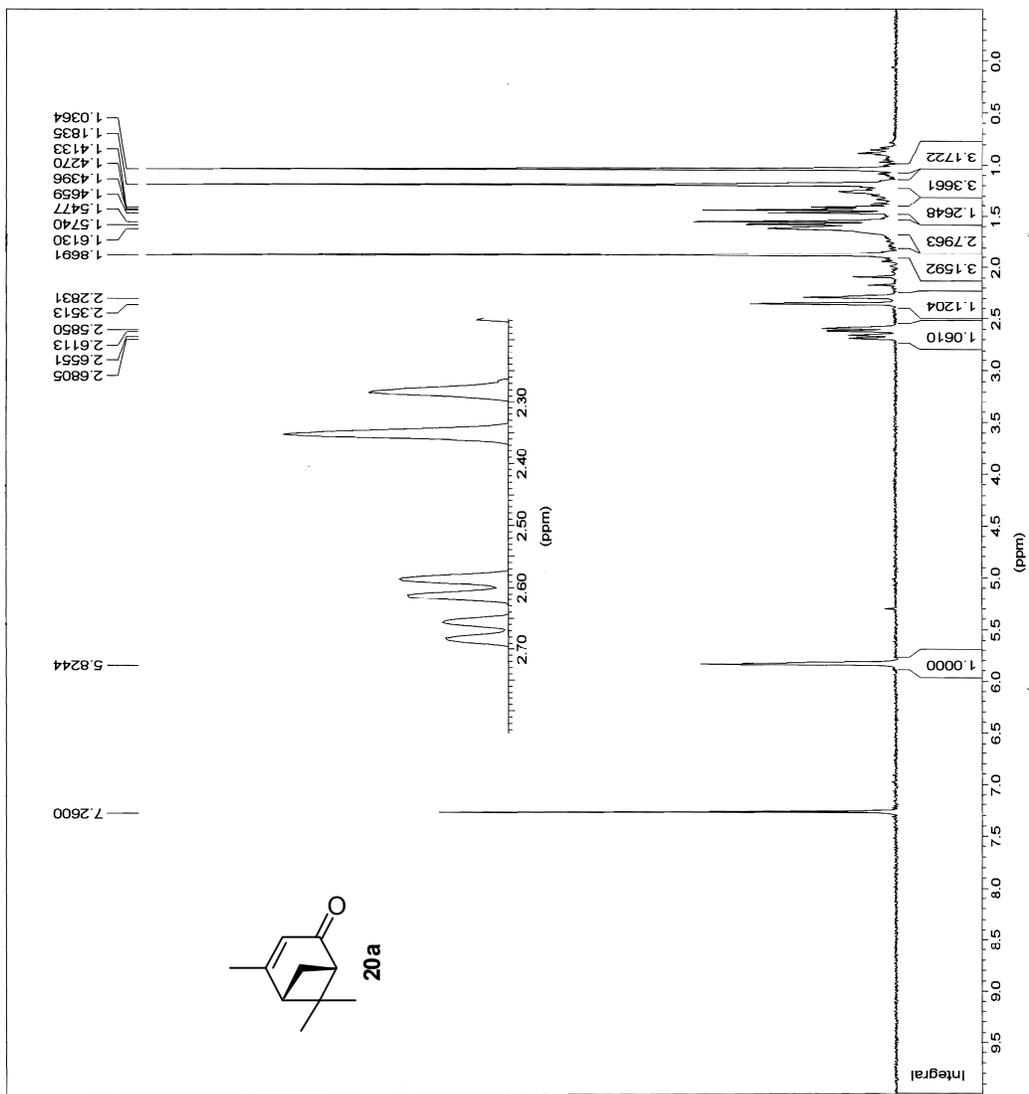
13C AMX500

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NAME : zfy0204a  
EXPNO : 2  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 125.7577890 MHz  
LOCHUC : 2H  
NS : 151  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709936 MHz  
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\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
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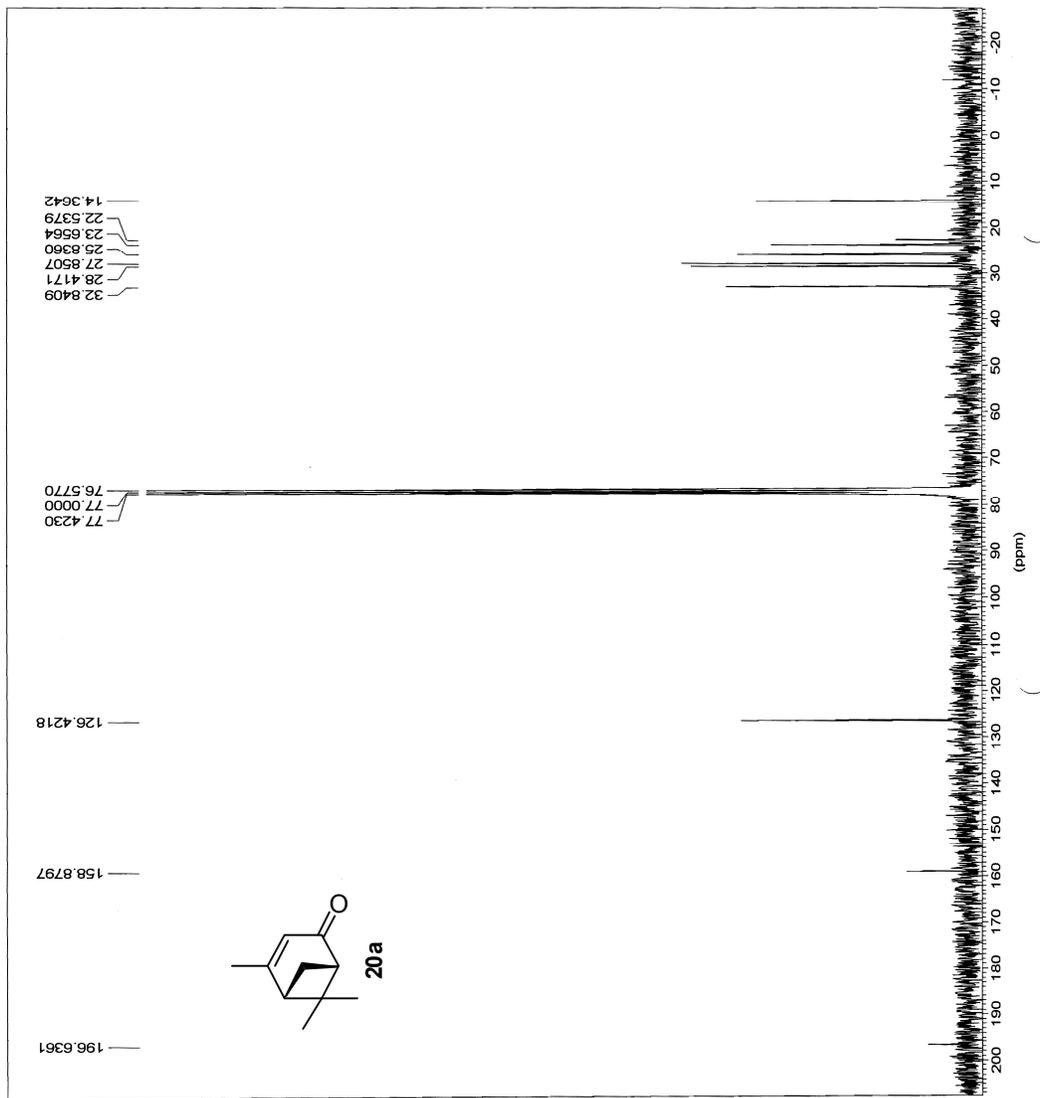


FY-2-193-1

\*\*\* Current Data Parameters \*\*\*  
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PROCNO : 1  
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BF1 : 300.130000 MHz  
LOCNUC : 2H  
NS : 8  
O1 : 1250.00 Hz  
PULPROG : zg30  
SFO1 : 300.1312500 MHz  
SOLVENT : CDCl3  
SW : 15.9573 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 0.10 Hz  
PHC0 : 778.701 degree  
PHC1 : 5.880 degree

