Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Stable and reactive diacetyliminoxyl radical in oxidative C–O coupling with β-dicarbonyl compounds and their complexes

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General

In all experiments RT stands for 22-25 °C. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AVANCE II 300 and Bruker Fourier 300HD (300.13 MHz for ^{1}H and 75.47 MHz for ^{13}C, respectively) spectrometers in $CDCl_3$. Chemical shifts were reported in parts per million(ppm), and the residual solvent peak was used as an internal reference: ^{1}H ($CDCl_3$ δ = 7.26 ppm), ^{13}C ($CDCl_3$ δ = 77.16 ppm). Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet). Coupling constants were reported in Hertz (Hz).

Ethyl 2-methylacetoacetate 95%, iron(III) perchlorate hydrate reagent grade ($Fe(ClO_4)_3 \cdot nH_2O$, Alfa Aesar, anhydrous basis purity ca. 65%), copper(II) hexafluoroacetylacetonate hydrate, copper(II) perchlorate hexahydrate 98%, copper(II) acetate 99%, nickel(II) acetate tetrahydrate 99%, cobalt(II) acetate tetrahydrate 98+%, cobalt(II) perchlorate hexahydrate 99%, manganese(II) acetate tetrahydrate 99%, manganese(III) acetate dihydrate 97%, $BF_3 \cdot Et_2O$, trifluoroacetic acid 99%, 4-dimethylaminopyridine 99%, 1,4-diazabicyclo[2.2.2]octane 97%, 2,6-lutidine 99%, potassium *tert*-butoxide 98+%, 2-acetylcyclopentanone 98%, 3-chloro-2,4-pentanedione 98% were used as is from commercial sources. CH_2Cl_2, DMSO, and pyridine were distilled prior to use. Glacial acetic acid was used as is from commercial sources. 3-Methylpentane-2,4-dione **1b**, 3-*n*-butylpentane-2,4-dione (for synthesis of copper complex **3d**), 3-benzylpentane-2,4-dione (for synthesis of copper complex **3c**), 2-methyl-1-phenylbutane-1,3-dione **1l**, 3-phenylpentane-2,4-dione **1m**, 2-methyl-1,3-diphenylpropane-1,3-dione **1n**, 2-benzyl-1,3-diphenylpropane-1,3-dione **1o**, ethyl 2-methyl-3-oxo-3-phenylpropanoate **1i**, ethyl 2-acetylpent-4-enoate **1j** and ethyl 2-benzyl-3-oxobutanoate **1k** were prepared as described in literature.[1–8] Anhydrous copper(II) hexafluoroacetylacetonate ($Cu(hfacac)_2$) was prepared from corresponding hydrate according the literature.[9] Iron (III) and nickel (II) acetylacetonates were prepared according literature procedures.[10,11] Copper (II) complexes **4b-h** were prepared according literature procedures.[12–17] It should be noted that diacetyliminoxyl radical is unstable in the neat state. Diacetyliminoxyl solutions in CH_2Cl_2 were prepared according published procedure[8]. To a stirred solution of diacetyl oxime (258 mg, 2 mmol) in 4 mL of CH_2Cl_2 was added $Pb(OAc)_4$ (469 mg, 1.0 mmol) with vigorous stirring. Stirring was continued for 10 min, then the reaction mixture was chromatographed on silica gel using CH_2Cl_2 as eluent. The fraction corresponding to the dark-red spot was collected, so that the volume of the fraction was 50 mL. For the reactions in Table 1 and Table 2 solutions were evaporated to an approximate volume of 15 mL. Transfer of diacetyliminoxyl from CH_2Cl_2 to DMSO was achieved by the addition of 15 mL of DMSO to a solution of the diacetyliminoxyl in 50 mL of CH_2Cl_2 followed by water-jet vacuum vacuum evaporation of the latter. A MeCN solution of diacetyliminoxyl radical was prepared by threefold co-evaporation of the solution of diacetyliminoxyl radical in 50 mL of CH_2Cl_2 with acetonitrile (30 mL) to an approximate volume of 20 mL.

Cyclic voltammetry of diacetyliminoxyl radical, di-*tert*-butyliminoxyl radical, and TEMPO

Cyclic voltammetry (CV) was implemented on an IPC-Pro M computer-assisted potentiostat manufactured by Econix (scan rate error 1.0%; potential setting 0.25 mV; scan rate 100 mV/s). The experiments were performed in a 25 mL five-neck glass conic electrochemical cell with a water jacket for thermostatting. CV curves (Fig. S1, S2) were recorded using a three-electrode scheme. The working electrode was a disc glassy-carbon electrode (d = 3 mm). A platinum wire served as an auxiliary electrode. An $Ag/AgNO_3$ in 0.1M *n*-Bu_4NBF_4/MeCN electrode which was linked to the solution under study by a porous glass diaphragm was used as the reference electrode. The solutions were kept under thermally controlled conditions at 25±0.5 °C and deaerated by bubbling argon. The working electrode was polished before recording each CV curve. In a typical case, 20 mL of 0.1M Bu_4NBF_4 solution in MeCN was utilized, the compound concentration was 0.05M. The initial potentials for di-*tert*-butyliminoxyl and diacetyliminoxyl radicals were set at 700 mV, for TEMPO – 0 mV. All the CV curves are composed of two separately recorded parts (cathodic and anodic region) in order to avoid unwanted side oxidation and reduction reactions.

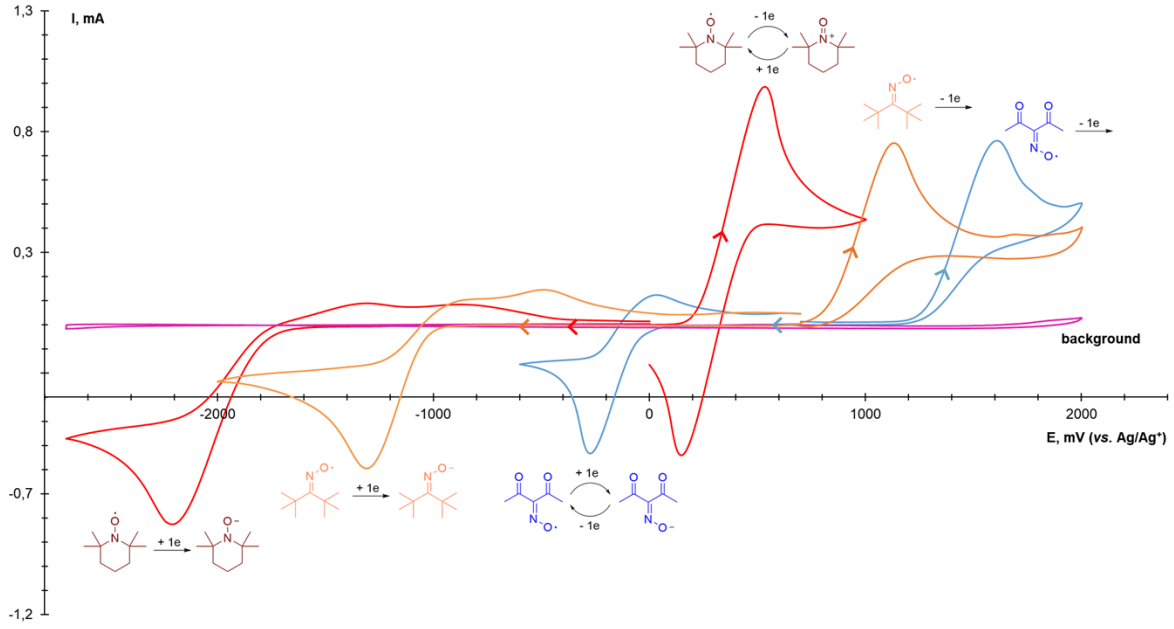


Fig. S1 CV-curves of 0.05M solutions of diacetyliminoxyl radical **2** (blue), di-*tert*-butyliminoxyl radical (orange) and TEMPO (red) in 0.1M Bu_4NBF_4 solution in MeCN on a working glassy-carbon electrode (d = 3 mm) under a scan rate of 0.1V/s at 298K.

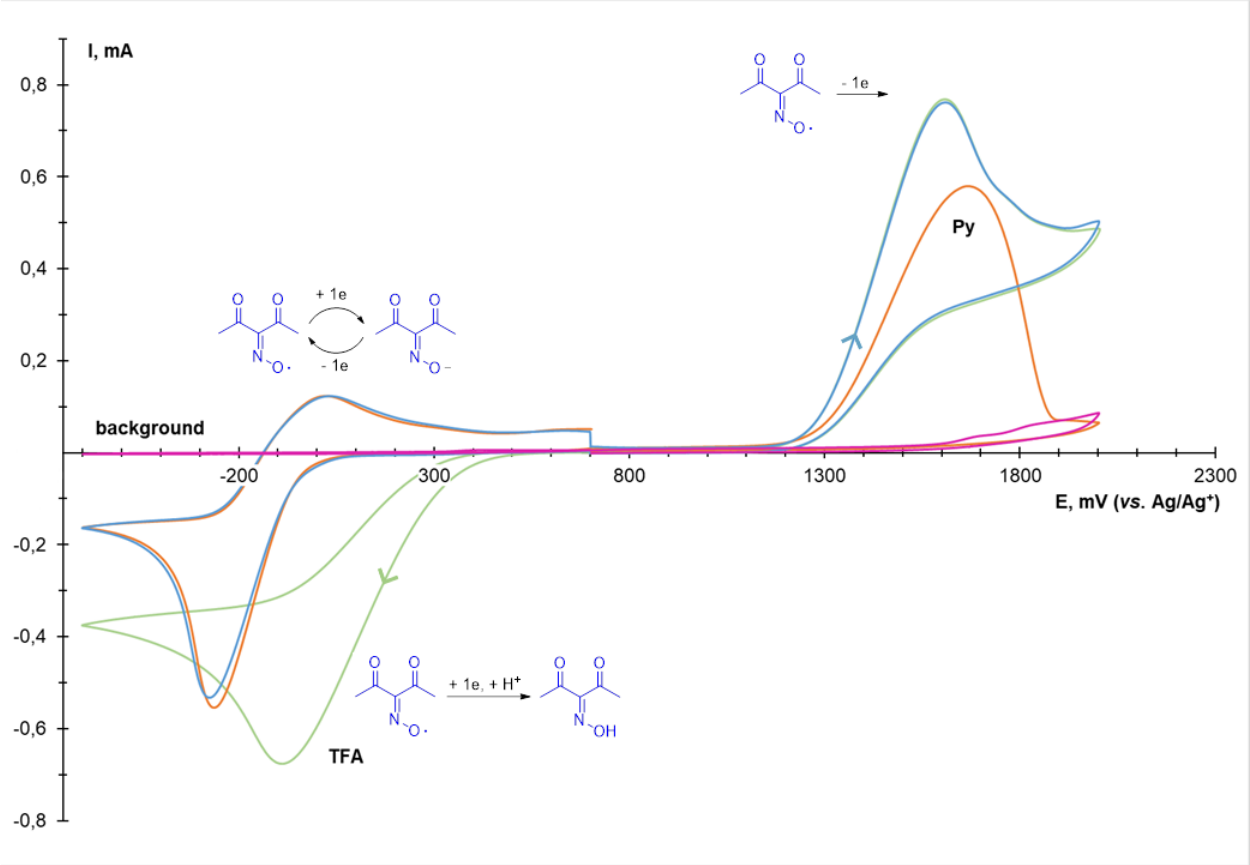


Fig. S2. CV-curves of 0.05M solutions of diacetyliminoxyl radical **2** (blue), **2** with 2 eq of pyridine (orange), **2** with 2 eq of TFA (green) in 0.1M Bu_4NBF_4 solution in MeCN on a working glassy-carbon electrode (d = 3 mm) under a scan rate of 0.1V/s at 298K.

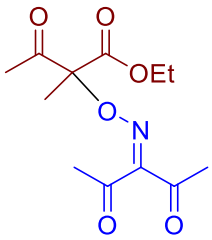
Experimental details and characterization data of synthesized compounds

^1H and ^{13}C NMR spectra of the synthesized compounds **3a-e,j,k** were in agreement with the literature data.[18,19]

Experimental details for Table 1.

Ethyl 2-methylacetoacetate **1a** (1 mmol, 144 mg) and an additive (0.5–1 mmol, 30–272 mg) were added to a stirred solution of diacetyliminoxyl radical **2** (2 mmol) in a solvent (15 mL, CH_2Cl_2 or DMSO) under air. The resulting solution was stirred at 23–25 °C for 24 hours. Then the reaction mixture was diluted with 0.05M solution (20 mL) of $Na_2S_2O_4$ and shaken. The organic layer was separated and washed with water (20 mL), dried over $MgSO_4$, and rotary evaporated under water-jet vacuum. In the case of DMSO reaction mixture was diluted with CH_2Cl_2 (10 mL), water (20 mL) and shaken. The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (2×10 mL), and all organic extracts were combined. Organic extract was washed with 0.05M solution of $Na_2S_2O_4$ (20 mL), water (20 mL), dried over $MgSO_4$ and rotary

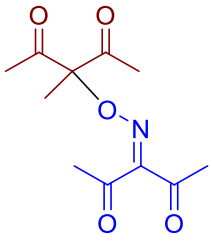
evaporated under water-jet vacuum. C–O coupling product **3a** was isolated by column chromatography on silica gel using the EtOAc/CH_2Cl_2 = 1/40 eluent.



Ethyl 2-(2,4-Dioxopentan-3-ylideneaminooxy)-2-methyl-3-oxobutanoate, 3a was synthesized as colorless oil.[18] **^{1}H NMR** (300.13 MHz, $CDCl_3$): δ = 4.33–4.11 (m, 2H), 2.35 (s, 3H), 2.29 (s, 3H), 2.19 (s, 3H), 1.67 (s, 3H), 1.23 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75.47 MHz, $CDCl_3$): δ = 201.2, 197.5, 193.8, 167.5, 157.4, 91.6, 62.5, 30.5, 25.9, 25.7, 19.4, 14.2.

Experimental details for Table 2.

3-Methyl-2,4-pentanedione **1b** (1 mmol, 114 mg) and an additive (0.5–1 mmol, 57–239 mg) were added to a stirred solution of diacetyliminoxyl radical **2** (2 mmol) in a solvent (15 mL, CH_2Cl_2 or DMSO) under air. The resulting solution was stirred at 23–25 °C for 24 hours. Then the reaction mixture was diluted with 0.05M solution (20 mL) of $Na_2S_2O_4$ and shaken. The organic layer was separated and washed with water (20 mL), dried over $MgSO_4$, and rotary evaporated under water-jet vacuum. In the case of DMSO reaction mixture was diluted with CH_2Cl_2 (10 mL), water (20 mL) and shaken. The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2 (2 × 10 mL), and all organic extracts were combined. Organic extract was washed with 20 mL 0.05M solution of $Na_2S_2O_4$, water (20 mL), dried over $MgSO_4$ and rotary evaporated under water-jet vacuum. C–O coupling product **3b** was isolated by column chromatography on silica gel using the EtOAc/CH_2Cl_2 = 1/40 eluent.



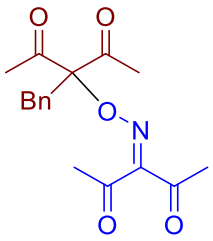
3-(((2,4-Dioxopentan-3-ylidene)amino)oxy)-3-methylpentane-2,4-dione, 3b was synthesized as yellow oil.[19] **^{1}H NMR** (300.13 MHz, $CDCl_3$): δ = 2.36 (s, 3H), 2.28 (s, 3H), 2.14 (s, 6H), 1.62 (s, 3H); **^{13}C NMR** (75.47 MHz, $CDCl_3$): δ = 201.9, 197.4, 193.6, 157.7, 96.5, 30.3, 26.0, 25.7, 19.0.

Experimental details for Table 3.

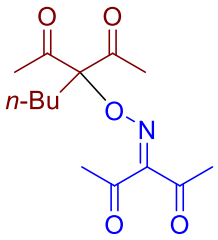
M(Meacac)$_n$ **4b, 4b', 4b''** (1 mmol, 284–395 mg) was added to a stirred solution of diacetyliminoxyl radical **2** (3 mmol) in CH_2Cl_2 (50 mL). The resulting solution was stirred at 23–25 °C for 1 hour under air. Then the reaction mixture was diluted with 0.05M solution of $Na_2S_2O_4$ (50 mL) and shaken. The organic layer was separated and washed with water (20 mL), dried over $MgSO_4$, and rotary evaporated under water-jet vacuum. Yields of **3b** were determined by ^{1}H NMR using 1,1,2,2-tetrachloroethane as an internal standard

Experimental details for Table 4.

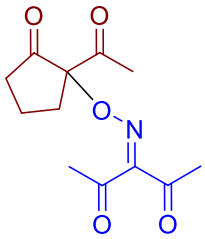
Cu(Racac)$_2$ **4b-h** (1 mmol, 261–441 mg) was added to a stirred solution of diacetyliminoxyl radical **2** (3 mmol) in CH_2Cl_2 (50 mL). The resulting solution was stirred at 23–25 °C for 1 hour. Then the reaction mixture was diluted with 0.05M solution of $Na_2S_2O_4$ (50 mL) and shaken. The organic layer was separated and washed with water (20 mL), dried over $MgSO_4$, and rotary evaporated under water-jet vacuum. C–O coupling products **3** were isolated by column chromatography on silica gel using the EtOAc/CH_2Cl_2 = 1/40 eluent or PE/EtOAc = 4/1 (in case of **3g**).



