

Experimental:

Commercial reagents were used as received unless stated otherwise. The catalyst **1** was prepared according to published procedures.^{1,2} ¹H NMR (300 or 500 MHz) and ¹³C NMR (75 or 125 MHz) spectra were recorded in CDCl₃ on JEOL (Eclipse-500 and ECX-300 respectively) instruments. High resolution electrospray mass spectra were obtained at the University of Florida using an APEX II instrument (Bruker Daltonics, Billerica, MA).

Ethyl 2-bromo-4-pentenoate (15a)³

EDA (114 mg, 2.00 mmol) in allyl bromide 1.5 mL was added by automatic syringe over 1 h to a stirred solution of the catalyst (40 mg, 0.050 mmol) in the allyl bromide (1.5 mL) under nitrogen in a flask shielded from light. The resulting solution was stirred 24 h at ambient temperature under nitrogen. The solution was concentrated and the residue was purified by flash chromatography (SiO₂, 98:3 pentane/ether), yielding the product as a colorless oil (147 mg, 75%).

¹H NMR (500 MHz): δ = 1.29 (t, J = 7.1 Hz, 3H), 2.76-2.69 (m, 1H), 2.89-2.83 (m, 1H), 4.22 (t, J = 7.4 Hz, 1H), 4.24 (q, J = 7.1 Hz, 2H), 5.20-5.13 (m, 2H), 5.80-5.71 (m, 1H)

***t*-Butyl 2-bromo-4-pentenoate (15b)**³

tert-Butyl diazoacetate (200 mg, 1.41 mmol) in the allyl bromide (1.5 mL) was added by automatic syringe over 1 h to a stirred solution of the catalyst (30 mg, 0.038 mmol) in the allyl bromide (1.5 mL) under nitrogen in a flask shielded from light. The resulting solution was stirred 24 h at ambient temperature under nitrogen. The solution was concentrated and the residue was purified by flash chromatography (SiO₂, 98:2 pentane/ether), yielding the product (215 mg, 65% yield) as a colorless oil.

IR (neat) cm⁻¹ = 2980, 1735, 1641, 1456.

¹H NMR (500 MHz): δ = 1.46 (s, 9H), 2.64-2.69 (m, 1H), 2.78-2.84 (m, 1H), 4.12 (t, J = 7.0 Hz, 1H), 5.13-5.18 (m, 2H), 5.70-5.79 (m, 1H).

¹³C NMR (125 MHz): δ = 28.2, 39.6, 46.5, 82.9, 119.3, 133.7, 168.8.

Ethyl 2-chloro-3-methyl-4-pentenoate (16a)⁴

Ethyl diazoacetate (218 mg, 2.00 mmol) in the crotyl chloride (1.5 mL) was added by automatic syringe over 1 h to a stirred solution of the catalyst (40 mg, 0.050 mmol) in the crotyl chloride (1.5 mL) under nitrogen in a flask shielded from light. The resulting solution was stirred 24 h at ambient temperature under nitrogen. The solution was concentrated and the residue was purified by flash chromatography (SiO₂, 98:6 pentane/ether), yielding the product (290 mg, 86% yield) as a colorless oil.

IR (neat) cm⁻¹ = 2982, 1748, 1642, 1457.

¹H NMR (500 MHz): δ = 1.15 (d, J = 6.7 Hz, 1.5H), 1.16 (d, J = 6.7 Hz, 1.5H), 1.28 (t, J = 7.2 Hz, 1.5H), 1.30 (t, J = 7.2 Hz, 1.5H), 2.82-2.90 (m, 1H), 4.26-4.15 (m, 3H), 5.17-5.11 (m, 2H), 5.87-5.72 (m, 1H), *syn/anti* ratio = 1/1.

1. Dias, H. V. R.; Jin, W. *Inorg. Chem.* **1996**, *35*, 267-268.

2. Dias, H. V. R.; Wang, Z.; Jin, W. *Inorg. Chem.* **1997**, *36*, 6205-6215.

3. Stotter, P.L.; Hill, K.A. *Tetrahedron Lett.* **1972**, *40*, 4067-4074.

4. Doyle, M.P.; Tamblyn, W.H.; Bagheri, V. *J. Org. Chem.* **1981**, *46*, 5094-5102

^{13}C NMR (125 MHz): δ = 14.13, 14.17, 16.0, 17.3, 42.00, 42.04, 61.9, 62.0, 62.1, 62.3, 116.82, 116.89, 137.7, 138.3, 168.8, 168.9.

***t*-Butyl 2-chloro-3-methyl-4-pentenoate (16b)**

tert-Butyl diazoacetate (166 mg, 1.17 mmol) in the crotyl chloride (1.5 mL) was added by automatic syringe over 1 h to a stirred solution of the catalyst (30 mg, 0.038 mmol) in the crotyl chloride (1.5 mL) under nitrogen in a flask shielded from light. The resulting solution was stirred 24 h at ambient temperature under nitrogen. The solution was concentrated and the residue was purified by flash chromatography (SiO_2 , 98:6 pentane/ether), yielding the product (230 mg, 96% yield) as a colorless oil.

IR (neat, cm^{-1}): = 2980, 1744, 1643, 1457.

^1H NMR (500 MHz): δ = 1.12, 1.4 (d, J = 2.0 Hz, 3H), 1.46, 1.45 (s, 9H), 2.79 (m, 1H), 4.04, 4.07 (d, J = 6.5 Hz, 1H), 5.08-5.11 (m, 2H), 5.72-5.77 (m, 1H).

^{13}C NMR (125 MHz): δ = 16.0, 17.4, 27.92, 27.96, 42.00, 42.04, 63.1, 63.3, 82.6, 82.7, 116.6, 116.7, 137.9, 138.5, 167.8, 167.9.

Ethyl 2-chloro-4-methyl-4-pentenoate (17a)

Ethyl diazoacetate (218 mg, 2.00 mmol) in the 3-chloro-2-methylpropene (1.5 mL) was added by automatic syringe over 1 h to a stirred solution of the catalyst (40 mg, 0.050 mmol) in the 3-chloro-2-methylpropene (1.5 mL) under nitrogen in a flask shielded from light. The resulting solution was stirred 24 h at ambient temperature under nitrogen. The solution was concentrated and the residue was purified by flash chromatography (SiO_2 , 98:3 pentane/Ether), yielding the product (200 mg, 59% yield) as a colorless oil.

IR (neat, cm^{-1}): = 2981, 2358, 1727, 1446, 1383.

^1H NMR (300 MHz): δ = 1.29 (m, 3H), 1.75 (s, 3H), 2.57 (dd, J = 3.0, 16.5 Hz, 1H), 2.75 (dd, J = 3.0, 16.5 Hz, 1H), 4.22 (m, 2H), 4.38 (m, 1H), 4.80 (s, 1H), 4.88 (s, 1H).

^{13}C NMR (75 MHz): δ = 14.1, 22.1, 42.9, 55.0, 62.1, 114.4, 139.9, 169.5.

HRMS (ESI): calcd for $\text{C}_8\text{H}_{14}\text{ClO}_2$ ($\text{M}+\text{H}$) $^+$ 177.0678, found 177.0674.

***t*-Butyl 2-chloro-4-methyl-4-pentenoate (17b)**

tert-Butyl diazoacetate (165 mg, 1.16 mmol) in the 3-chloro-2-methylpropene (1.5 mL) was added by automatic syringe over 1 h to a stirred solution of the catalyst (30 mg, 0.038 mmol) in 3-chloro-2-methylpropene (1.5 mL) under nitrogen in a flask shielded from light. The resulting solution was stirred 24 h at ambient temperature under nitrogen. The solution was concentrated and the residue was purified by flash chromatography (SiO_2 , 98:3 pentane/ether), yielding the product (200 mg, 84% yield) as a colorless oil.

IR (neat, cm^{-1}) = 2982, 1744, 1652, 1455, 1395.

^1H NMR (500 MHz): δ = 1.45 (s, 9H), 1.74 (s, 3H), 2.52 (dd, J = 7.3, 14.7 Hz, 1H), 2.71 (dd, J = 7.3 and 14.7 Hz, 1H), 4.25 (t, J = 7.3 Hz, 1H), 4.78 (s, 1H), 4.85 (s, 1H).

^{13}C NMR (125 MHz): δ = 22.1, 27.8, 43.0, 56.0, 82.6, 114.2, 140.2, 168.5.

HRMS (ESI): calcd for $\text{C}_{10}\text{H}_{18}\text{ClO}_2$ ($\text{M}+\text{H}$) $^+$ 205.0990, found 205.0989.

Ethyl 2-bromo-4-methyl-4-pentenoate (18a)

Ethyl diazoacetate (218 mg, 2.00 mmol) in the 3-bromo-2-methylpropene (1.5 mL) was added by automatic syringe over 1 h to a stirred solution of the catalyst (40 mg, 0.050 mmol) in the 3-bromo-2-methylpropene (1.5 mL) under nitrogen in a flask shielded from light. The resulting solution was stirred 24 h at ambient temperature under nitrogen. The

solution was concentrated and the residue was purified by flash chromatography (SiO₂, 97:3 pentane/ether), yielding the product (240 mg, 57% yield) as a colorless oil.

IR (neat, cm⁻¹): = 2925, 2854, 1741, 1457.

¹H NMR (300 MHz): δ = 1.29 (t, *J* = 7.2 Hz, 3H), 1.74 (s, 3H), 2.65 (dd, *J* = 7.2, 15.0 Hz, 1H), 2.85 (dd, *J* = 8.1, 14.7 Hz, 1H), 4.22 (q, *J* = 7.2 Hz, 2H), 4.34 (dd, *J* = 7.2, 8.4 Hz, 1H), 4.78 (s, 1H), 4.88 (s, 1H).

¹³C NMR (75 MHz) δ = 14.0, 22.2, 42.8, 43.6, 62.0, 114.1, 140.8, 169.6.

HRMS (ESI): calcd for C₈H₁₄BrO₂ (M+H)⁺ 221.0172, found 221.0174.

***t*-Butyl 2-bromo-4-methyl-4-pentenoate (18b)**

tert-Butyl diazoacetate (166 mg, 1.17 mmol) in the 3-bromo-2-methylpropene (1.5 mL) was added by automatic syringe over 1 h to a stirred solution of the catalyst (30 mg, 0.038 mmol) in the 3-bromo-2-methylpropene (1.5 mL) under nitrogen in a flask shielded from light. The resulting solution was stirred 24 h at ambient temperature under nitrogen. The solution was concentrated and the residue was purified by flash chromatography (SiO₂, 97:3 pentane/ether), yielding the product (260 mg, 89% yield) as a colorless oil.

IR (neat, cm⁻¹): = 3081, 2980, 1743, 1651, 1455, 1394.

¹H NMR (500 MHz): δ = 1.48 (s, 9H), 1.77 (s, 3H), 2.55 (dd, *J* = 7.5, 14.5 Hz, 1H), 2.74 (dd, *J* = 7.5, 14.5 Hz, 1H), 4.28 (t, *J* = 8.0 Hz, 1H), 4.81 (s, 1H), 4.88 (s, 1H).

¹³C NMR (125 MHz): δ = 22.2, 27.9, 43.1, 56.1, 82.6, 114.3, 140.2, 168.5.

Ethyl 2-bromo-3-bromomethyl-4-pentenoate (19a)

Ethyl diazoacetate (218 mg, 2.00 mmol) and 1,4-dibromo-2-butene (1.00 g), dissolved in dichloromethane (1 mL) was added by automatic syringe over 1 h to a stirred solution of the catalyst (40 mg, 0.050 mmol) and 1,4-dibromo-2-butene (1.00 g) in dichloromethane (1 mL) under nitrogen in a flask shielded from light. The resulting solution was stirred 24 h at ambient temperature under nitrogen. The solution was concentrated and the residue was purified by flash chromatography (SiO₂, 98:3 pentane/ether), yielding the product as a colorless oil (400 mg, 70% yield).