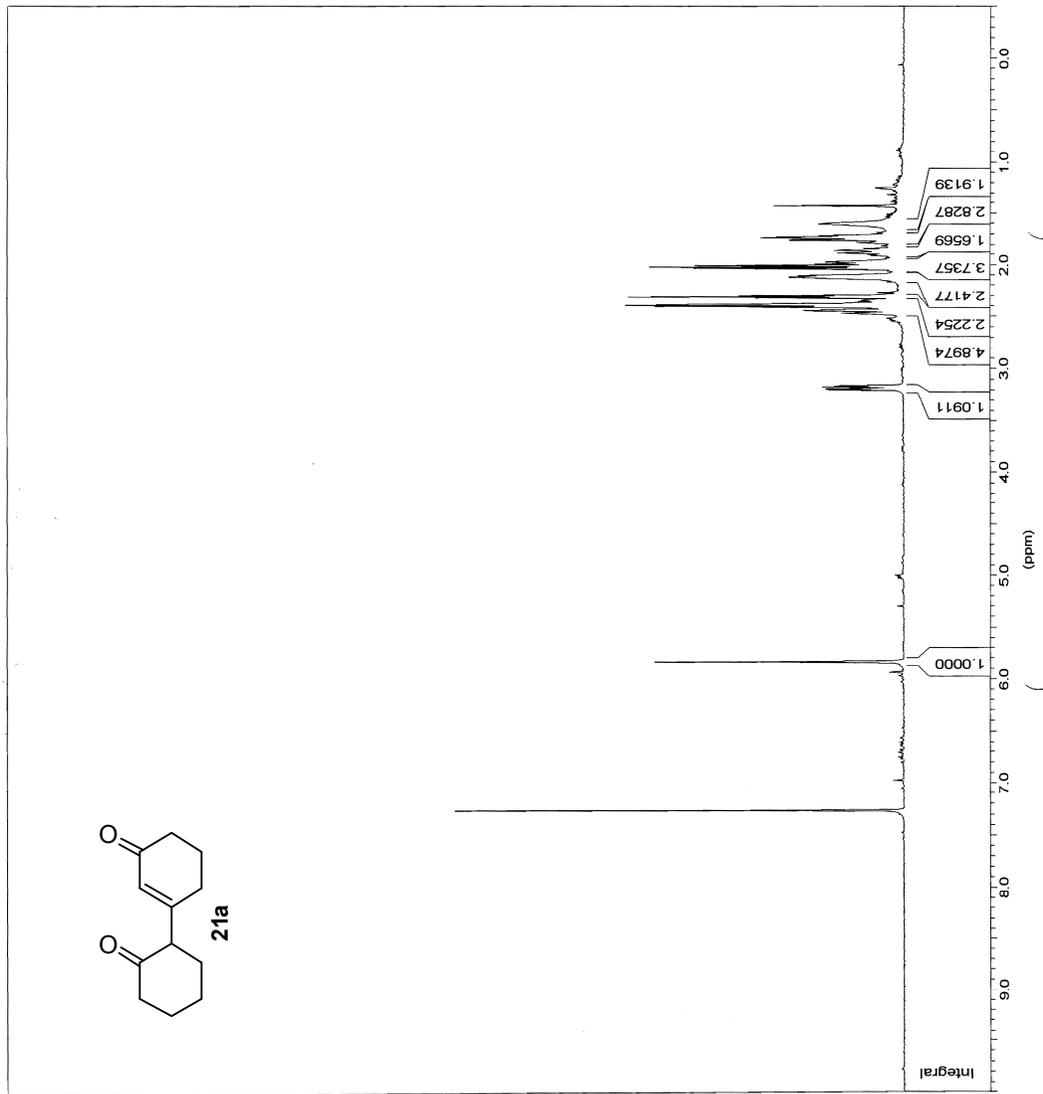
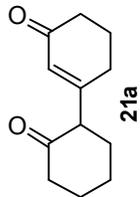


FY-2-193-1



\*\*\* Current Data Parameters \*\*\*  
NAME : zfy0206  
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PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 75.4677190 MHz  
LOCNUC : 2H  
NS : 1068  
O1 : 6792.09 Hz  
PULPROG : zgpg  
SFO1 : 75.4745111 MHz  
SOLVENT : CDCl3  
SW : 234.9203 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 3.00 Hz  
PHC0 : 181.966 degree  
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1H AMX500  
FY-2-185-1



\*\*\* Current Data Parameters \*\*\*

NAME : zfy0204  
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PROCNO : 1

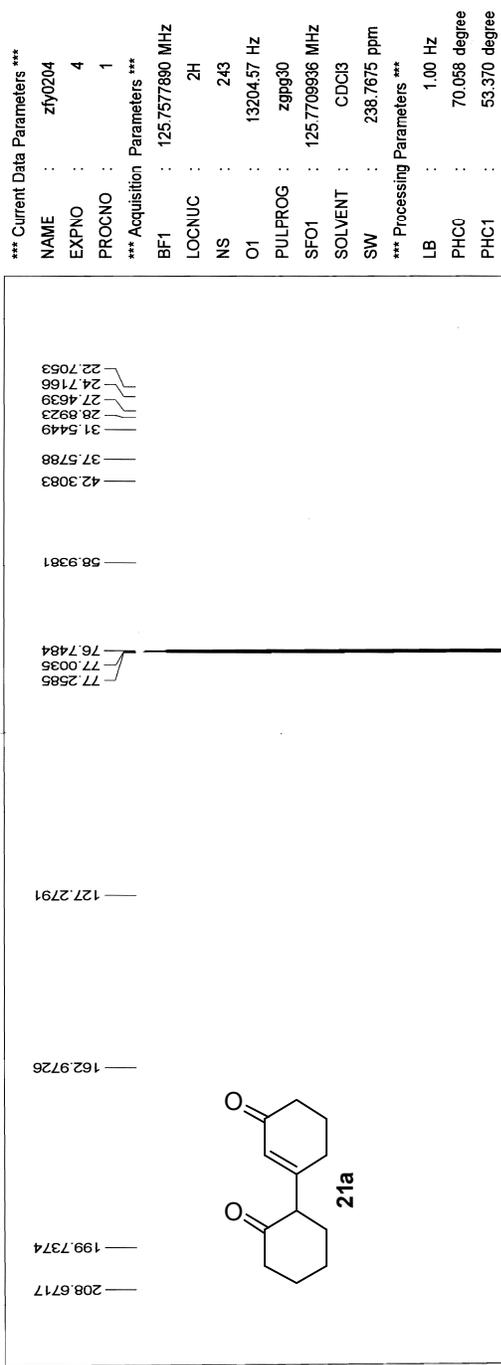
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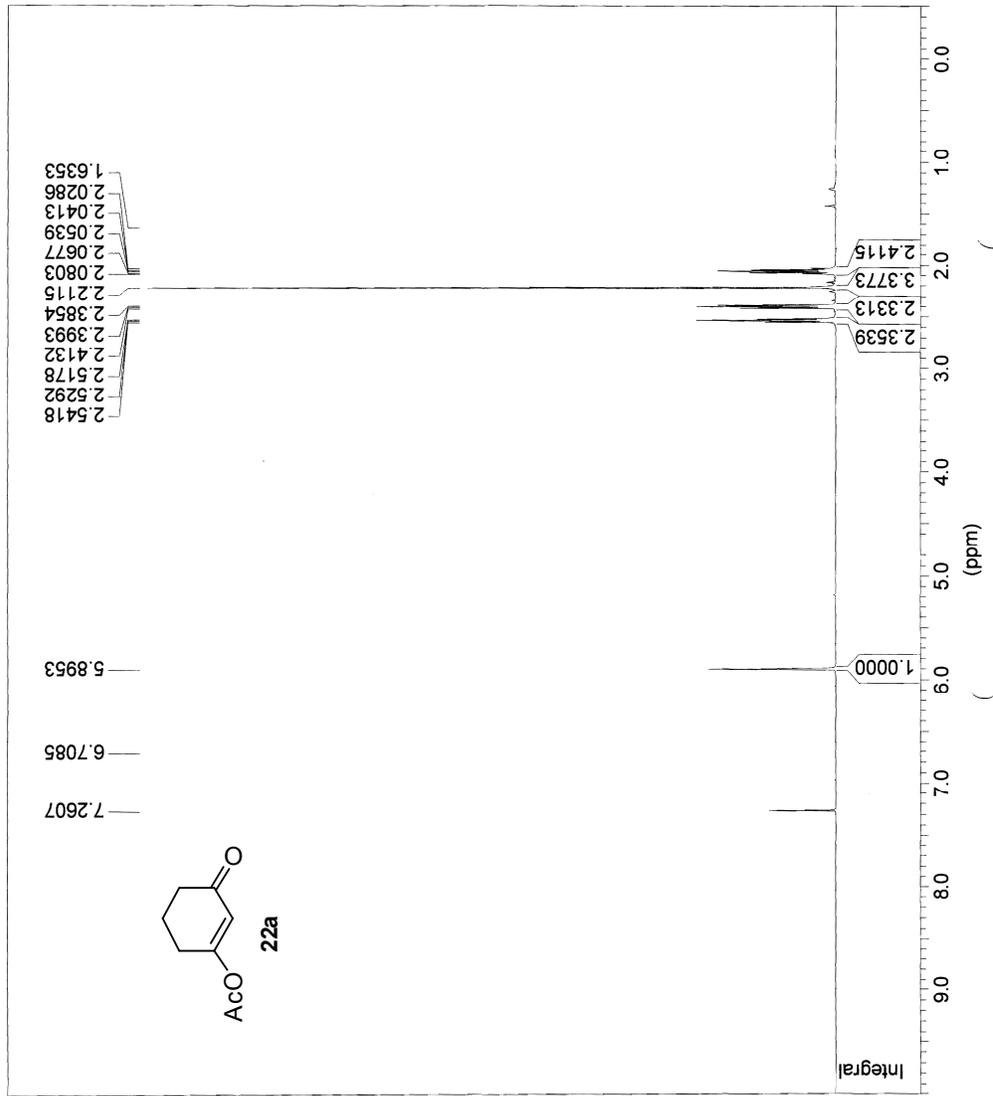
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13C AMX500  
FY-2-185-1