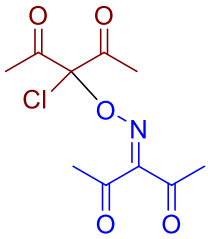
3-Benzyl-3-(((2,4-dioxopentan-3-ylidene)amino)oxy)pentane-2,4-dione, **3c** was synthesized as colorless oil.[19] **^{1}H NMR** (300.13 MHz, $CDCl_3$): δ = 7.36–7.18 (m, 3H), 7.09–6.95 (m, 2H), 3.51 (s, 2H), 2.40 (s, 3H), 2.26 (s, 3H), 2.02 (s, 6H). **^{13}C NMR** (75.47 MHz, $CDCl_3$): δ = 201.4, 197.3, 193.5, 158.1, 133.8, 130.2, 128.7, 127.6, 99.6, 39.1, 30.0, 27.4, 26.0.



3-Butyl-3-(((2,4-dioxopentan-3-ylidene)amino)oxy)pentane-2,4-dione, **3d** was synthesized as colorless oil.[18] **^{1}H NMR** (300.13 MHz, $CDCl_3$): δ = 2.43 (s, 3H), 2.31 (s, 3H), 2.24–2.11 (m, 8H), 1.38–1.22 (m, 2H), 1.22–1.08 (m, 2H), 0.86 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (75.47 MHz, $CDCl_3$): δ = 201.9, 197.7, 193.7, 157.7, 100.0, 32.9, 30.4, 27.0, 25.9, 25.2, 22.8, 13.8.



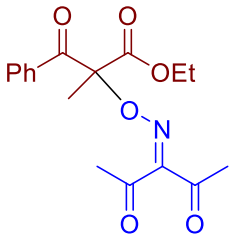
3-(((1-Acetyl-2-oxocyclopentyl)oxy)imino)pentane-2,4-dione, **3e** was synthesized as colorless oil.[18] **^{1}H NMR** (300.13 MHz, $CDCl_3$): δ = 2.74–2.57 (m, 1H), 2.51–2.14 (m, 3H), 2.37 (s, 3H), 2.33 (s, 3H), 2.24 (s, 3H), 2.13–1.96 (m, 2H). **^{13}C NMR** (75.47 MHz, $CDCl_3$): δ = 209.3, 203.3, 197.3, 193.6, 157.9, 95.9, 37.0, 31.9, 30.6, 26.7, 25.9, 18.2.



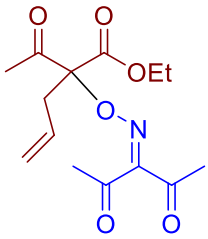
3-Chloro-3-(((2,4-dioxopentan-3-ylidene)amino)oxy)pentane-2,4-dione, 3g was synthesized as pale yellow oil. **^{1}H NMR** (300.13 MHz, $CDCl_3$): δ = 2.41 (s, 3H), 2.38 (s, 3H), 2.37 (s, 6H); **^{13}C NMR** (75.47 MHz, $CDCl_3$): δ = 195.9, 195.3, 193.0, 159.6, 101.6, 30.5, 26.2, 25.8. **FT-IR** (thin layer): ν_{max} = 1752, 1731, 1702, 1361, 1221, 846. **HR-MS (ESI)**: *m/z* = 262.0485, 264.0456, calcd. for $C_{10}H_{12}ClNO_5$+H^+: 262.0477, 264.0447.

Experimental details for Table 5.

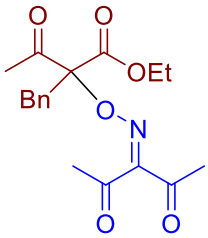
Dicarbonyl compound **1** (1 mmol, 114-314 mg) and an additive (0.5 mmol, 57–239 mg, except for additive-free experiments) were added to a stirred solution of diacetyliminoxyl radical **2** (2 mmol) in CH_2Cl_2 (15 mL). The resulting solution was stirred at 23–25 °C for 24 hours. Then the reaction mixture was diluted with 0.05M solution of $Na_2S_2O_4$ (20 mL) and shaken. The organic layer was separated and washed with water (20 mL), dried over $MgSO_4$, and rotary evaporated under water-jet vacuum. C–O coupling products **3** were isolated by column chromatography on silica gel.



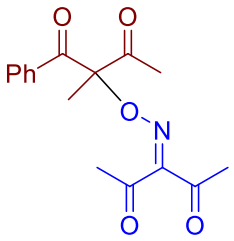
Ethyl 2-(((2,4-dioxopentan-3-ylidene)amino)oxy)-2-methyl-3-oxo-3-phenylpropanoate, 3i was isolated by column chromatography (PE/EtOAc = 4/1 eluent) as pale yellow oil. **^{1}H NMR** (300.13 MHz, $CDCl_3$): δ = 7.98–7.85 (m, 2H), 7.59–7.47 (m, 1H), 7.46–7.35 (m, 2H), 4.22 (q, *J* = 7.1 Hz, 2H), 2.31 (s, 3H), 2.16 (s, 3H), 1.91 (s, 3H), 1.16 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (75.47 MHz, $CDCl_3$): δ = 197.1, 193.7, 192.0, 168.3, 157.0, 133.9, 133.7, 129.4, 128.7, 90.7, 62.5, 30.5, 25.8, 21.0, 13.9; **FT-IR** (thin layer): ν_{max} = 1754, 1727, 1694, 1598, 1448, 1364, 1274, 1237, 1115, 986, 957, 697. **HR-MS (ESI)**: *m/z* = 356.1105, calcd. for $C_{17}H_{19}NO_6$+Na^+: 356.1105.



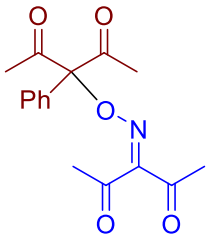
Ethyl 2-acetyl-2-(((2,4-dioxopentan-3-ylidene)amino)oxy)pent-4-enoate, 3j was was isolated by column chromatography (EtOAc/CH_2Cl_2 = 1/40 eluent) as pale yellow oil.[18] **^{1}H NMR** (300.13 MHz, $CDCl_3$): δ = 5.71–5.43 (m, 1H), 5.19–4.97 (m, 2H), 4.20 (q, *J* = 7.2 Hz, 2H), 2.92 (d, *J* = 7.1 Hz, 2H), 2.35 (s, 3H), 2.29 (s, 3H), 2.16 (s, 3H), 1.22 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (75.47 MHz, $CDCl_3$): δ = 200.7, 197.1, 193.5, 166.4, 157.4, 130.0, 120.5, 93.5, 62.4, 37.5, 30.3, 26.6, 25.8, 14.1.



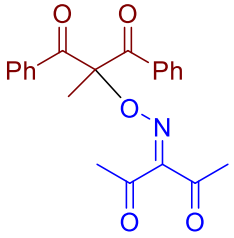
Ethyl 2-benzyl-2-(((2,4-dioxopentan-3-ylidene)amino)oxy)-3-oxobutanoate, 3k was isolated by column chromatography (EtOAc/CH_2Cl_2 = 1/40 eluent) as pale yellow oil.[19] **^{1}H NMR** (300.13 MHz, $CDCl_3$): δ = 7.25–7.15 (m, 3H), 7.09–6.98 (m, 2H), 4.30–4.10 (m, 2H), 3.51 (s, 2H), 2.34 (s, 3H), 2.18 (s, 3H), 2.00 (s, 3H), 1.21 (t, *J* = 7.1 Hz, 1H); **^{13}C NMR** (75.47 MHz, $CDCl_3$): δ = 201.4, 197.2, 193.5, 166.4, 157.4, 133.6, 130.3, 128.4, 127.4, 94.2, 62.4, 38.9, 30.0, 26.9, 25.8, 14.0.



3-(((2-Methyl-1,3-dioxo-1-phenylbutan-2-yl)oxy)imino)pentane-2,4-dione, 3l was isolated by column chromatography (PE/EtOAc = 4/1 eluent) as pale yellow solid. Mp = 71–73 °C. **^{1}H NMR** (300.13 MHz, $CDCl_3$): δ = 7.88–7.78 (m, 2H), 7.59–7.49 (m, 1H), 7.48–7.37 (m, 2H), 2.30 (s, 3H), 2.28 (s, 3H), 2.17 (s, 3H), 1.86 (s, 3H); **^{13}C NMR** (75.47 MHz, $CDCl_3$): δ = 202.0, 197.4, 194.0, 193.7, 157.3, 134.0, 133.8, 129.5, 128.8, 96.3, 30.6, 26.0, 25.8, 20.6. **FT-IR** (thin layer): ν_{max} = 1719, 1688, 1356, 1299, 1257, 1228, 1109, 971, 938, 700. **HR-MS (ESI)**: *m/z* = 304.1180, calcd. for $C_{16}H_{17}NO_5$+H^+: 304.1179.

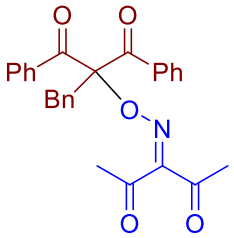


3-(((2,4-Dioxopentan-3-ylidene)amino)oxy)-3-phenylpentane-2,4-dione, 3m was was isolated by column chromatography (PE/EtOAc = 4/1 eluent) as pale yellow solid. Mp = 139–140 °C. **^{1}H NMR** (300.13 MHz, $CDCl_3$): δ = 7.47–7.37 (m, 3H), 7.35–7.27 (m, 2H), 2.54 (s, 3H), 2.32 (s, 3H), 2.20 (s, 6H); **^{13}C NMR** (75.47 MHz, $CDCl_3$): δ = 201.2, 197.6, 193.8, 157.6, 132.4, 129.7, 129.3, 126.3, 98.3, 30.4, 27.2, 26.0. **FT-IR** (thin layer): ν_{max} = 1737, 1721, 1691, 1354, 1298, 1210, 1198, 1176, 1049, 983, 931, 766, 702, 586. **HR-MS (ESI)**: *m/z* = 304.1178, calcd. for $C_{16}H_{17}NO_5$+H^+: 304.1179.



3-(((2-Methyl-1,3-dioxo-1,3-diphenylpropan-2-yl)oxy)imino)pentane-2,4-dione, 3n was was isolated by column chromatography (PE/EtOAc = 4/1 eluent) as pale yellow oil. **^{1}H NMR** (300.13 MHz, $CDCl_3$): δ = 7.97–7.83 (m, 4H), 7.60–7.48 (m, 2H), 7.48–7.33 (m, 4H), 2.16 (s, 6H),

2.09 (s, 3H); **^{13}C NMR** (75.47 MHz, $CDCl_3$): δ = 197.4, 194.2, 193.6, 157.4, 133.9, 133.8, 129.8, 128.8, 95.8, 30.4, 25.8, 22.2. **FT-IR** (thin layer): ν_{max} = 1726, 1703, 1597, 1366, 1273, 985, 970, 941, 697. **HR-MS (ESI)**: *m/z* = 388.1153, calcd. for $C_{21}H_{19}NO_5$+Na^+: 388.1155.



3-(((2-Benzyl-1,3-dioxo-1,3-diphenylpropan-2-yl)oxy)imino)pentane-2,4-dione, 3o was was isolated by column chromatography (PE/EtOAc = 4/1 eluent) as pale yellow oil. **^{1}H NMR** (300.13 MHz, $CDCl_3$): δ = 7.57–7.43 (m, 6H), 7.35–7.16 (m, 7H), 7.13–7.00 (m, 2H), 3.91 (s, 2H), 2.16 (s, 3H), 2.09 (s, 3H); **^{13}C NMR** (75.47 MHz, $CDCl_3$): δ = 197.4, 194.0, 193.7, 157.3, 134.6, 134.1, 133.6, 131.0, 129.7, 128.7, 128.5, 127.6, 99.4, 41.7, 30.1, 26.0. **FT-IR** (thin layer): ν_{max} = 1728, 1710, 1695, 1670, 1238, 936, 762, 693. **HR-MS (ESI)**: *m/z* = 442.1649, calcd. for $C_{27}H_{23}NO_5$+H^+: 442.1649.

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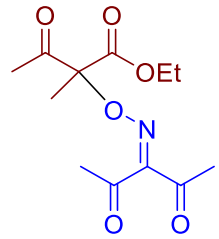
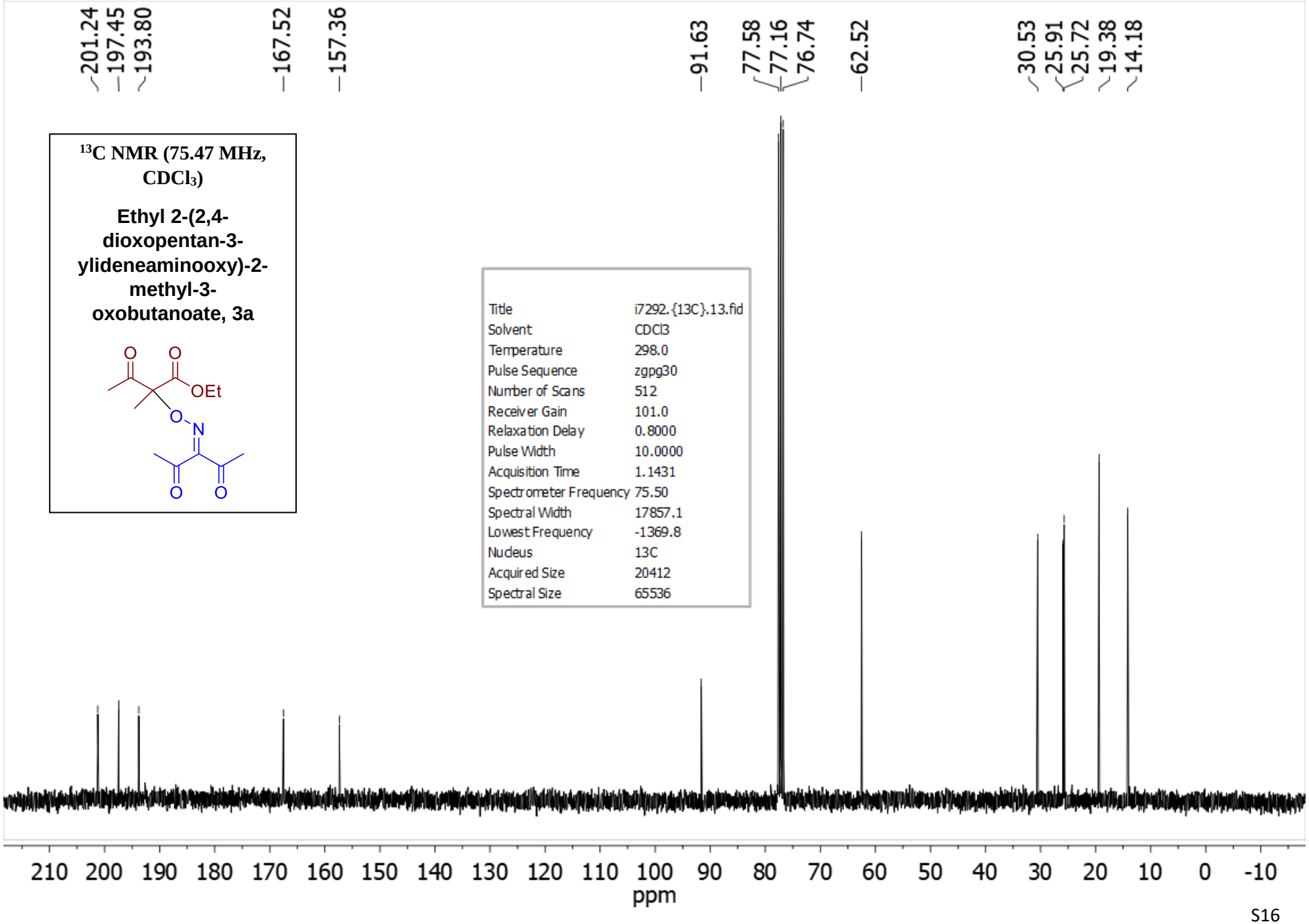
The ^{1}H and ^{13}C spectra of synthesized compounds

^{1}H NMR (300.13 MHz, $CDCl_3$)