IR (neat, cm⁻¹): = 2983, 2359, 1740, 1370.

¹H NMR (300 MHz): δ = 1.25-1.27 (m, 3H), 2.98-3.01 (m, 1H), 3.48-3.49 (m, 1H), 3.61-3.66, 3.72-3.78 (m, 1H), 4.14-4.25 (m, 2H), 4.30, 4.70 (dd, *J* = 3.0, 16.5 Hz, 1H), 5.26-5.27 (m, 2H), 5.71-5.74 (m, 1H), *syn/anti* ratio = 1/1.

¹³C NMR (75 MHz): δ = 14.0, 14.3, 34.3, 36.0, 47.61, 47.64, 48.4, 50.0, 62.1, 62.5, 120.4, 120.9, 133.7, 134.0, 168.1, 168.4.

HRMS (ESI): calcd for C₈H₁₃Br₂O₂ (M+H)⁺ 300.9261, found 300.9260.

***t*-Butyl 2-bromo-3-bromomethyl-4-pentenoate (19b)**

tert-Butyl diazoacetate (130 mg, 0.915 mmol) and 1,4-dibromo-2-butene (1.00 g), dissolved in dichloromethane (1 mL) was added by automatic syringe over 1 h to a stirred solution of the catalyst (30 mg, 0.038 mmol) and 1,4-dibromo-2-butene (1.00 g) in dichloromethane (1 mL) under nitrogen in a flask shielded from light. The resulting solution was stirred 24 h at ambient temperature under nitrogen. The solution was concentrated and the residue was purified by flash chromatography (SiO₂, 98:3 pentane/ether), yielding the product (260 mg, 87% yield) as a colorless oil.

IR (neat, cm⁻¹): = 2979, 1734, 1476, 1426.

^1H NMR (500 MHz) δ = 1.44, 1.46 (s, 9H), 2.92-3.00 (m, 1H), 3.5 (m, 1H), 3.64 (dd, J = 3.2, 10.0 Hz), 3.75 (dd, J = 5.3, 10.0 Hz, 1H), 4.2 (d, J = 9.9 Hz), 4.61 (d, J = 5.2 Hz, 1H), 5.25-5.29 (m, 2H), 5.71-5.75 (m, 1H), *syn/anti* ratio = 1/1.

^{13}C NMR (75 MHz): δ = 27.8, 27.9, 34.5, 36.3, 47.6, 48.6, 49.2, 51.4, 82.9, 83.3, 120.2, 120.8, 133.8, 134.2, 167.0, 167.4.

HRMS (ESI): calcd for $\text{C}_{10}\text{H}_{16}\text{Br}_2\text{O}_2\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 350.9394, found 350.9396.

***tert*-Butyl 2-bromo-3,4-pentadienoate (23b)**

tert-Butyl diazoacetate (166 mg, 1.17 mmol) in distilled propargyl bromide (1.5 mL) was added by automatic syringe over 1 h to a stirred solution of the catalyst (30 mg, 0.038 mmol) in the propargyl bromide (1.5 mL) under nitrogen in a flask shielded from light. The resulting solution was stirred 24 h at ambient temperature under nitrogen. The solution was concentrated and the residue was purified by flash chromatography (SiO_2 , 98:2 pentane/ether), yielding the product as a colorless oil (180 mg, 66% yield).

IR (neat, cm^{-1}): = 2979, 1951, 1734, 1369.

^1H NMR (500 MHz): δ = 1.48 (s, 9H), 4.60 (dt, J = 6.5, 10.0 Hz, 1H), 4.73 (d, J = 9.5 Hz, 1H), 4.96 (d, J = 6.5 Hz, 1H), 4.97 (d, J = 6.5 Hz, 1H)

^{13}C NMR (125 MHz): δ = 27.8, 44.6, 78.3, 83.0, 89.6, 167.0, 209.2.

HRMS (ESI): calcd for $\text{C}_9\text{H}_{13}\text{BrO}_2\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 254.991, found 254.991.

Ethyl 2-bromo-3-ethyl-3,4-pentadienoate (24a)

EDA (218 mg, 2.00 mmol) in the 1-bromo-2-pentyne (1.5 mL) was added by automatic syringe over 1 h to a stirred solution of the catalyst (40 mg, 0.050 mmol) in the 1-bromo-2-pentyne (1.5 mL) under nitrogen in a flask shielded from light. The resulting solution was stirred 24 h at ambient temperature under nitrogen. The solution was concentrated and the residue was purified by flash chromatography (SiO_2 , 94:6 pentane/ether), yielding the product (316 mg, 71% yield) as a colorless oil.

IR (neat, cm^{-1}): = 2974, 2935, 1951, 1744, 1456.

^1H NMR (500 MHz): δ = 1.16 (m, 3H), 0.92 (m, 3H), 2.00-2.14 (m, 2H), 4.06-4.16 (m, 2H), 4.74-4.86 (m, 3H)

^{13}C NMR (75 MHz): δ = 11.9, 14.1, 21.8, 48.9, 62.3, 79.4, 103.5, 168.2, 206.8.

HRMS (ESI): calcd for $\text{C}_9\text{H}_{14}\text{BrO}_2$ ($\text{M}+\text{H}$) $^+$ 233.0172, found 233.0174.

***tert*-Butyl 2-bromo-3-ethyl-3,4-pentadienoate (24a)**

tert-Butyl diazoacetate (176 mg, 1.24 mmol) in the 1-bromo-2-pentyne 1.5 mL was added by automatic syringe over 1 h to a stirred solution of the catalyst (30 mg, 0.038 mmol) in the 1-bromo-2-pentyne (1.5 mL) under nitrogen in a flask shielded from light. The resulting solution was stirred 24 h at ambient temperature under nitrogen. The solution was concentrated and the residue was purified by flash chromatography (SiO_2 , 94:6 pentane/ether), yielding the product as a colorless oil (240 mg, 74% yield).

IR (neat, cm^{-1}): = 2975, 2935, 1951, 1742, 1457, 1369.

^1H NMR (300 MHz): δ = 1.03 (t, J = 7.5 Hz, 3H), 1.46 (s, 9H), 2.17 (m, 2H), 4.81 (s, 1H), 4.89 (m, 2H)

^{13}C NMR (75 MHz): δ = 11.9, 21.9, 27.9, 50.2, 79.3, 82.9, 103.4, 166.4, 206.6.

HRMS (ESI): calcd for $\text{C}_{22}\text{H}_{34}\text{Br}_2\text{O}_4\text{Na}$ ($2\text{M}+\text{Na}$) $^+$ 545.0706, found 545.0713.

***tert*-Butyl 2-chloro-4-hexenoate (26)**

tert-Butyl diazoacetate (166 mg, 1.17 mmol) in 3-chloro-1-butene (1.5 mL) was added by automatic syringe over 1 h to a stirred solution of the catalyst (30 mg, 0.038 mmol) in the 3-chloro-1-butene (1.5 mL) under nitrogen in a flask shielded from light. The resulting solution was stirred 24 h at ambient temperature under nitrogen. The solution was concentrated and the residue was purified by flash chromatography (SiO₂, 19:1 hexane/dichloromethane), yielding the product as a colorless oil (77% yield, 184 mg).

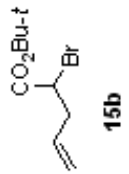
IR (neat, cm⁻¹): = 2977, 1740, 1464, 1426.

¹H NMR (300 MHz): δ = 5.59 (m, 1H), 5.39 (m, 1H), 4.13 (t, J = 6.9 Hz, 1H), 2.50-2.70 (m, 2H), 1.682 (m, 3H), 1.47 (s, 9H).

¹³C NMR (75 MHz): δ = 168.4, 130.0, 124.8, 82.5, 57.9, 38.4, 27.9, 18.0.

HRMS (ESI): calcd for C₁₀H₁₈ClO₂ (M+H)⁺ 205.0990, found 205.0983.

pashu_ii_150-8.jdf



9

(Millions)

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5.7393

5.1796
5.1530
5.1457

4.1333
4.1196
4.1040

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2.6830

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0.961

2.024

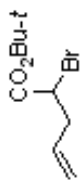
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0.998

1.016

X : parts per Million : 1H

pashu_ii_150-3.jdf



15b

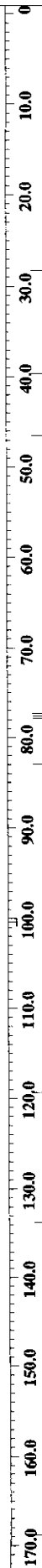
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2.0

1.0

0

(Millions)

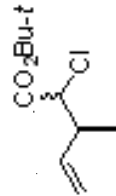


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46.5436
39.6531
28.2796

X : parts per Million : 13C



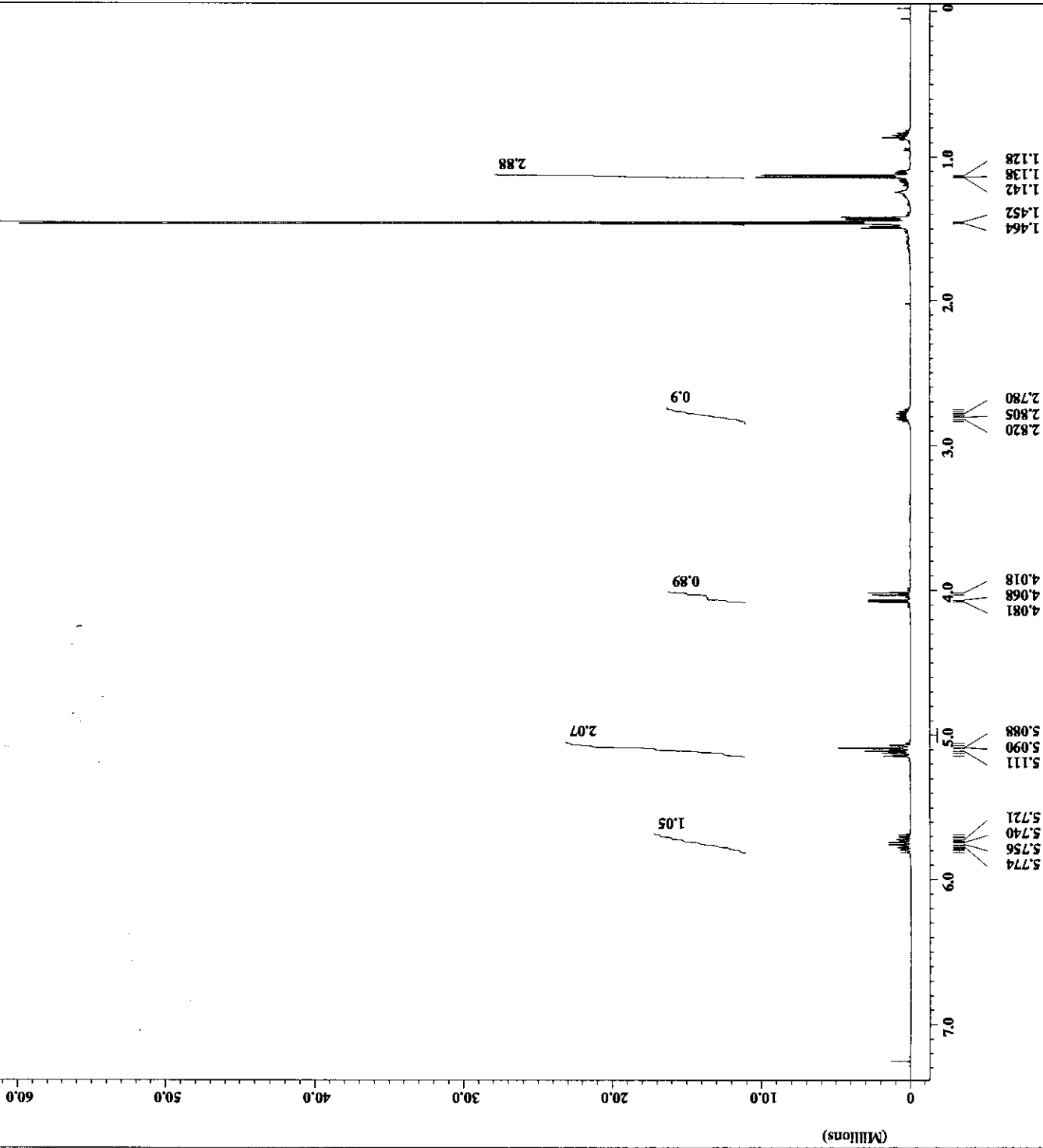
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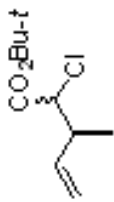
16b