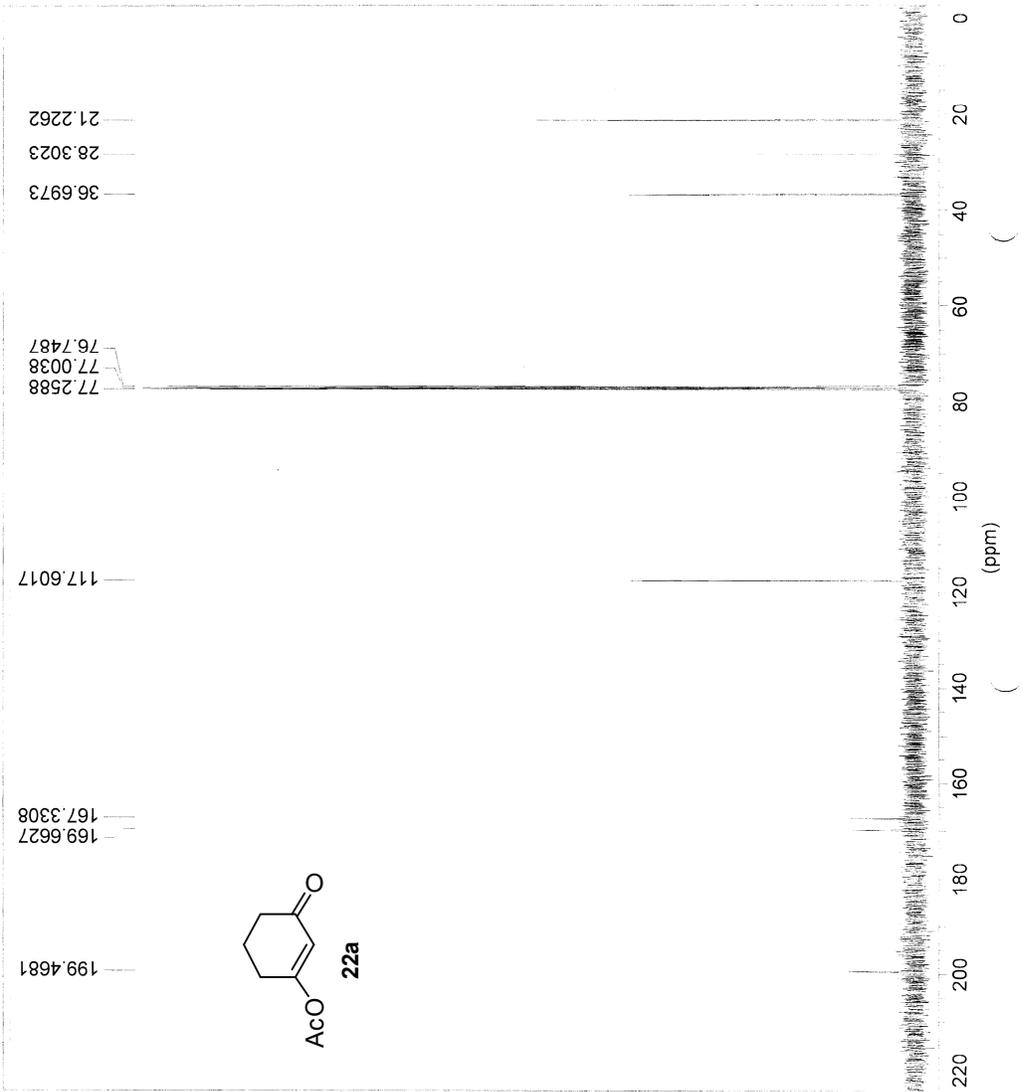


1H AMX500  
FY-2-67-1



\*\*\* Current Data Parameters \*\*\*  
NAME : zfy0126  
EXPNO : 4  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
INSTRUM : av500  
LOCNUC : 2H  
NS : 8  
NUCLEUS : off  
O1 : 3088.51 Hz  
PULPROG : zg30  
SFO1 : 500.1330885 MHz  
SOLVENT : CDCl3  
SW : 20.6557 ppm  
TD : 32768  
TE : 300.0 K  
\*\*\* Processing Parameters \*\*\*  
LB : 0.30 Hz  
\*\*\* 1D NMR Plot Parameters \*\*\*  
NUCLEUS : off

13C AMX500



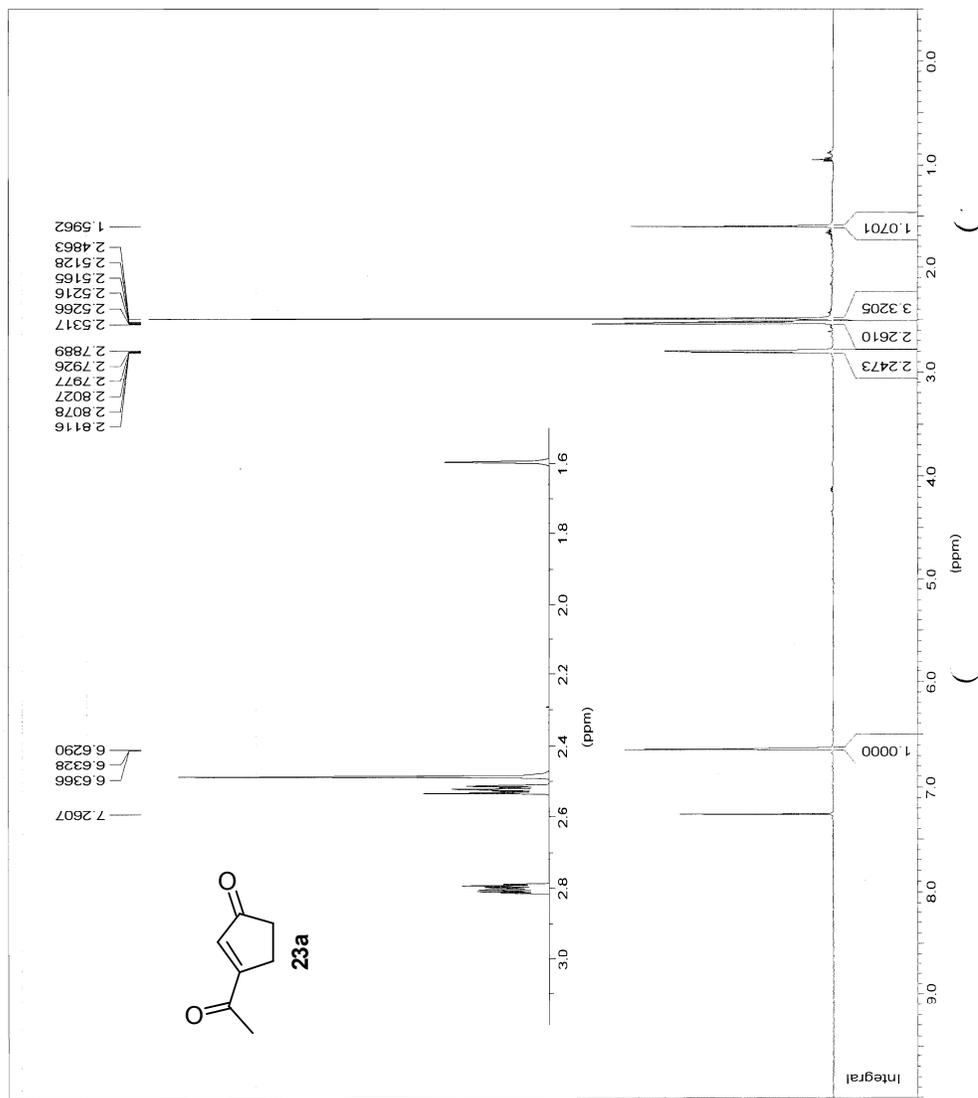
\*\*\* Current Data Parameters \*\*\*

NAME : zfy0126  
EXPNO : 2  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
INSTRUM : av500  
LOCNUC : 2H  
NS : 86  
NUCLEUS : off  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709936 MHz  
SOLVENT : CDCl3  
SW : 238.7675 ppm  
TD : 65536  
\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
\*\*\* 1D NMR Plot Parameters \*\*\*  
NUCLEUS : off

1H AMX500

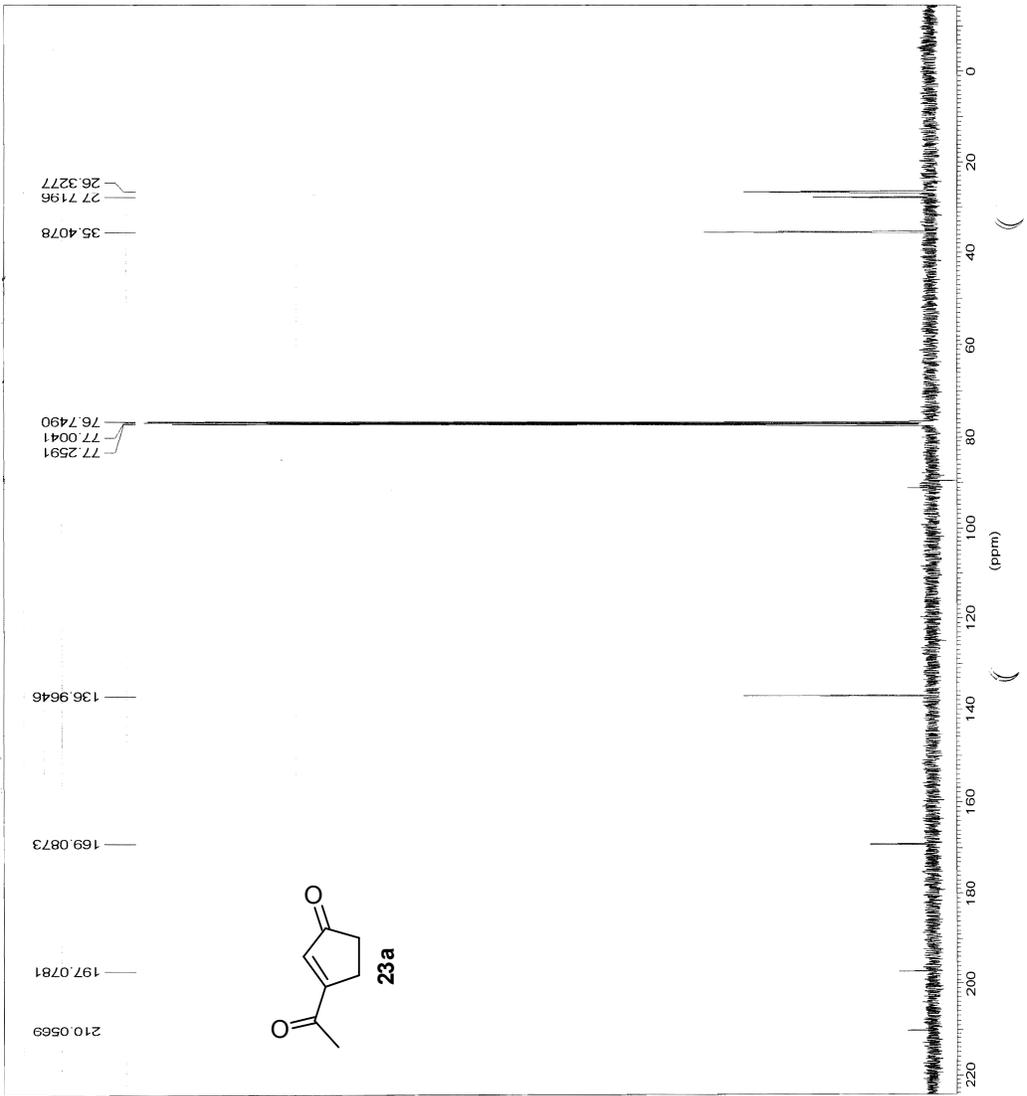
FY-2-25-1

\*\*\* Current Data Parameters \*\*\*  
NAME : zfy1220  
EXPNO : 2  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 500.1300000 MHz  
LOCNUC : 2H  
NS : 16  
O1 : 3088.51 Hz  
PULPROG : zg30  
SFO1 : 500.1330885 MHz  
SOLVENT : CDC8  
SW : 20.6557 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 0.30 Hz  
PHC0 : 107.308 degree  
PHC1 : -0.667 degree

