Supporting Information

Experimental

General procedures

The product distribution of the reaction of PCl_3 for the synthesis of chlorophosphoramidites was examined in situ by ^{31}P NMR and ^{1}H-^{31}P coupled NMR. The chlorophosphoramidites were prepared in either 1-butyl-3-methylimidazolium bis{(trifluoromethyl)sulfonyl}imide ($[C_4mim][NTf_2]$) or 1-butyl-2,3-dimethylimidazolium bis{(trifluoromethyl)sulfonyl}imide ($[C_4dmim][NTf_2]$). The ionic liquids were prepared in house using standard literature methods[1] from the appropriate bromide salt. 1-ethyl-3-methylimidazolium tris(pentafluoroethyl)trifluorophosphate ($[C_2mim][FAP]$), 1-butyl-3-methylimidazolium tris(pentafluoroethyl)trifluorophosphate ($[C_4mim][FAP]$) and 1-hexyl-3-methylimidazolium tris(pentafluoroethyl)trifluorophosphate ($[C_6mim][FAP]$) were supplied by Merck KGaA. All ionic liquids were dried under high vacuum for 2 h prior to use. The water content and bromide content were measured for each ionic liquid using Karl Fischer titration and ion chromatography, respectively. In each case the bromide levels were below 5 ppm and the water content for the dried ionic liquids were <0.04 wt%.

PCl_3, 3-hydroxypropionitrile, Hünigs base, diisopropylamine, diethylamine, ethylmethylamine and morpholine were obtained from Aldrich and used as supplied. DCM was distilled over calcium hydride prior to use. Each nucleoside was azeotroped with toluene three times then dried under high vacuum before use.

Spectroscopic details

All the nuclear magnetic resonance spectra were recorded on a Bruker Avance 300, 400 or 500 at 25 ^{o}C. For ionic liquid samples an aliquot was transferred directly into the NMR tube with no addition of deuterated solvents. The ^{31}P NMR chemical shifts were recorded in parts per million (ppm) relative to an external probe (sealed capillary inside the NMR tube sample) of triethylphosphonate ($PO(OEt)_3$) in $CDCl_3$ (solvent used for locking/shimming optimisation). The $PO(OEt)_3$ probe was referenced to 0.2 ppm. For the nucleotides the NMR was recorded in $CDCl_3$ referenced to 0.00 ppm using TMS for the ^{1}H NMR and 77.0 ppm using $CDCl_3$ for the ^{13}C NMR.

General experimental conditions

Chlorophosphoramidites

To a stirred solution of PCl_3 (1 eq) in dried $[NTf_2]^-$-based ionic liquid (1eq) under an atmosphere of argon was added Hünig's base (1 eq). The solution was stirred vigorously for 5 min and 3-hydroxypropionitrile (1 eq) was added. The reaction mixture was stirred for 30 min then either nucleophilic amine (2 eq) was added or Hünig's base (1 eq) and nucleophilic amine (1 eq). Reactions were generally complete after a further 40 min. Isolation was achieved by extraction with dry diethyl ether. $[C_nmim][FAP]$ was added to the extraction mixture after separation but before concentration for stabilising purposes.

(2-Cyanoethoxy)-*N*-*N*-diisopropylamino-chlorophosphoramidite (1)

^{1}H NMR (300 MHz, $CDCl_3$) 1.26 (12H, d, J 6.8 Hz), 2.73 (2H, t, J, 6.25 Hz), 3.68-3.86 (2H, m), 4.05 (2H, dt, J 8.17, 6.28 Hz). ^{13}C NMR (75 MHz, $CDCl_3$) 19.7, 46.5 (d), 47.8, 60.8 (d), 117.3. ^{31}P NMR (121 MHz, $CDCl_3$) 181.

(2-Cyanoethoxy)-*N*-*N*-diethylamino-chlorophosphoramidite (2)

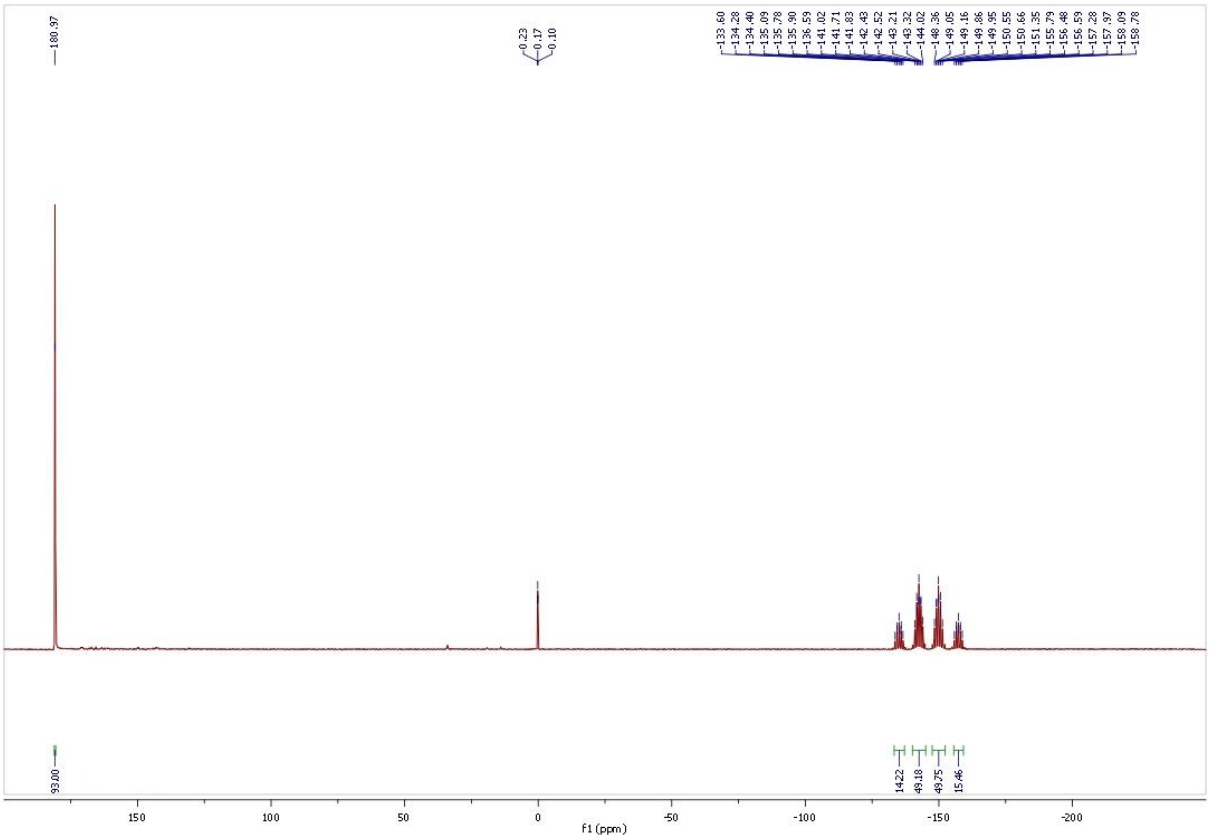
^{1}H NMR (300 MHz, $CDCl_3$) 1.12 (6H, t, J 9.0 Hz), 2.72 (2H, t, J, 6.0 Hz), 3.09-3.16 (4H, m), 4.03 (2H, dt, J 8.0, 6.0 Hz). ^{13}C NMR (75 MHz, $CDCl_3$) 18.2, 39.8 (d), 42.5, 61.5 (d), 118.3. ^{31}P NMR (121 MHz, $CDCl_3$) 176.9 ($P(OR)(NEt_2)Cl$).

(2-Cyanoethoxy)-*N*-*N*-ethylmethylamino-chlorophosphoramidite (3)

^{1}H NMR (300 MHz, $CDCl_3$) 1.03 (3H, t, J 9.0 Hz, $CH_2C\boldsymbol{H}_3$), 2.57 (3H, m, N-CH_3), 2.72 (2H, t, J, 6.0 Hz), 3.09-3.16 (2H, m), 4.03 (2H, dt, J 8.0, 6.0 Hz). ^{13}C NMR (75 MHz, $CDCl_3$) 16.3, 20.1 (d), 31.2 (d), 43.6 (d), 61.4 (d), 118.3. ^{31}P NMR (121 MHz, $CDCl_3$) 174.9.

The ^{31}P NMR spectra for **1-3** stabilised in C_6mim FAP are shown below. In each case, the mole ratio of chlorophosphoramidite to ionic liquid was typically 1:1. Each of the spectra show the chlorophosphoramidite ~174-180ppm, FAP anion at -150 ppm and an internal standard at 0.2 ppm.

1



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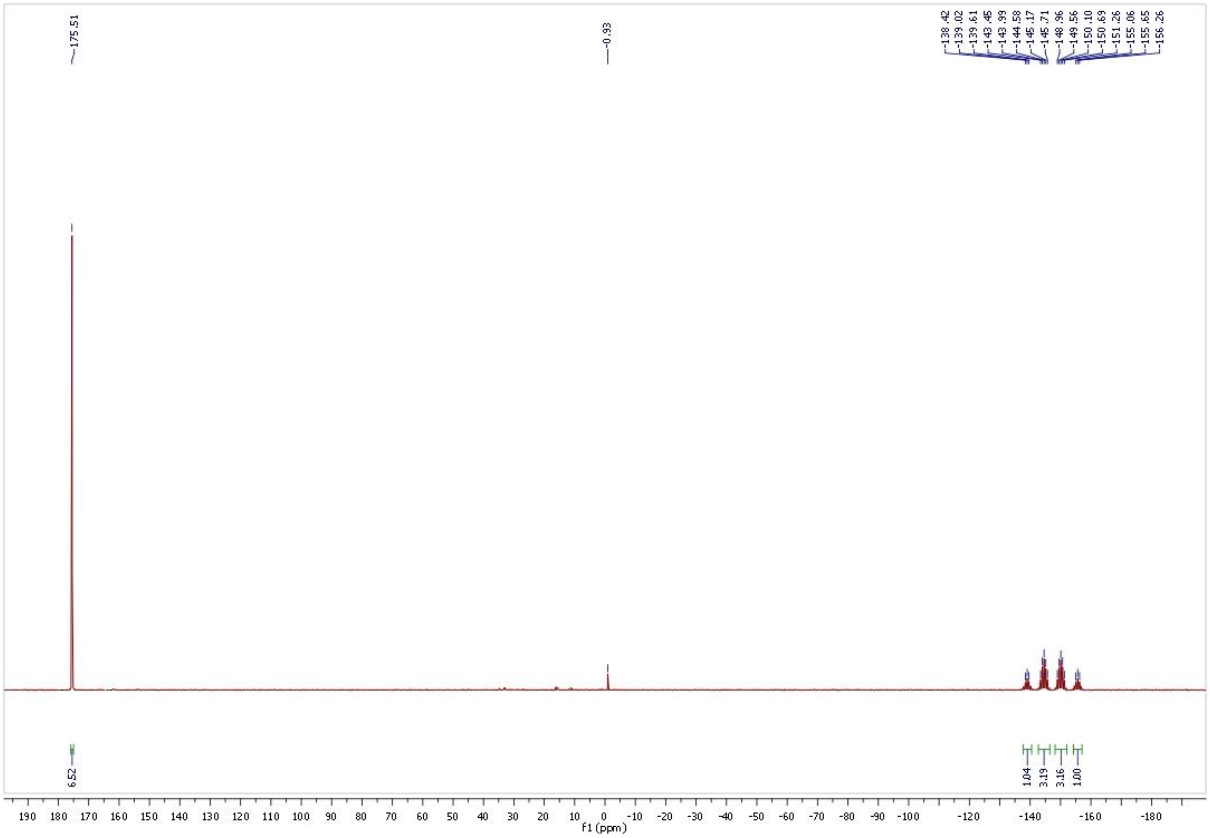
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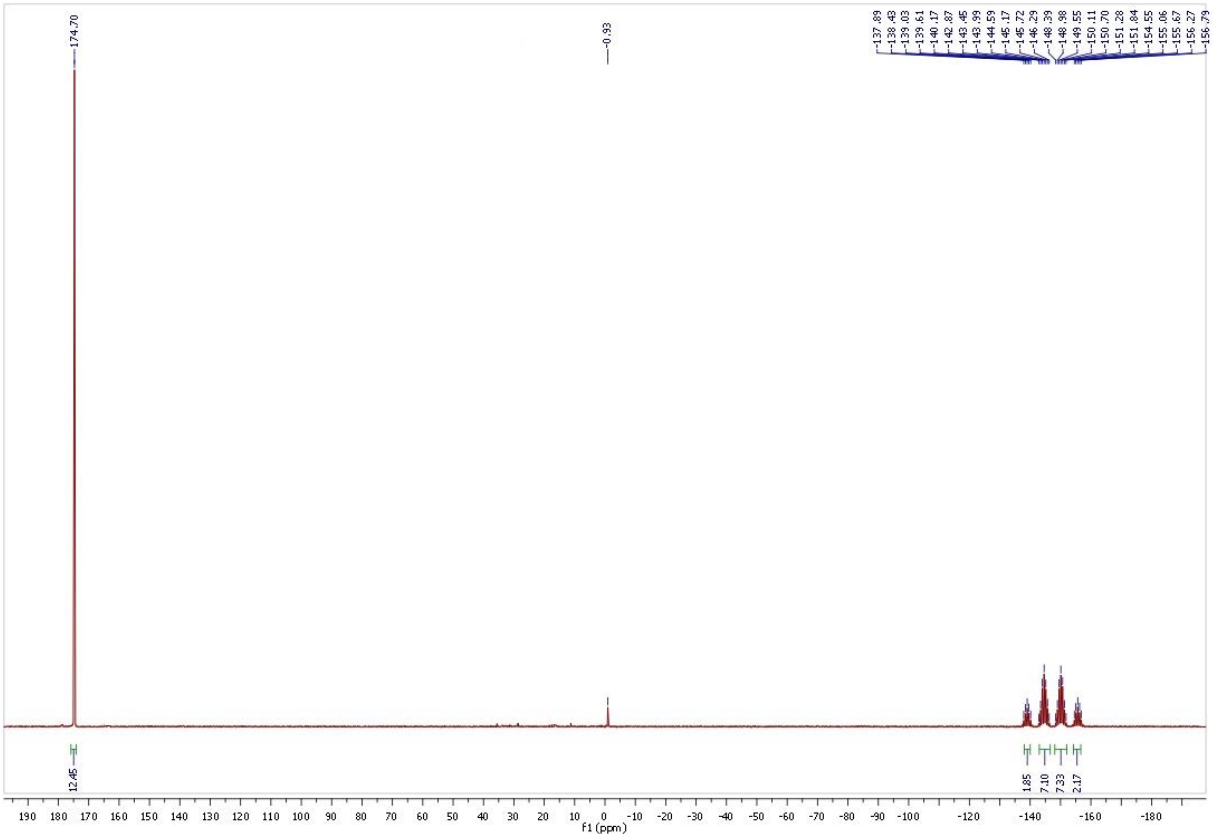
139.02
139.61
143.45
143.99
144.58
145.17
145.71
148.96
149.56
150.10
150.69
151.26
155.06
155.65
156.26
6.52
1.04
3.19
3.16
1.00
f1 (ppm)

3


174.70
0.93
137.89
138.43
139.03
139.61
140.17
142.87
143.45
143.99
144.59
145.17
145.72
146.29
148.39
148.98
149.55
150.11
150.70
151.28
151.84
154.55
155.06
155.67
156.27
156.79
12.45
1.85
7.10
7.33
2.17
f1 (ppm)

Phosphitylation Reactions
All reactions were carried out in a 50-60 mg scale based on the amount of protected nucleoside. This generally equated to 50 μL of Hünig's base and 100 μL of the ionic liquid stabilised chlorophosphoramidite.