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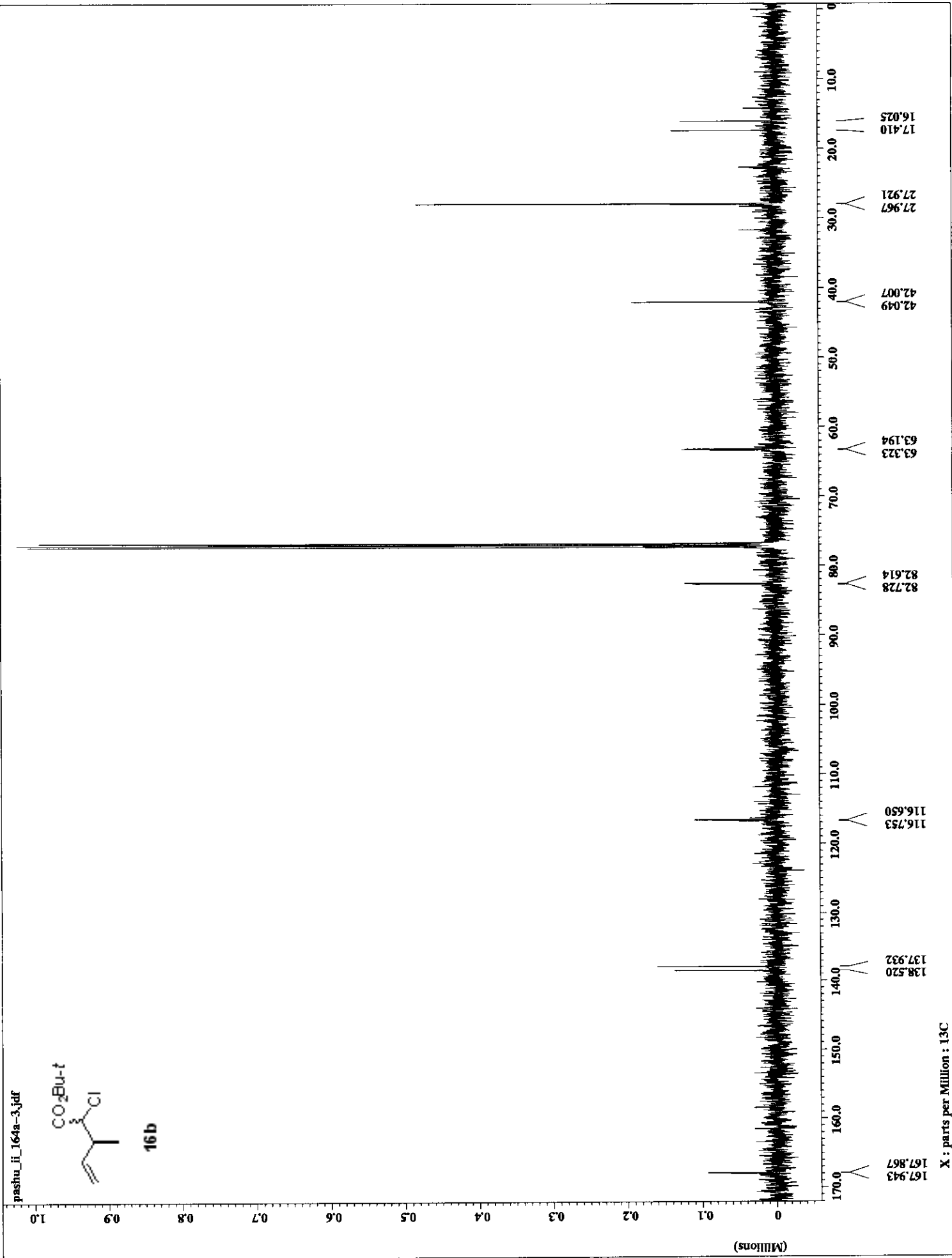
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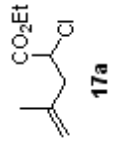
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16b



pashu_ii_113-3.jdf



abundance

3.73

2.79

1

0.99

1.83

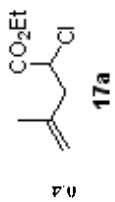
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2.09

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X : parts per Million : 1H



0.4

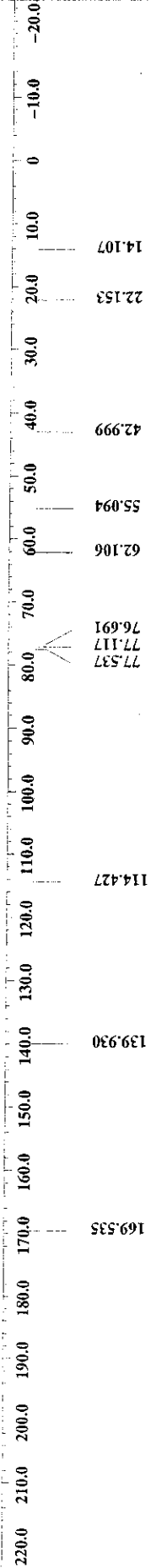
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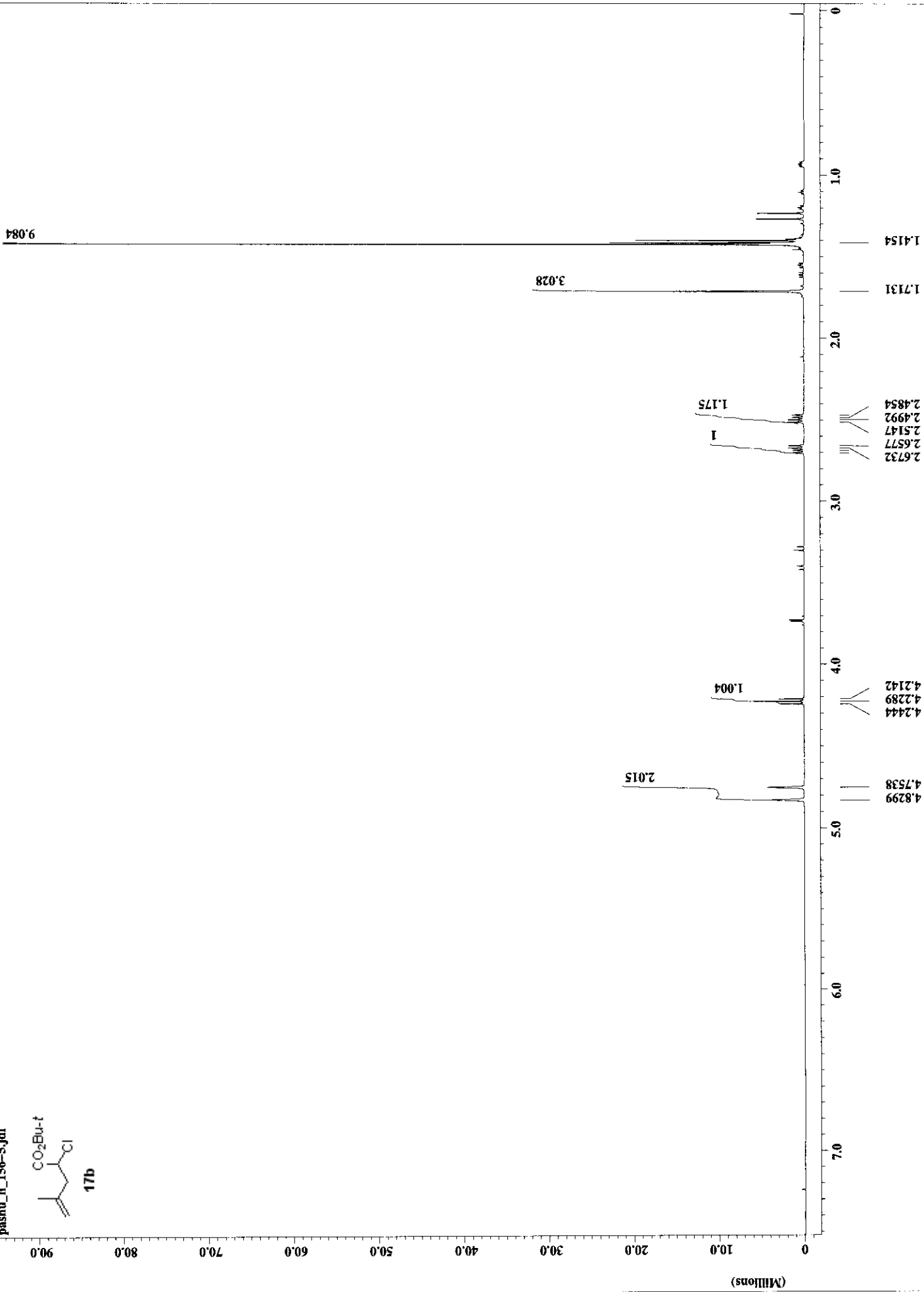
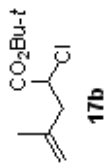
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abundance