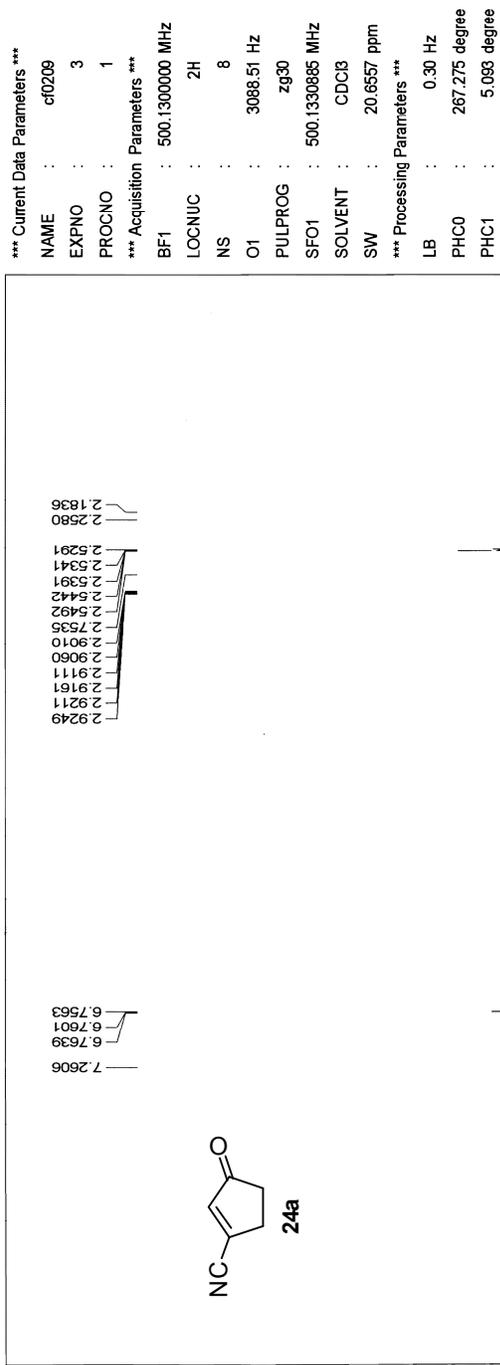


13C AMX500  
FY-2-25-1

\*\*\* Current Data Parameters \*\*\*  
NAME : zfy1220  
EXPNO : 3  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 125.7577890 MHz  
LOCNUC : 2H  
NS : 323  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709936 MHz  
SOLVENT : CDCl3  
SW : 238.7675 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
PHC0 : 251.665 degree  
PHC1 : 47.899 degree

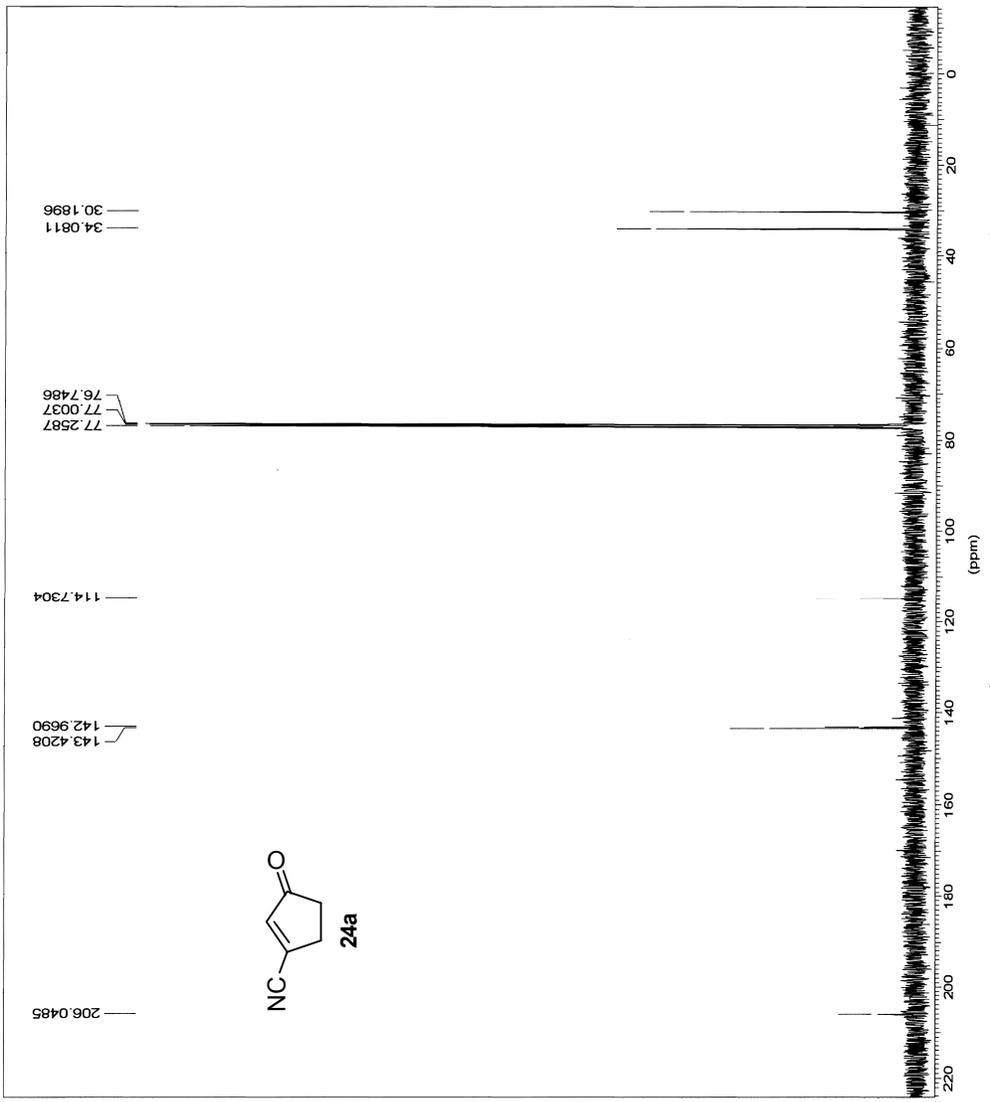


1H AMX500  
fy-2-195-1



13C AMX500  
fy-2-195-1

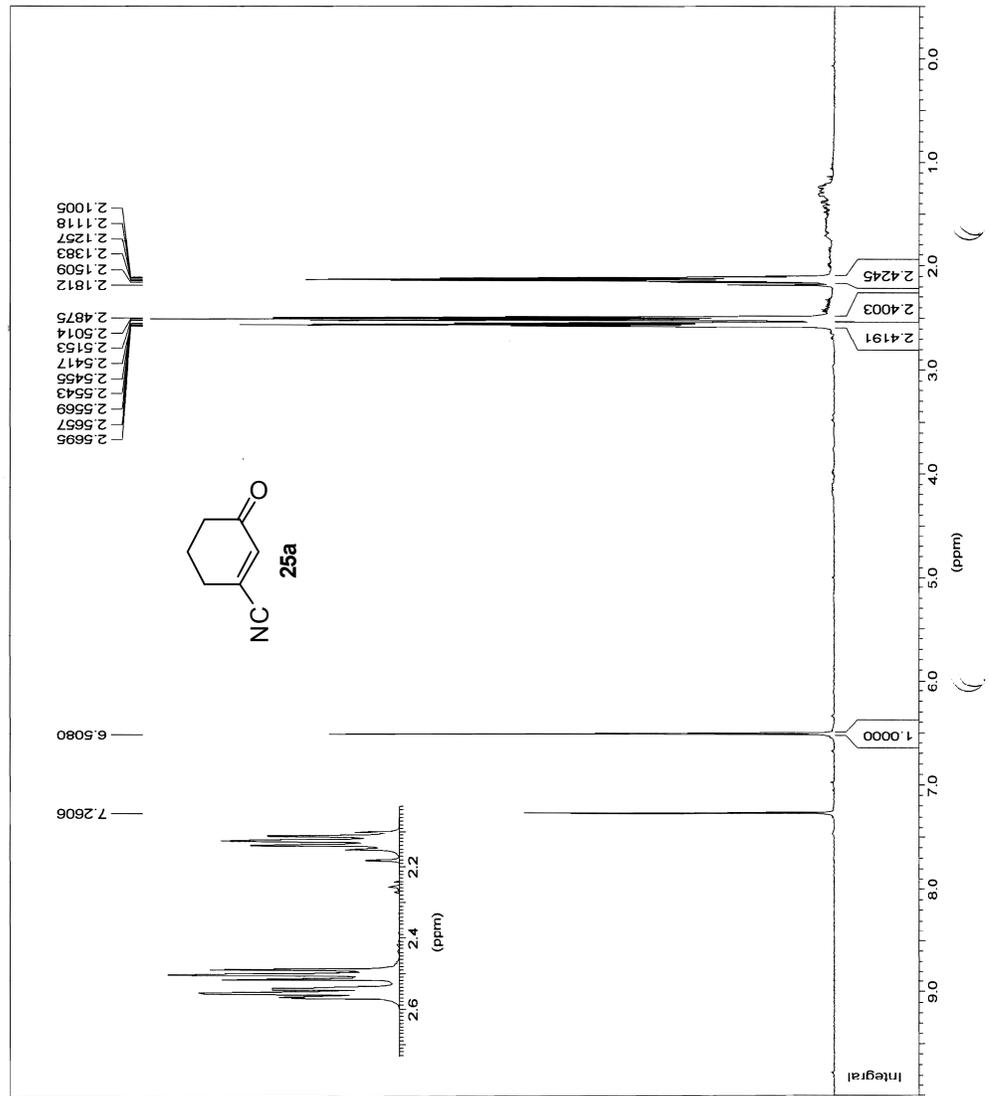
\*\*\* Current Data Parameters \*\*\*  
NAME : c10209  
EXPNO : 4  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 125.7577890 MHz  
LOCNUC : 2H  
NS : 158  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709836 MHz  
SOLVENT : CDCl3  
SW : 238.7675 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
PHC0 : 99.192 degree  
PHC1 : -17.471 degree