Method 1 – Mixed Solvent System
To a stirred solution of the protected nucleoside (1 eq) in dry DCM was added Hünig's base (4 eq). The mixture was stirred for 5 min then the stabilised chlorophosphoramidite in [C$_n$mim][FAP] (2.5 eq) was added. The reaction was monitored by tlc over time, and on completion the reaction mixture was concentrated in vacuo.

Method 2 – Mixed Solvent System with DMAP
To a stirred solution of the protected nucleoside (1 eq) in dry DCM was added Hünig's base (4 eq) and DMAP (0.1 eq). The mixture was stirred for 5 min then the stabilised chlorophosphoramidite in [C$_n$mim][FAP] (2.5 eq) was added. The reaction was monitored by tlc over time, and on completion the reaction mixture was concentrated in vacuo.

Method 3 – Solventless Reaction
To the stabilised chlorophosphoramidite in [C$_n$mim][FAP] was added Hunigs base followed by the partially protected nucleoside. The reaction was monitored by tlc over time, and on equilibrium the reaction mixture was directly submitted to purification.

Method 4 – Sonication
To the partically protected nucleoside (1 eq) and Hünig's base (4 eq) was added stabilised chlorophosphoramidite in [C$_n$mim][FAP] (2.5 eq). The mixture was sonicated in a Decon F5100b sonic bath for 1.5 h.

Method 5 – Ball Milling
To the partically protected nucleoside (1 eq) and Hünigs base (4 eq) was added stabilised chlorophosphoramidite in [C$_n$mim][FAP] (1.5 eq). The mixture was shaken in a 1.5 ml steel vessel with a 5 mm diameter steel ball bearing in a Retsch MM400 mixer mill at 30 Hz for 0.5 h. In bulk physical form, the ball-milled reactions were pastes, consisting of a small amount of liquid phase (primarily the IL, chlorophosphoramidite and Hünig's base) in which there was a dispersion of solid particles (primarily the nucleoside reagent and/or product depending on the extent of reaction).

For all methods, purification was achieved by concentration of solvent (where necessary) then filtration of the crude residue through a short pad of silica gel (1:1 hexane/ethyl acetate, 1% NEt_3). Recovery of unreacted nucleoside could be achieved by subsequent washing of the silica with ethyl acetate.

All compounds are a diastereoisomeric mixture, hence the complex ^{1}H and ^{13}C NMR spectra, and 2 peaks in each of the ^{31}P NMR spectra.

N-Benzoyl-5'-*O*-(4,4'-dimethoxytrityl)-2'-deoxyadenosine-3'-*O*-[*O*-(2-cyanoethyl)-*N*,*N*'-diisopropylphosphoramidite (1a)
^{1}H NMR (500 MHz, $CDCl_3$) 1.12-1.22 (12H, m), 2.48 (2H, t, J 6.4 Hz), 2.63 (2H, t, J 6.3 Hz), 2.75-2.78 (1H, m), 3.31-3.3.63 (7H, m), 3.76 (3H, s), 3.78 (3H, s), 4.28-4.35 (1H, m), 4.76-4.82 (1H, m), 6.49-6.55 (1H, m), 6.76-6.82 (4H, m), 7.17-7.30 (6H, m), 7.38-7.7.41 (2H, m), 7.51-7.62 (4H, m), 8.02 (2H, d, J 12.0 Hz), 8.20 (1H, s), 8.22 (1H, s), 8.75 (1H, s), 8.76 (1H, s), 8.97 (1H, br s). ^{13}C NMR (125 MHz, $CDCl_3$) 22.9-23.1, 24.6-24.6, 39.6, 43.3-43.3, 50.37, 55.28, 58.1, 63.4, 77.2, 84.7, 86.5, 113.1, 117.5,

122.0-132.8, 135.6, 135.7, 141.8, 144.4, 149.4, 152.6, 158.5. ^{31}P NMR (121 MHz, $CDCl_3$) 150.0, 150.1. HRMS (ES, M+H$^+$) calculated for $C_{47}H_{53}N_7O_7P$ 858.3744, found 858.3756.

N-Benzoyl-5'-*O*-(4,4'-dimethoxytrityl)-2'-deoxycytidine-3'-*O*-[*O*-(2-cyanoethyl)-*N*,*N*'-diisopropylphosphoramidite (1b)

^{1}H NMR (500 MHz, $CDCl_3$) 1.03-1.55 (12H, m CH_3), 2.13-2.35 (1H, m), 2.34-2.38 (1H, m), 2.60-2.66 (1H, m), 2.70-2.80 (2H, m), 3.50-3.86 (12H, m), 4.06-4.26 (1H, m), 4.53-4.58 (1H, m), 5.30 (2H, br s), 6.29 (1H, dd, 5.1, 10.9 Hz), 6.86 (1H, dt, J 6.8, 3.4 Hz), 7.11-7.67 (13H, m), 7.89 (1H, d, J 7.0 Hz), 8.31 (1H, d, J 7.2 Hz), 8.72 (1H, br s). ^{13}C NMR (125 MHz, $CDCl_3$) 22.6, 23.2, 24.9, 26.1, 30.2, 31.2, 36.6, 42.3, 50.7, 55.6, 62.9, 71.1, 86.6, 87.3, 113.7, 123.7, 123.8, 127.5, 127.9, 128.4, 128.5, 129.4, 130.4, 133.5, 136.2, 144.5, 144.6, 145.1, 154.7, 159.1, 162.4. ^{31}P NMR (121 MHz, $CDCl_3$) 150.0, 150.5. HRMS (ES, M+H$^+$) calculated for $C_{46}H_{53}N_5O_8P$ 834.3632, found 834.3660.

N-Isobutyryl-5'-*O*-(4,4'-dimethoxytrityl)-2'-*O*-TBDMS-guanosine-3'-*O*-[*O*-(2-cyanoethyl)-*N*,*N*'-diisopropylphosphoramidite (1c)

^{1}H NMR (400 MHz, $CDCl_3$) -0.04 (3H, s), -0.00 (3H, s), 0.78 (9H, s), 0.79-1.35 (12H, m), 1.38 (6H, d, J 6.8 Hz), 1.77-1.89 (1H, m), 2.62-2.93 (3H, m), 3.11-3.20 (1H, m), 3.46-3.80 (4H, m), 3.89 (6H, s), 3.90-4.34 (2H, m), 4.88-4.92 (1H, m), 5.14-5.20 (1H, m), 5.72 (1H, d, J 7.5 Hz), 5.96 (1H, J 7.8 Hz), 6.78-6.85 (1H, m), 7.18-7.64 (13H, m), 7.75 (1H, s), 7.85 (1H, s), 7.95 (1H, s), 8.64 (2H, br s). ^{13}C NMR (100 MHz, $CDCl_3$) -5.2, -4.6, 17.8, 18.2, 18.2, 19.9, 24.6, 24.7, 25.5, 35.8, 43.3, 43.5, 55.3, 57.2, 63.1, 72.9, 73.2, , 84.7, 86.0, 86.6, 113.2, 117.2, 122.9, 127.2, 127.9, 128.0, 128.1, 129.9, 130.0, 135.8, 136.2, 139.5, 144.5, 145.2, 146.8, 148.1, 155.5, 158.8, 178.2. ^{31}P NMR (121 MHz, $CDCl_3$) 150.0, 150.1. HRMS (ES, M+H$^+$) calculated for $C_{50}H_{69}N_7O_9PSi$ 970.4664, found 970.4701.

N-Benzoyl-5'-*O*-(4,4'-dimethoxytrityl)-2'-deoxyadenosine-3'-*O*-[*O*-(2-cyanoethyl)-*N*,*N*'-diethylphosphoramidite (2a)

^{1}H NMR (400 MHz, $CDCl_3$) 0.97-1.37 (6H, m), 2.47-2.66 (4H, m), 2.70-2.76 (1H, m), 2.85-3.21 (4H, m), 3.29-3.48 (2H, m), 3.68-3.90 (4H, m), 3.77 (3H, s), 3.78 (3H, s), 4.28-4.32 (1H, m), 4.73-4.86 (1H, m), 6.73-6.86 (1H, m), 7.16-7.42 (12H, m), 7.57 (3H, t, J 7.2 Hz), 8.03 (2H, d, J 7.3 Hz), 8.15-8.22 (2H, m), 8.72 (1H, s), 8.73 (1H, s), 8.99 (1H, br s, NH). ^{13}C NMR (100 MHz, $CDCl_3$) 20.4, 20.9, 22.6, 29.7, 32.4, 37.4, 37.6, 39.8, 46.0 55.2, 55.2, 57.9, 60.5, 63.5, 76.7, 85.0, 86.5, 113.1, 117.2, 123.2, 127.8, 128.1, 128.2, 128.8, 130.0, 132.7, 141.1, 144.6, 158.5, 158.6. ^{31}P NMR (121 MHz, $CDCl_3$) 149.4, 149.5. HRMS (ES, M+H$^+$) calculated for $C_{45}H_{49}N_7O_7P$ 830.3431, found 830.3444.

N-Benzoyl-5'-*O*-(4,4'-dimethoxytrityl)-2'-deoxycytidine-3'-*O*-[*O*-(2-cyanoethyl)-*N*,*N*'-diethylphosphoramidite (2b)

^{1}H NMR (400 MHz, $CDCl_3$) 1.20-1.46 (6H, m CH_3), 2.79-2.89 (2H, m), 2.24-2.37 (2H, m), 2.48 (2H, t, J 6.4 Hz), 2.61 (2H, t, J 6.4 Hz), 2.94-3.16 (2H, m), 3.37-3.60 (6H, m), 3.89 (6H, s), 4.03-4.25 (5H, m), 4.60-4.98 (2H, m), 6.29 (1H, m), 6.86 (1H, m), 7.16-7.68 (13H, m), 7.87 (1H, d, J 7.0 Hz), 8.53 (1H, br s). ^{13}C NMR (100 MHz, $CDCl_3$) 20.1, 21.0, 22.2, 25.7, 29.8, 30.8, 31.2, 36.2, 37.4, 38.1, 39.7, 40.7, 46.8, 50.4, 55.2, 58.0, 58.2, 59.5, 60.4, 61.9, 76.7, 85.6, 87.0, 113.3, 117.3, 122.1, 123.5, 127.2, 127.4, 127.6, 127.8, 127.9, 128.4, 128.2, 129.0, 130.1, 133.1, 135.2, 144.1, 158.7, 158.7. ^{31}P NMR (121 MHz, $CDCl_3$) 149.4, 149.9. HRMS (ES, M+H$^+$) calculated for $C_{44}H_{49}N_5O_8P$ 806.3319, found 806.3318.

N-Isobutyryl-5'-*O*-(4,4'-dimethoxytrityl)-2'-*O*-TBDMS-guanosine-3'-*O*-[*O*-(2-cyanoethyl)-*N*,*N*'-diethylphosphoramidite (2c)

^{1}H NMR (400 MHz, $CDCl_3$) -0.08 (3H, s), -0.07 (3H, s), 0.75 (9H, s), 0.80-1.37 (12H, m), 2.56-3.13 (3H, m), 3.46-3.72 (4H, m), 3.77 (6H, s), 3.94-4.41 (2H, m), 4.84-4.95 (1H, m), 4.97-5.07 (1H, m), 5.66 (1H, d, J 7.2 Hz), 5.83 (1H, J 7.2 Hz), 6.69-6.77 (1H, m), 7.10-7.75 (13H, m), 7.77 (1H, s), 7.87 (1H, s), 8.17 (2H, br s).^{13}C NMR (100 MHz, $CDCl_3$) -5.2, -4.7, -0.0, 15.0, 15.1, 17.8, 18.5, 23.0, 25.4, 35.8, 35.9, 55.2, 58.7, 76.6, 76.9, 88.3, 113.3, 118.1, 127.2, 127.9, 128.1, 130.0, 135.9, 147.0, 147.3, 152.1, 154.3, 155.4, 158.8,

161.1, 177.9, 178.5. ^{31}P NMR (121 MHz, $CDCl_3$) 150.1, 150.5. HRMS (ES, M+H$^+$) calculated for $C_{48}H_{65}N_7O_9PSi$ 942.4351, found 942.4380.

N-Benzoyl-5'-*O*-(4,4'-dimethoxytrityl)-2'-deoxyadenosine-3'-*O*-[*O*-(2-cyanoethyl)-*N*,*N*'-ethylmethylphosphoramidite (3a)

^{1}H NMR (400 MHz, $CDCl_3$) 1.00-1.51 (3H, m), 1.80-1.90 (1H, m), 2.46-2.66 (2H, m), 2.70-2.76 (1H, m), 2.85-3.21 (2H, m), 3.29-3.48 (2H, m), 3.68-3.82 (10H, m), 3.89 (3H, s), 4.28-4.32 (1H, m), 6.76-6.82 (1H, m), 7.16-7.42 (12H, m), 7.47-67 (4H, t, J 7.2 Hz), 8.03 (2H, d, J 7.2 Hz), 8.15-8.22 (2H, m), 8.74 (1H, s), 8.98 (1H, br s, NH). ^{13}C NMR (100 MHz, $CDCl_3$) 22.2, 22.2, 25.7, 29.7, 30.8, 36.2, 43.6, 50.4, 54.0, 55.2, 55.8, 60.5, 66.0, 68.1, 76.6, 83.1, 86.7, 113.1, 117.8, 122.1, 123.5, 127.8, 128.1, 128.2, 128.8, 130.0, 132.7, 141.1, 144.6, 157.3, 158.6. ^{31}P NMR (121 MHz, $CDCl_3$) 149.4, 149.8. HRMS (ES, M+H$^+$) calculated for $C_{44}H_{47}N_7O_7P$ 816.3275, found 816.3265.