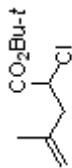


pashu_ii_156-5.jdf



X : parts per Million : 1H

pashu_i1_156_copy-3.jdf



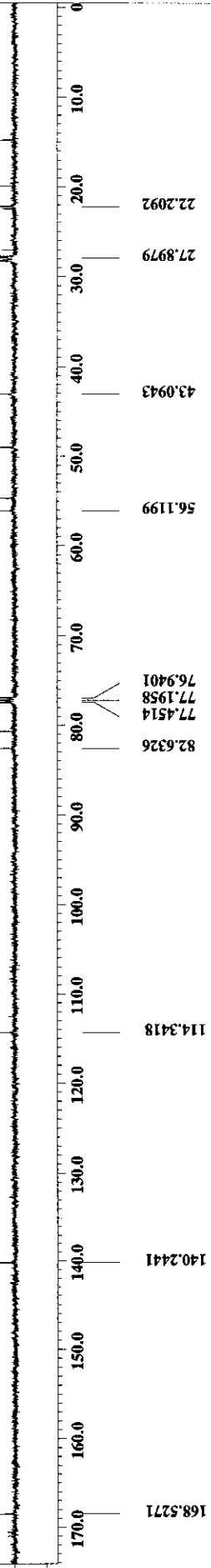
17b

30.0

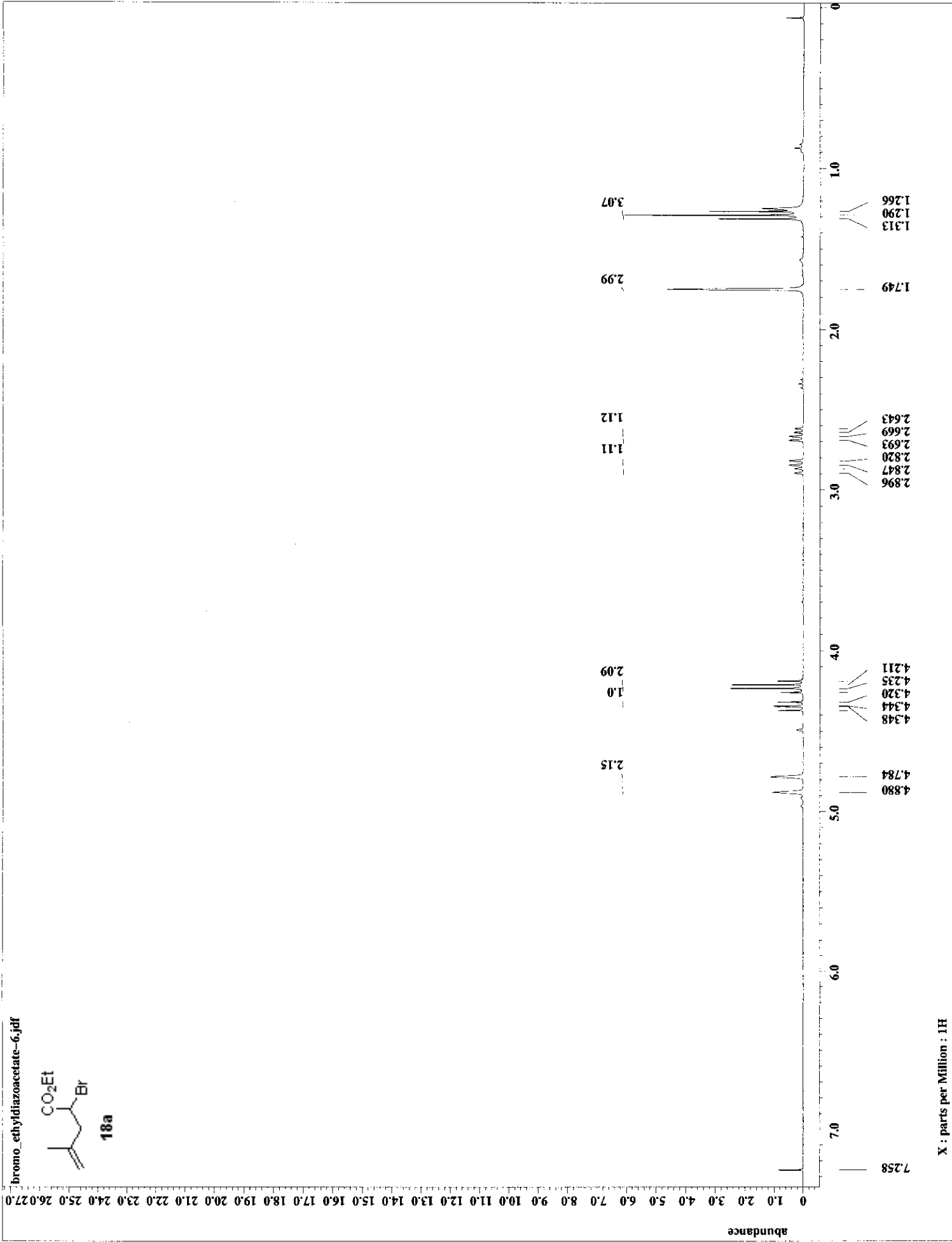
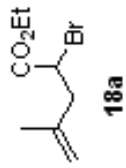
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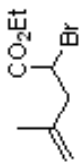
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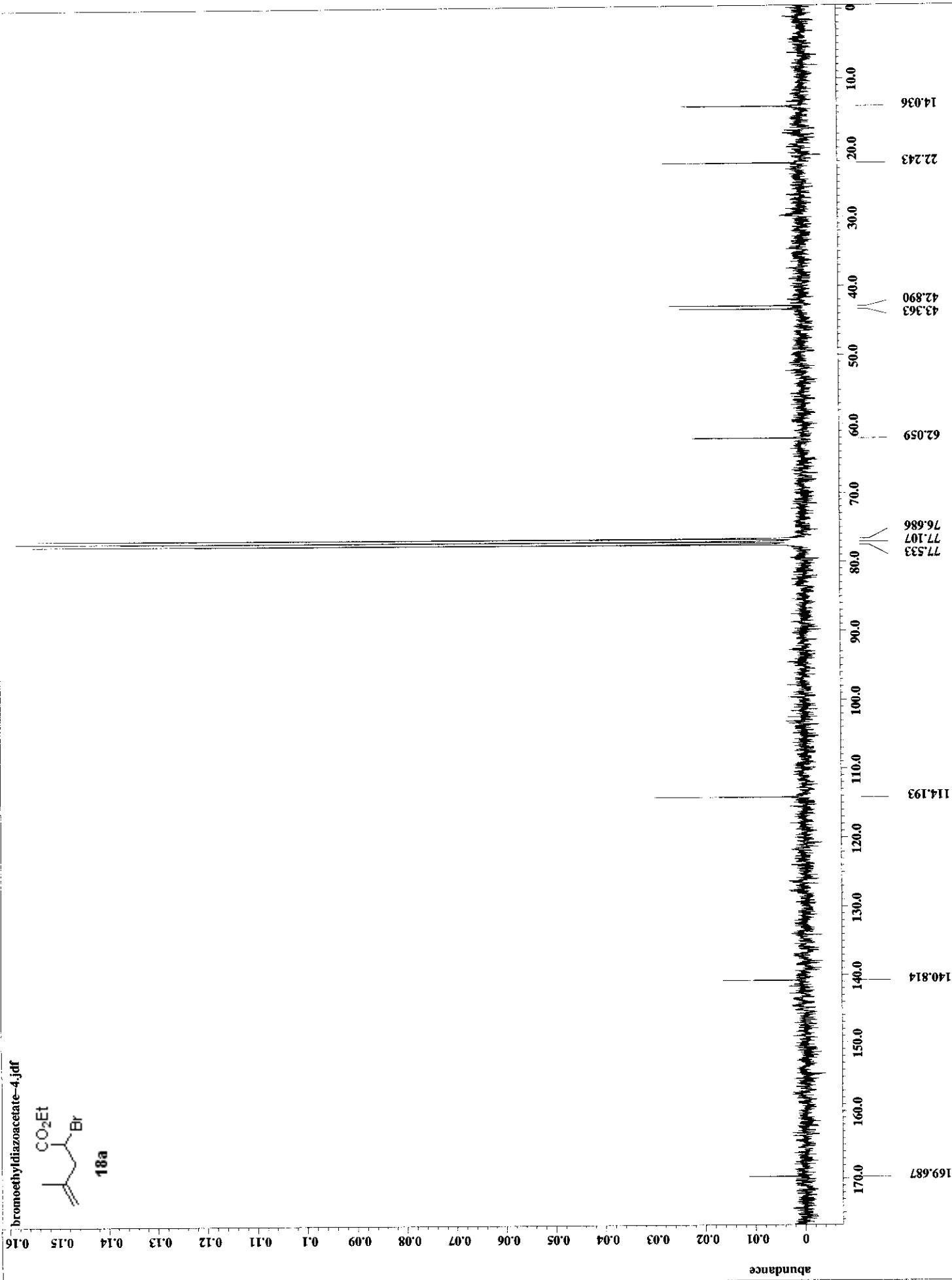
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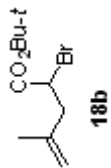


18a

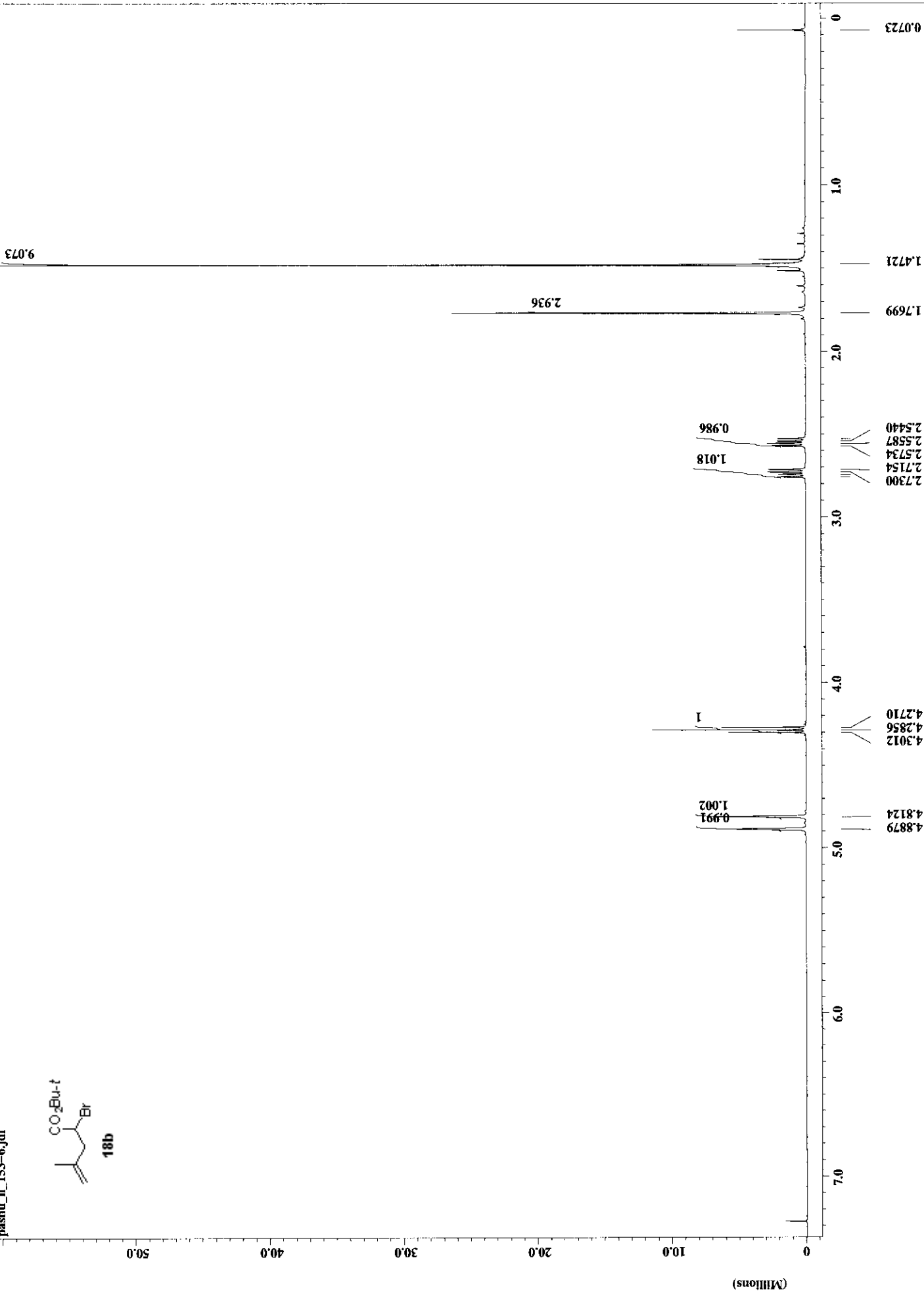


X : parts per Million : 13C

pashu_ii_153-9.jdf

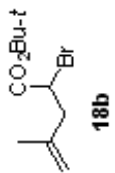


9.073



X : parts per Million : 1H

pashu_ii_153-4.jdf

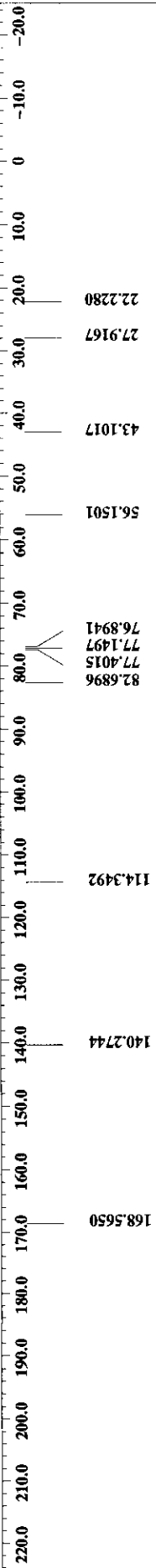


3.0

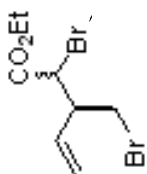
2.0

1.0

(Millions)