Ethyl 2-(2,4-dioxopentan-3-ylideneaminooxy)-2-methyl-3-oxobutanoate, 3a

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197.45
193.80
167.52
157.36
91.63
77.58
77.16
76.74
62.52
30.53
25.91
25.72
19.38
14.18
^{13}C NMR (75.47 MHz, $CDCl_3$)
Ethyl 2-(2,4-dioxopentan-3-ylideneaminooxy)-2-methyl-3-oxobutanoate, 3a
O
O
OEt
O
N
O
O
Title i7292.{13C}.13.fid
Solvent CDCl3
Temperature 298.0
Pulse Sequence zgpg30
Number of Scans 512
Receiver Gain 101.0
Relaxation Delay 0.8000
Pulse Width 10.0000
Acquisition Time 1.1431
Spectrometer Frequency 75.50
Spectral Width 17857.1
Lowest Frequency -1369.8
Nucleus 13C
Acquired Size 20412
Spectral Size 65536
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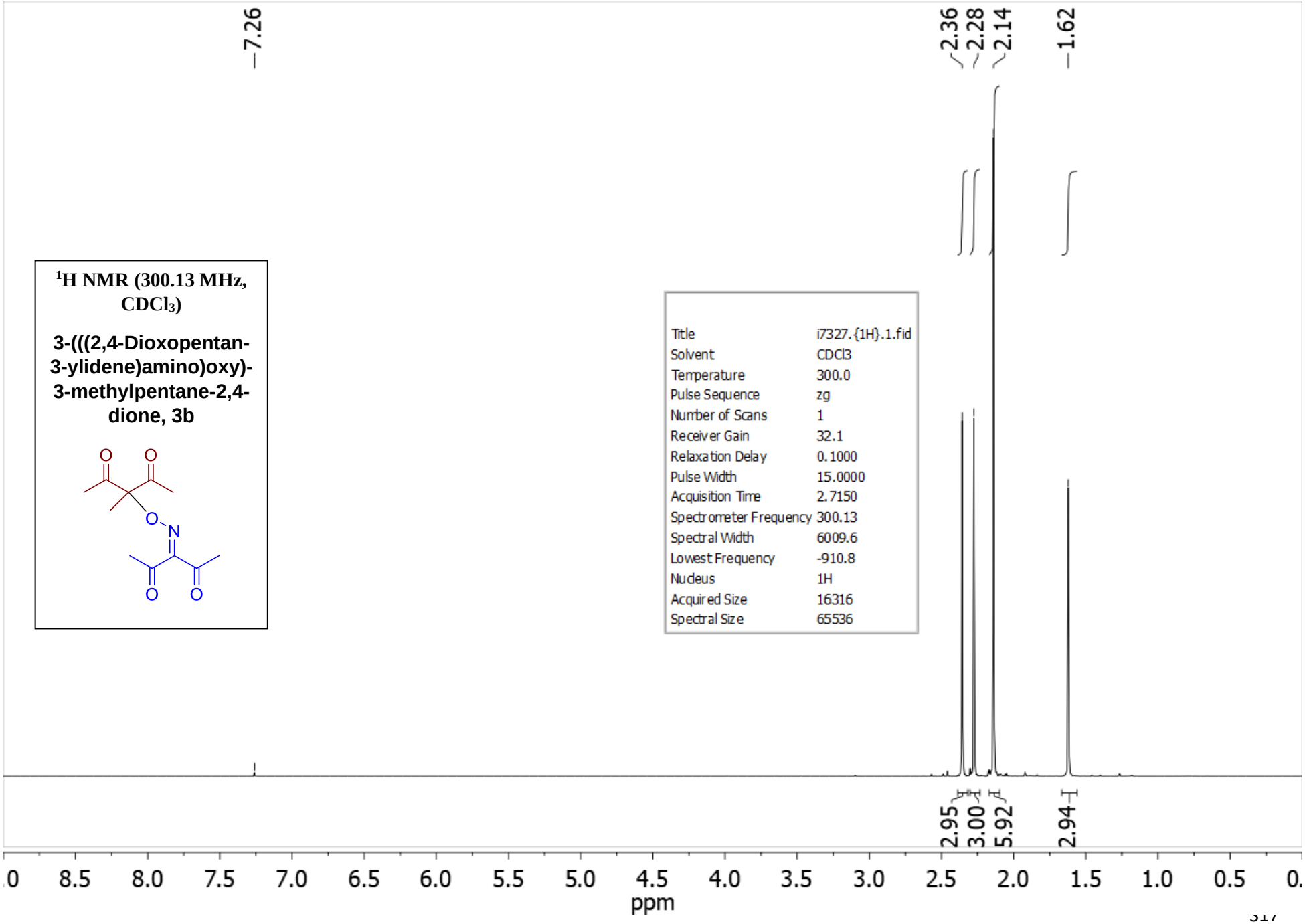
ppm


7.26
2.36
2.28
2.14
1.62
^{1}H NMR (300.13 MHz, $CDCl_3$)
3-(((2,4-Dioxopentan-3-ylidene)amino)oxy)-3-methylpentane-2,4-dione, 3b
Title i7327.{1H}.1.fid
Solvent CDCl3
Temperature 300.0
Pulse Sequence zg
Number of Scans 1
Receiver Gain 32.1
Relaxation Delay 0.1000
Pulse Width 15.0000
Acquisition Time 2.7150
Spectrometer Frequency 300.13
Spectral Width 6009.6
Lowest Frequency -910.8
Nucleus 1H
Acquired Size 16316
Spectral Size 65536
2.95
3.00
5.92
2.94
8.5
8.0
7.5
7.0
6.5
6.0
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4.5
4.0
3.5
3.0
2.5
2.0
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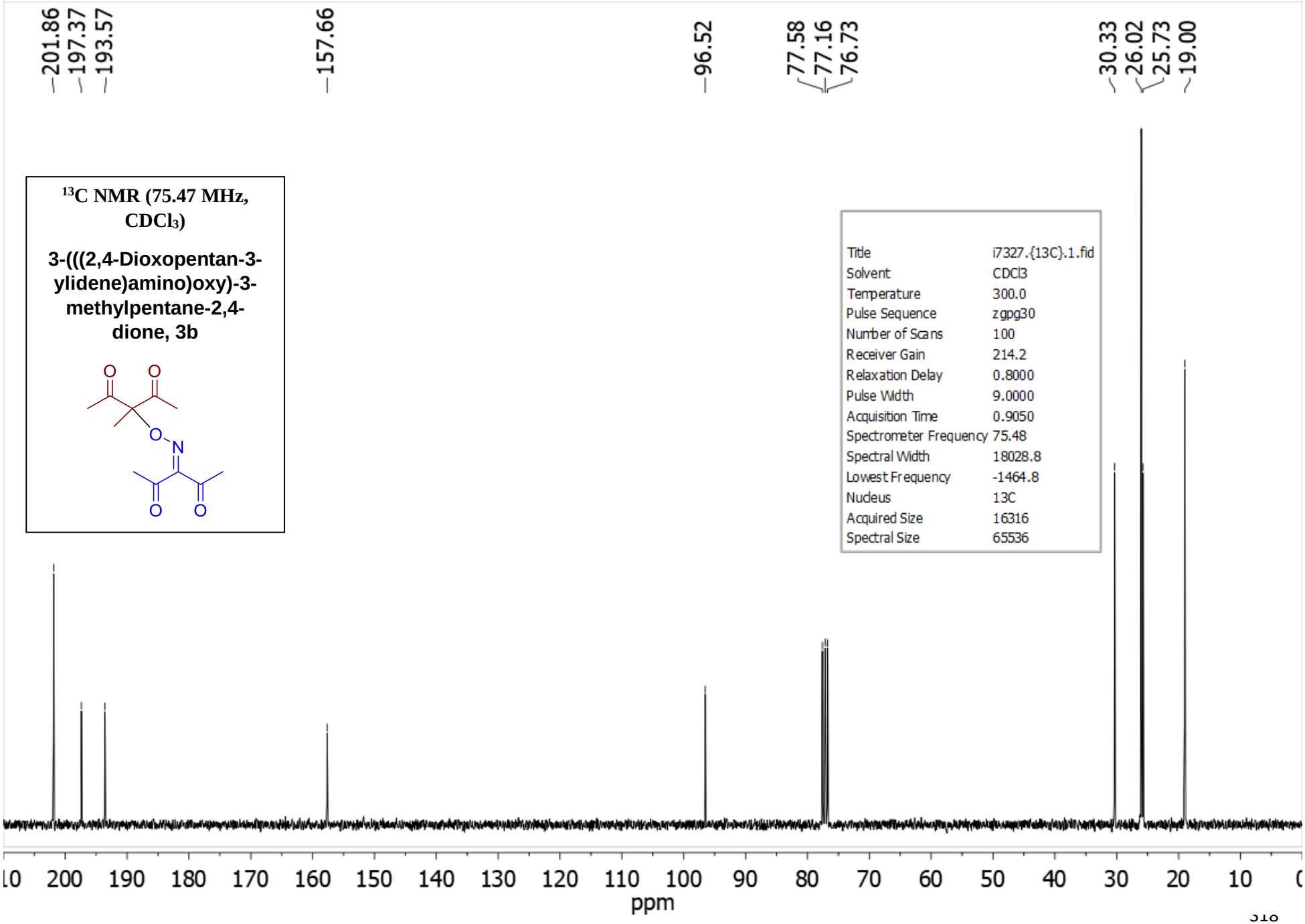
1.0
0.5
ppm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197.37
193.57
157.66
96.52
77.58
77.16
76.73
30.33
26.02
25.73
19.00
^{13}C NMR (75.47 MHz, $CDCl_3$)
3-(((2,4-Dioxopentan-3-ylidene)amino)oxy)-3-methylpentane-2,4-dione, 3b
Title i7327.{13C}.1.fid
Solvent CDCl3
Temperature 300.0
Pulse Sequence zgpg30
Number of Scans 100
Receiver Gain 214.2
Relaxation Delay 0.8000
Pulse Width 9.0000
Acquisition Time 0.9050
Spectrometer Frequency 75.48
Spectral Width 18028.8
Lowest Frequency -1464.8
Nucleus 13C
Acquired Size 16316
Spectral Size 65536
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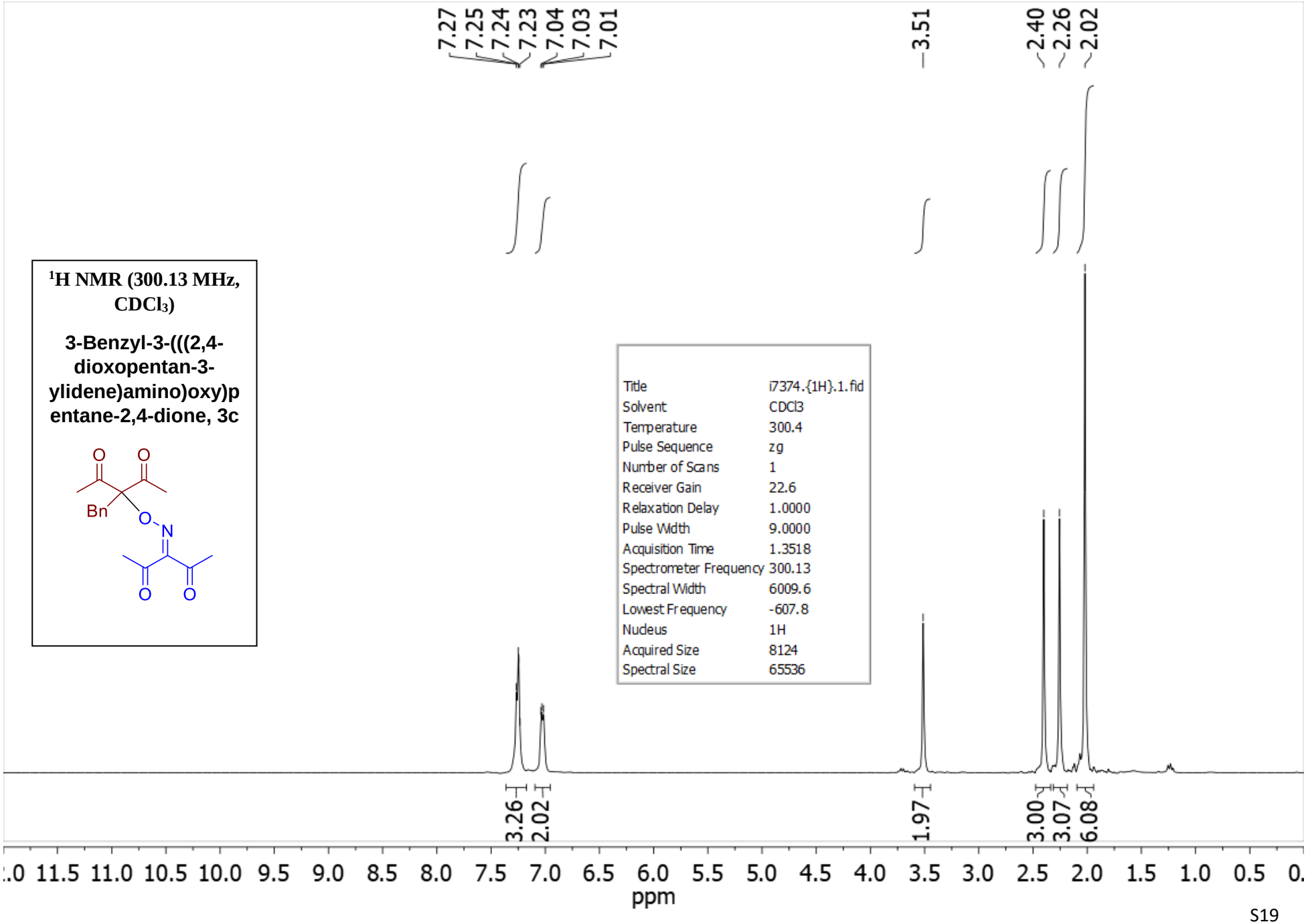
ppm


7.27
7.25
7.24
7.23
7.04
7.03
7.01
3.51
2.40
2.26
2.02
^{1}H NMR (300.13 MHz, $CDCl_3$)
3-Benzyl-3-(((2,4-dioxopentan-3-ylidene)amino)oxy)pentane-2,4-dione, 3c
Bn
Title i7374.{1H}.1.fid
Solvent CDCl3
Temperature 300.4
Pulse Sequence zg
Number of Scans 1
Receiver Gain 22.6
Relaxation Delay 1.0000
Pulse Width 9.0000
Acquisition Time 1.3518
Spectrometer Frequency 300.13
Spectral Width 6009.6
Lowest Frequency -607.8
Nucleus 1H
Acquired Size 8124
Spectral Size 65536
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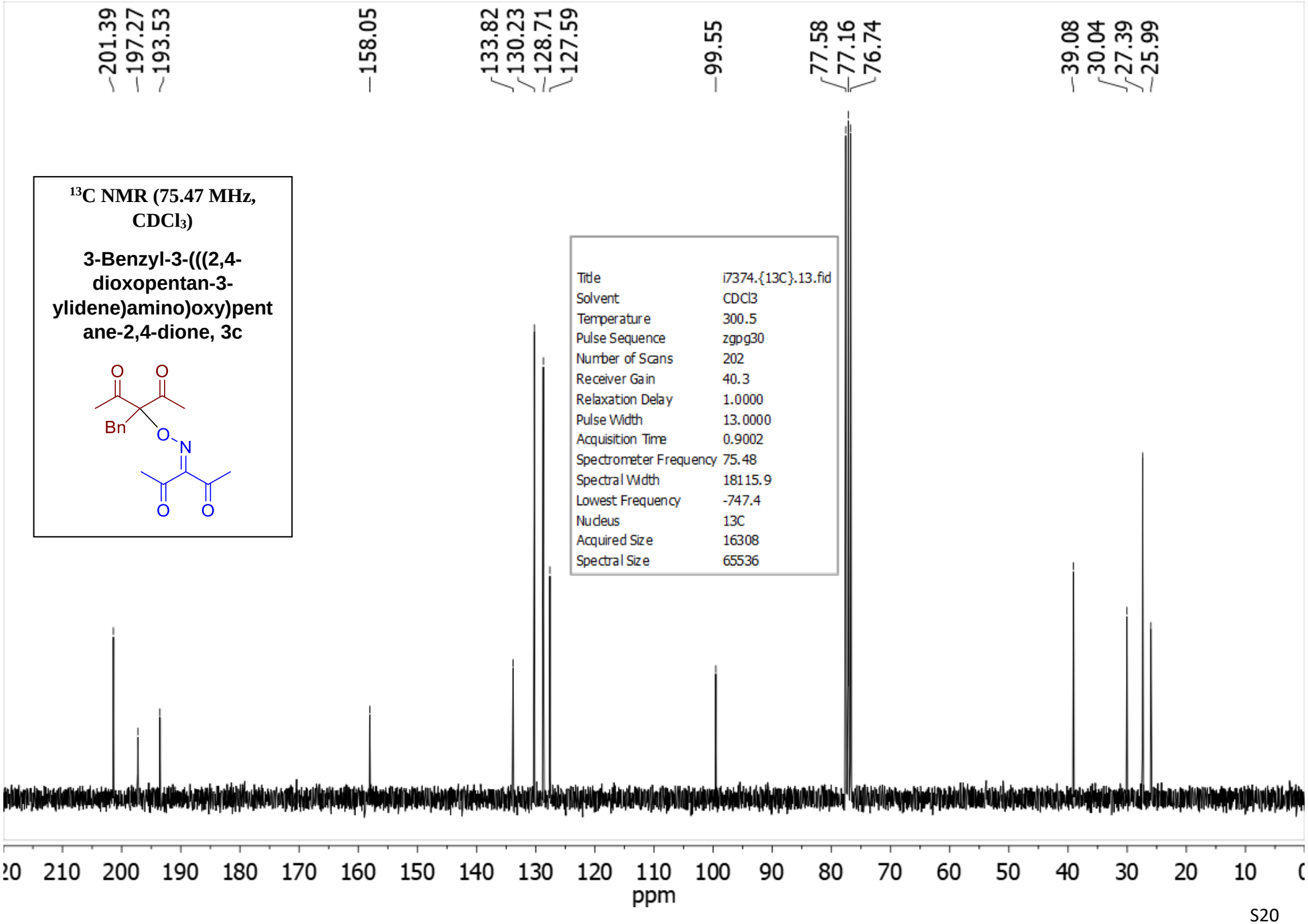
2.02
1.97
3.00
3.07
6.08
ppm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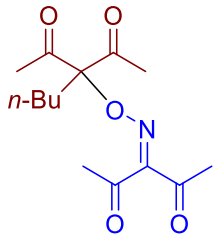
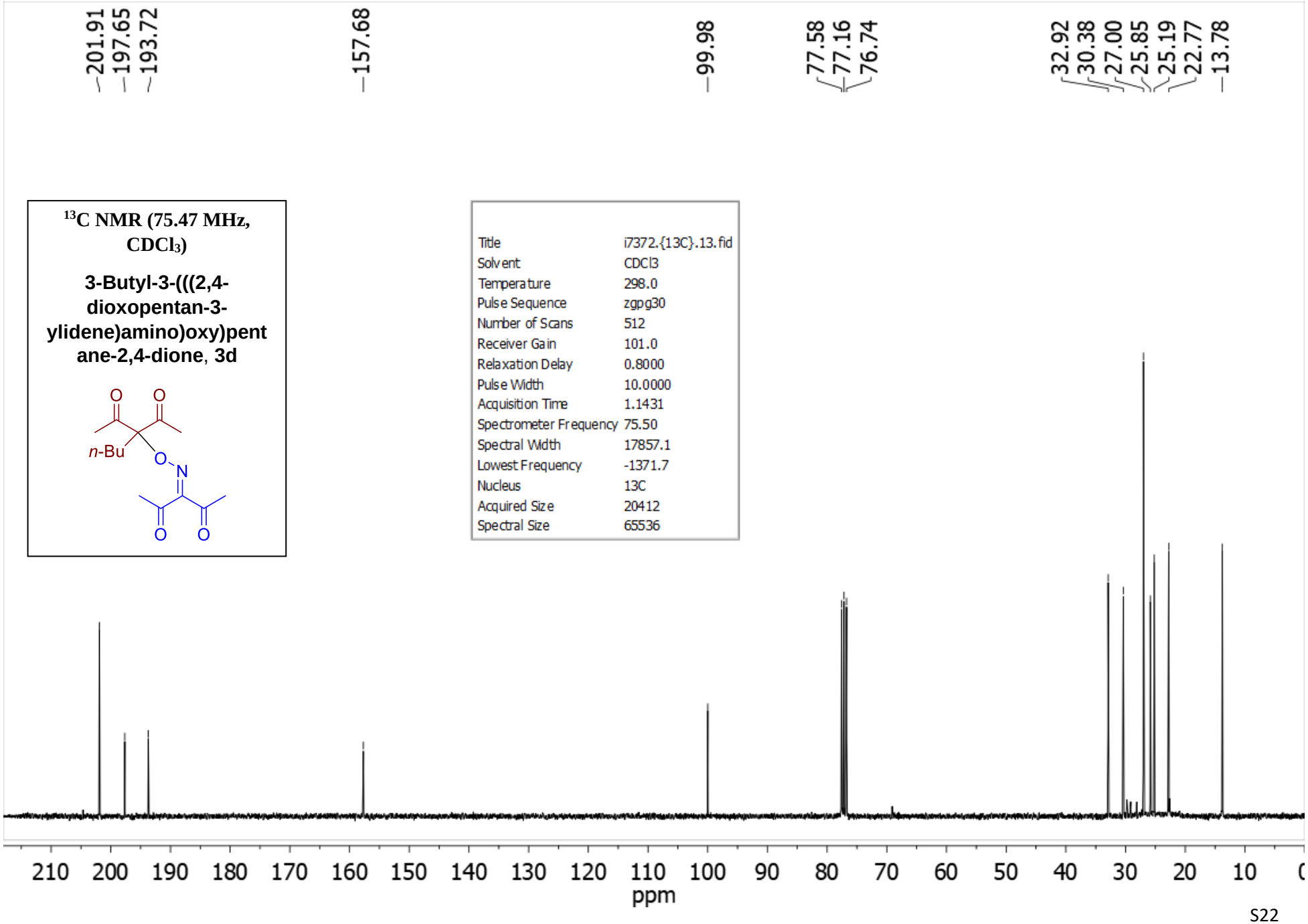
197.27
193.53
158.05
133.82
130.23
128.71
127.59
99.55
77.58
77.16
76.74
39.08
30.04
27.39
25.99
^{13}C NMR (75.47 MHz, $CDCl_3$)
3-Benzyl-3-(((2,4-dioxopentan-3-ylidene)amino)oxy)pentane-2,4-dione, 3c
Bn
O
N
Title i7374.{13C}.13.fid
Solvent CDCl3
Temperature 300.5
Pulse Sequence zgpg30
Number of Scans 202
Receiver Gain 40.3
Relaxation Delay 1.0000
Pulse Width 13.0000
Acquisition Time 0.9002
Spectrometer Frequency 75.48
Spectral Width 18115.9
Lowest Frequency -747.4
Nucleus 13C
Acquired Size 16308
Spectral Size 65536
ppm