1H AMX500

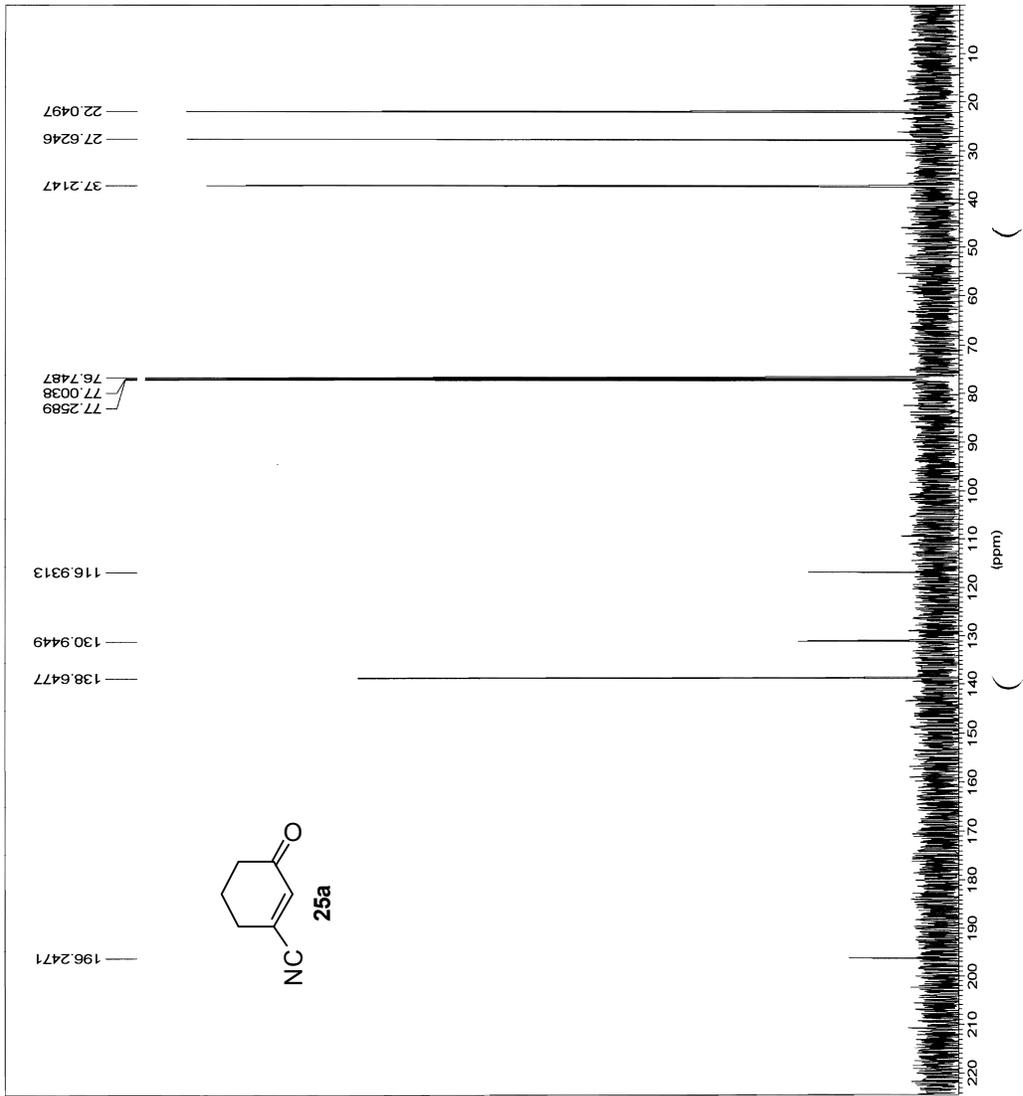
FY-279-1

\*\*\* Current Data Parameters \*\*\*  
NAME : zfy12  
EXPNO : 2  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 500.130000 MHz  
LOGNJC : 2H  
NS : 16  
O1 : 3088.51 Hz  
PULPROG : zg30  
SFO1 : 500.1300885 MHz  
SOLVENT : CDC13  
SW : 20.6557 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 0.30 Hz  
PHC0 : 111.238 degree  
PHC1 : 0.493 degree



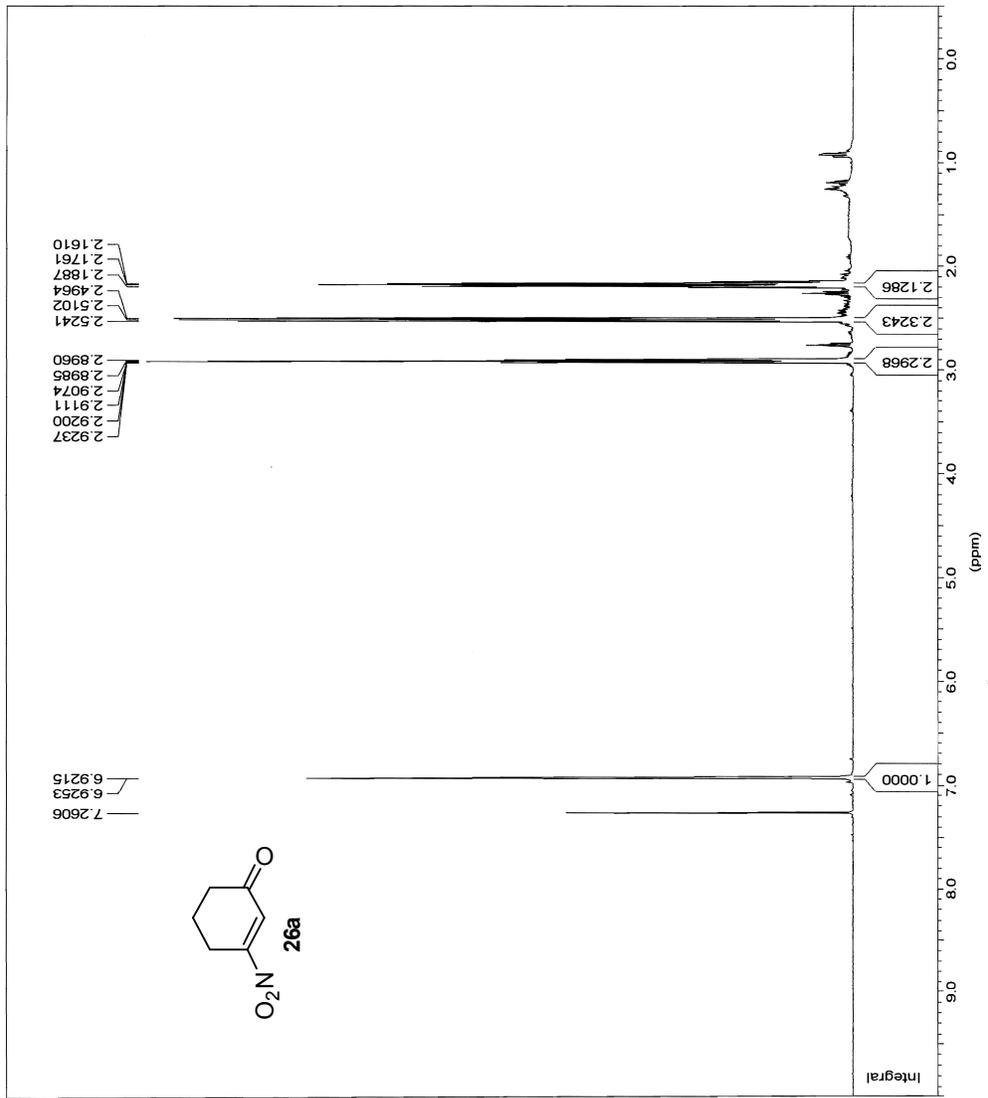
13C AMX500

\*\*\* Current Data Parameters \*\*\*  
NAME : zfy12  
EXPNO : 3  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 125.7577690 MHz  
LOCNUC : 2H  
NS : 213  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709936 MHz  
SOLVENT : CDCl3  
SW : 238.7675 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
PHC0 : 247.364 degree  
PHC1 : 57.033 degree