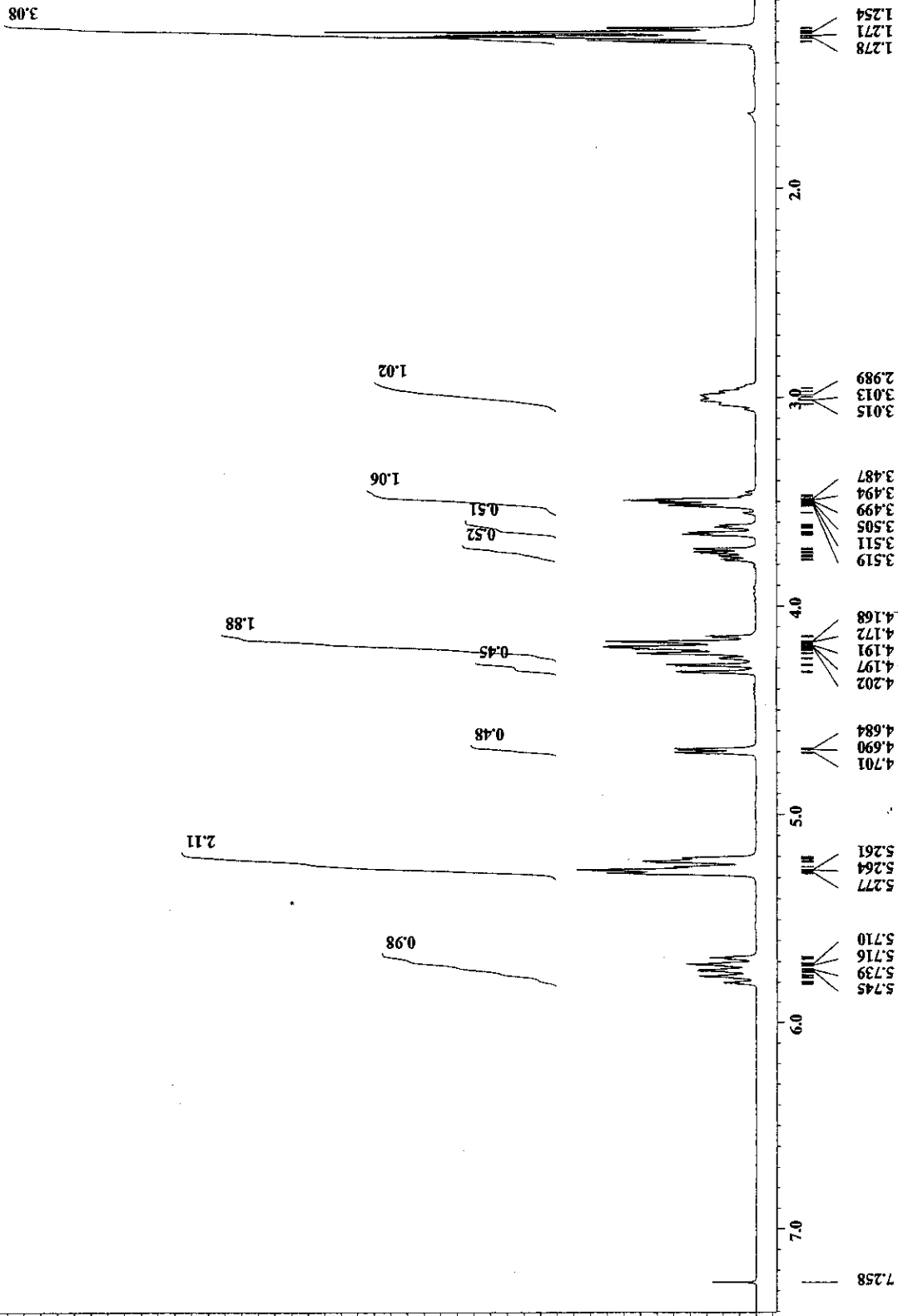


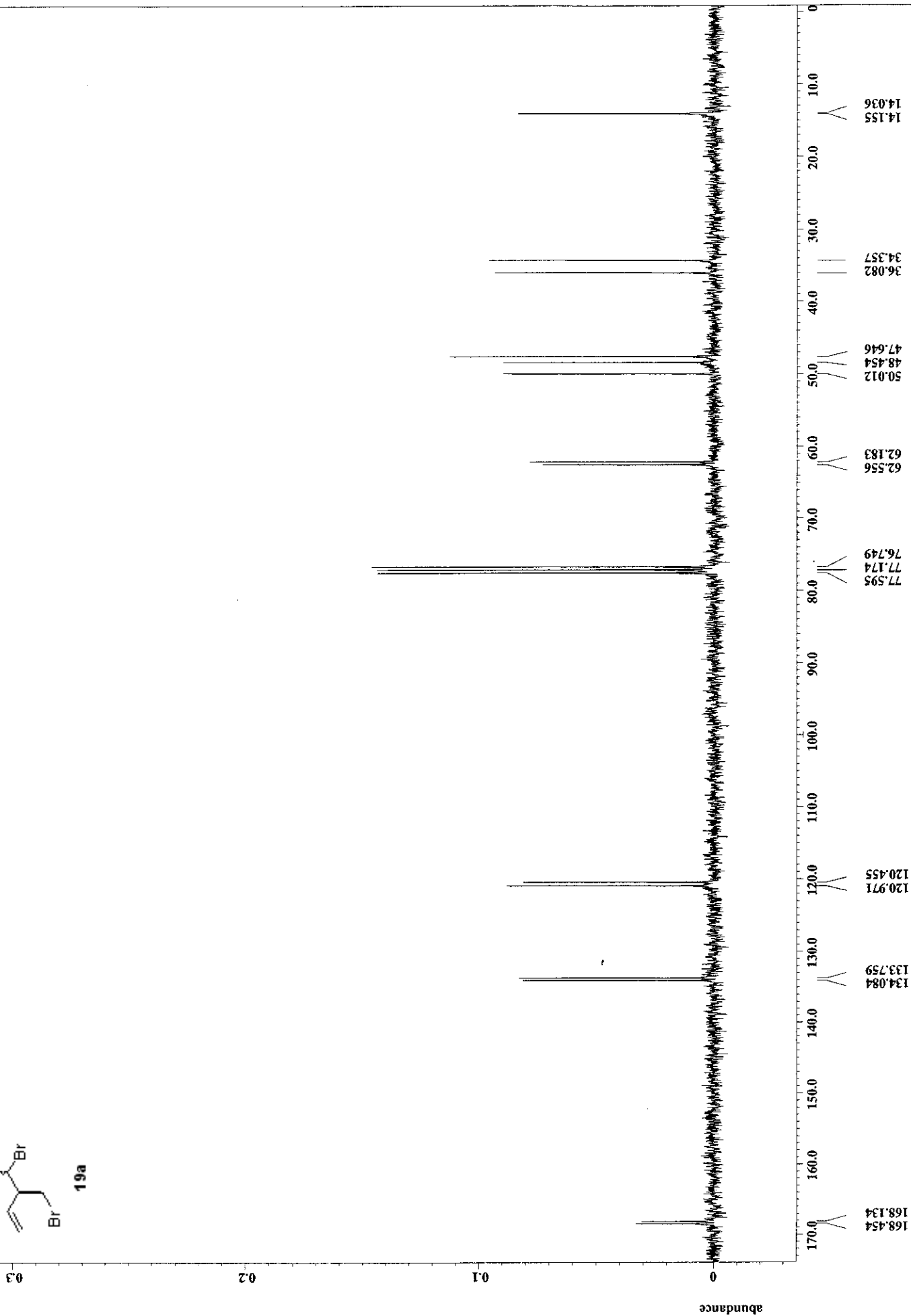
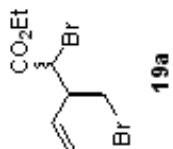
X : parts per Million : 13C

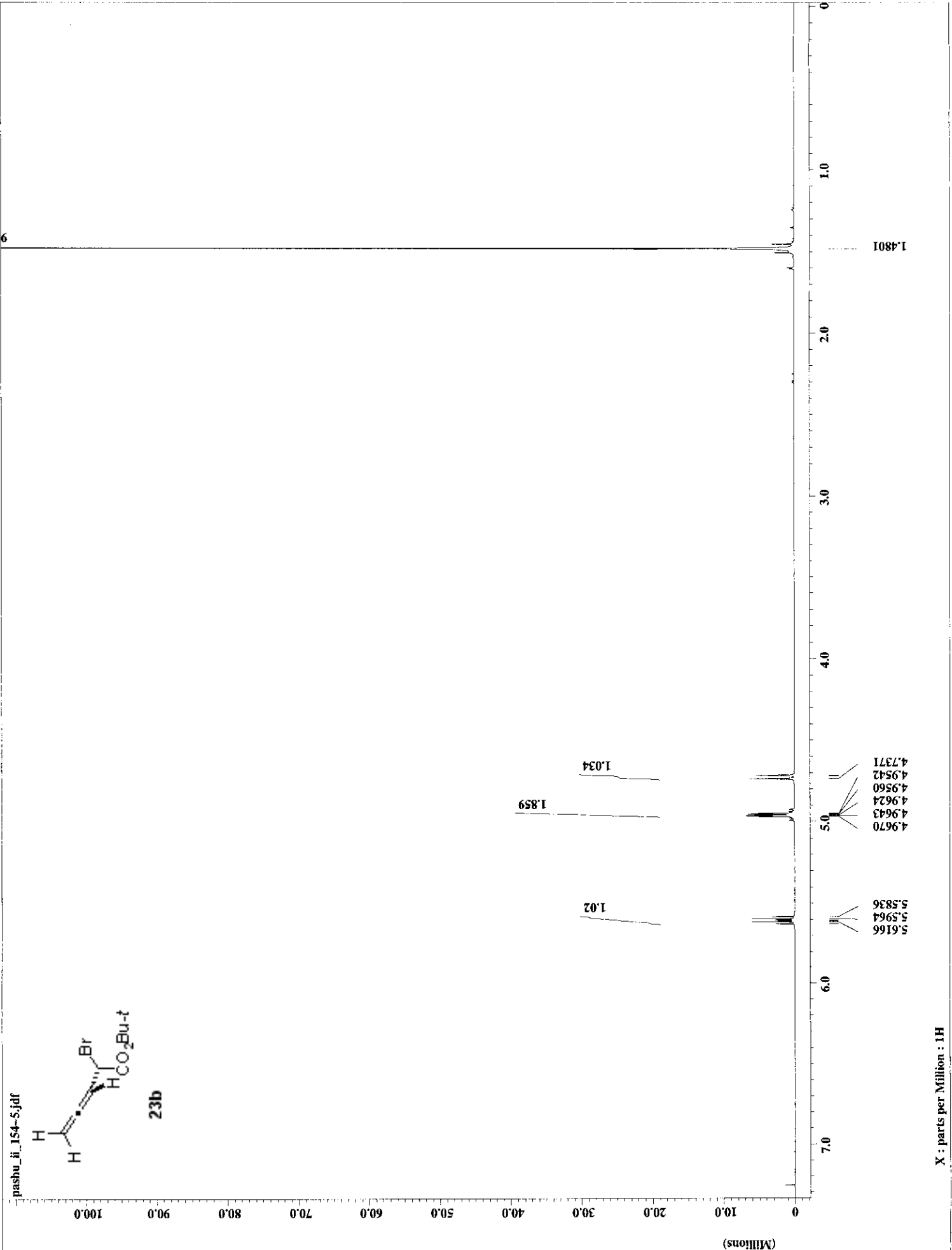
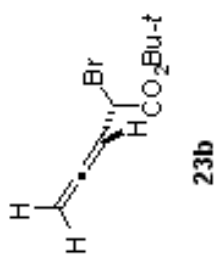