N-Benzoyl-5'-*O*-(4,4'-dimethoxytrityl)-2'-deoxycytidine-3'-*O*-[*O*-(2-cyanoethyl)-*N*,*N*'-ethylmethylphosphoramidite (3b)

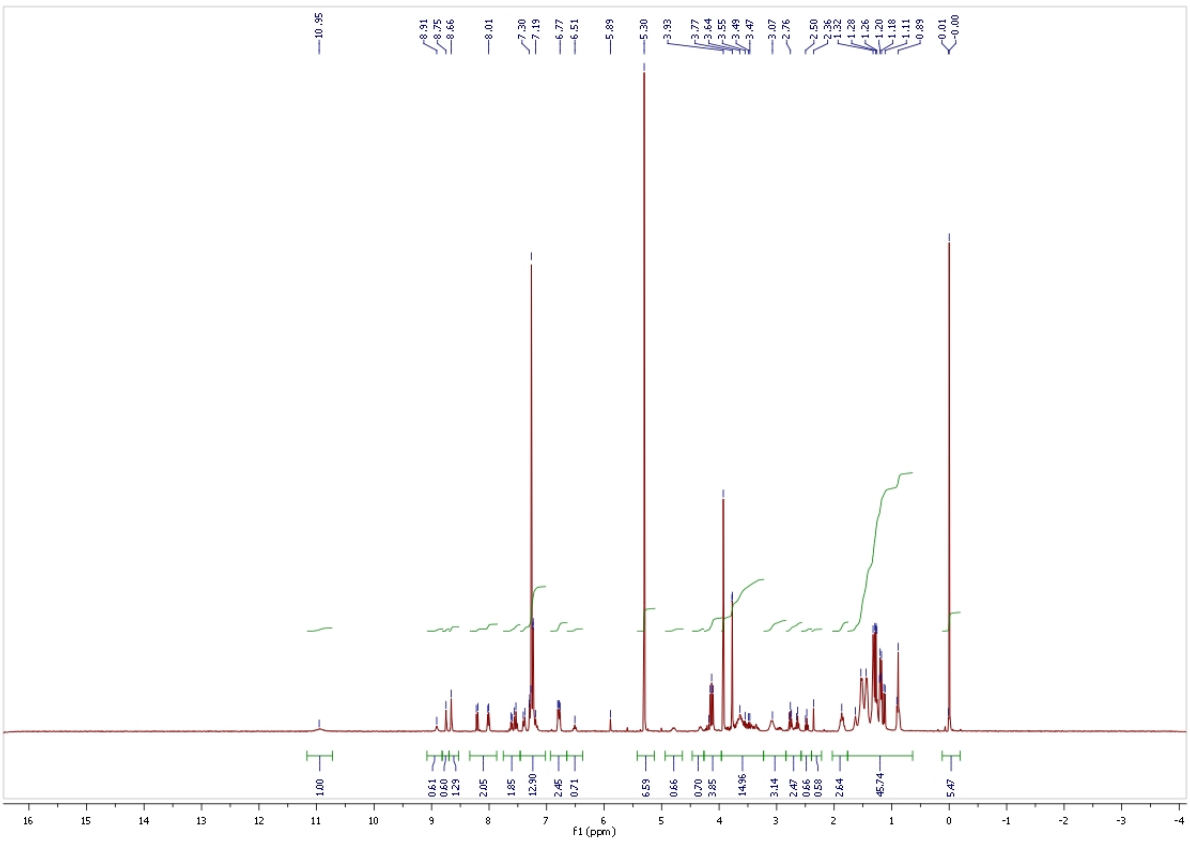
^{1}H NMR (400 MHz, $CDCl_3$) 1.10-1.44 (3H, m), 1.90-2.06 (2H, m), 2.36-2.66 (4H, m), 2.89-3.03 (2H, m), 3.48-3.57 (2H, m), 3.68 (2H, br s), 3.72-3.75 (2H, m), 3.86 (6H, s), 3.92 (2H, t, J 7.5Hz), 3.99-4.06 (5H, m), 6.29 (1H, m), 6.74-6.74 (1H, m), 6.97-7.56 (13H, m), 7.82 (1H, d, J 7.6 Hz), 7.98 (1H, s), 8.25 (1H, br s). ^{13}C NMR (100 MHz, $CDCl_3$) 12.4, 20.9, 21.2, 24.3, 28.4, 29.6, 30.2, 30.6, 34.5, 35.2, 41.2, 45.1, 49.0, 52.9, 54.2, 57.9, 66.3, 73.9, 76.2, 84.5, 86.2, 112.3, 117.7, 120.4, 120.7, 121.1, 122.5, 126.4, 127.2, 128.1, 130.1, 133.4, 134.2, 158.6, 158.7. ^{31}P NMR (121 MHz, $CDCl_3$) 148.1, 148.6. HRMS (ES, M+H$^+$) calculated for $C_{43}H_{47}N_5O_8P$ 792.3173, found 792.3162.

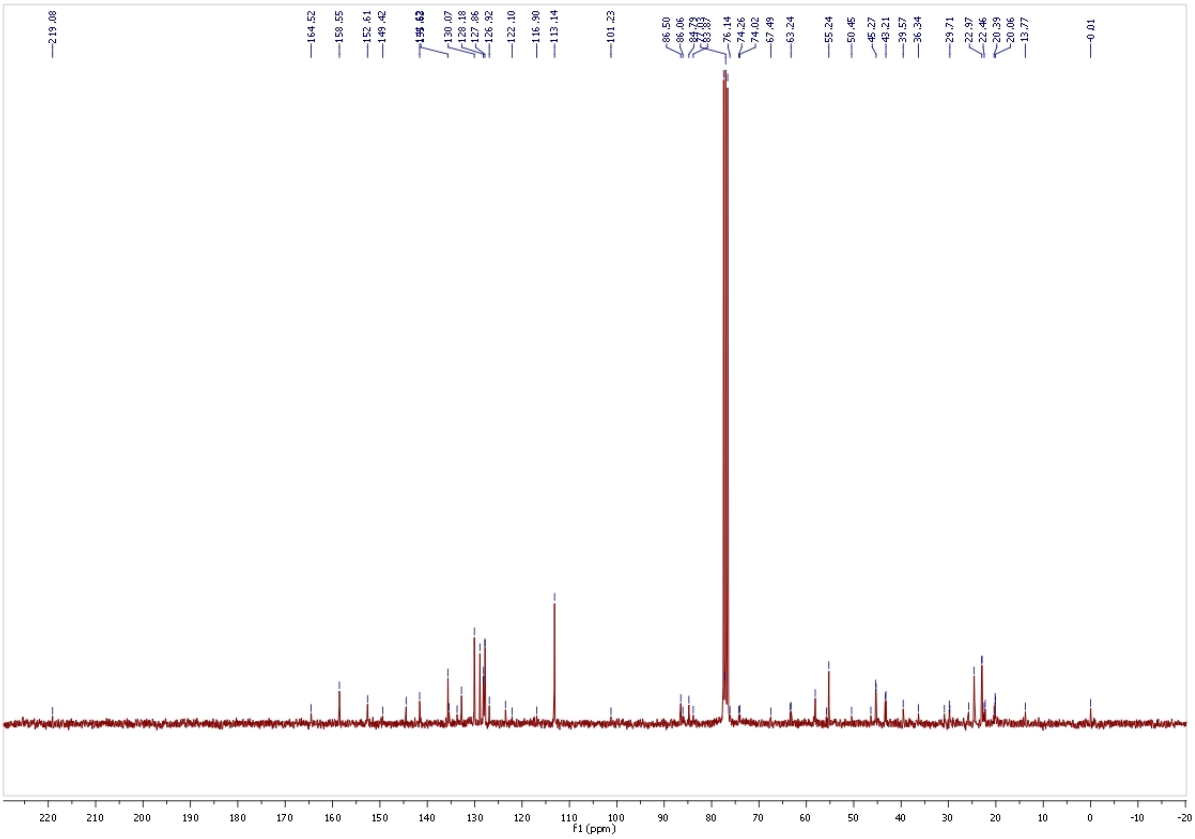
N-Isobutyryl-5'-*O*-(4,4'-dimethoxytrityl)-2'-*O*-TBDMS-guanosine-3'-*O*-[*O*-(2-cyanoethyl)-*N*,*N*'-ethylmethylphosphoramidite (3c)

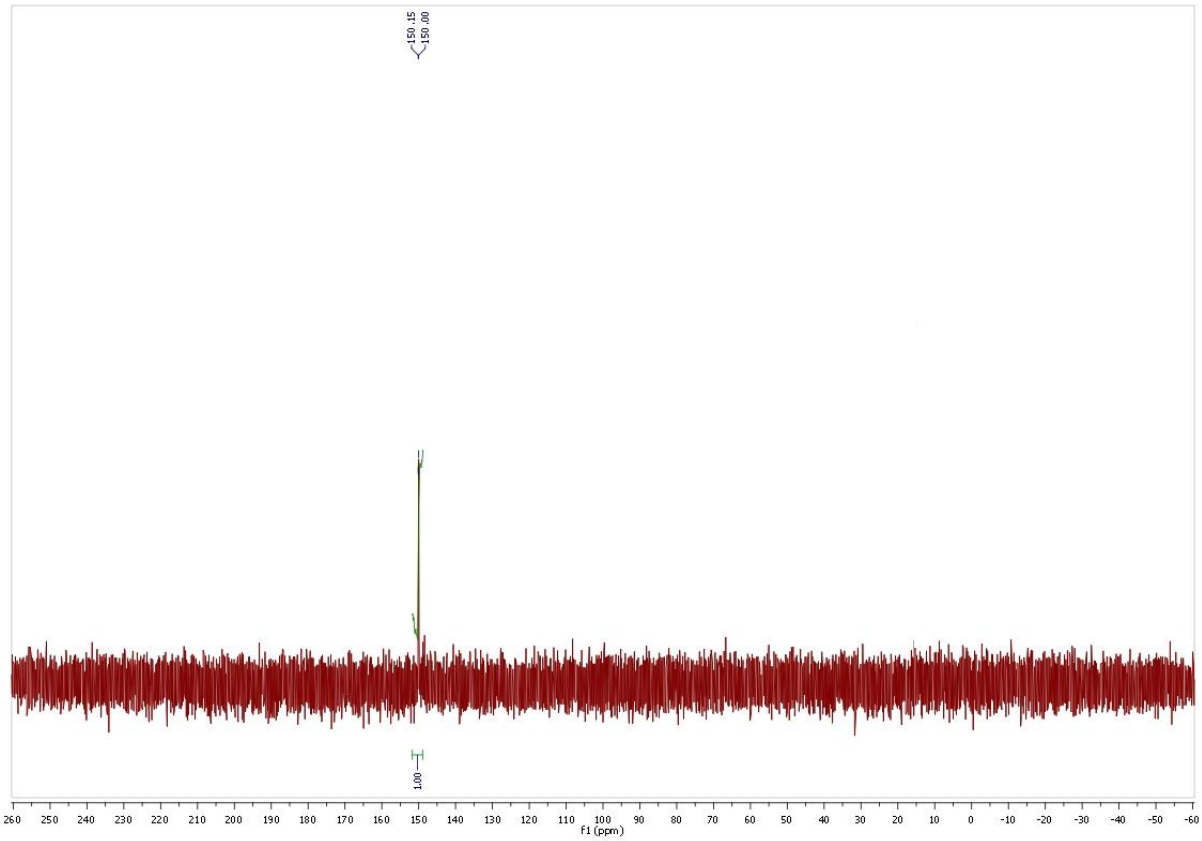
^{1}H NMR (400 MHz, $CDCl_3$) -0.08 (3H, s), -0.07 (3H, s), 0.81 (9H, s), 0.80-1.38 (9H, m), 2.56-3.13 (5H, m), 3.46-3.84 (10H, m), 3.94-4.50 (4H, m), 5.00-5.04 (1H, m), 5.18-5.22 (1H, m), 5.70-5.91 (2H, m), 6.73-6.88 (1H, m), 7.15-7.75 (13H, m), 7.79-7.84 (1H, m), 7.88 (1H, s). ^{13}C NMR (100 MHz, $CDCl_3$) -4.8, -4.7, 14.6, 18.2, 18.7, 25.9, 36.2, 55.6, 60.8, 63.5, 64.1, 71.4, 74.1, 84.8, 86.4, 105.0, 113.7, 117.7, 123.3, 127.6, 128.3, 128.5, 130.4, 136.0, 136.2, 136.6, 139.8, 145.7, 147.3, 148.5, 155.9, 159.2, 178.6, 178.9. ^{31}P NMR (121 MHz, $CDCl_3$) 148.0, 148.5. HRMS (ES, M+Na$^+$) calculated for $C_{47}H_{62}N_7O_9NaSiP$ 950.4014, found 950.4051.

The NMR spectra for each of the compounds formed are shown below. In each case, the figures are in the order ^{1}H NMR, ^{13}C NMR, ^{31}P NMR.