^{1}H NMR (300.13 MHz, $CDCl_3$)

3-Butyl-3-(((2,4-dioxopentan-3-ylidene)amino)oxy)pentane-2,4-dione, 3d




201.91
197.65
193.72
157.68
99.98
77.58
77.16
76.74
32.92
30.38
27.00
25.85
25.19
22.77
13.78
^{13}C NMR (75.47 MHz, $CDCl_3$)
3-Butyl-3-(((2,4-dioxopentan-3-ylidene)amino)oxy)pentane-2,4-dione, 3d
n-Bu
Title i7372.{13C}.13.fid
Solvent CDCl3
Temperature 298.0
Pulse Sequence zgpg30
Number of Scans 512
Receiver Gain 101.0
Relaxation Delay 0.8000
Pulse Width 10.0000
Acquisition Time 1.1431
Spectrometer Frequency 75.50
Spectral Width 17857.1
Lowest Frequency -1371.7
Nucleus 13C
Acquired Size 20412
Spectral Size 65536
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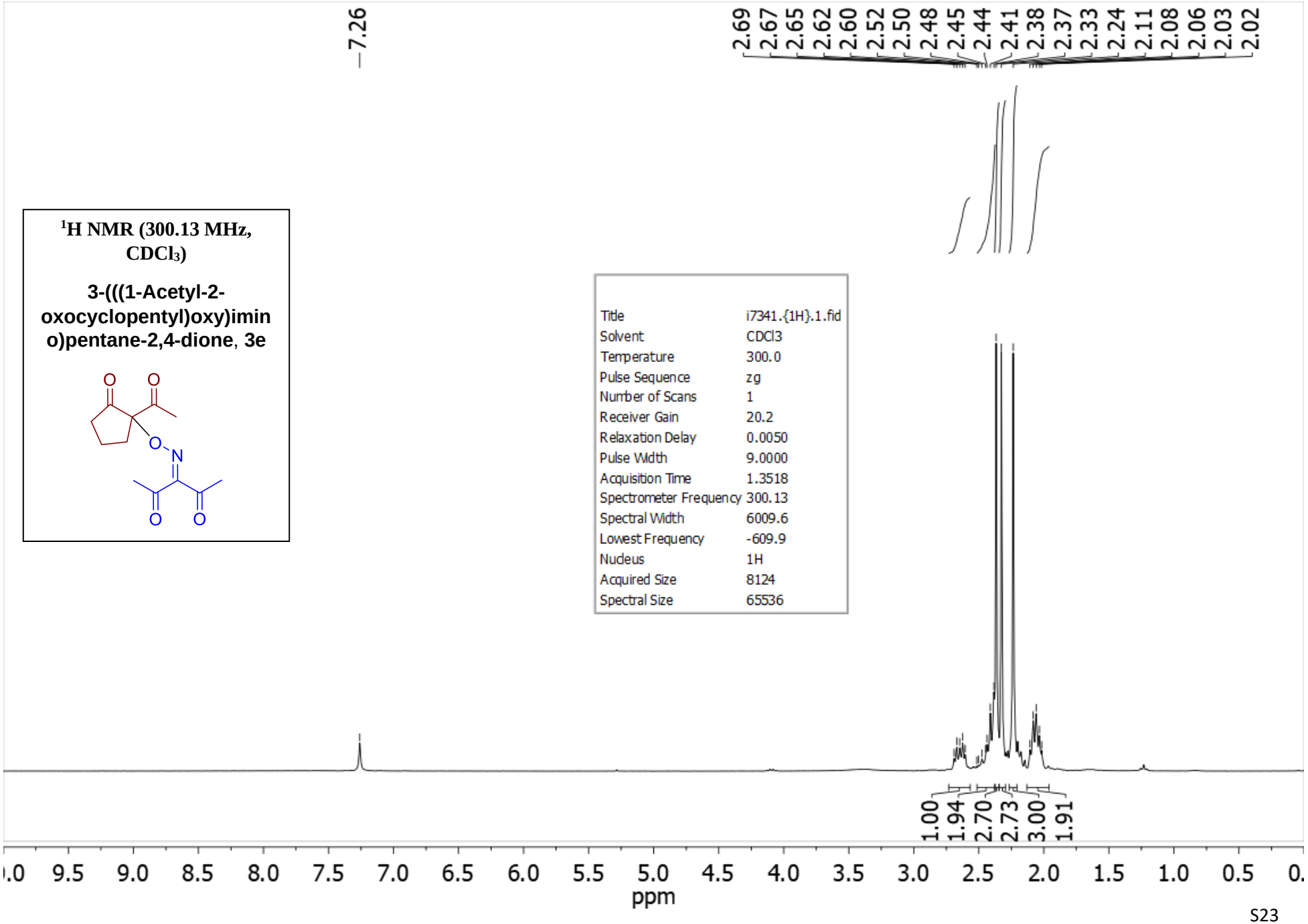
ppm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2.69
2.67
2.65
2.62
2.60
2.52
2.50
2.48
2.45
2.44
2.41
2.38
2.37
2.33
2.24
2.11
2.08
2.06
2.03
2.02
1H NMR (300.13 MHz, CDCl3)
3-(((1-Acetyl-2-oxocyclopentyl)oxy)imino)pentane-2,4-dione, 3e
Title i7341.{1H}.1.fid
Solvent CDCl3
Temperature 300.0
Pulse Sequence zg
Number of Scans 1
Receiver Gain 20.2
Relaxation Delay 0.0050
Pulse Width 9.0000
Acquisition Time 1.3518
Spectrometer Frequency 300.13
Spectral Width 6009.6
Lowest Frequency -609.9
Nucleus 1H
Acquired Size 8124
Spectral Size 65536
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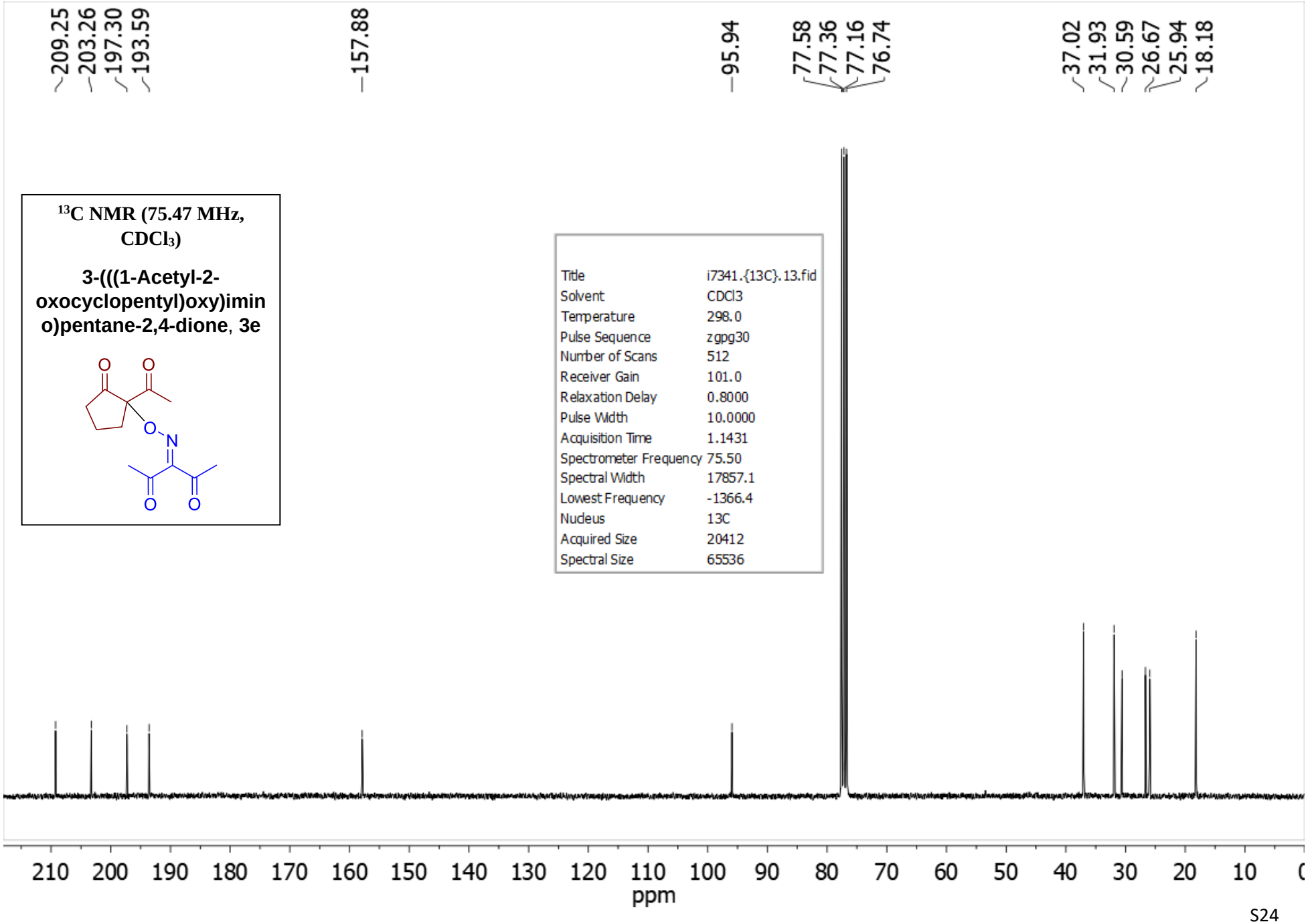
3.00
1.91
ppm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193.59
157.88
95.94
77.58
77.36
77.16
76.74
37.02
31.93
30.59
26.67
25.94
18.18
^{13}C NMR (75.47 MHz, $CDCl_3$)
3-(((1-Acetyl-2-oxocyclopentyl)oxy)imino)pentane-2,4-dione, 3e
Title i7341.{13C}.13.fid
Solvent CDCl3
Temperature 298.0
Pulse Sequence zgpg30
Number of Scans 512
Receiver Gain 101.0
Relaxation Delay 0.8000
Pulse Width 10.0000
Acquisition Time 1.1431
Spectrometer Frequency 75.50
Spectral Width 17857.1
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Nucleus 13C
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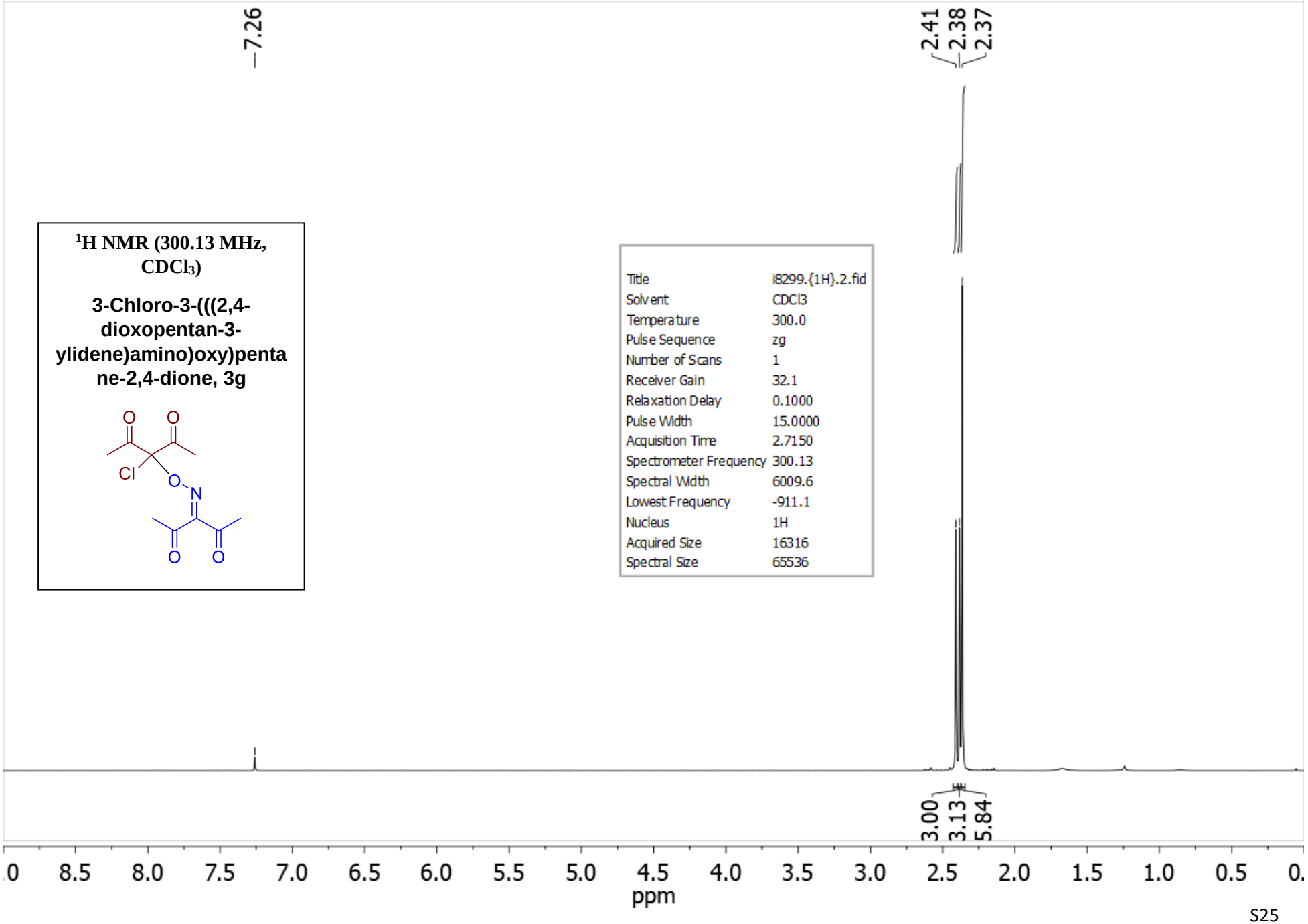
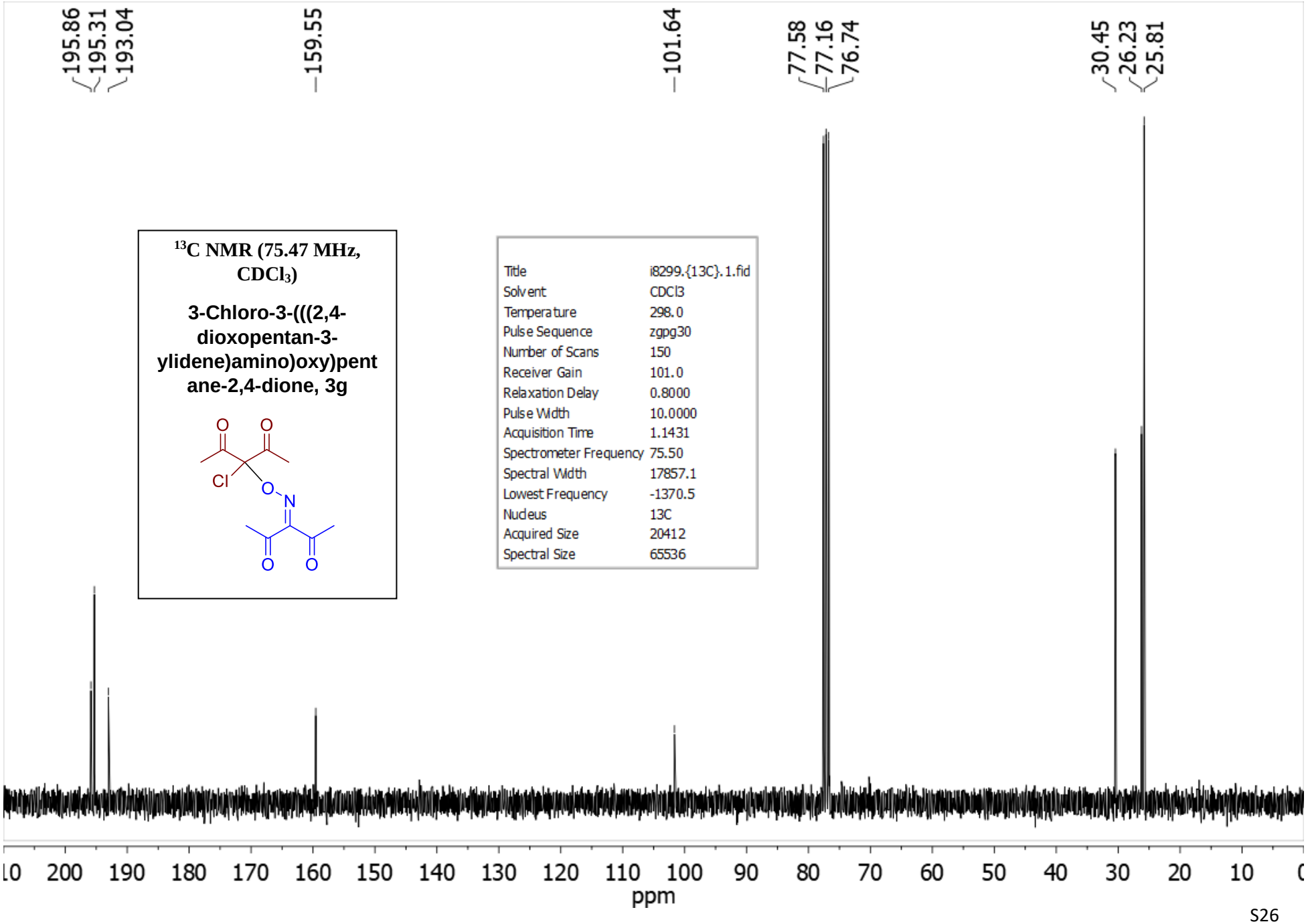
ppm