19a

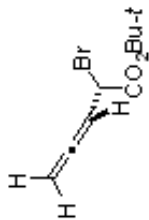
abundance





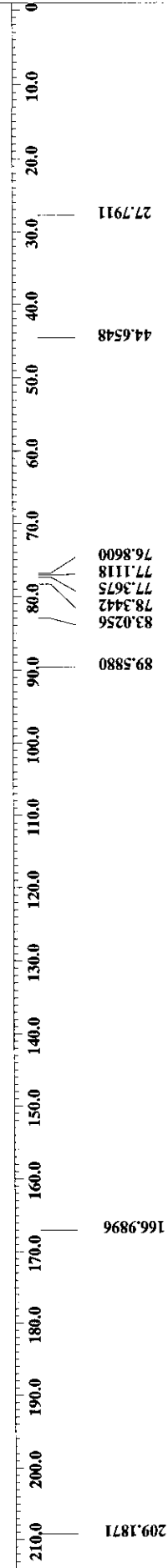


pashu_ii_154-3.jdt

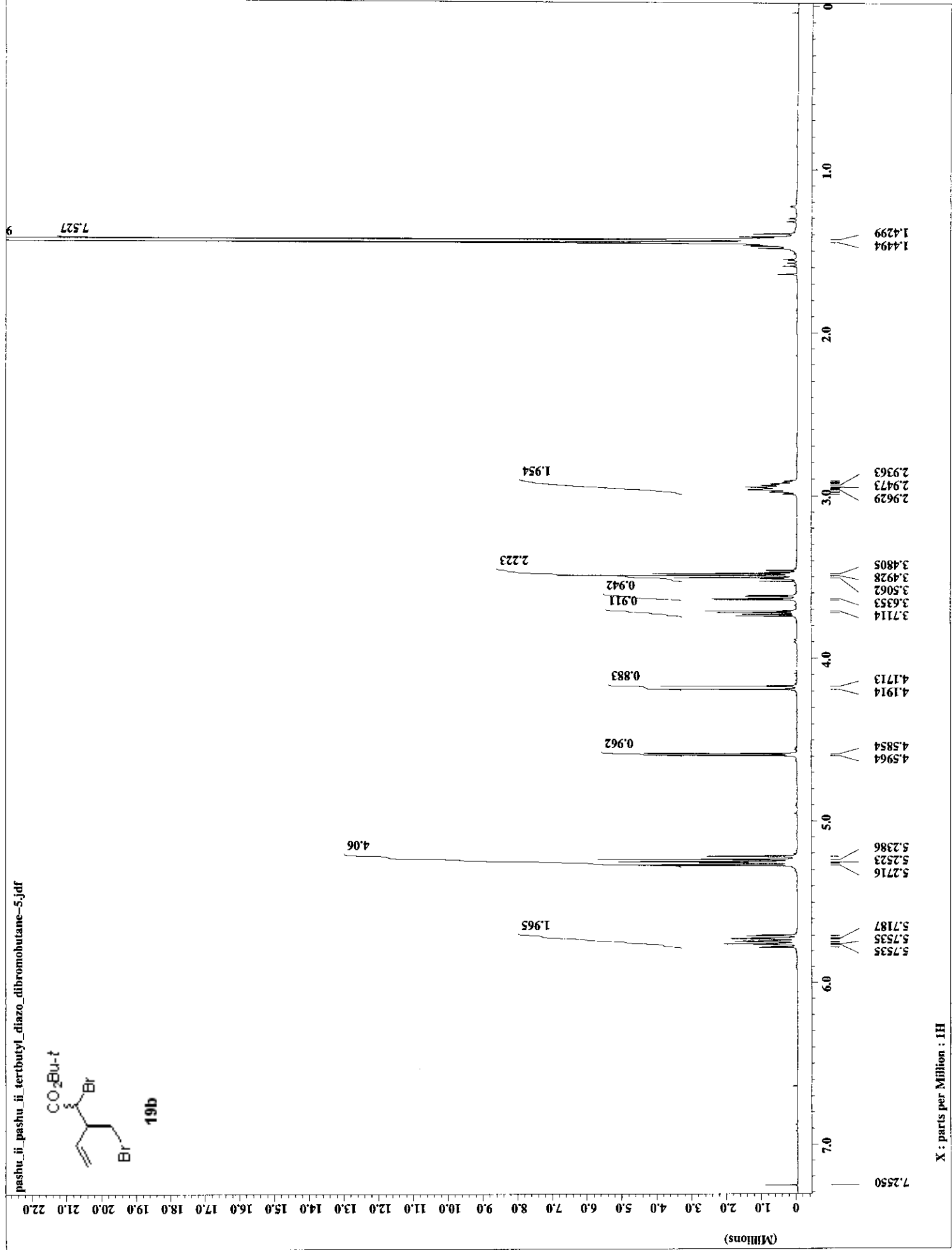
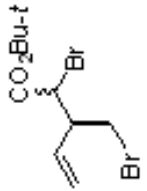


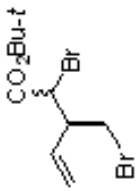
23b

(Millions)

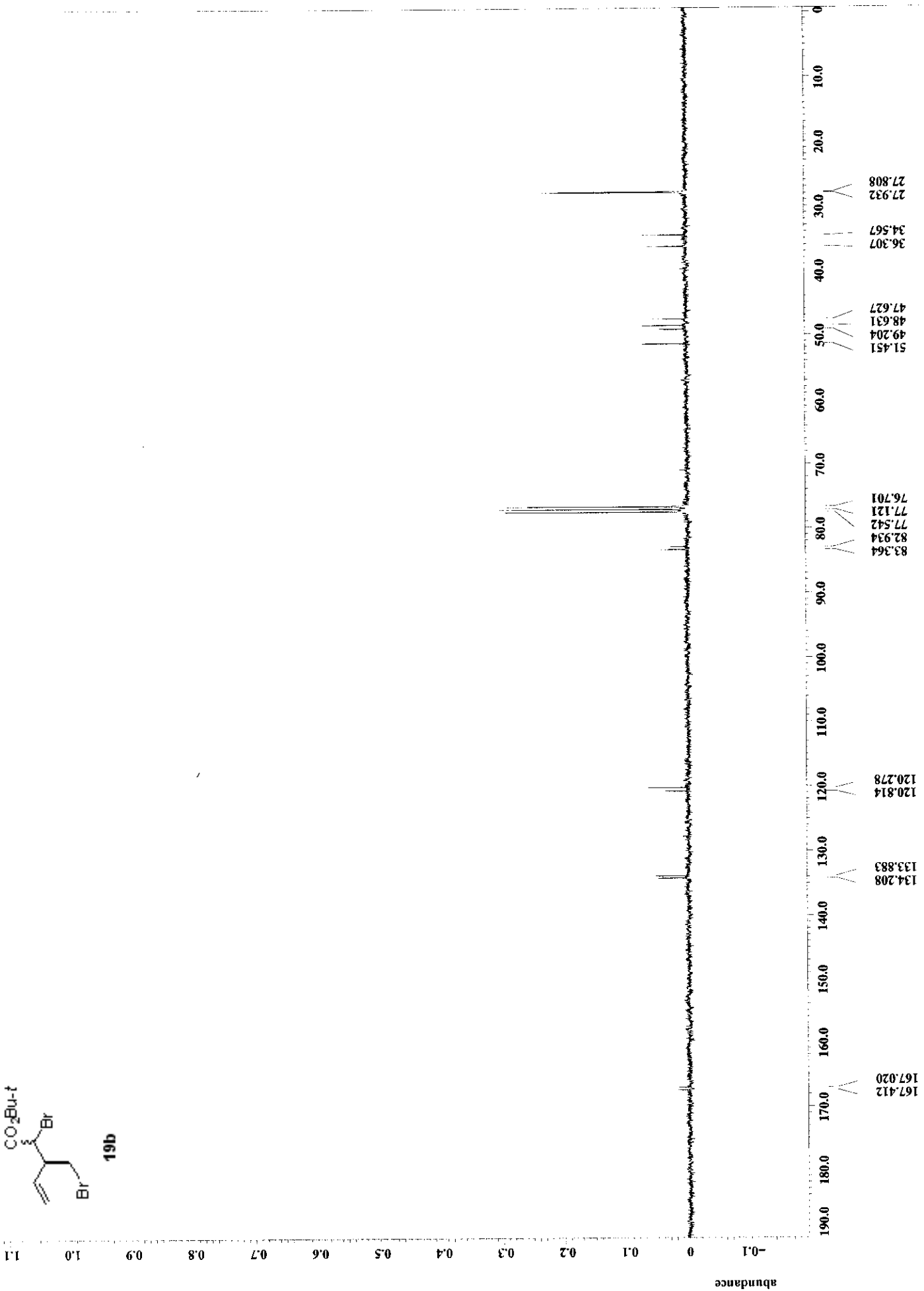


X : parts per Million : 13C

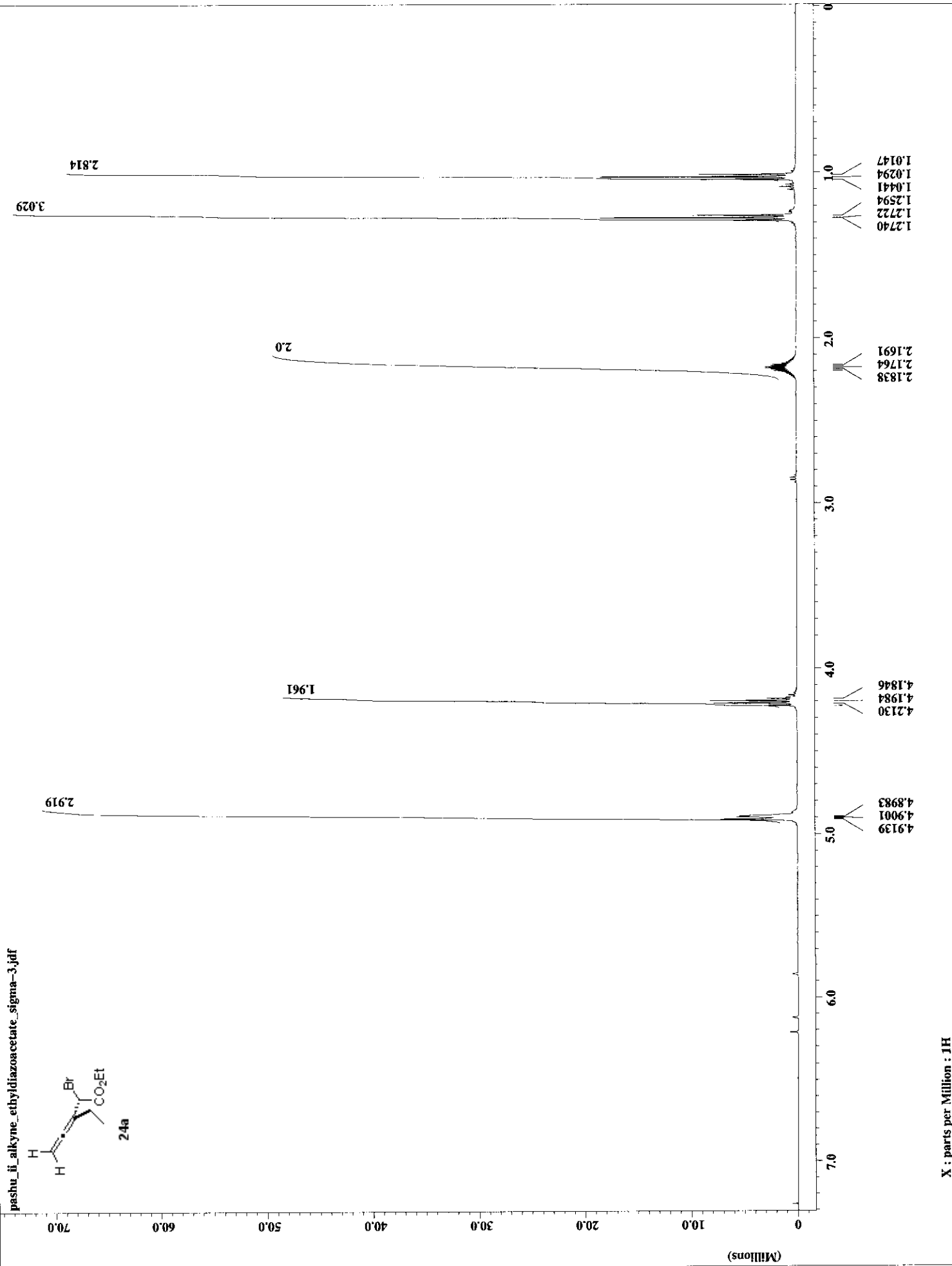
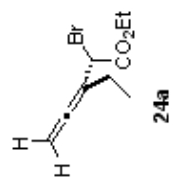