1a



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1.00
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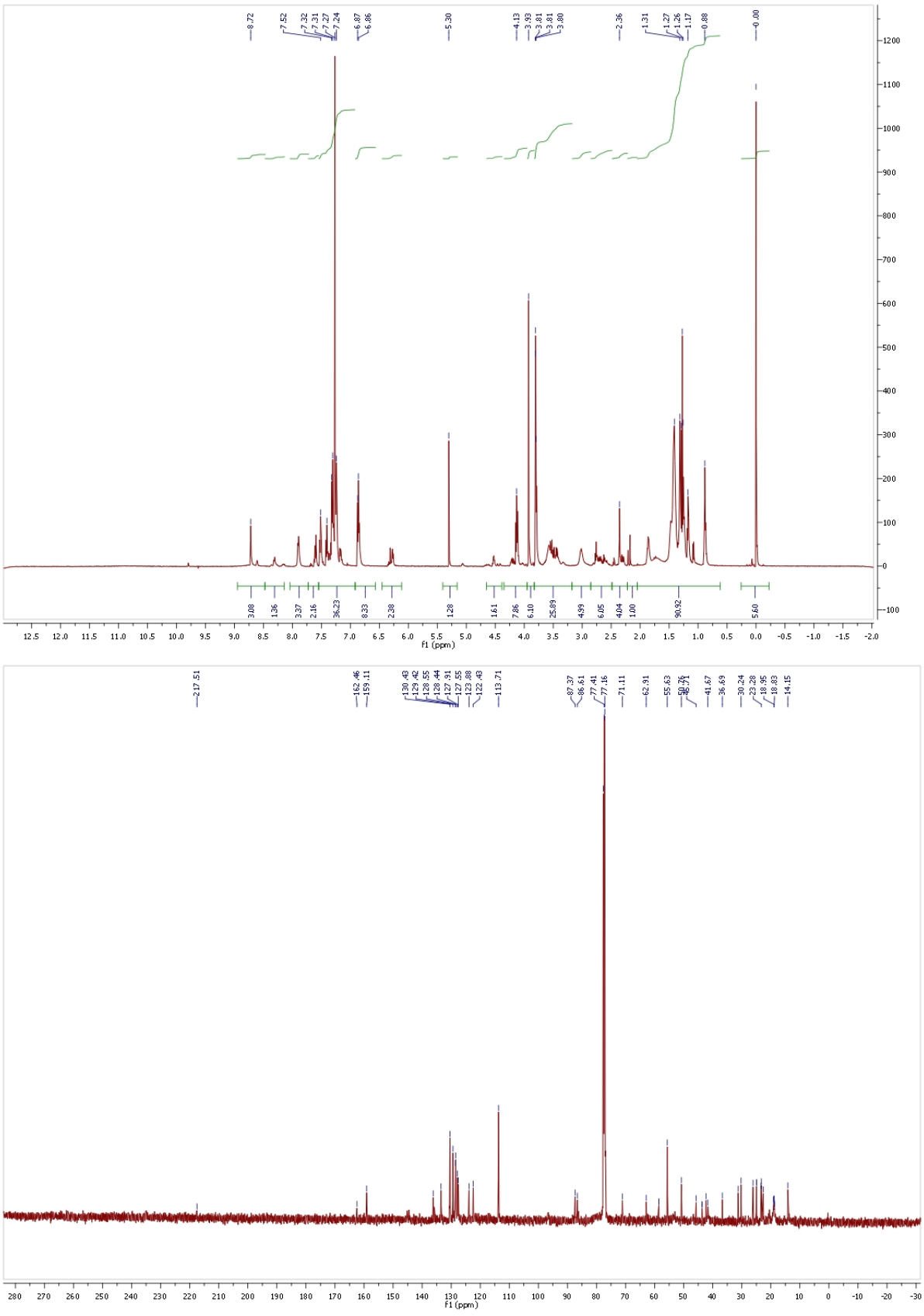
f1 (ppm)

1b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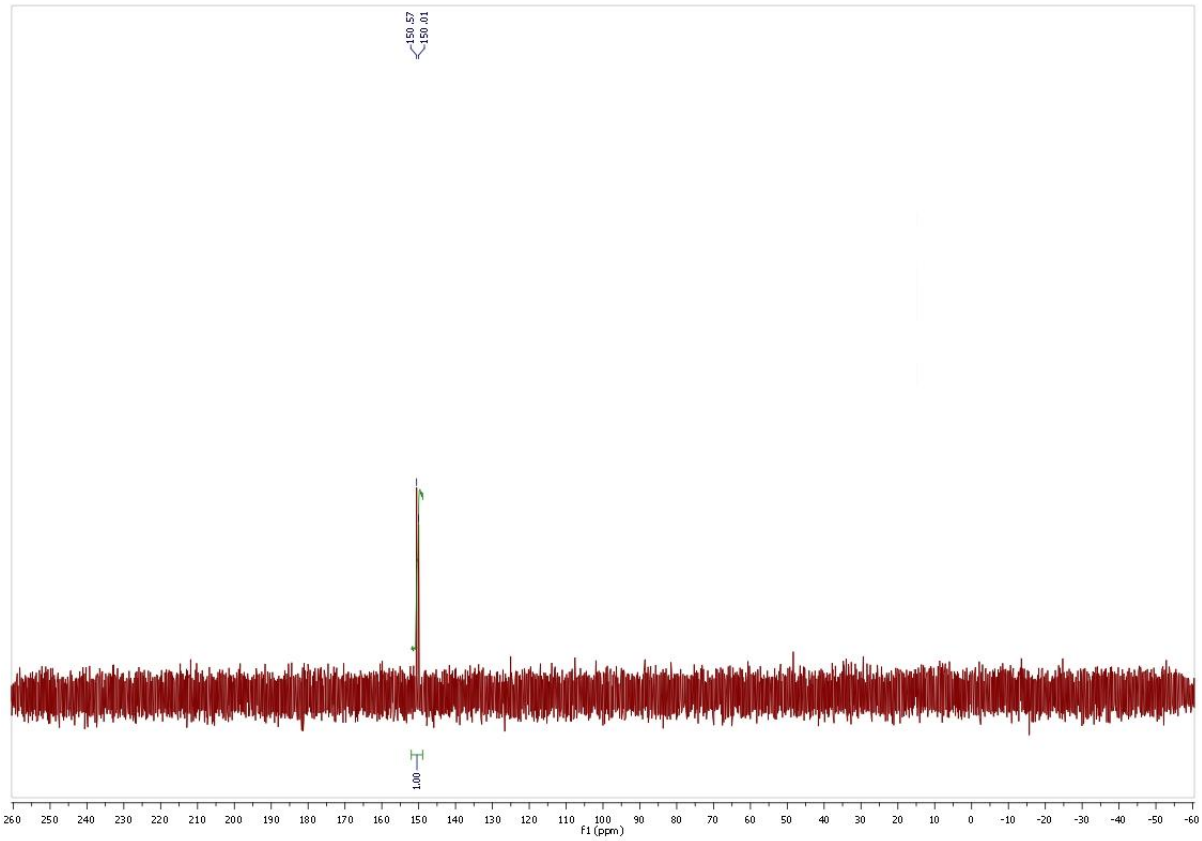
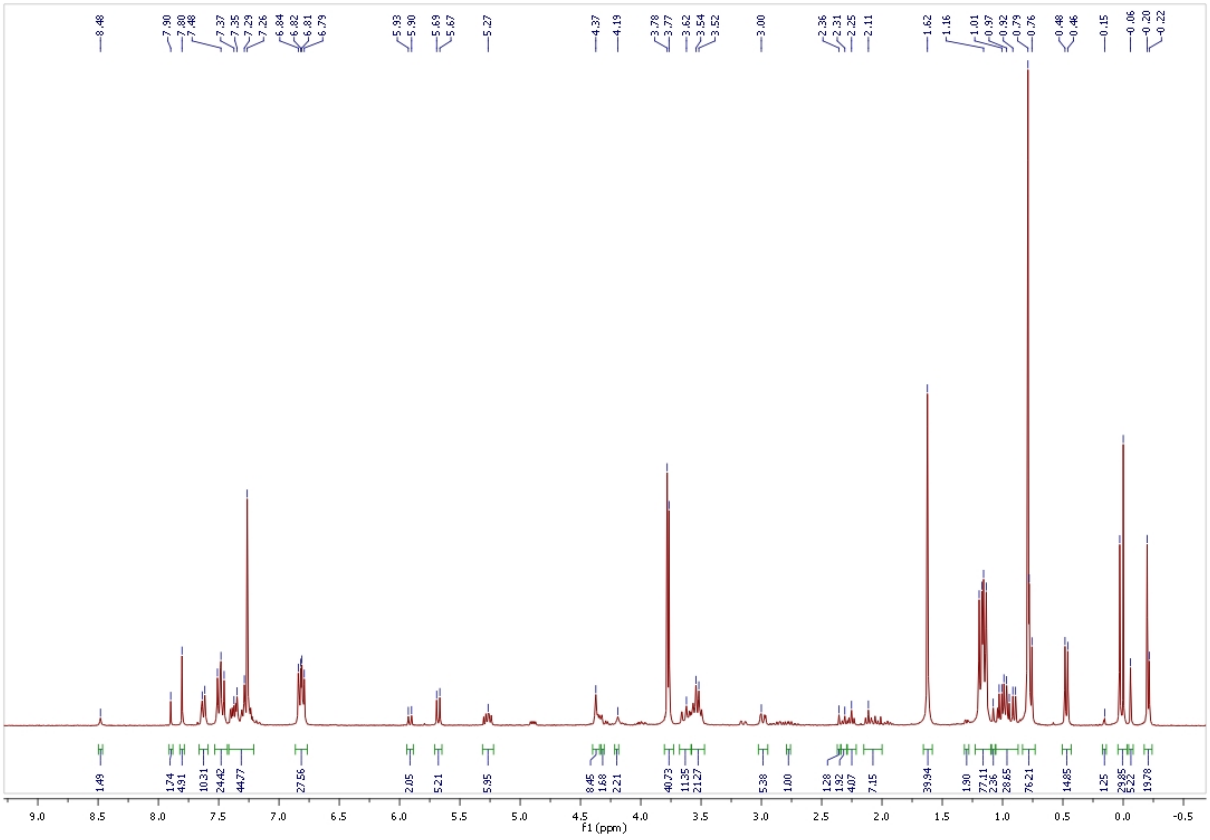
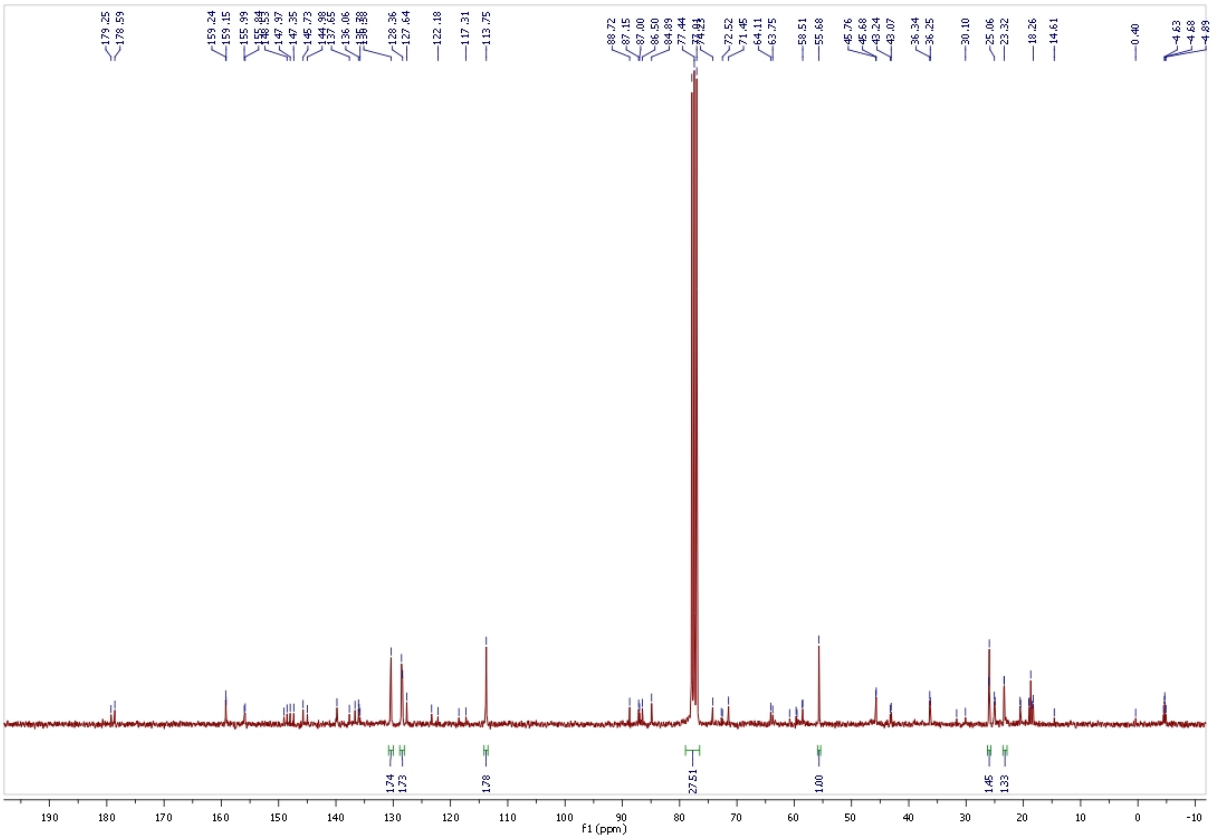
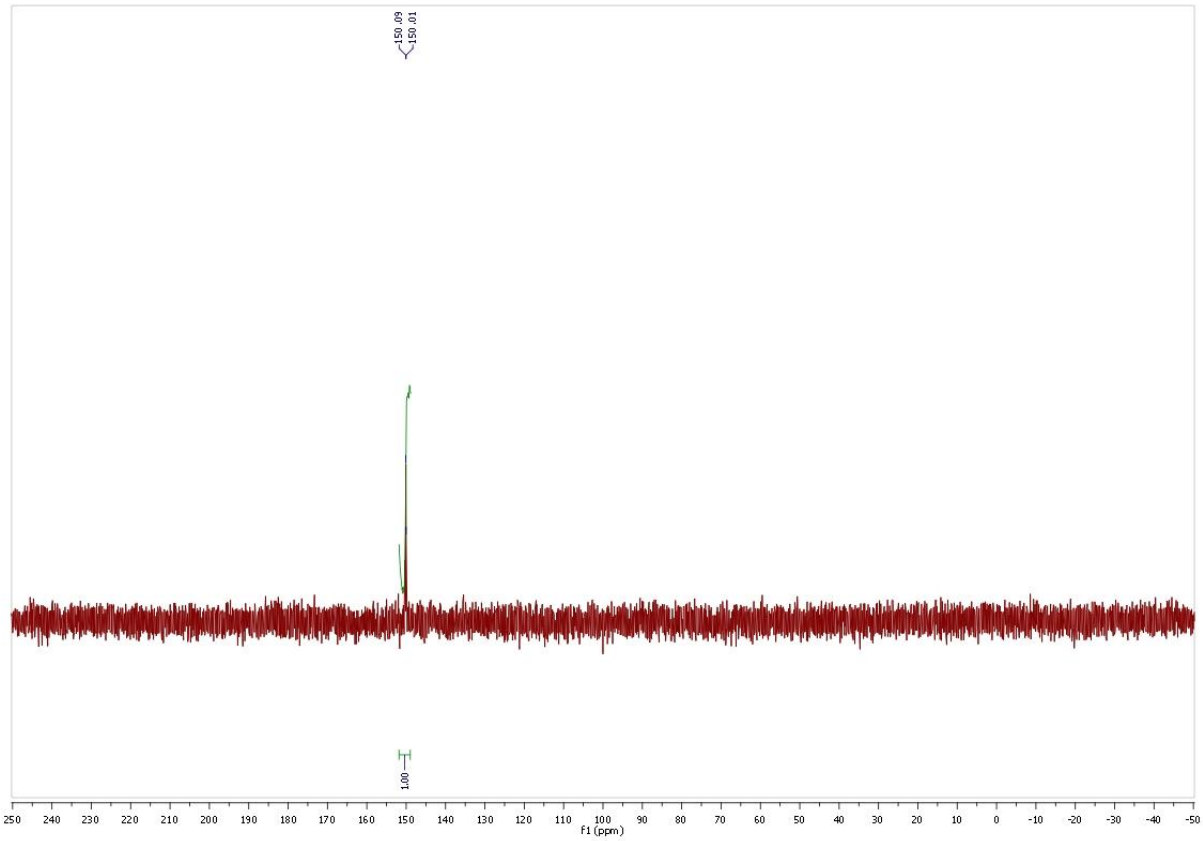
7.52
7.32
7.31
7.27
7.24
6.87
6.86
5.30
4.13
3.93
3.81
3.81
3.80
2.36
1.31
1.27
1.26
1.17
0.88
0.00
f1 (ppm)
217.51
162.46
159.11
130.43
129.42
128.55
128.44
127.91
127.55
123.88
122.43
113.71
97.37
86.61
77.41
77.16
71.11
62.91
55.63
41.67
36.69
30.24
23.28
18.95
18.83
14.15
f1 (ppm)