1H AMX500

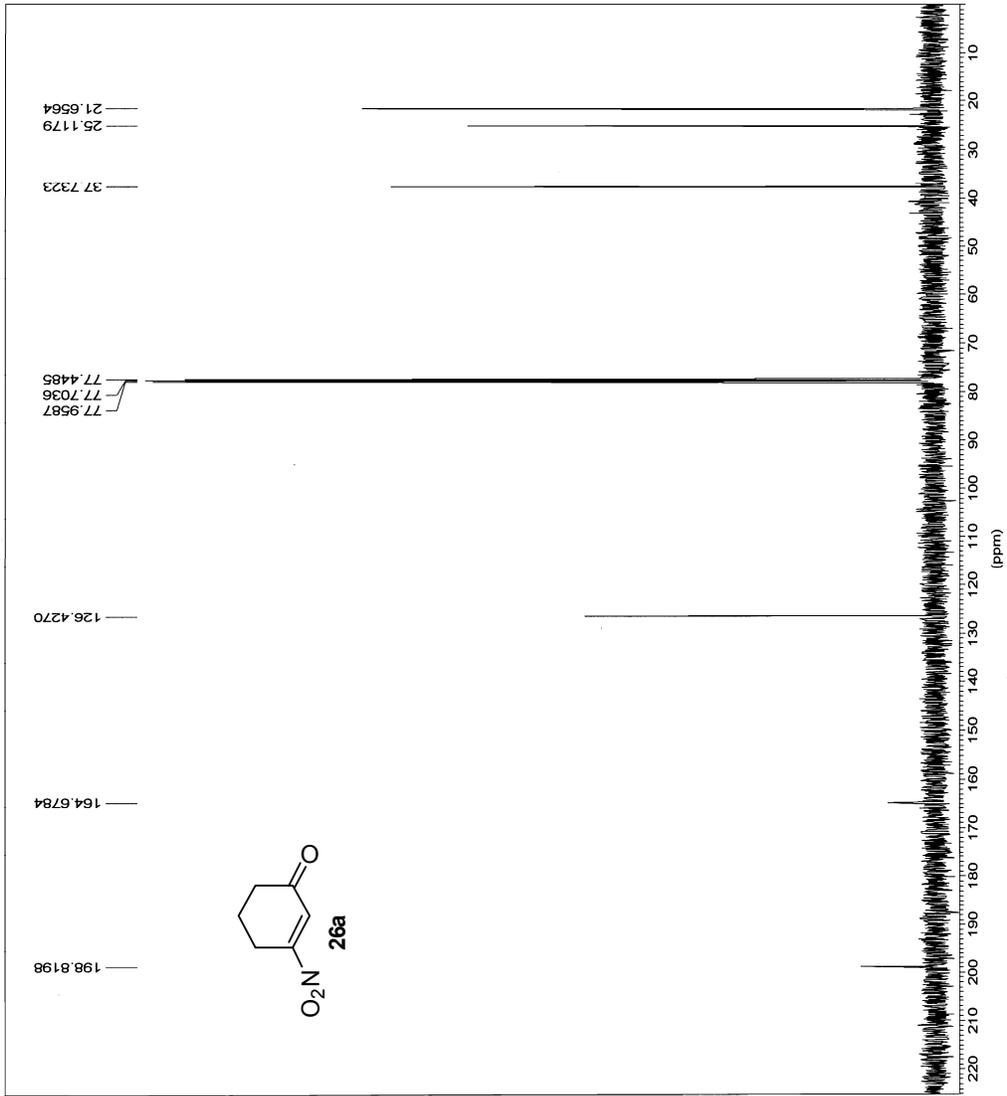
FY-2-9-1



\*\*\* Current Data Parameters \*\*\*

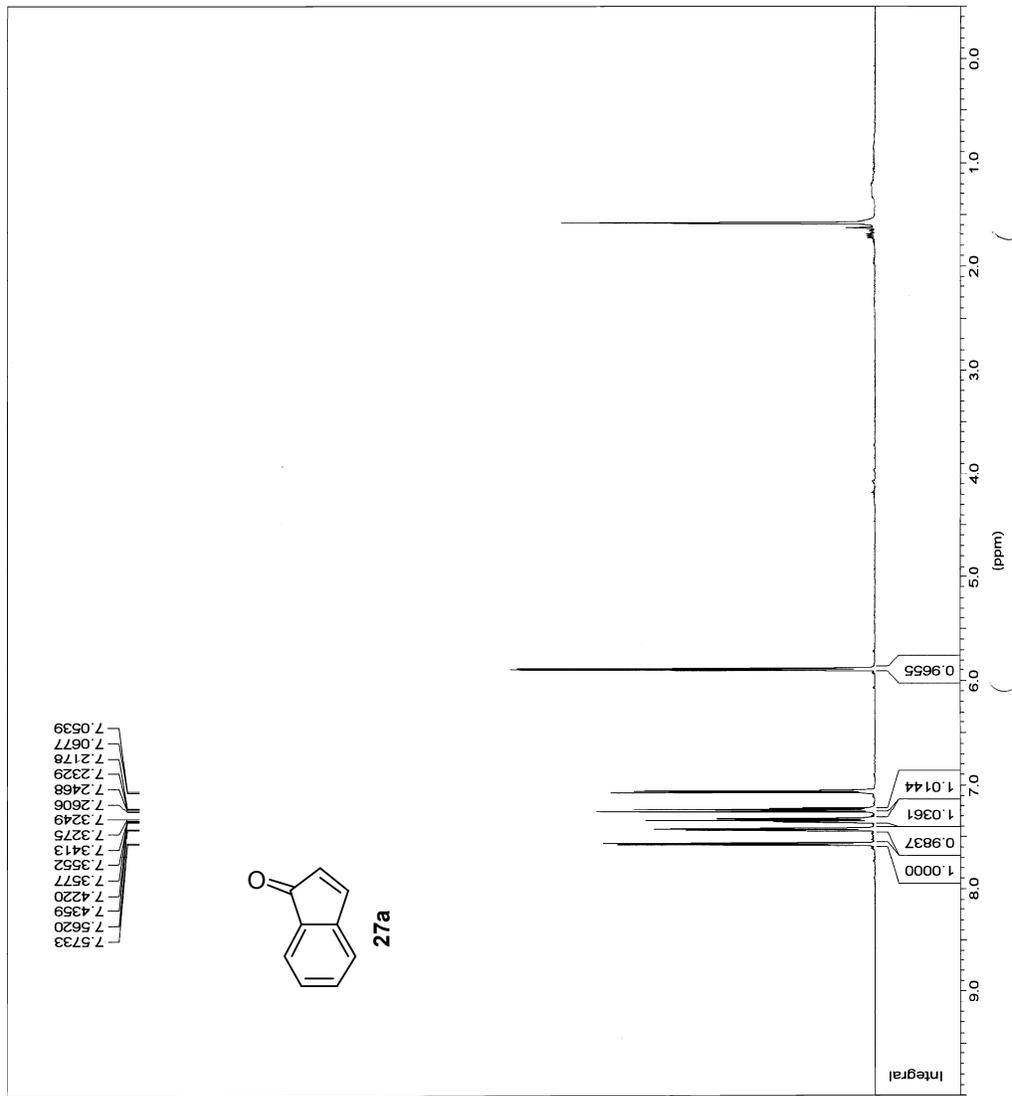
NAME : zfy0105  
EXPNO : 1  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 500.130000 MHz  
LOCNUC : 2H  
NS : 8  
OI : 3088.51 Hz  
PULPROG : zg30  
SFO1 : 500.1330885 MHz  
SOLVENT : CDCl3  
SW : 20.6557 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 0.30 Hz  
PHC0 : 99.701 degree  
PHC1 : 3.532 degree

13C AMX500  
FY-2-5-1



\*\*\* Current Data Parameters \*\*\*  
NAME : zfy0105  
EXPNO : 2  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 125.7577890 MHz  
LOCNUC : 2H  
NS : 159  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709936 MHz  
SOLVENT : CDC13  
SW : 238.7675 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
PHC0 : 258.588 degree  
PHC1 : 33.528 degree