19b

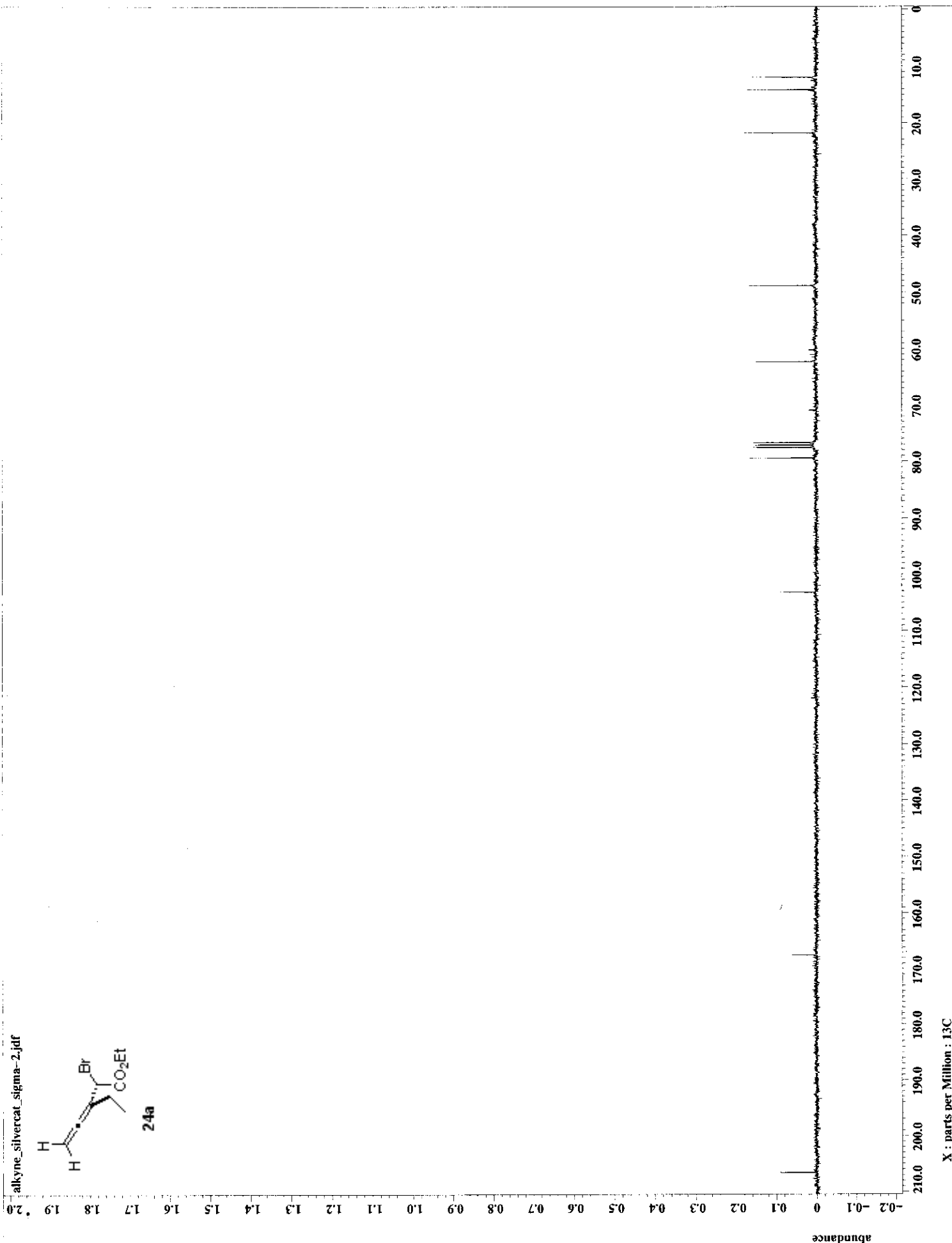
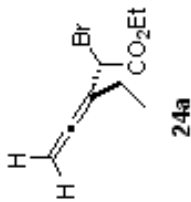