^{1}H NMR (300.13 MHz, $CDCl_3$)

3-Chloro-3-(((2,4-dioxopentan-3-ylidene)amino)oxy)pentane-2,4-dione, 3g


195.86
195.31
193.04
159.55
101.64
77.58
77.16
76.74
30.45
26.23
25.81
^{13}C NMR (75.47 MHz, $CDCl_3$)
3-Chloro-3-(((2,4-dioxopentan-3-ylidene)amino)oxy)pentane-2,4-dione, 3g
Title i8299.{13C}.1.fid
Solvent CDCl3
Temperature 298.0
Pulse Sequence zgpg30
Number of Scans 150
Receiver Gain 101.0
Relaxation Delay 0.8000
Pulse Width 10.0000
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Nucleus 13C
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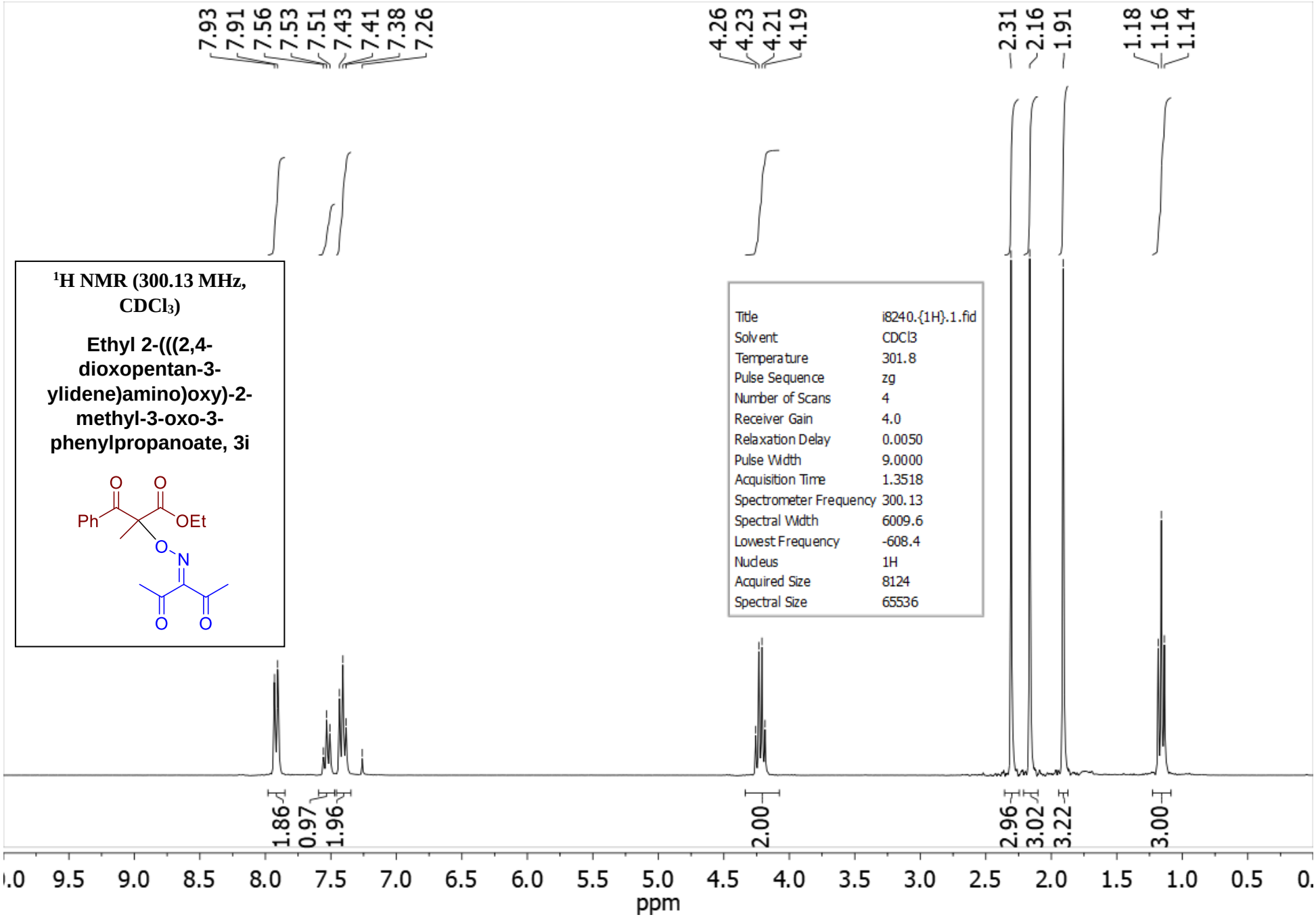
Spectral Size 65536
ppm


7.93
7.91
7.56
7.53
7.51
7.43
7.41
7.38
7.26
4.26
4.23
4.21
4.19
2.31
2.16
1.91
1.18
1.16
1.14
1H NMR (300.13 MHz, CDCl3)
Ethyl 2-(((2,4-dioxopentan-3-ylidene)amino)oxy)-2-methyl-3-oxo-3-phenylpropanoate, 3i
Ph
OEt
O
N
Title i8240.{1H}.1.fid
Solvent CDCl3
Temperature 301.8
Pulse Sequence zg
Number of Scans 4
Receiver Gain 4.0
Relaxation Delay 0.0050
Pulse Width 9.0000
Acquisition Time 1.3518
Spectrometer Frequency 300.13
Spectral Width 6009.6
Lowest Frequency -608.4
Nucleus 1H
Acquired Size 8124
Spectral Size 65536
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0.97
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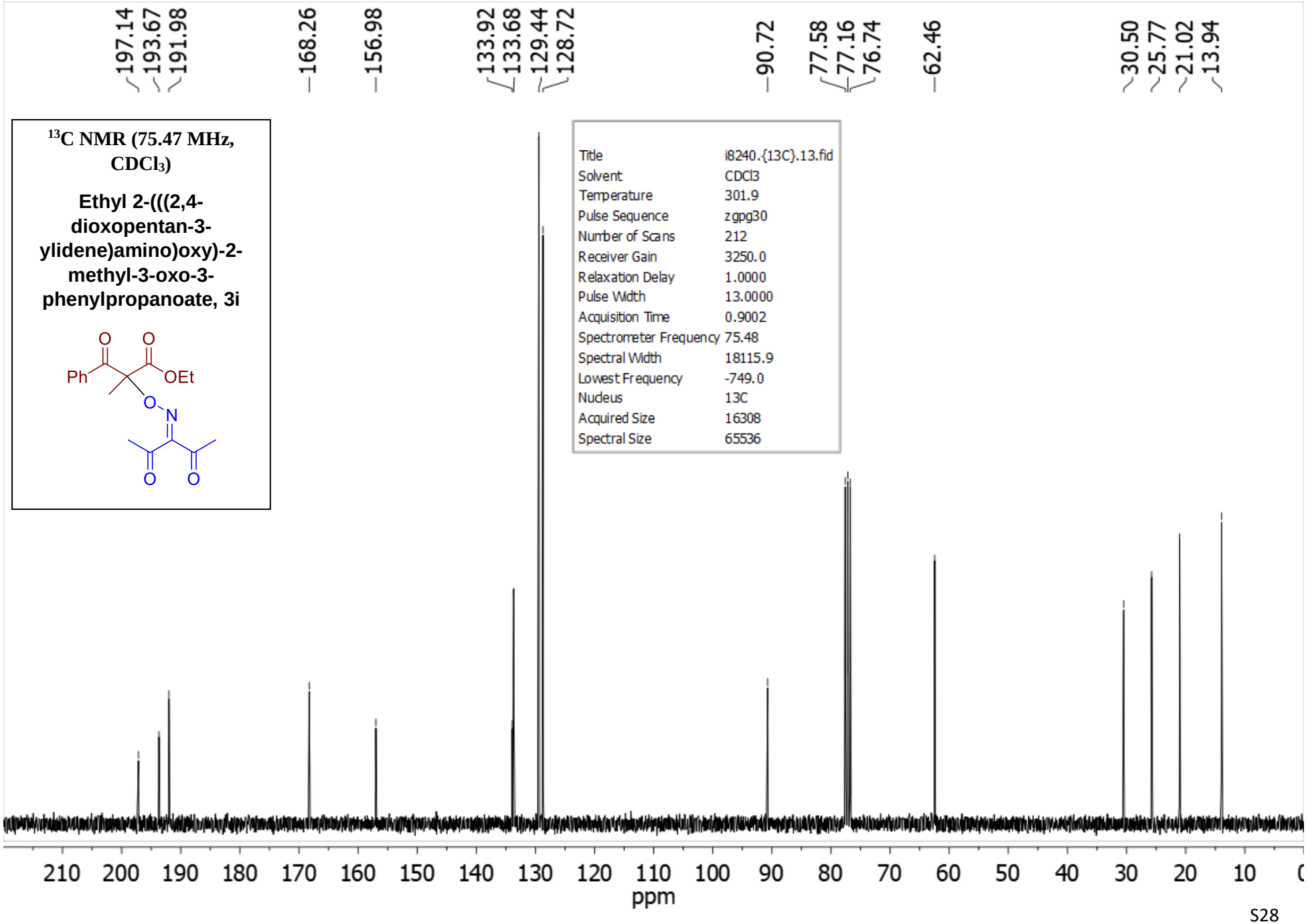
2.00
2.96
3.02
3.22
3.00
ppm