150.57
150.01
1.00
260 250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60
f1 (ppm)

1c



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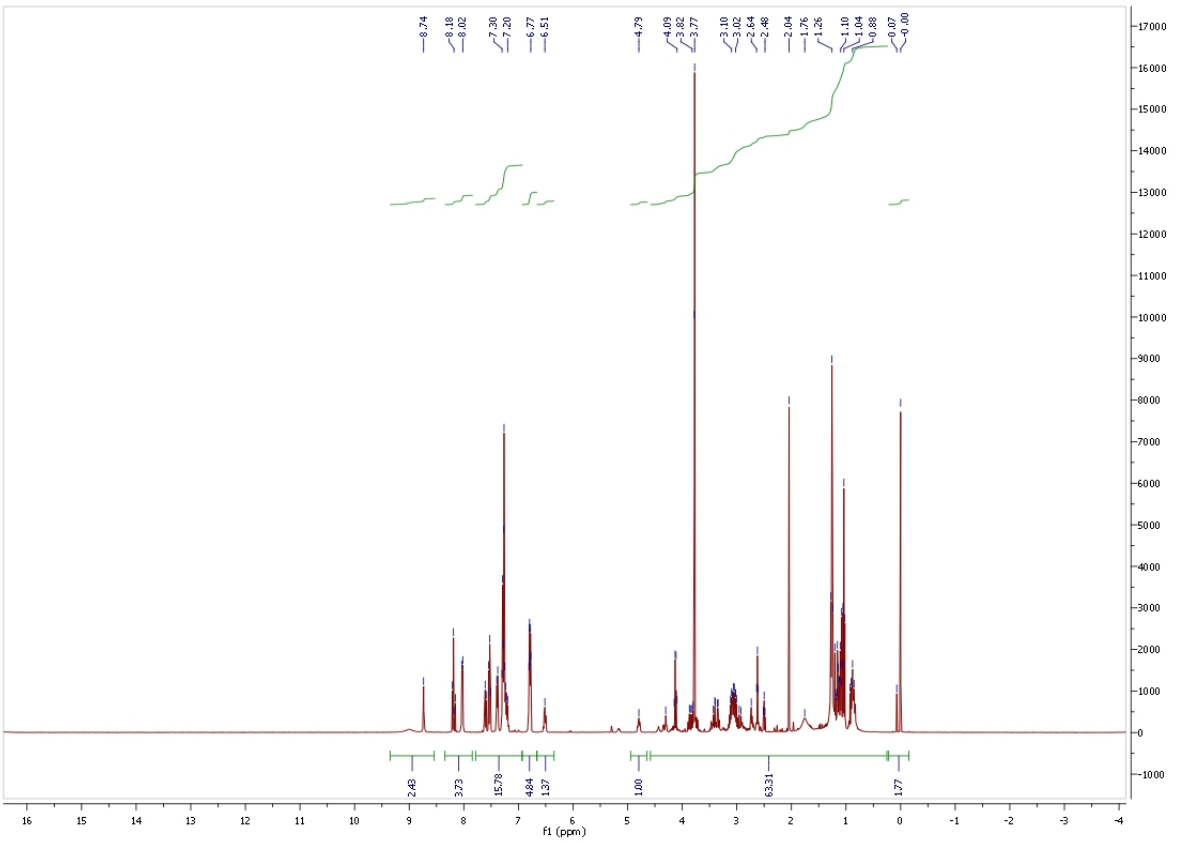
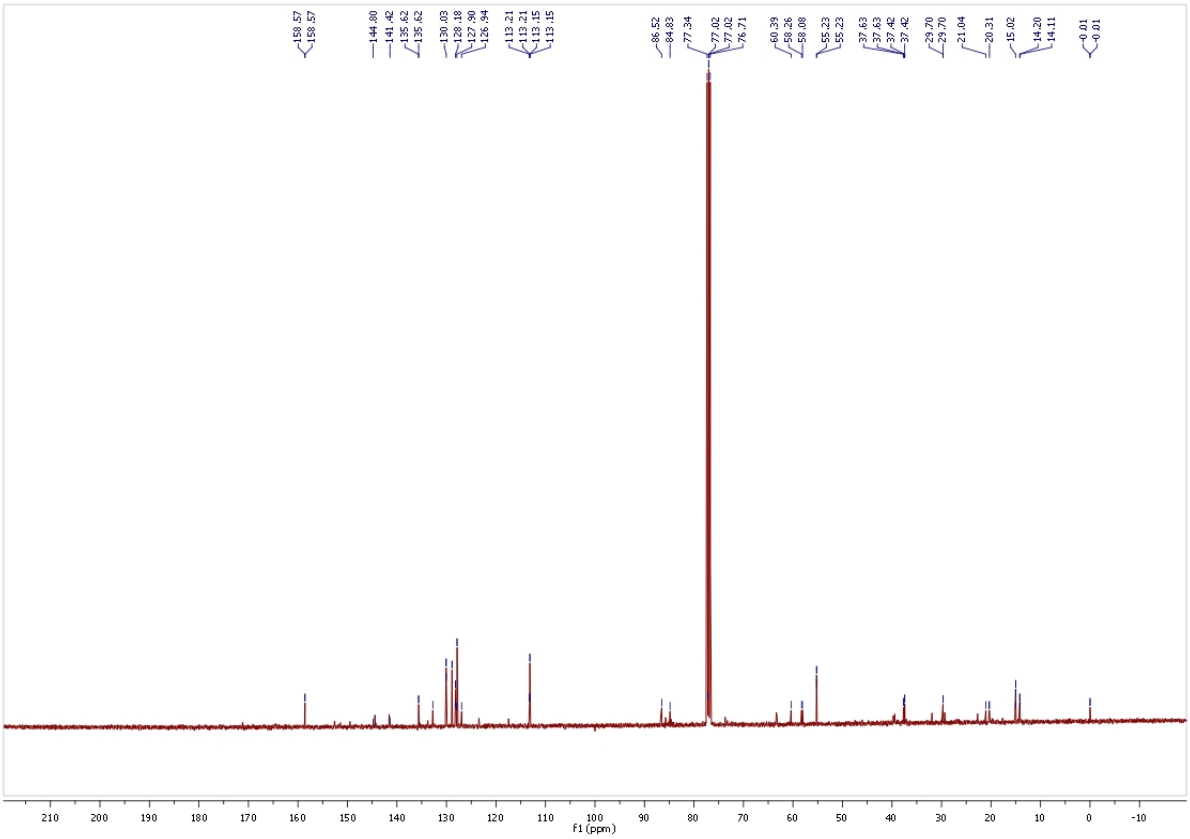
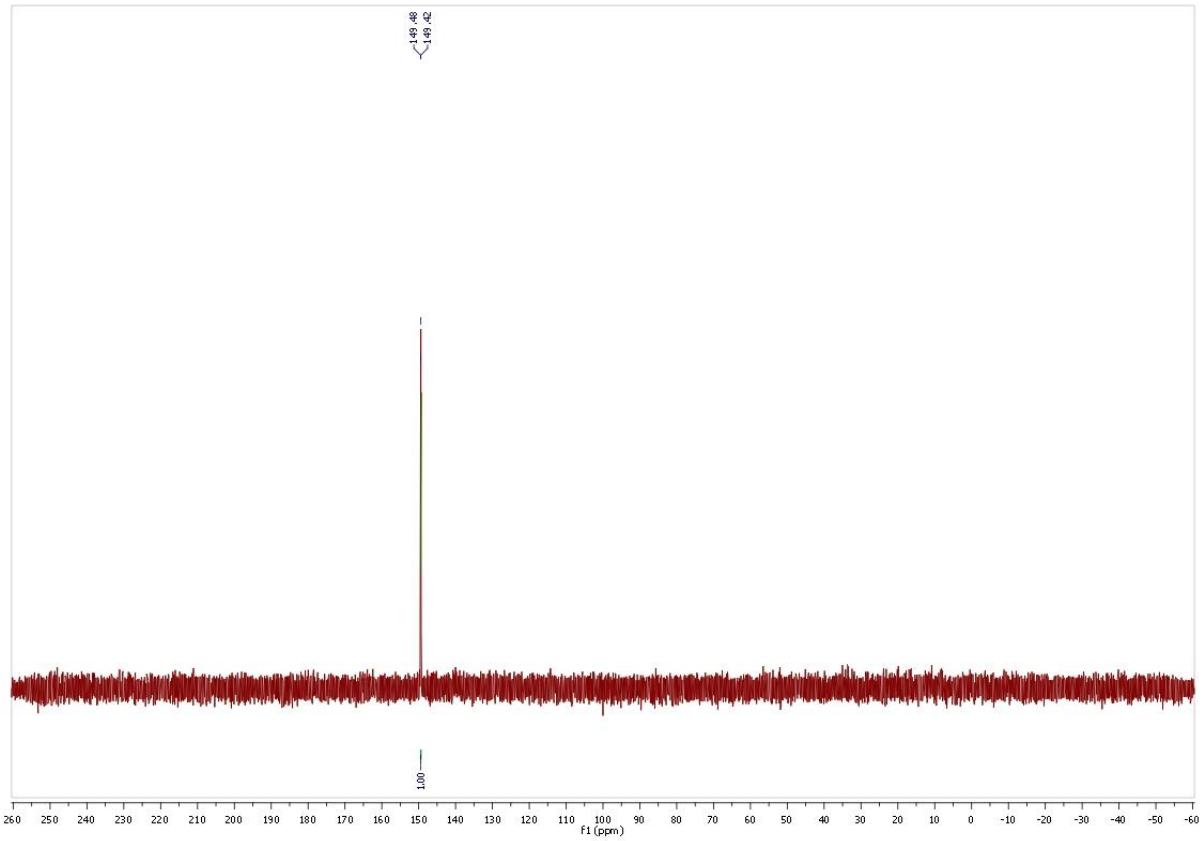
150.01
100
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50
f1 (ppm)

2a


8.74
8.18
8.02
7.30
7.20
6.77
6.51
4.79
4.09
3.82
3.77
3.10
3.02
2.64
2.48
2.04
1.76
1.26
1.10
1.04
0.88
0.07
0.00
2.49
3.73
15.78
4.84
1.37
1.00
63.31
1.77
f1 (ppm)


158.57
158.57
144.80
141.42
135.62
135.62
130.03
128.18
127.90
126.94
113.21
113.21
113.15
113.15
86.52
84.83
77.34
77.02
77.02
76.71
60.39
58.26
58.08
55.23
55.23
37.63
37.63
37.42
37.42
29.70
29.70
21.04
20.31
15.02
14.20
14.11
0.01
0.01
f1 (ppm)


149.48
149.42
1.00
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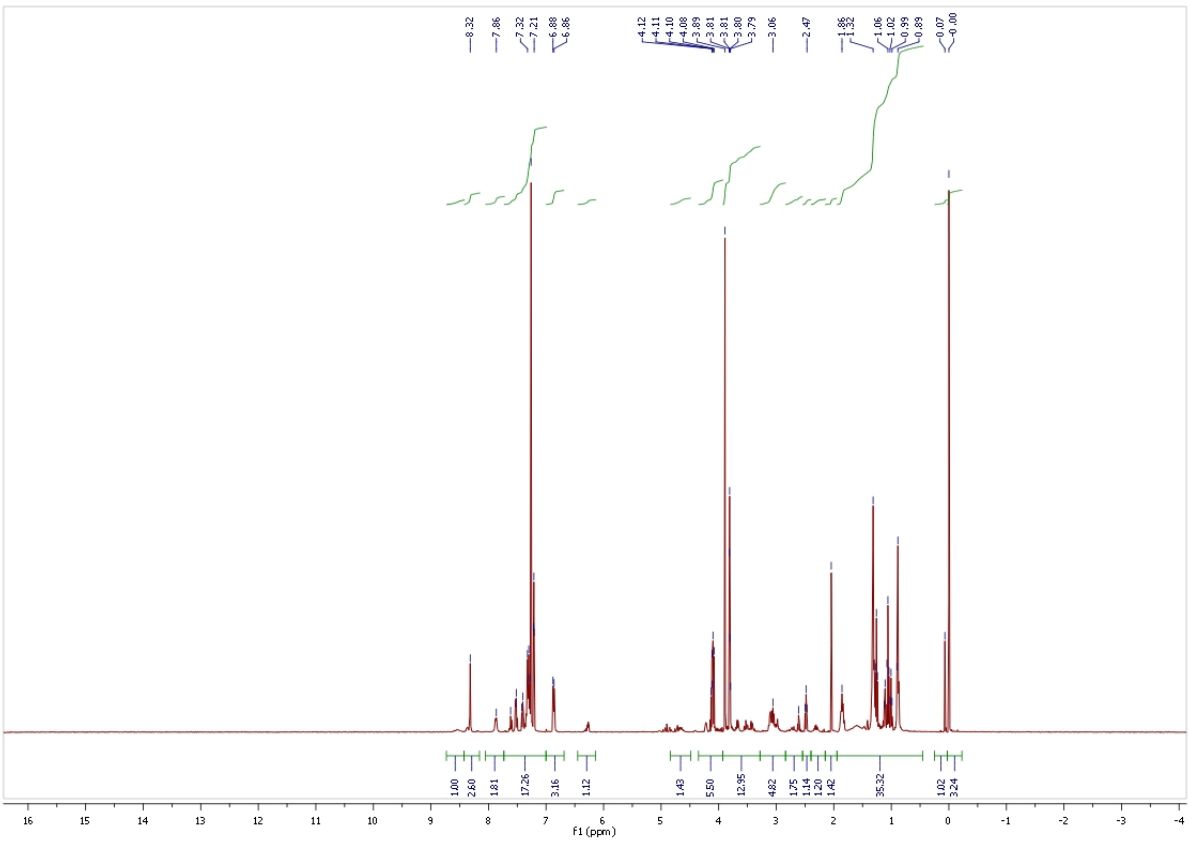
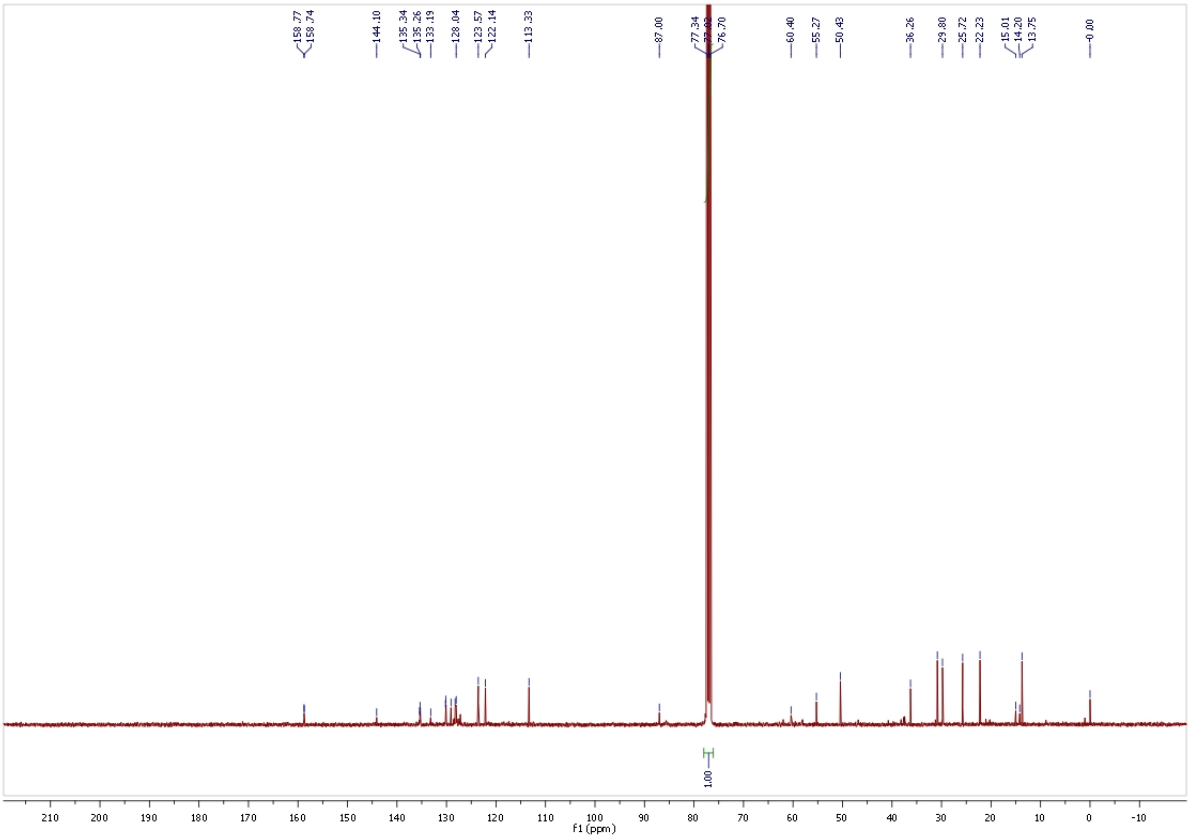
f1 (ppm)