pashu_ii_alkyne_ethylidiazacetate_sigma-3.jdf



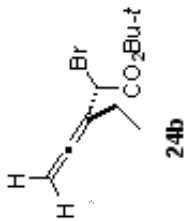
X : parts per Million : 1H

alkyne_silvercat_sigma-2.jdf

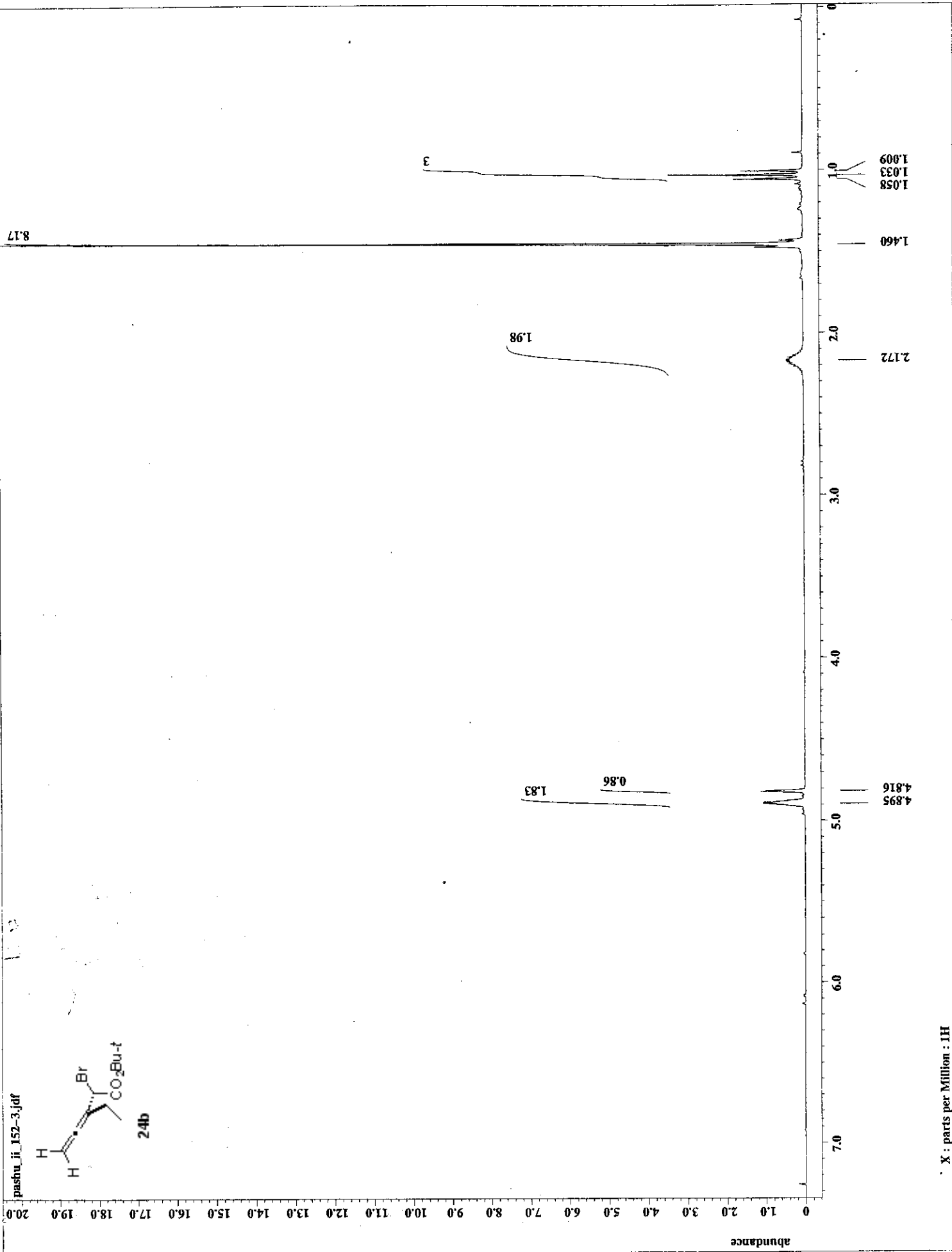


X : parts per Million : 13C

pashu_jf_152-3.jdf

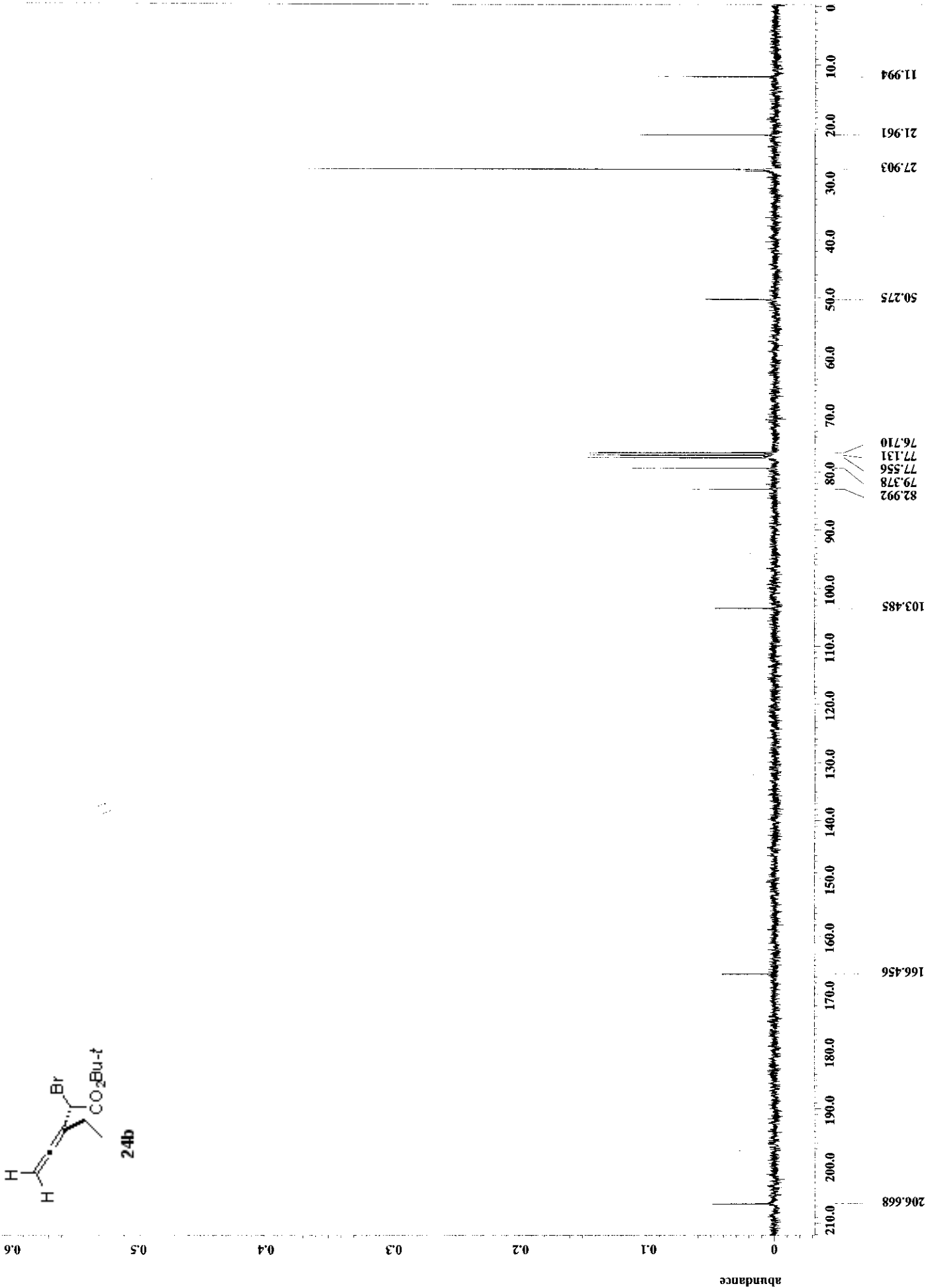
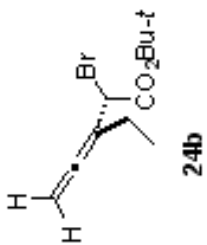


8.17



X : parts per Million : 1H

pashu_ii_152-3.jdf



X : parts per Million : 13C



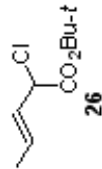
```

Filename = pashu_ii_3 chlorobutene
Author = delta
Experiment = single_pulse.exp
Sample_id = S#335649
Solvent = CHLOROFORM-D
Creation_time = 8-MAY-2006 09:31:52
Revision_time = 8-MAY-2006 12:18:47
Current_time = 8-MAY-2006 12:20:04

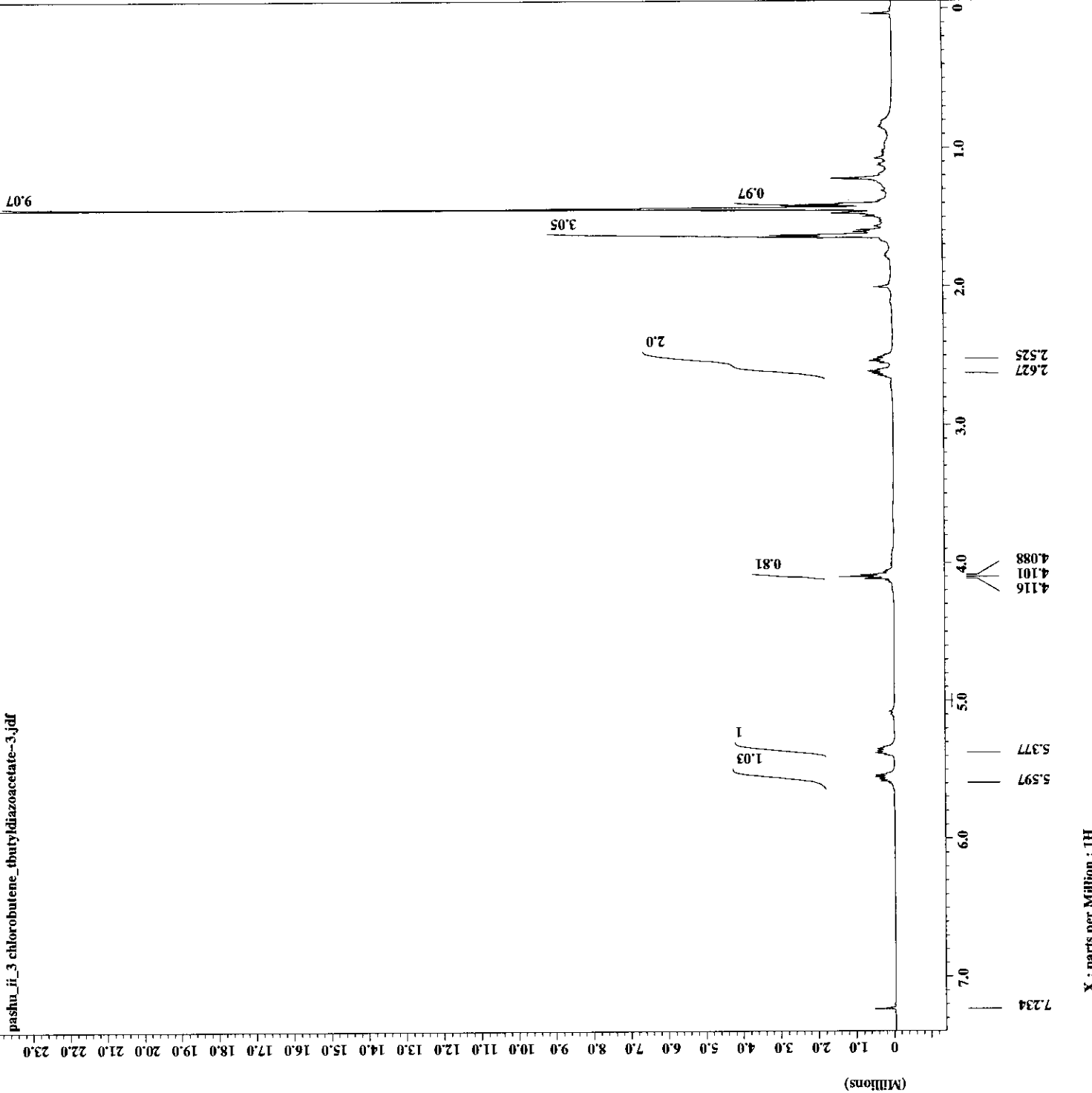
Content = Single Pulse Experiment
Data_format = 1D_COMPLEX
Dir_size = 16384
Dir_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR

Field_strength = 11.7473579 [T] (500 [MH]
X_acq_duration = 2.1823488 [s]
X_domain = 1H
X_freq = 500.15991521 [MHz]
X_offset = 16384
X_points = 0
X_prescans = 0
X_resolution = 0.45822189 [Hz]
X_sweep = 7.50750751 [kHz]
Clipped = FALSE
Mod_return = 1
Scans = 24
Total_scans = 24

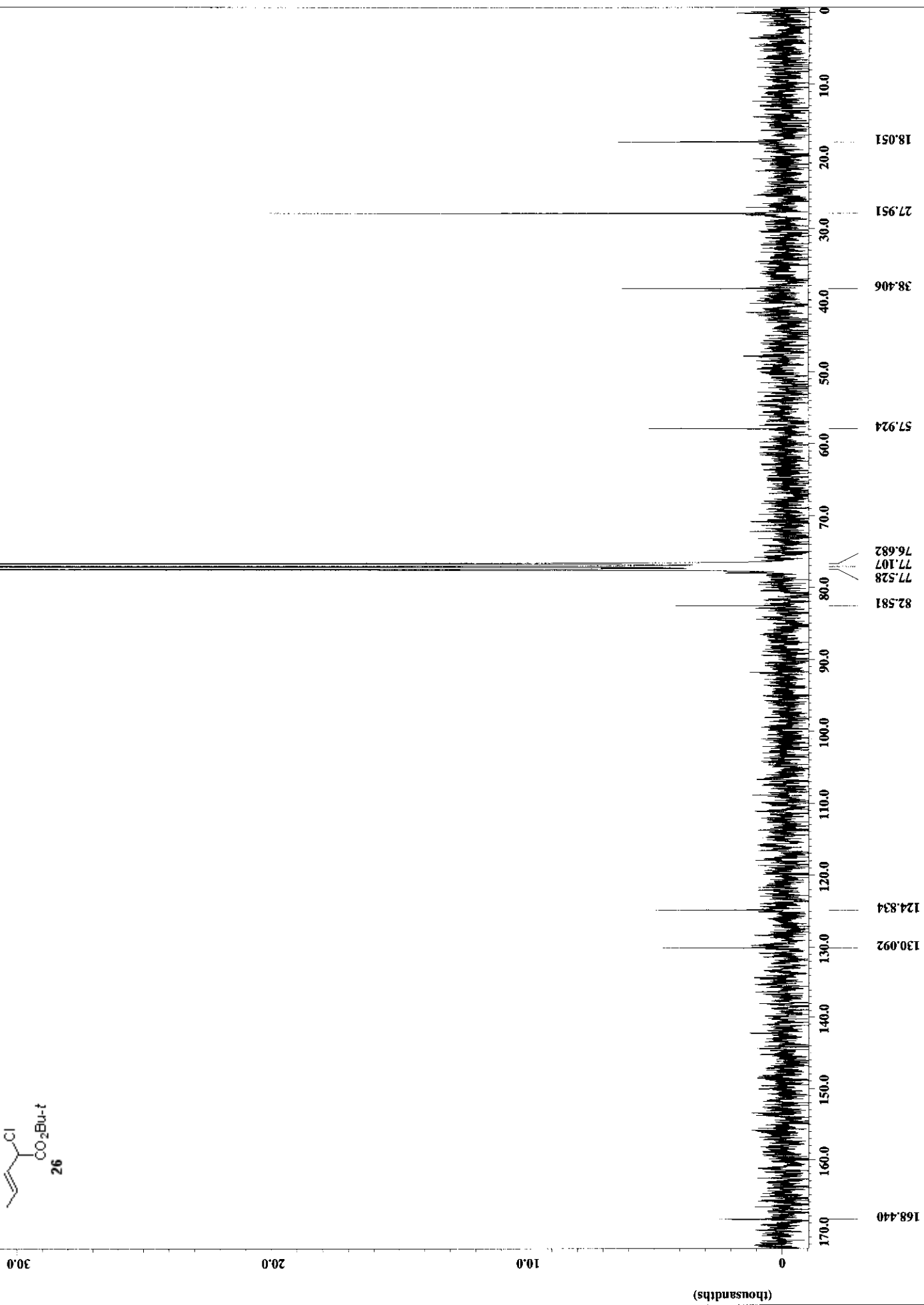
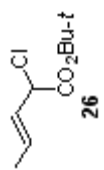
X_90_width = 18.5 [us]
X_acq_time = 2.1823488 [s]
X_angle = 45 [deg]
X_pulse = 9.25 [us]
Initial_wait = 1 [s]
Phase_preset = 3 [us]
Recvr_gain = 15
Relaxation_delay = 4 [s]
Temp_get = 25.8 [dC]
Unblank_time = 2 [us]
  
```



pashu_ii_3 chlorobutene_tbutyldiazoacetate-3.jdf



pashu_ii_sec chloro_tbutyl-4.jdf



X : parts per Million : 13C