197.14
193.67
191.98
168.26
156.98
133.92
133.68
129.44
128.72
90.72
77.58
77.16
76.74
62.46
30.50
25.77
21.02
13.94
^{13}C NMR (75.47 MHz, $CDCl_3$)
Ethyl 2-(((2,4-dioxopentan-3-ylidene)amino)oxy)-2-methyl-3-oxo-3-phenylpropanoate, 3i
Ph
OEt
O
N
Title i8240.{13C}.13.fid
Solvent CDCl3
Temperature 301.9
Pulse Sequence zgpg30
Number of Scans 212
Receiver Gain 3250.0
Relaxation Delay 1.0000
Pulse Width 13.0000
Acquisition Time 0.9002
Spectrometer Frequency 75.48
Spectral Width 18115.9
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Spectral Size 65536
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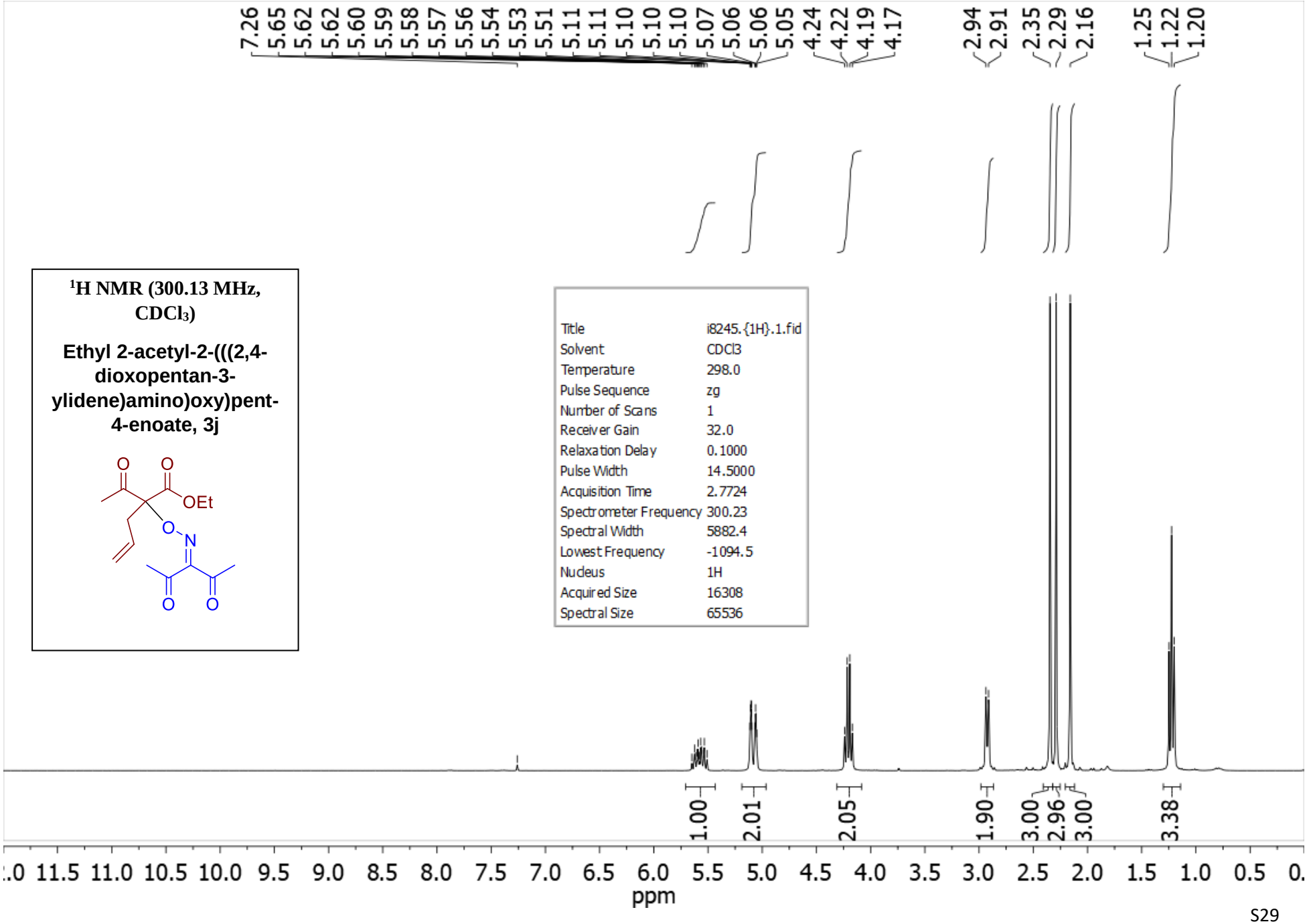
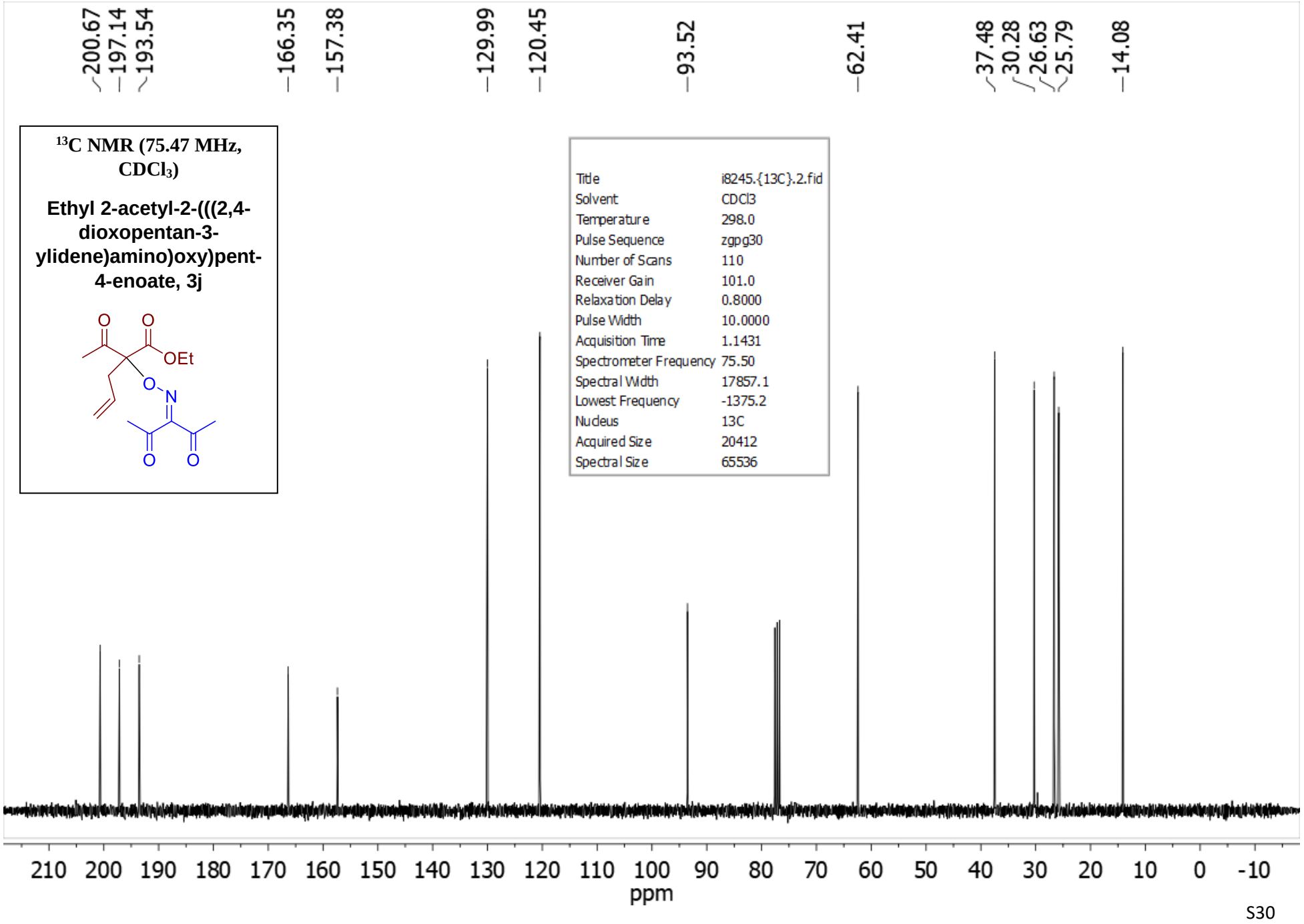
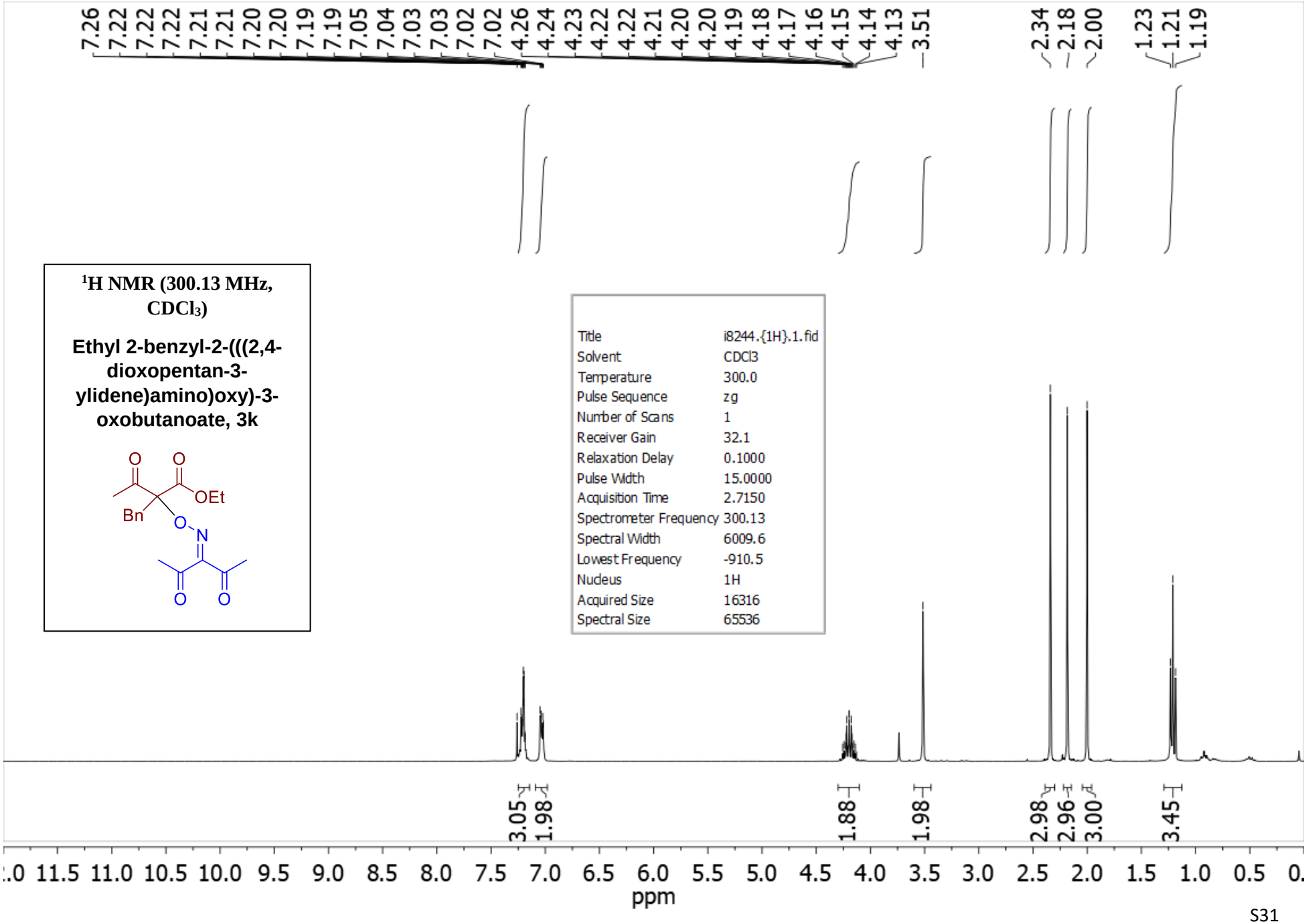


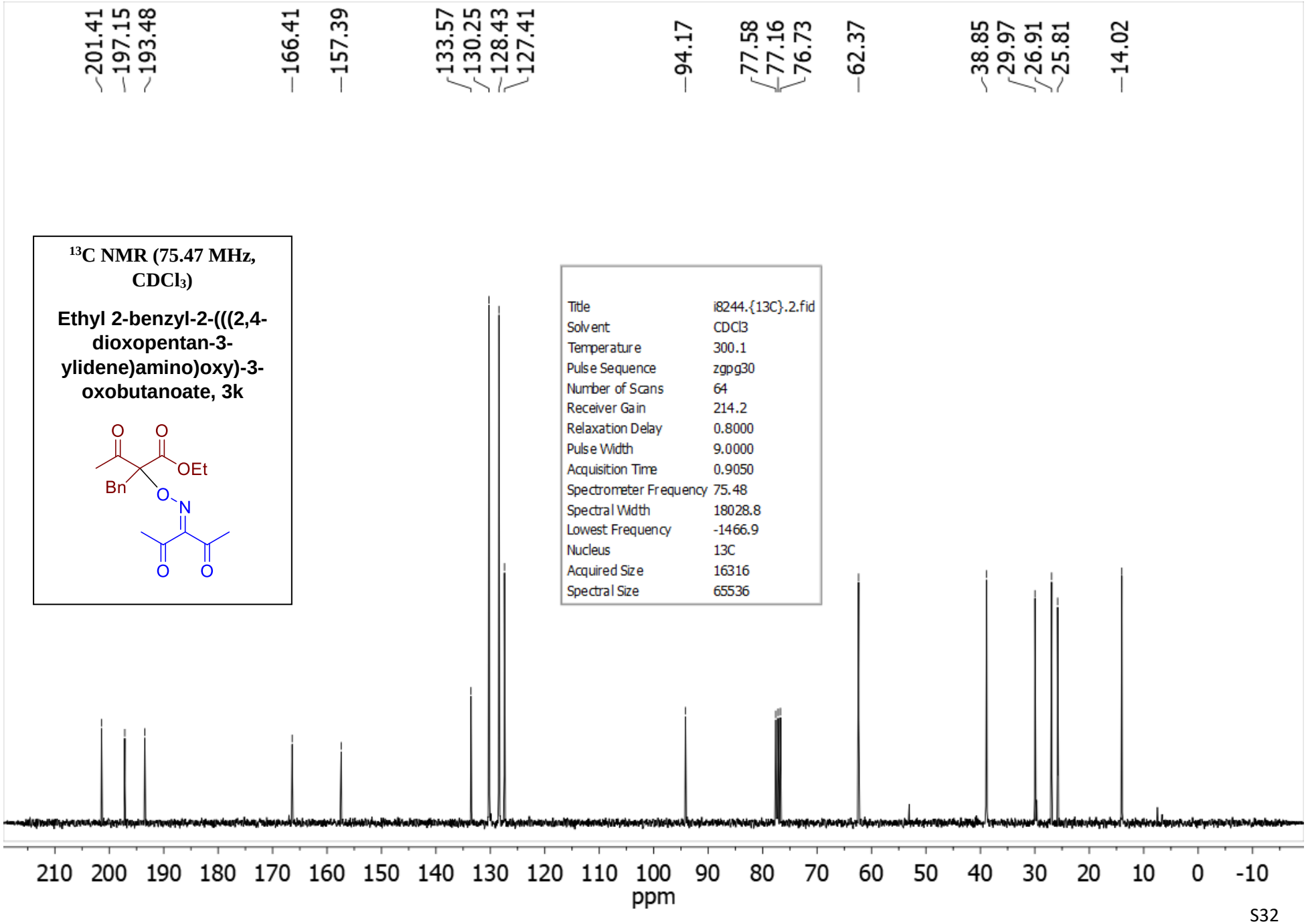
^{1}H NMR (300.13 MHz, $CDCl_3$)

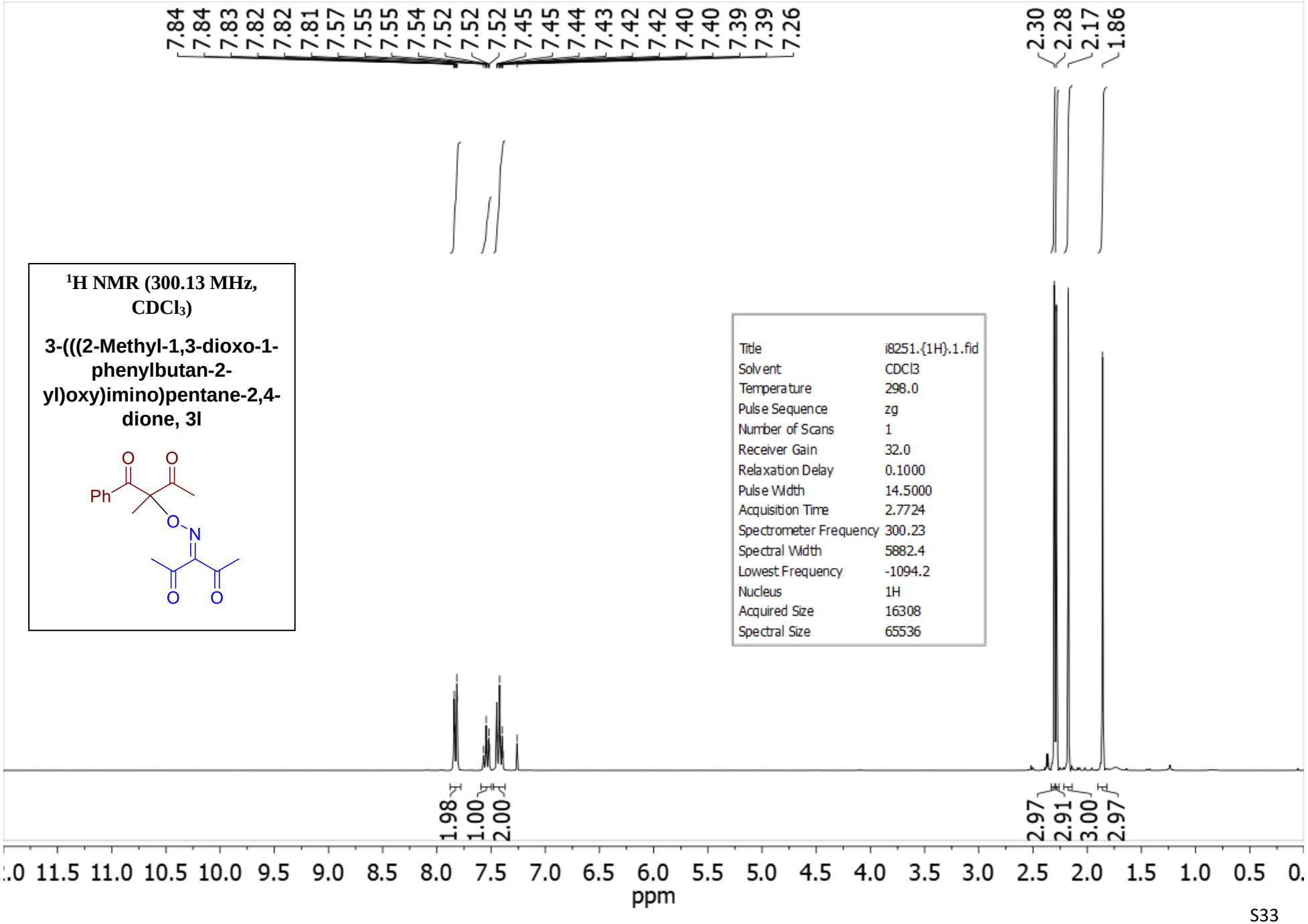
Ethyl 2-acetyl-2-(((2,4-dioxopentan-3-ylidene)amino)oxy)pent-4-enoate, 3j