1H AMX500

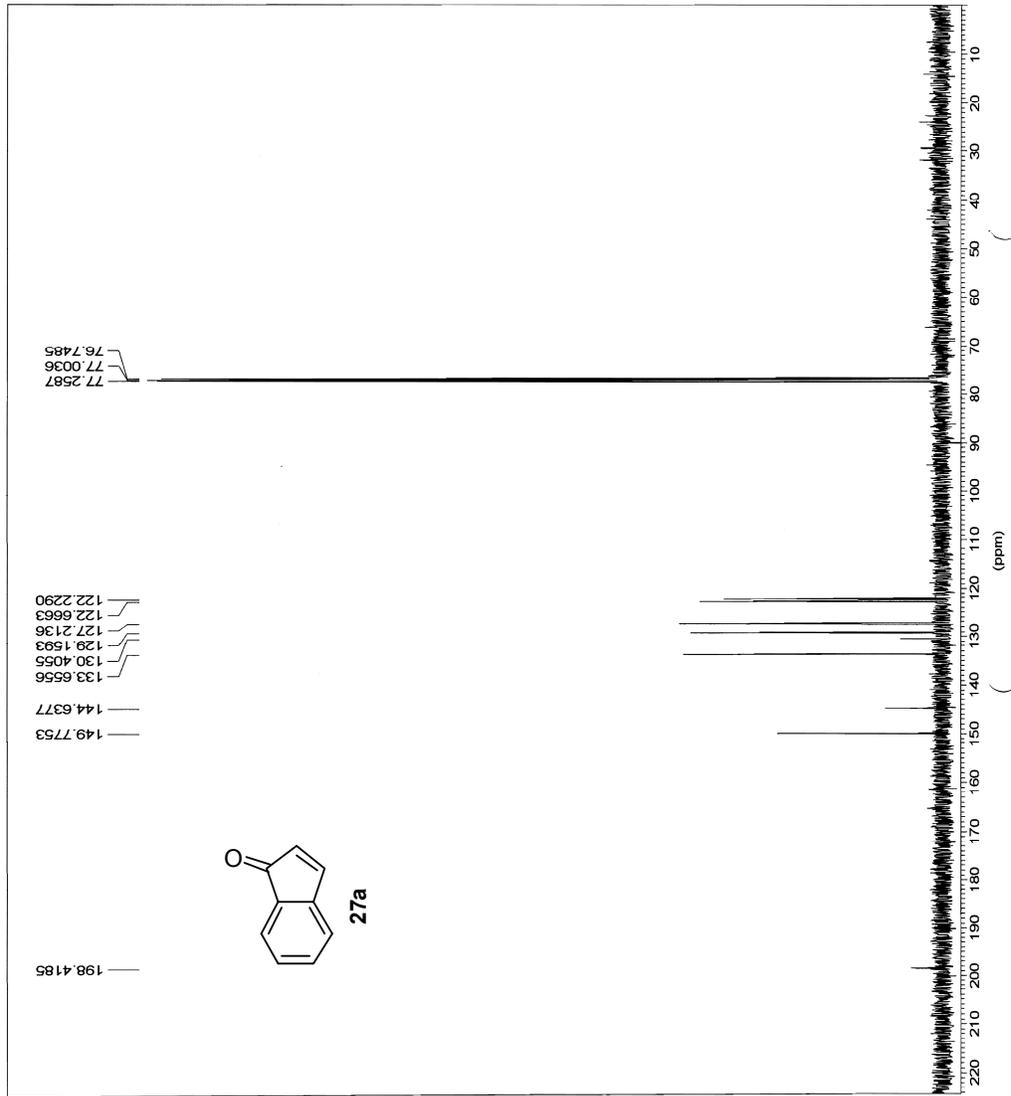


\*\*\* Current Data Parameters \*\*\*

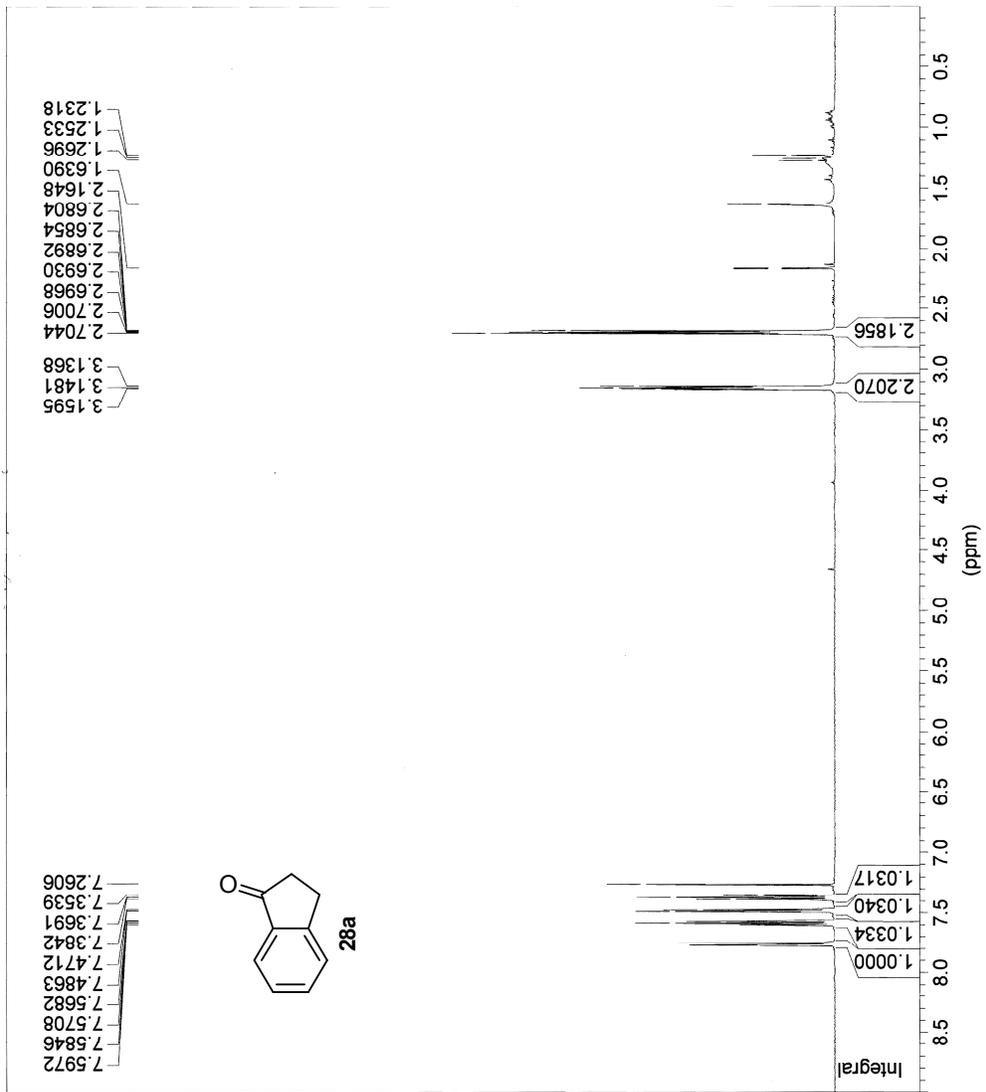
NAME : zfy0210  
EXPNO : 1  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 500.1300000 MHz  
LOCNUC : 2H  
NS : 8  
O1 : 3088.51 Hz  
PULPROG : zg30  
SFO1 : 500.1330885 MHz  
SOLVENT : CDCl3  
SW : 20.6557 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 0.30 Hz  
PHC0 : 282.248 degree  
PHC1 : 3.456 degree

13C AMX500

\*\*\* Current Data Parameters \*\*\*  
NAME : zfy0210  
EXPNO : 2  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
BF1 : 125.7577690 MHz  
LOCHUC : 2H  
NS : 213  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709936 MHz  
SOLVENT : CDC13  
SW : 238.7675 ppm  
\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
PHC0 : 72.669 degree  
PHC1 : 59.425 degree

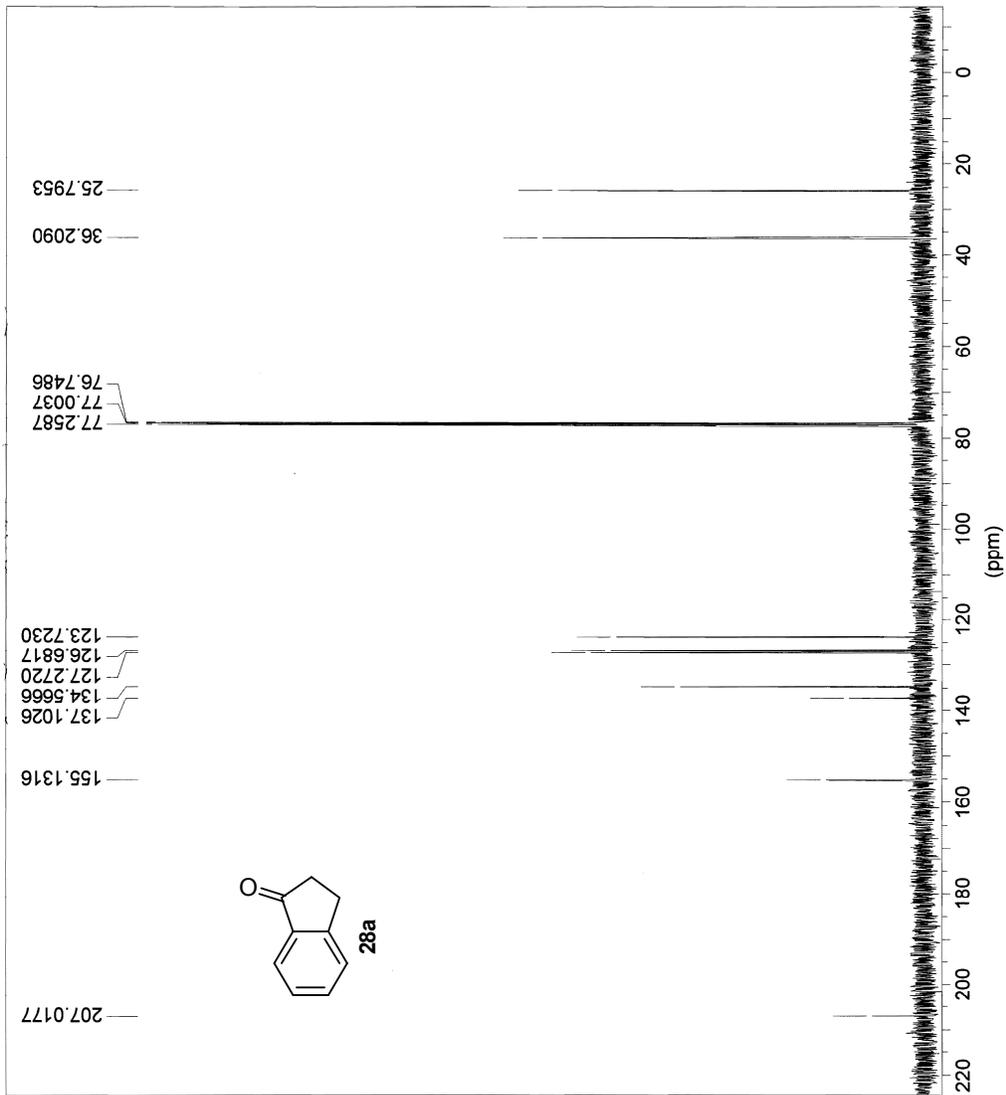


1H AMX500  
fr-2-155-1



\*\*\* Current Data Parameters \*\*\*  
NAME : c0129  
EXPNO : 4  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
INSTRUM : av500  
LOCNUC : 2H  
NS : 8  
NUCLEUS : off  
O1 : 3088.51 Hz  
PULPROG : zg30  
SFO1 : 500.1330885 MHz  
SOLVENT : CDCl3  
SW : 20.6557 ppm  
TD : 32768  
TE : 300.0 K  
\*\*\* Processing Parameters \*\*\*  
LB : 0.30 Hz  
\*\*\* 1D NMR Plot Parameters \*\*\*  
NUCLEUS : off

13C AMX500  
fy-2-155-1

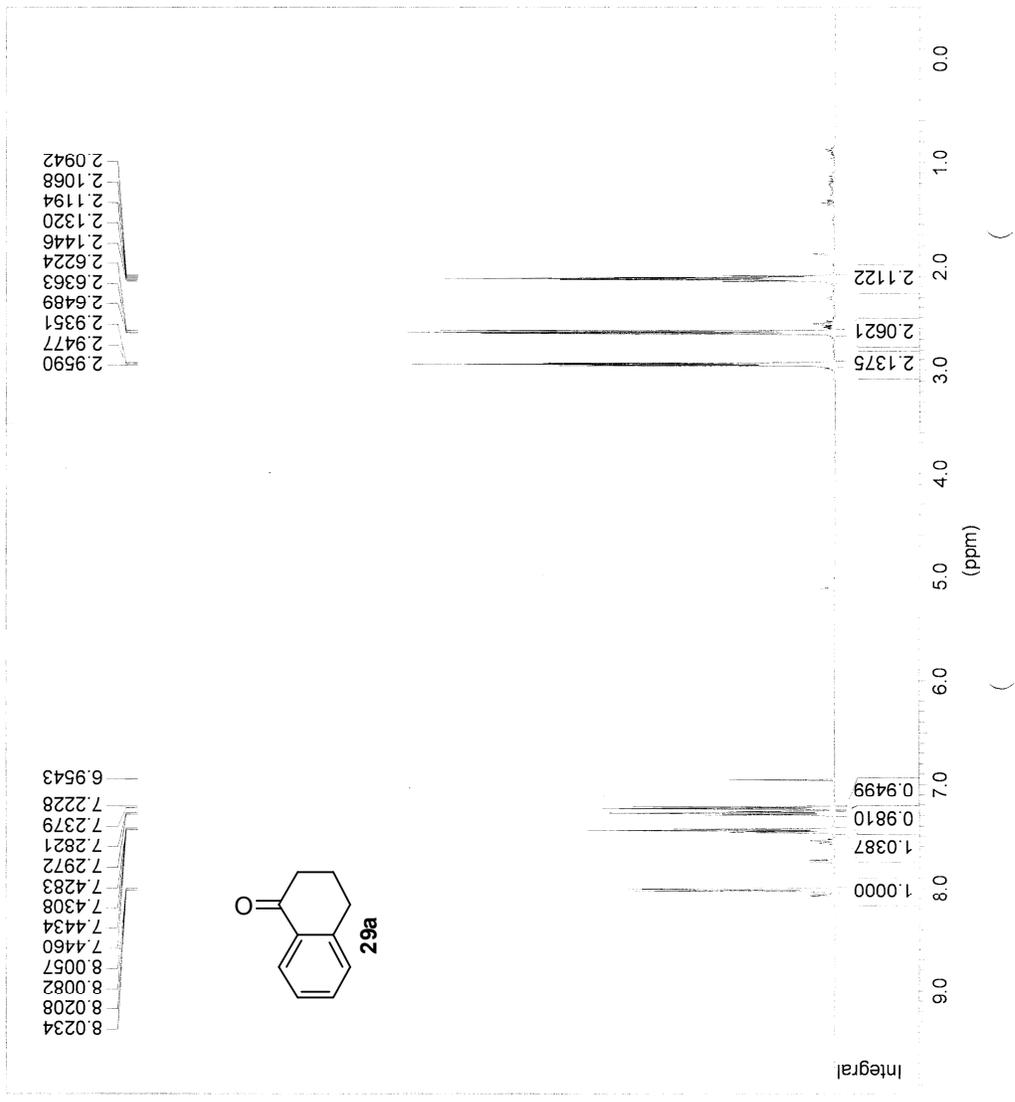


\*\*\* Current Data Parameters \*\*\*  
NAME : cf0129  
EXPNO : 5  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
INSTRUM : av600  
LOGNUC : 2H  
NS : 148  
NUCLEUS : off  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFC1 : 125.7709836 MHz  
SOLVENT : CDCl3  
SW : 238.7675 ppm  
TD : 65536  
TE : 300.0 K  
\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
\*\*\* 1D NMR Plot Parameters \*\*\*  
NUCLEUS : off

1H AMX500

FY-2-143-1

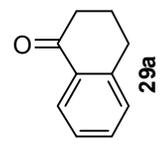
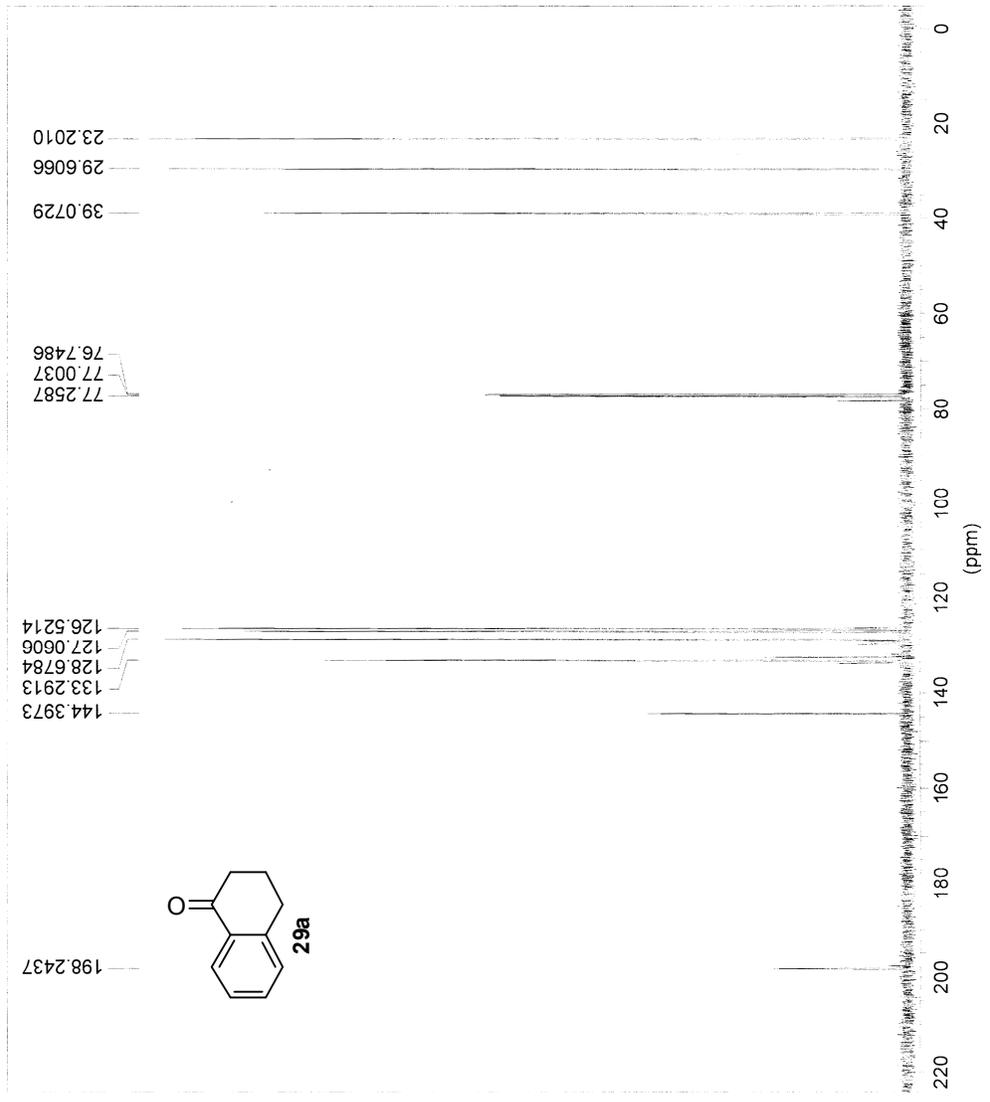
• **Puber** •



\*\*\* Current Data Parameters \*\*\*  
NAME : zfy0128  
EXPNO : 1  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
INSTRUM : av500  
LOCNUC : 2H  
NS : 8  
NUCLEUS : off  
O1 : 3088.51 Hz  
PULPROG : zg30  
SFO1 : 500.1330885 MHz  
SOLVENT : CDCl3  
SW : 20.6557 ppm  
TD : 32768  
\*\*\* Processing Parameters \*\*\*  
LB : 0.30 Hz  
\*\*\* 1D NMR Plot Parameters \*\*\*  
NUCLEUS : off



\*\*\* Current Data Parameters \*\*\*  
NAME : zfy0128  
EXPNO : 2  
PROCNO : 1  
\*\*\* Acquisition Parameters \*\*\*  
INSTRUM : av600  
LOCNUC : 2H  
NS : 91  
NUCLEUS : off  
O1 : 13204.57 Hz  
PULPROG : zgpg30  
SFO1 : 125.7709836 MHz  
SOLVENT : CDCl3  
SW : 238.7675 ppm  
TD : 65536  
\*\*\* Processing Parameters \*\*\*  
LB : 1.00 Hz  
\*\*\* 1D NMR Plot Parameters \*\*\*  
NUCLEUS : off



13C AMX500

FY-2-143-1