2b


8.32
7.86
7.32
7.21
6.88
6.86
4.12
4.11
4.10
4.08
3.89
3.81
3.81
3.80
3.79
3.06
2.47
1.86
1.32
1.06
1.02
0.99
0.89
0.07
0.00
1.00
2.60
1.81
17.26
3.16
1.12
1.43
5.50
12.95
4.82
1.75
1.14
1.20
1.42
35.32
1.02
3.24
f1 (ppm)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158.74
144.10
135.34
135.26
133.19
128.04
123.57
122.14
113.33
87.00
77.34
77.00
76.70
60.40
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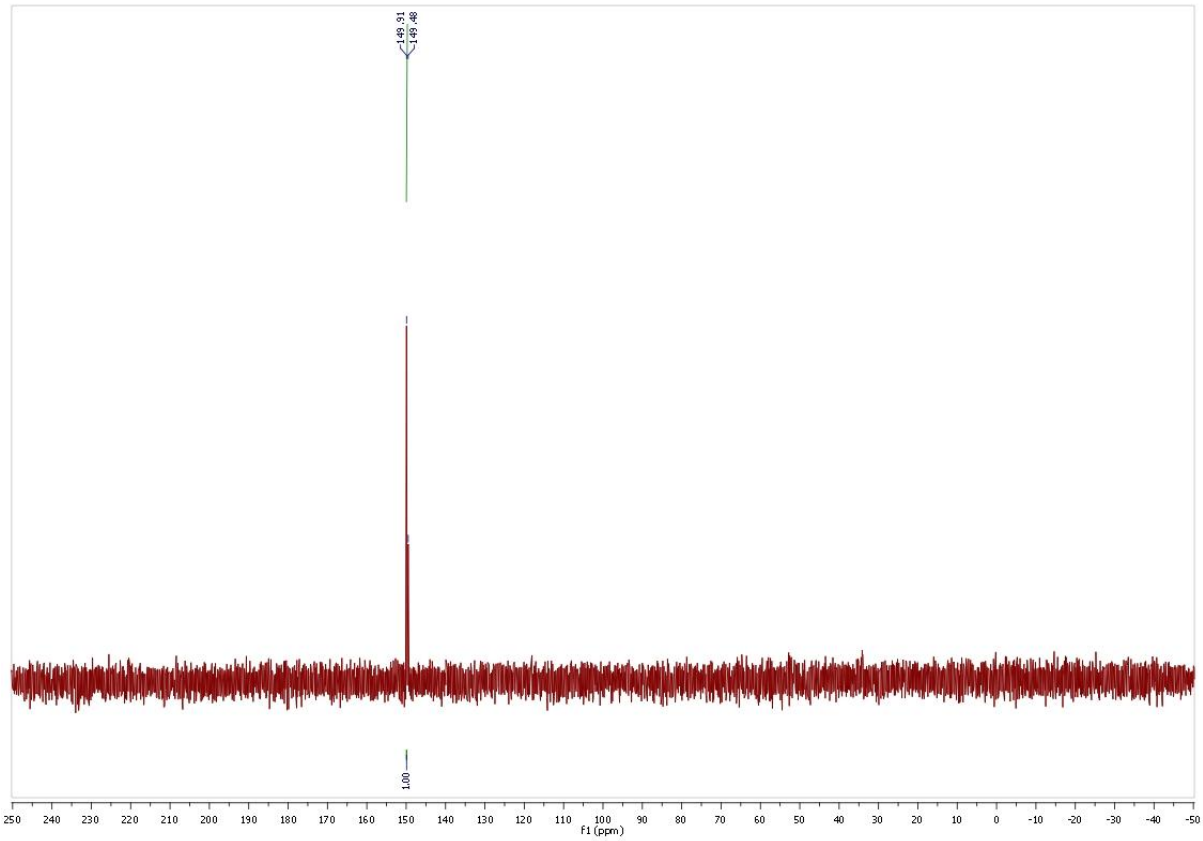
14.20
13.75
0.00
1.00
f1 (ppm)


149.91
149.48
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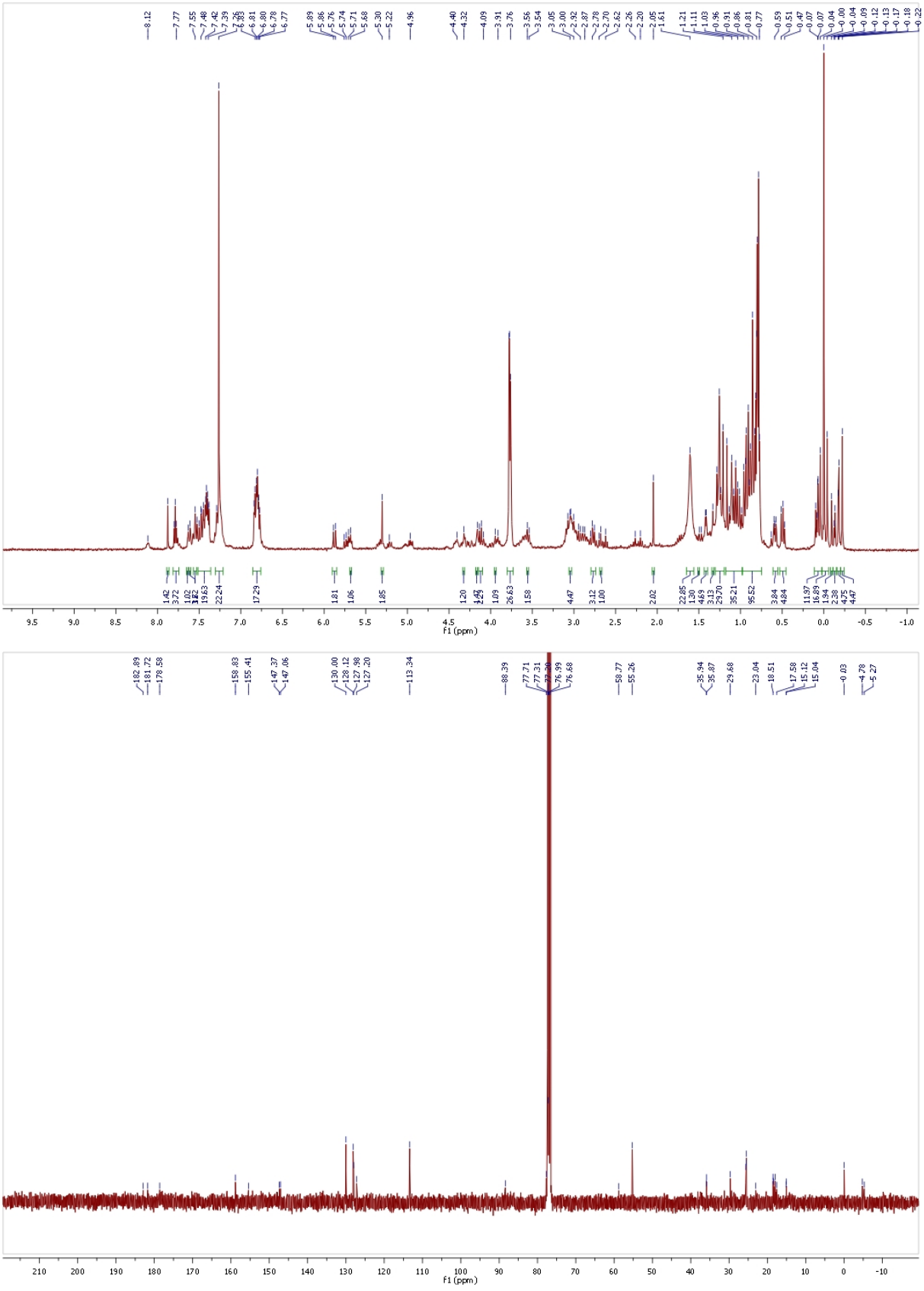
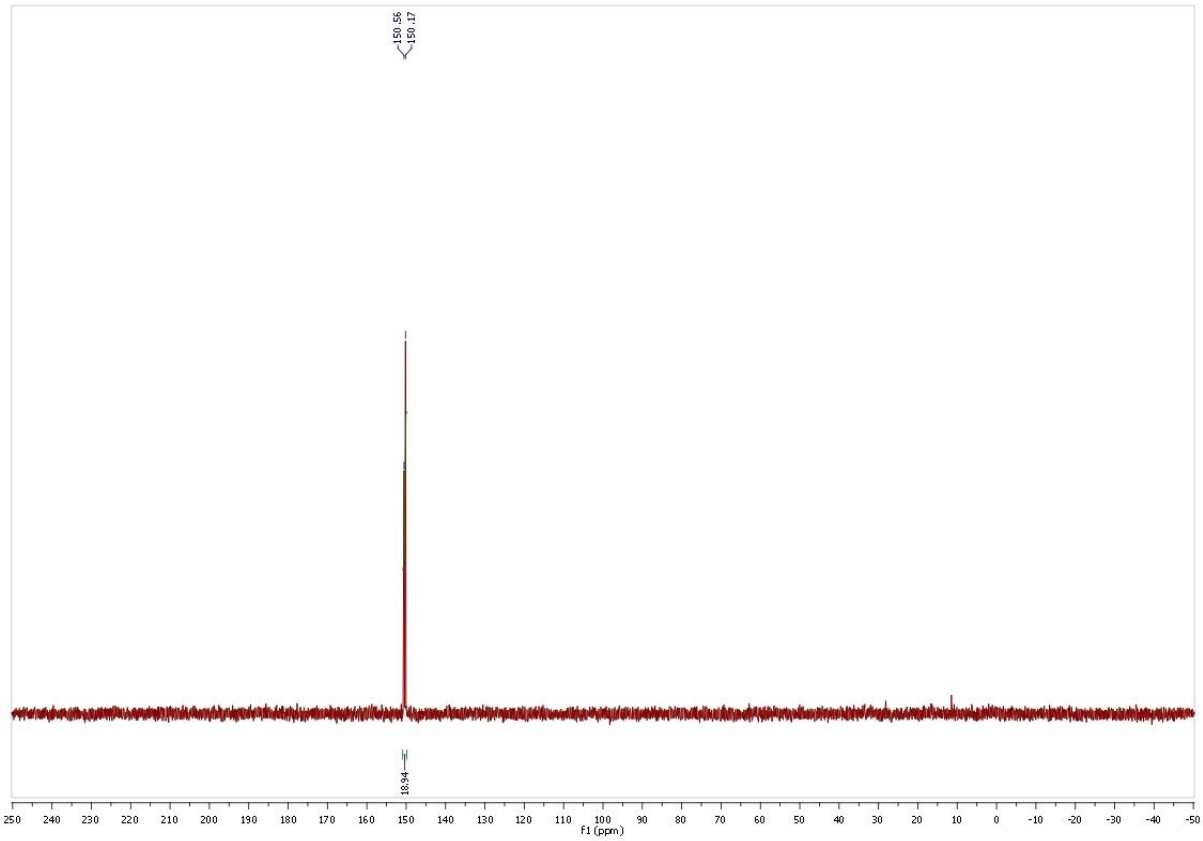
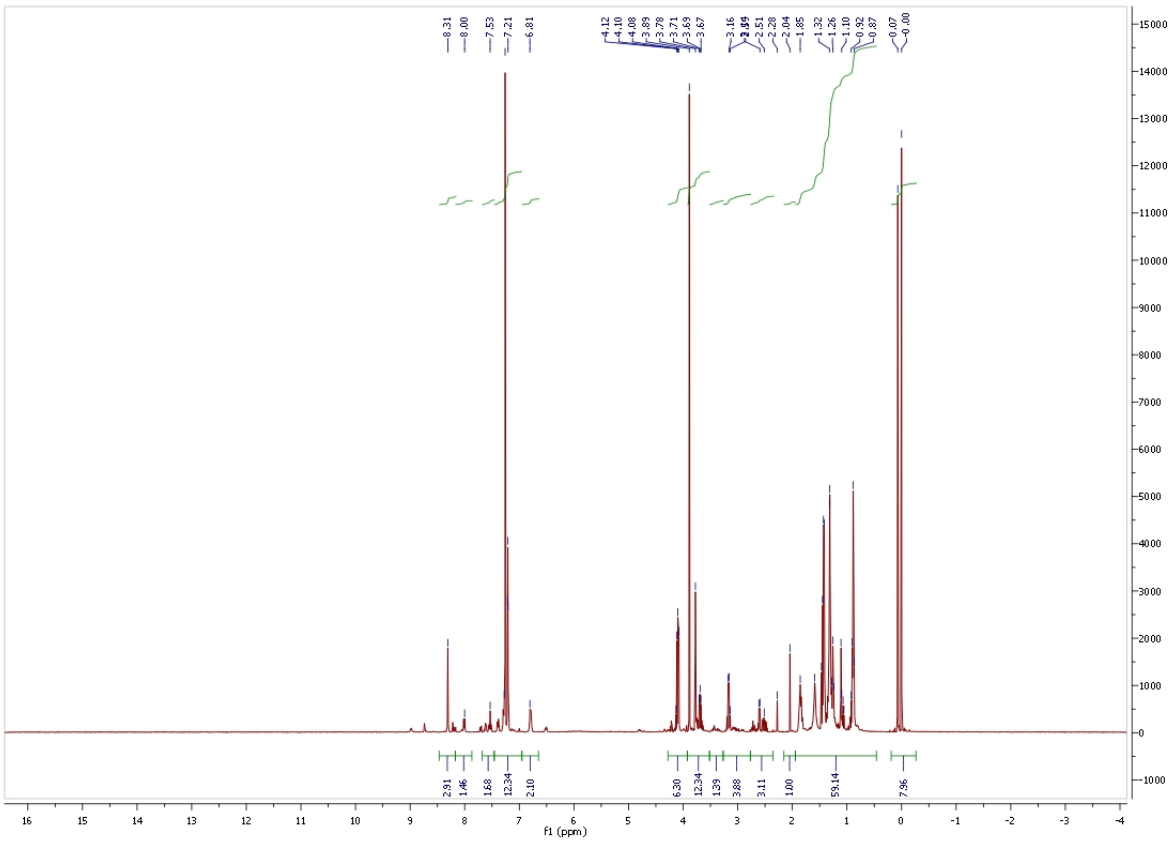
f1 (ppm)

2c




150.56
150.17
18.94
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50
f1 (ppm)

3a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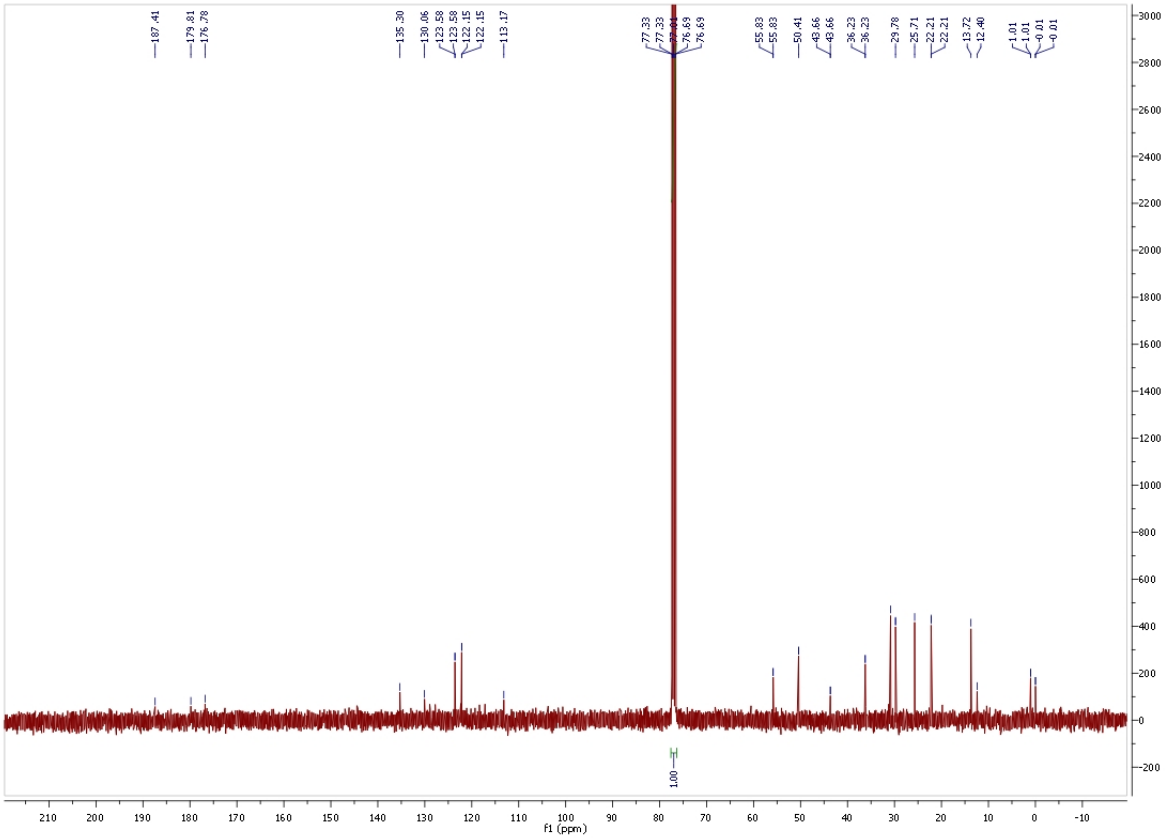
4.12
4.10
4.08
3.89
3.78
3.71
3.69
3.67
3.16
2.51
2.28
2.04
1.85
1.32
1.26
1.10
0.92
0.87
0.07
0.00
2.91
1.46
1.68
12.34
2.10
6.30
12.34
1.39
3.88
3.11
1.00
59.14
7.96
f1 (ppm)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130.06
123.58
123.58
122.15
122.15
113.17
77.33
77.33
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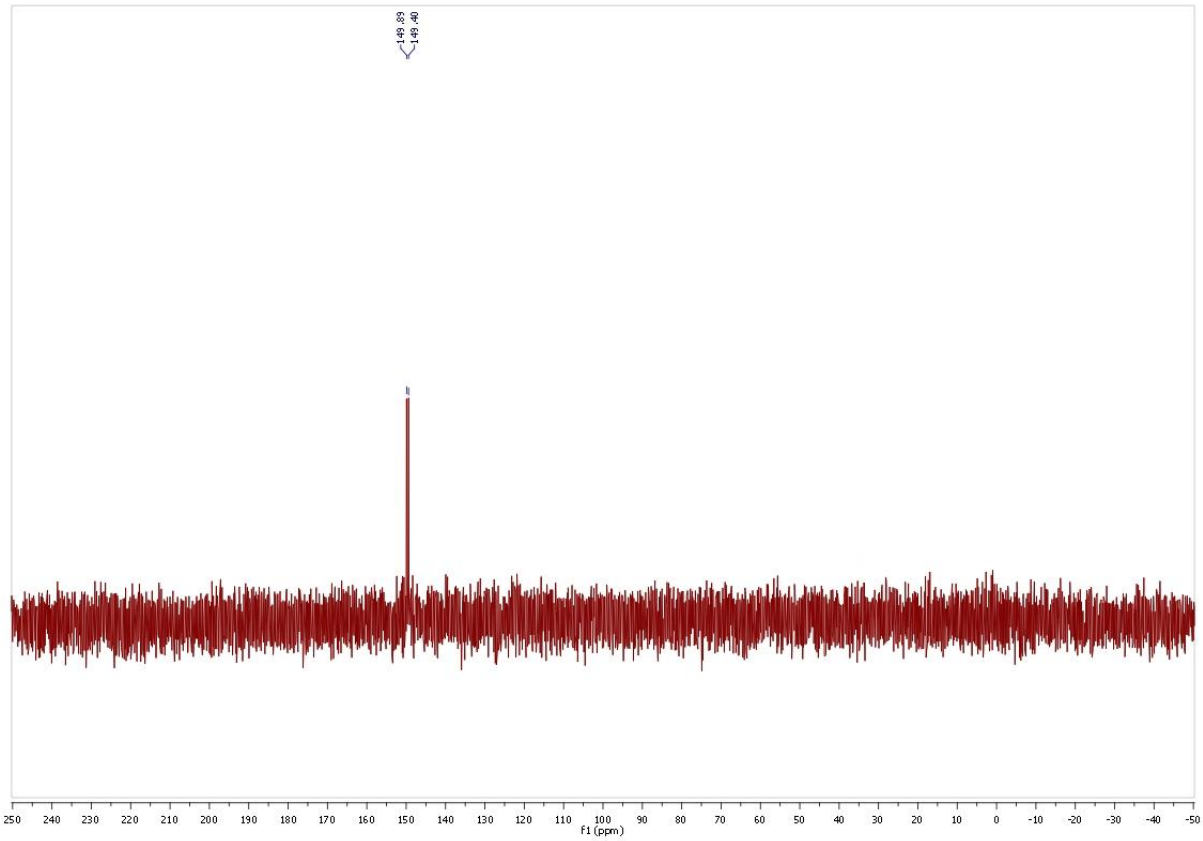
76.69
55.83
55.83
50.41
43.66
43.66
36.23
36.23
29.78
25.71
22.21
22.21
13.72
12.40
1.01
1.01
0.01
0.01
1.00
f1 (ppm)


149.89
149.40
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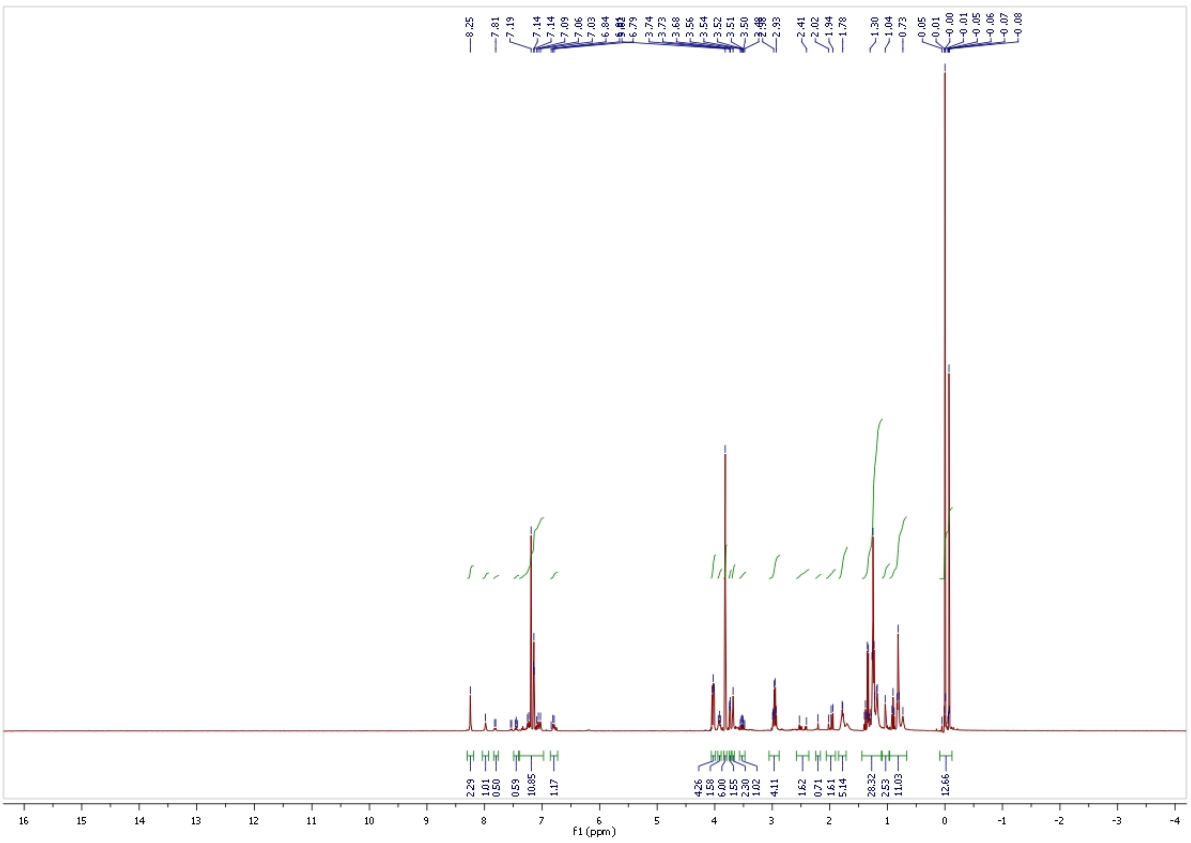
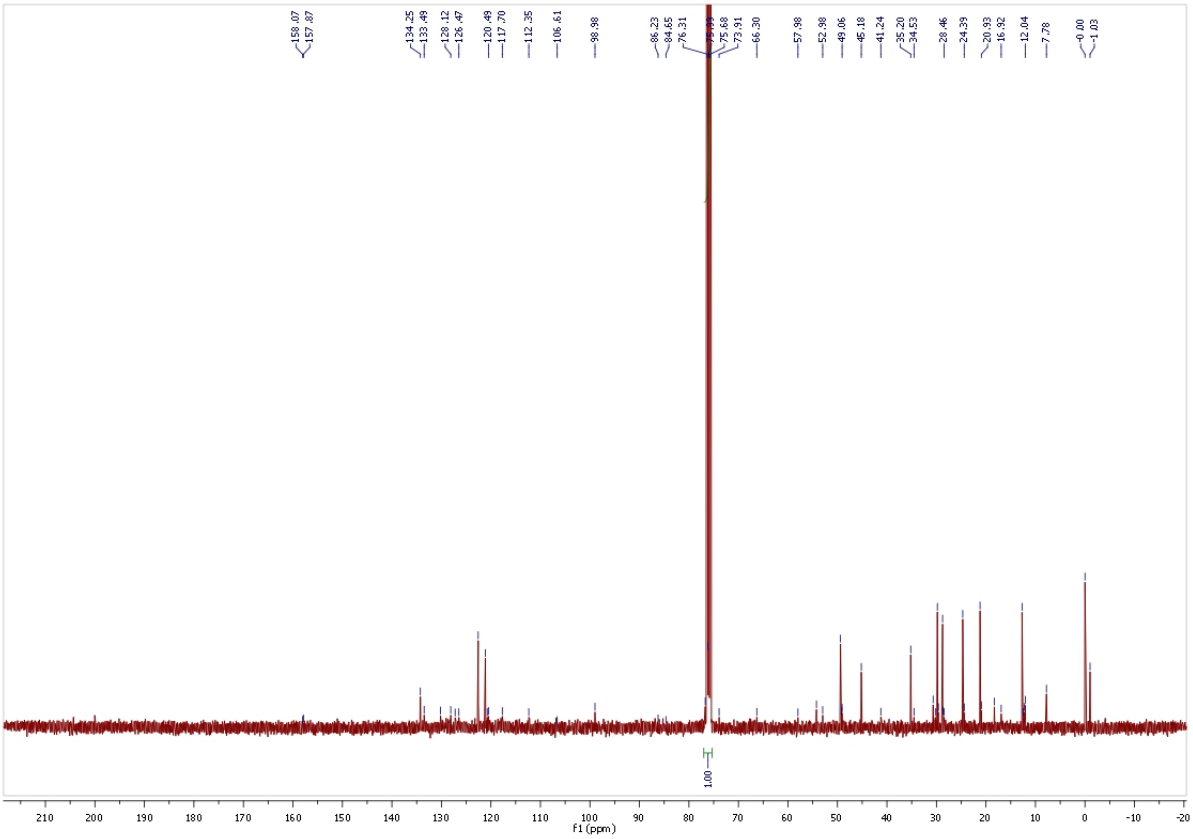
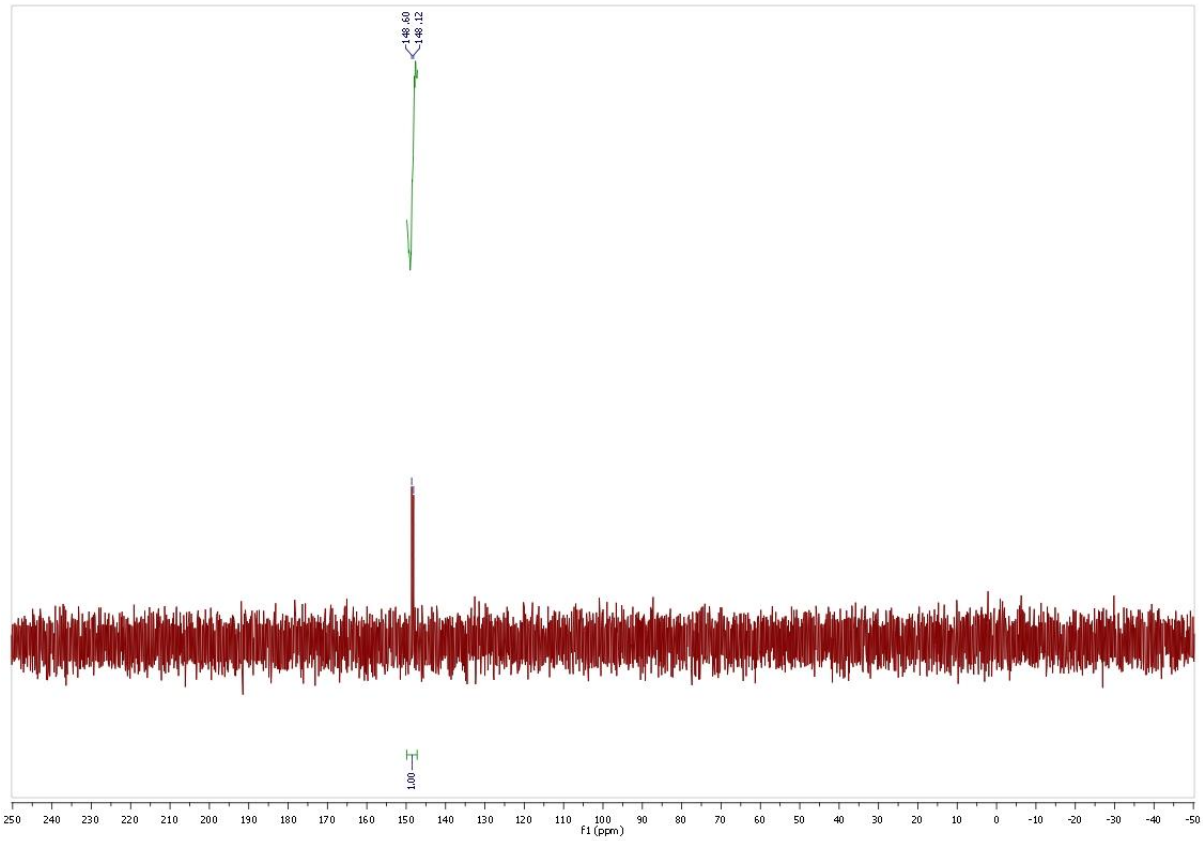
f1 (ppm)

3b






148.60
148.12
1.00
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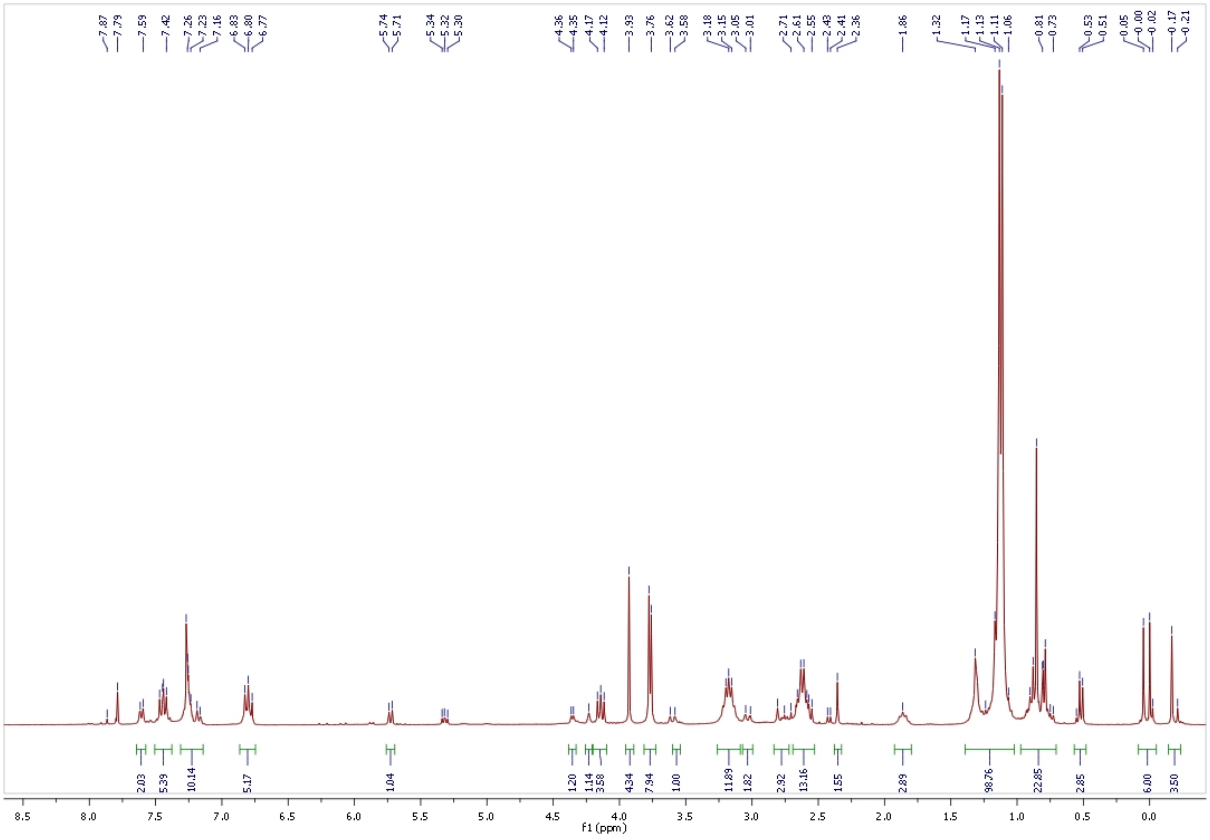
f1 (ppm)

3c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7.23
7.16
6.83
6.80
6.77
5.74
5.71
5.34
5.32
5.30
4.36
4.35
4.17
4.12
3.93
3.76
3.62
3.58
3.18
3.15
3.05
3.01
2.71
2.61
2.55
2.43
2.41
2.36
1.86
1.32
1.17
1.13
1.11
1.06
0.81
0.73
0.53
0.51
0.05
0.00
-0.02
-0.17
-0.21
2.03
5.39
10.14
5.17
1.04
1.20
1.14
3.58
4.34
7.94
1.00
11.89
1.82
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13.16
1.55
2.89
98.76
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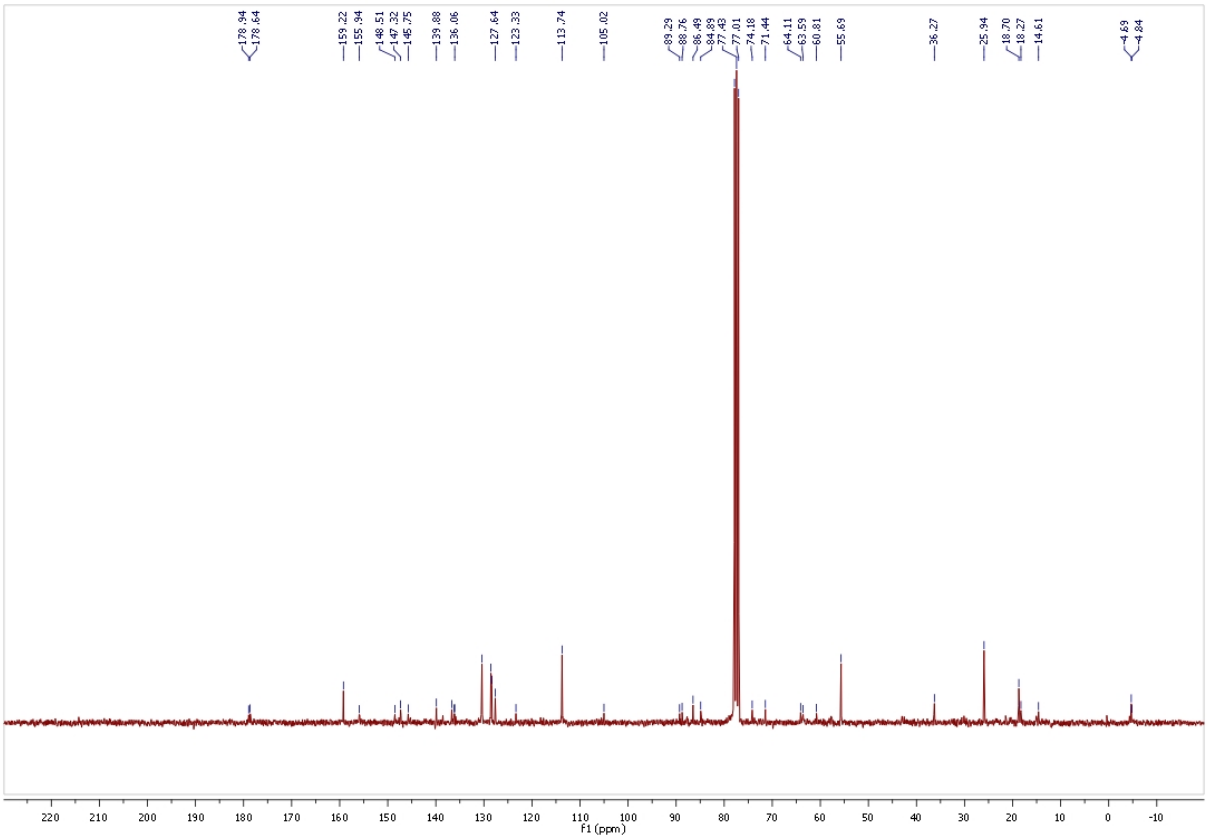
2.85
6.00
3.50
f1 (ppm)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155.94
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139.88
136.06
127.64
123.33
113.74
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88.76
86.49
84.89
77.43
77.01
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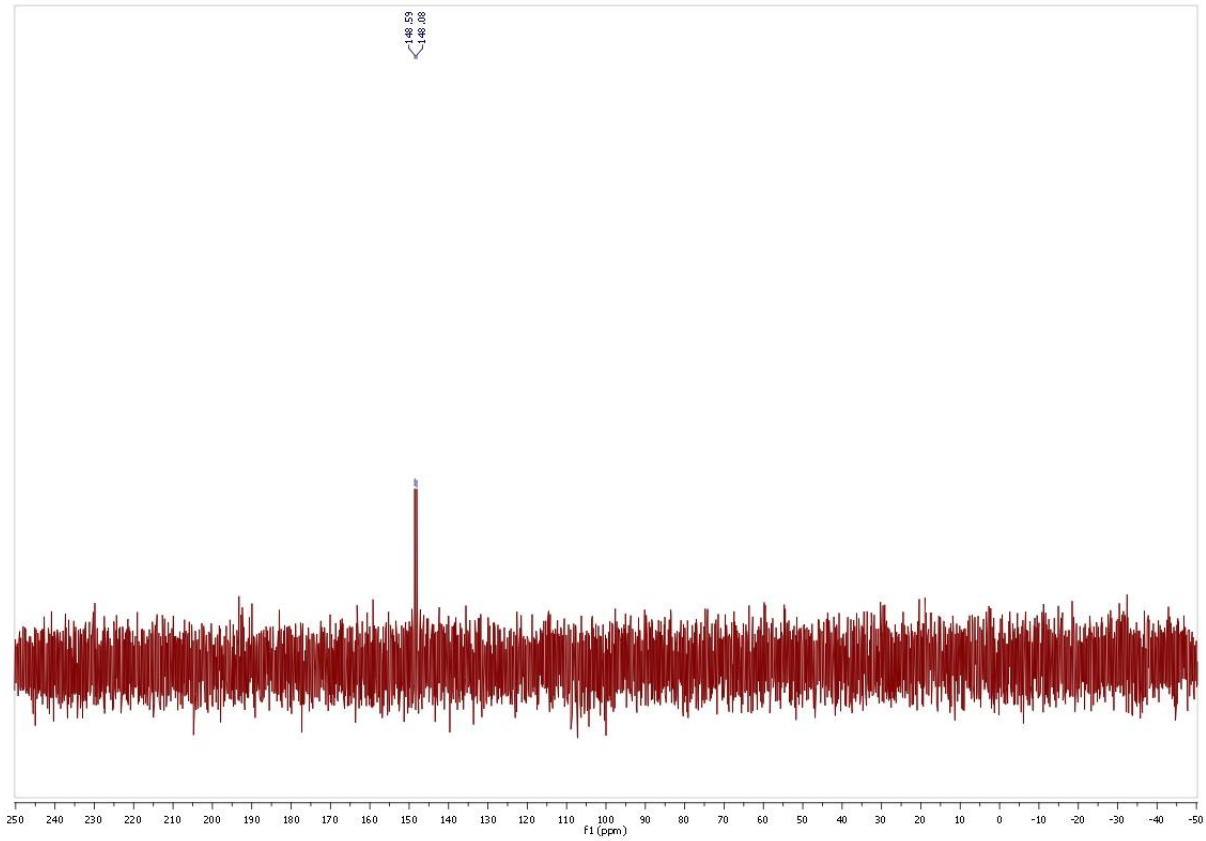
18.27
14.61
4.69
4.84
f1 (ppm)



References

[1] P. Bonhôte, A. Dias, N. Papageorgiou, K. Kalyanasundaram and M. Grätzel, *Inorg. Chem.,* **1996**, 35, 1168–1178.