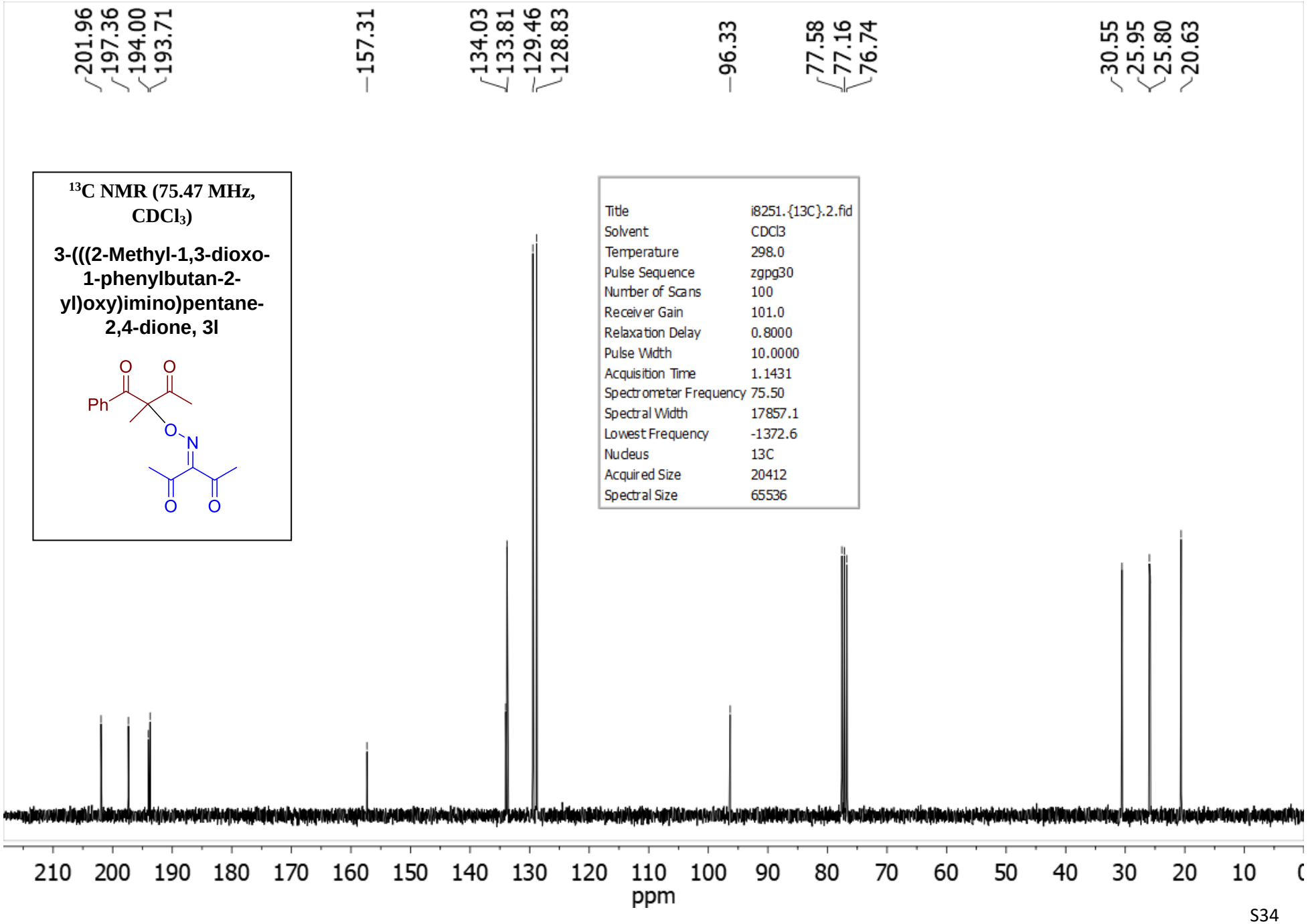
Title	i8245.{1H}.1.fid
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg
Number of Scans	1
Receiver Gain	32.0
Relaxation Delay	0.1000
Pulse Width	14.5000
Acquisition Time	2.7724
Spectrometer Frequency	300.23
Spectral Width	5882.4
Lowest Frequency	-1094.5
Nucleus	1H
Acquired Size	16308
Spectral Size	65536

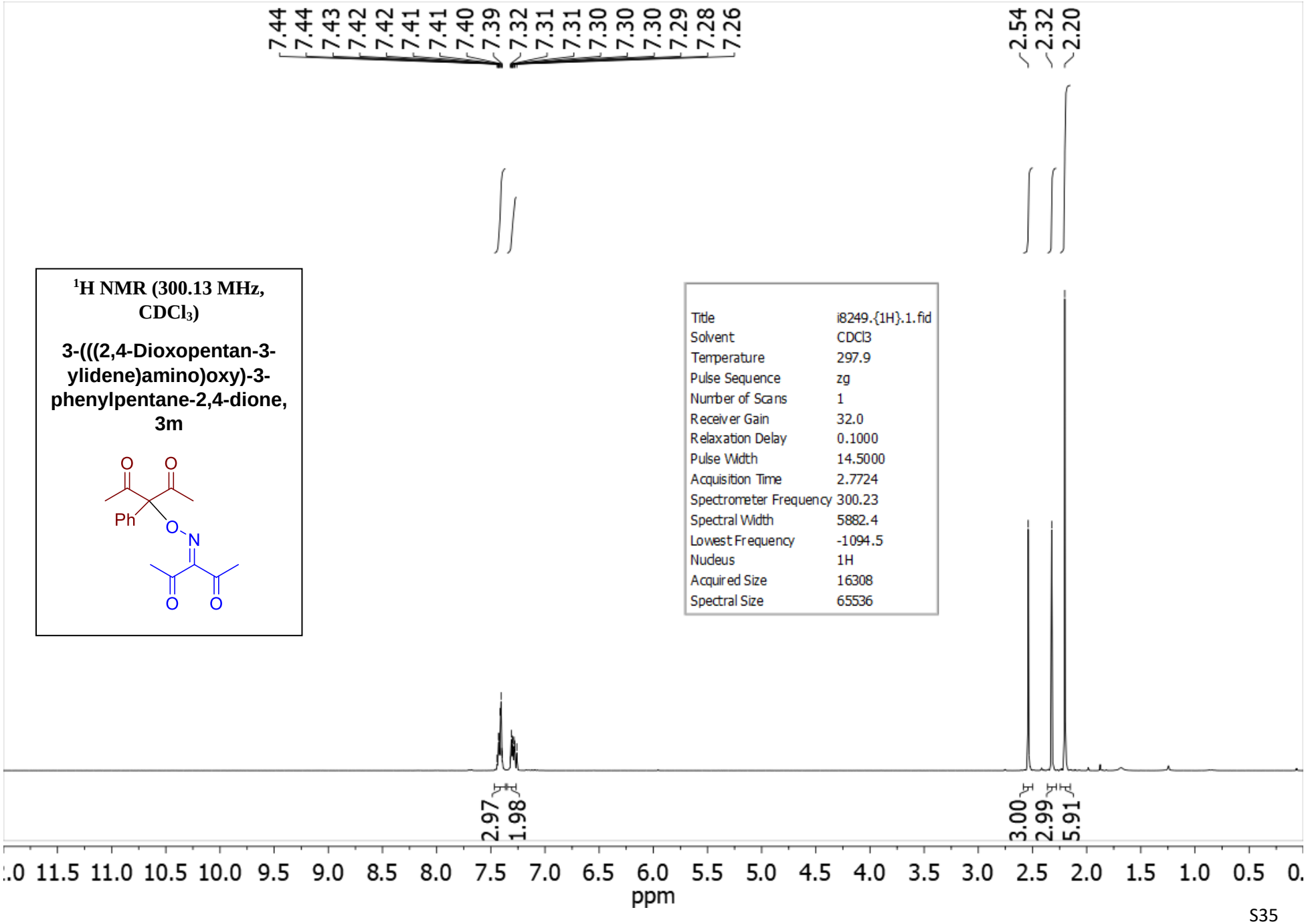

200.67
197.14
193.54
166.35
157.38
129.99
120.45
93.52
62.41
37.48
30.28
26.63
25.79
14.08
^{13}C NMR (75.47 MHz, $CDCl_3$)
Ethyl 2-acetyl-2-(((2,4-dioxopentan-3-ylidene)amino)oxy)pent-4-enoate, 3j
OEt
Title i8245.{13C}.2.fid
Solvent CDCl3
Temperature 298.0
Pulse Sequence zgpg30
Number of Scans 110
Receiver Gain 101.0
Relaxation Delay 0.8000
Pulse Width 10.0000
Acquisition Time 1.1431
Spectrometer Frequency 75.50
Spectral Width 17857.1
Lowest Frequency -1375.2
Nucleus 13C
Acquired Size 20412
Spectral Size 65536
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm

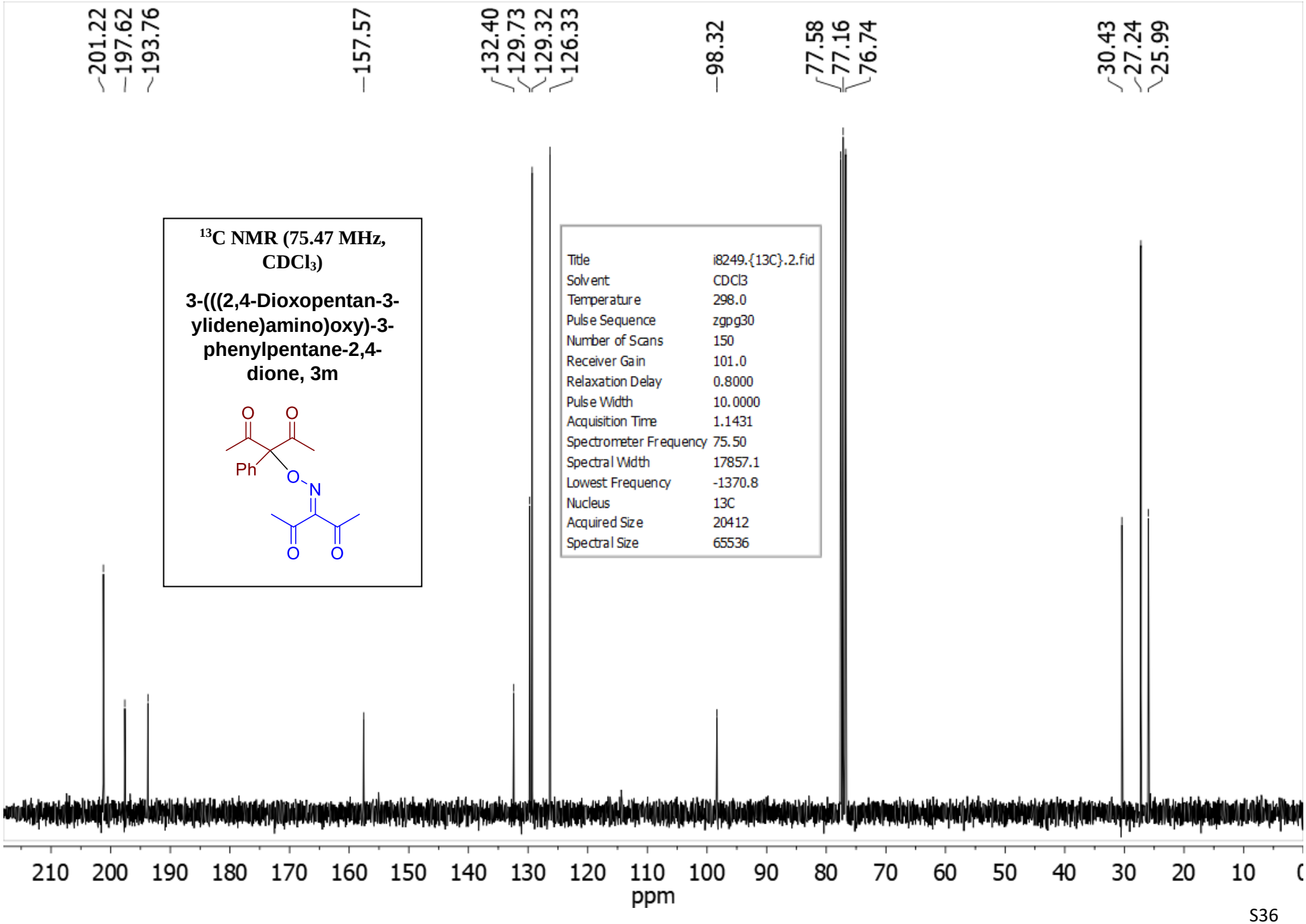

7.26 7.22 7.22 7.22 7.21 7.21 7.20 7.20 7.19 7.19 7.05 7.04 7.03 7.03 7.02 7.02 4.26 4.24 4.23 4.22 4.22 4.21 4.20 4.20 4.19 4.18 4.17 4.16 4.15 4.14 4.13 3.51
2.34 2.18 2.00
1.23 1.21 1.19
¹H NMR (300.13 MHz, CDCl3)
Ethyl 2-benzyl-2-(((2,4-dioxopentan-3-ylidene)amino)oxy)-3-oxobutanoate, 3k
O O OEt Bn O N O O
Title i8244.{1H}.1.fid
Solvent CDCl3
Temperature 300.0
Pulse Sequence zg
Number of Scans 1
Receiver Gain 32.1
Relaxation Delay 0.1000
Pulse Width 15.0000
Acquisition Time 2.7150
Spectrometer Frequency 300.13
Spectral Width 6009.6
Lowest Frequency -910.5
Nucleus 1H
Acquired Size 16316
Spectral Size 65536
3.05 1.98 1.88 1.98 2.98 2.96 3.00 3.45
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
ppm

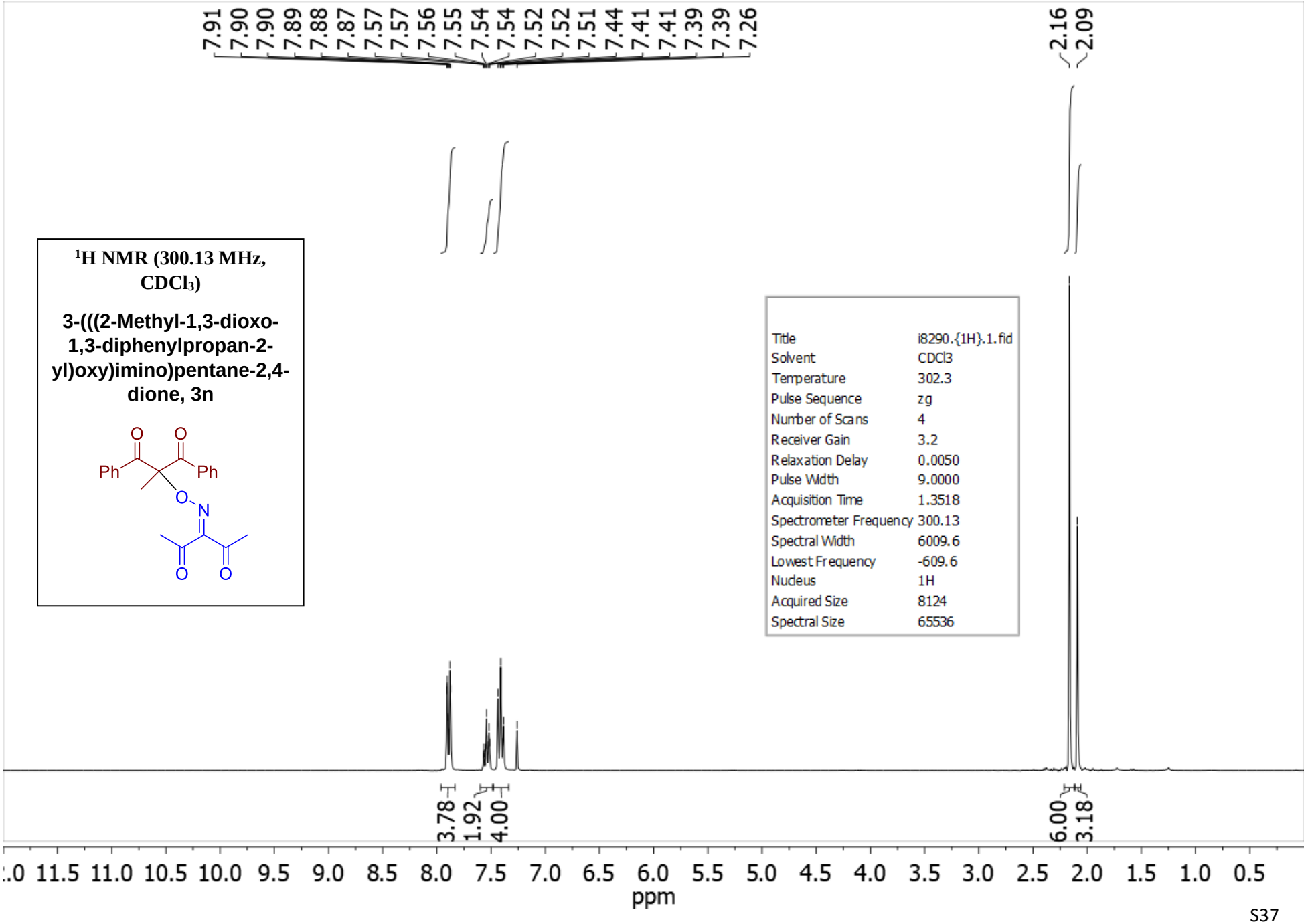

201.41
197.15
193.48
166.41
157.39
133.57
130.25
128.43
127.41
94.17
77.58
77.16
76.73
62.37
38.85
29.97
26.91
25.81
14.02
^{13}C NMR (75.47 MHz, $CDCl_3$)
Ethyl 2-benzyl-2-(((2,4-dioxopentan-3-ylidene)amino)oxy)-3-oxobutanoate, 3k
O
O
OEt
Bn
O
N
O
O
Title i8244.{13C}.2.fid
Solvent CDCl3
Temperature 300.1
Pulse Sequence zgpg30
Number of Scans 64
Receiver Gain 214.2
Relaxation Delay 0.8000
Pulse Width 9.0000
Acquisition Time 0.9050
Spectrometer Frequency 75.48
Spectral Width 18028.8
Lowest Frequency -1466.9
Nucleus 13C
Acquired Size 16316
Spectral Size 65536
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm

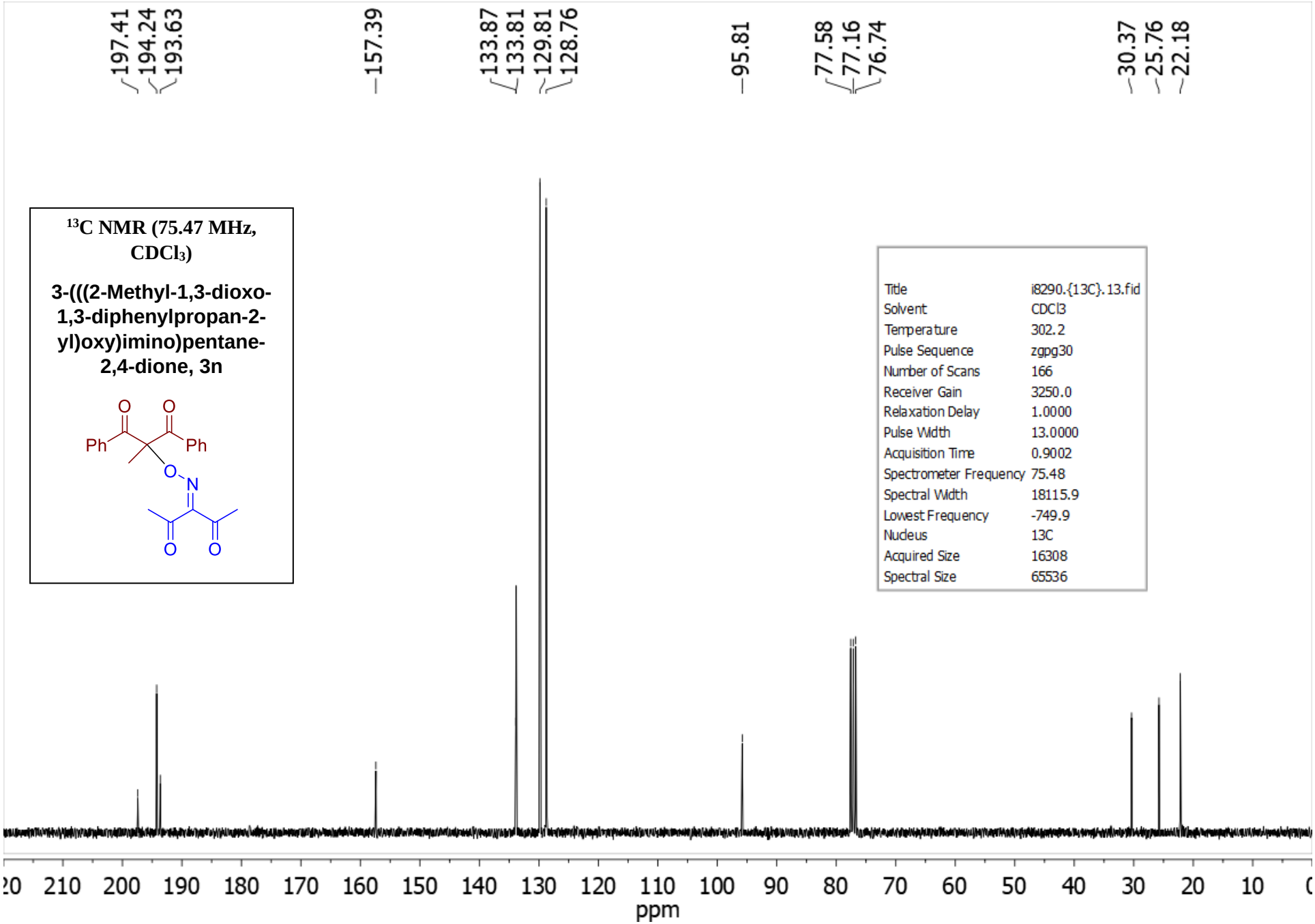

7.84
7.84
7.83
7.82
7.82
7.81
7.57
7.55
7.55
7.54
7.52
7.52
7.52
7.45
7.45
7.44
7.43
7.42
7.42
7.40
7.40
7.39
7.39
7.26
2.30
2.28
2.17
1.86
1H NMR (300.13 MHz, CDCl3)
3-(((2-Methyl-1,3-dioxo-1-phenylbutan-2-yl)oxy)imino)pentane-2,4-dione, 3l
Ph
O
O
O
N
O
O
Title i8251.{1H}.1.fid
Solvent CDCl3
Temperature 298.0
Pulse Sequence zg
Number of Scans 1
Receiver Gain 32.0
Relaxation Delay 0.1000
Pulse Width 14.5000
Acquisition Time 2.7724
Spectrometer Frequency 300.23
Spectral Width 5882.4
Lowest Frequency -1094.2
Nucleus 1H
Acquired Size 16308
Spectral Size 65536
1.98
1.00
2.00
2.97
2.91
3.00
2.97
ppm

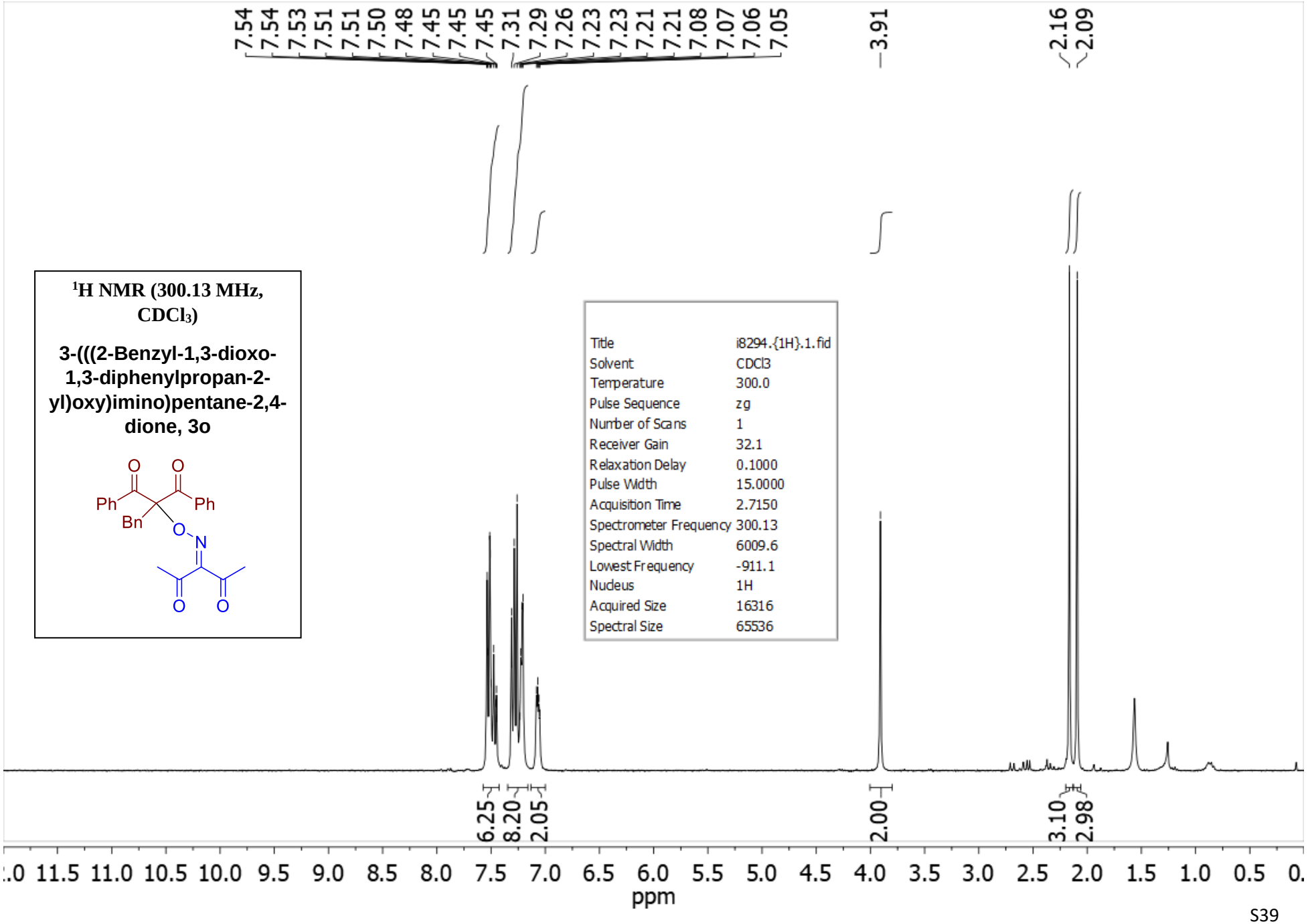

201.96
197.36
194.00
193.71
157.31
134.03
133.81
129.46
128.83
96.33
77.58
77.16
76.74
30.55
25.95
25.80
20.63
^{13}C NMR (75.47 MHz, $CDCl_3$)
3-(((2-Methyl-1,3-dioxo-1-phenylbutan-2-yl)oxy)imino)pentane-2,4-dione, 3l
Ph
O
O
O
N
O
O
Title i8251.{13C}.2.fid
Solvent CDCl3
Temperature 298.0
Pulse Sequence zgpg30
Number of Scans 100
Receiver Gain 101.0
Relaxation Delay 0.8000
Pulse Width 10.0000
Acquisition Time 1.1431
Spectrometer Frequency 75.50
Spectral Width 17857.1
Lowest Frequency -1372.6
Nucleus 13C
Acquired Size 20412
Spectral Size 65536
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm

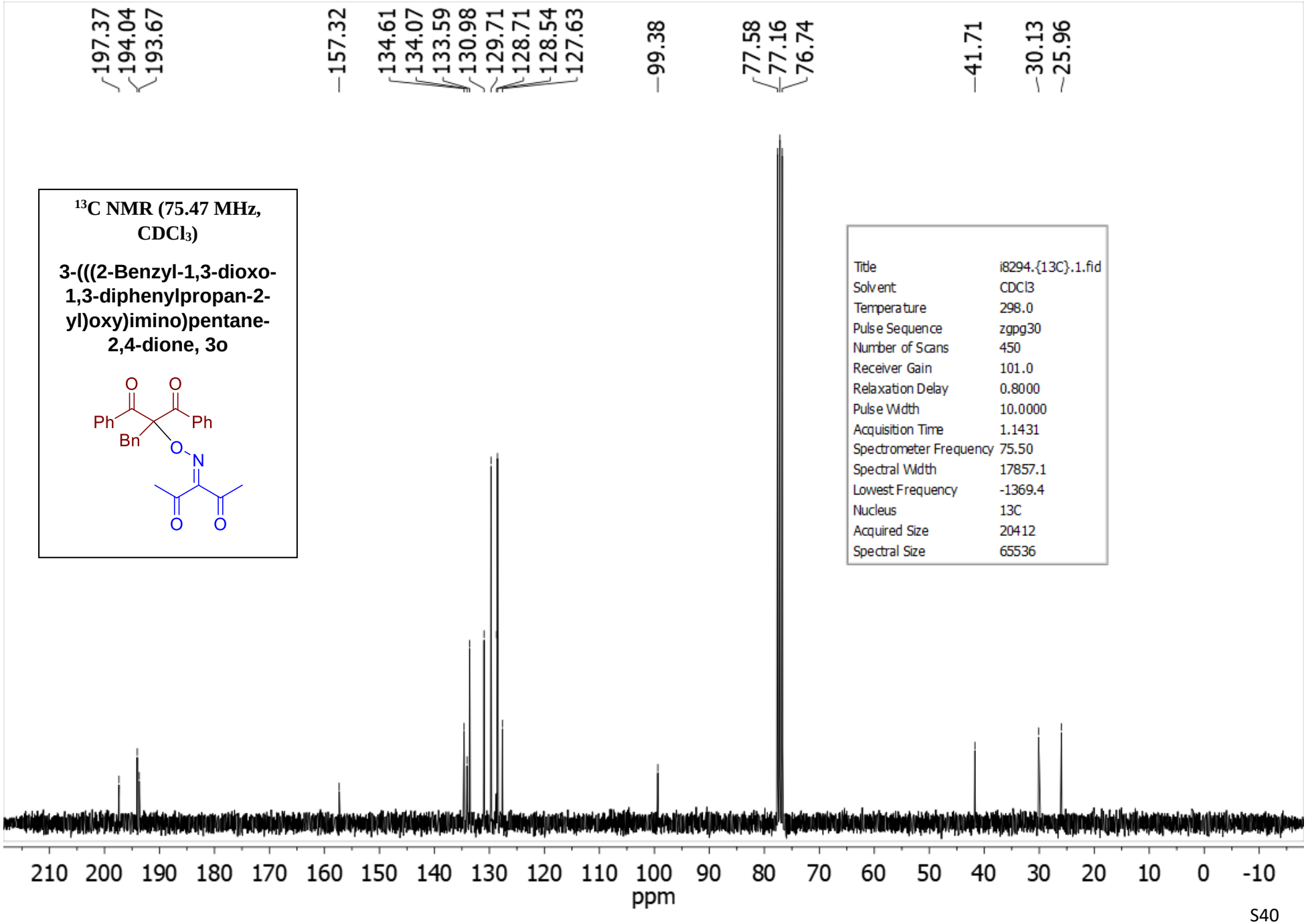

7.44
7.44
7.43
7.42
7.42
7.41
7.41
7.40
7.39
7.32
7.31
7.31
7.30
7.30
7.30
7.29
7.28
7.26
2.54
2.32
2.20
^{1}H NMR (300.13 MHz, CDCl$_3$)
3-(((2,4-Dioxopentan-3-ylidene)amino)oxy)-3-phenylpentane-2,4-dione, 3m
Ph
O
N
Title i8249.{1H}.1.fid
Solvent CDCl3
Temperature 297.9
Pulse Sequence zg
Number of Scans 1
Receiver Gain 32.0
Relaxation Delay 0.1000
Pulse Width 14.5000
Acquisition Time 2.7724
Spectrometer Frequency 300.23
Spectral Width 5882.4
Lowest Frequency -1094.5
Nucleus 1H
Acquired Size 16308
Spectral Size 65536
2.97
1.98
3.00
2.99
5.91
ppm


201.22
197.62
193.76
157.57
132.40
129.73
129.32
126.33
98.32
77.58
77.16
76.74
30.43
27.24
25.99
^{13}C NMR (75.47 MHz, $CDCl_3$)
3-(((2,4-Dioxopentan-3-ylidene)amino)oxy)-3-phenylpentane-2,4-dione, 3m
Ph
O
N
Title i8249.{13C}.2.fid
Solvent CDCl3
Temperature 298.0
Pulse Sequence zgpg30
Number of Scans 150
Receiver Gain 101.0
Relaxation Delay 0.8000
Pulse Width 10.0000
Acquisition Time 1.1431
Spectrometer Frequency 75.50
Spectral Width 17857.1
Lowest Frequency -1370.8
Nucleus 13C
Acquired Size 20412
Spectral Size 65536
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm


7.91
7.90
7.90
7.89
7.88
7.87
7.57
7.57
7.56
7.55
7.54
7.54
7.52
7.52
7.51
7.44
7.41
7.41
7.39
7.39
7.26
2.16
2.09
^{1}H NMR (300.13 MHz, $CDCl_3$)
3-(((2-Methyl-1,3-dioxo-1,3-diphenylpropan-2-yl)oxy)imino)pentane-2,4-dione, 3n
Ph
Ph
O
N
O
O
O
O
Title i8290.{1H}.1.fid
Solvent CDCl3
Temperature 302.3
Pulse Sequence zg
Number of Scans 4
Receiver Gain 3.2
Relaxation Delay 0.0050
Pulse Width 9.0000
Acquisition Time 1.3518
Spectrometer Frequency 300.13
Spectral Width 6009.6
Lowest Frequency -609.6
Nucleus 1H
Acquired Size 8124
Spectral Size 65536
3.78
1.92
4.00
6.00
3.18
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
ppm


197.41
194.24
193.63
157.39
133.87
133.81
129.81
128.76
95.81
77.58
77.16
76.74
30.37
25.76
22.18
^{13}C NMR (75.47 MHz, $CDCl_3$)
3-(((2-Methyl-1,3-dioxo-1,3-diphenylpropan-2-yl)oxy)imino)pentane-2,4-dione, 3n
Ph
Ph
O
N
Title i8290.{13C}.13.fid
Solvent CDCl3
Temperature 302.2
Pulse Sequence zgpg30
Number of Scans 166
Receiver Gain 3250.0
Relaxation Delay 1.0000
Pulse Width 13.0000
Acquisition Time 0.9002
Spectrometer Frequency 75.48
Spectral Width 18115.9
Lowest Frequency -749.9
Nucleus 13C
Acquired Size 16308
Spectral Size 65536
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm


7.54
7.54
7.53
7.51
7.51
7.50
7.48
7.45
7.45
7.45
7.31
7.29
7.26
7.23
7.23
7.21
7.21
7.08
7.07
7.06
7.05
3.91
2.16
2.09
^{1}H NMR (300.13 MHz, $CDCl_3$)
3-(((2-Benzyl-1,3-dioxo-1,3-diphenylpropan-2-yl)oxy)imino)pentane-2,4-dione, 3o
Ph
Bn
Ph
O
N
Title i8294.{1H}.1.fid
Solvent CDCl3
Temperature 300.0
Pulse Sequence zg
Number of Scans 1
Receiver Gain 32.1
Relaxation Delay 0.1000
Pulse Width 15.0000
Acquisition Time 2.7150
Spectrometer Frequency 300.13
Spectral Width 6009.6
Lowest Frequency -911.1
Nucleus 1H
Acquired Size 16316
Spectral Size 65536
6.25
8.20
2.05
2.00
3.10
2.98
ppm


197.37
194.04
193.67
157.32
134.61
134.07
133.59
130.98
129.71
128.71
128.54
127.63
99.38
77.58
77.16
76.74
41.71
30.13
25.96
^{13}C NMR (75.47 MHz, $CDCl_3$)
3-(((2-Benzyl-1,3-dioxo-1,3-diphenylpropan-2-yl)oxy)imino)pentane-2,4-dione, 3o
Ph
Bn
Ph
O
N
Title i8294.{13C}.1.fid
Solvent CDCl3
Temperature 298.0
Pulse Sequence zgpg30
Number of Scans 450
Receiver Gain 101.0
Relaxation Delay 0.8000
Pulse Width 10.0000
Acquisition Time 1.1431
Spectrometer Frequency 75.50
Spectral Width 17857.1
Lowest Frequency -1369.4
Nucleus 13C
Acquired Size 20412
Spectral Size 65536
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm