Electronic Supplementary Material (ESI) for Chemical Communications

Supplementary Information

Regioselectivity among six secondary hydroxyl groups: Selective acylation of the least reactive hydroxyl groups of inositol

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Materials and General Procedures

Chromatograms were visualized under UV light and by dipping plates into either phosphomolybdic acid in MeOH or anisaldehyde in ethanol, followed by heating. The ^{1}H NMR, COSY, and HMQC spectra were recorded on a Bruker (500 MHz) NMR spectrometer. Proton chemical shifts are reported in ppm (δ) relative to internal tetramethylsilane (TMS, δ 0.0 ppm) or with the solvent reference relative to TMS employed as the internal standard ($CDCl_3$, δ 7.26 ppm; D_2O, δ 4.79 ppm). Data are reported as follows: chemical shift (multiplicity [singlet (s), doublet (d), triplet (t), quartet (q), and multiplet (m)], coupling constants [Hz], integration and peak identification). All NMR signals were assigned on the basis of ^{1}H NMR, ^{13}C NMR, COSY and HMQC experiments. ^{13}C spectra were recorded with complete proton decoupling. Carbon chemical shifts are reported in ppm (δ) relative to TMS with the respective solvent resonance as the internal standard. All NMR data were collected at 25 °C. Melting points were determined using Stuart SMP30 melting point apparatus and are uncorrected. Flash column chromatography was performed using Silica Gel (200-400 mesh). All reactions were carried out under argon or nitrogen atmosphere employing oven dried glassware.

Preparation of H_2SO_4-silica

A slurry of 20 g of silica gel (200-400 mesh, FINAR make) with 150 mL of diethyl ether was made in a 250 mL RB flask. To this slurry, 1 mL of conc. H_2SO_4 was added very slowly with vigorous shaking over a period of 30 min. The ether was evaporated under reduced pressure and the free flowing silica was heated at 120 °C in an oven for 1h. The resulting solid was kept under high vaccum at room temperature for 2h and was stored in a desicator.

General procedure for the synthesis of 2-*O*-acyl-*myo*-inositol

A mixture of *myo*-inositol (2 mmol) and trialkylorthoester (2.4 mmol) in dry DMF / DMSO (10 mL) was stirred in presence of H_2SO_4-silica (50 mg) at 70 ^{o}C for 6-12 h under argon atmosphere. The resulting solution was neutralized by adding solid $NaHCO_3$, filtered and washed successively with reaction solvent. The combined filtrate was evaporated under reduced pressure (freeze drying in case of DMSO). The residue obtained was chromatographed using isopropanol and ethyl acetate (1:9 v/v) to yield respective 2-*O*-acyl-*myo*-inositol.

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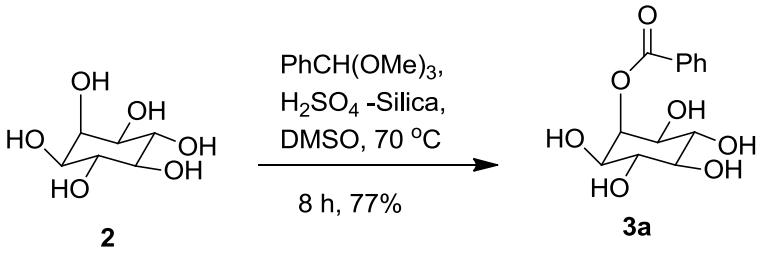
Synthetic Procedures and Characterization

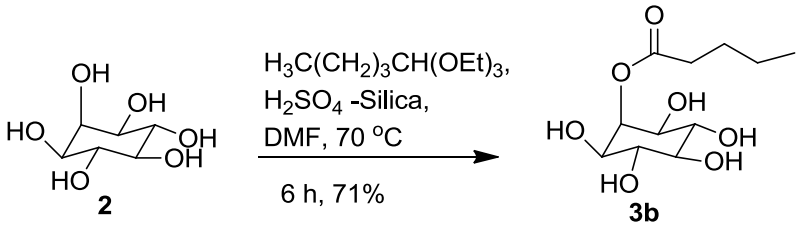
2-*O*-benzoyl-*myo*-inositol (3a)



A mixture of *myo*-inositol **2** (0.36 g, 2 mmol) and trimethylorthobenzoate (0.42 mL, 2.4 mmol) in dry DMSO (10 mL) was stirred in presence of H_2SO_4-silica (50 mg) at 70 ^oC for 8 h under argon atmosphere. The resulting solution was neutralized by adding solid $NaHCO_3$, filtered and washed with DMSO. The DMSO was removed by freeze drying. The residue thus obtained was chromatographed using isopropanol and ethyl acetate (1:9 v/v) to yield compound **3a** (0.44 g, 77%) as a white solid.

mp: 232-234 ^oC. ^1H NMR (500 MHz, DMSO-d6) δ 7.96-7.94 (m, 2H, Ar-H), 7.66 (t, *J* = 7.3 Hz, 1H, Ar-H), 7.55 (t, *J* = 7.7 Hz, 2H, Ar-H), 5.44 (s, 1H,), 4.91-4.79 (m, -OH), 3.49-3.15 (m, 4H), 3.06 (t, *J* = 6.4 Hz, H-5); ^{13}C NMR (125 MHz, DMSO-d6) δ 165.10, 132.93, 130.60, 129.14, 12.53, 75.75, 74.83, 73.21, 69.89. Elemental analysis: calcd for $C_{13}H_{16}O_7$: C, 54.93; H, 5.67%. Found: C, 54.76; H, 5.39%.

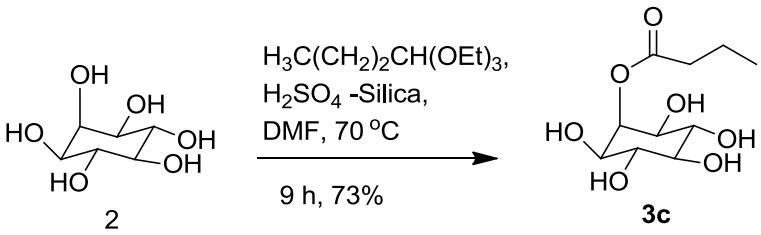
2-*O*-pentanoyl-*myo*-inositol (3b)



A mixture of *myo*-inositol (0.36 g, 2 mmol) and triethylorthovalerate (0.57 mL, 2.4 mmol) in dry DMF (10 mL) was stirred in presence of H_2SO_4-silica (50 mg) at 70 ^oC for 6 h under argon atmosphere. The resulting solution was neutralized by adding solid $NaHCO_3$, filtered and washed with DMF. The DMF was evaporated under reduced pressure. The residue thus obtained was chromatographed using isopropanol and ethyl acetate (1:9 v/v) to yield compound **3b** (0.37 g, 71%) as a white solid.

mp: 155-157 °C. ^{1}H NMR (500 MHz, DMSO-d6) δ 5.20 (t, *J* = 2.6 Hz, H-2), 4.73 (bs, -OH), 3.34-3.26 (m, 4H, Ins-H), 2.97 (t, *J* = 8.6 Hz, H-5), 2.28 (t, *J* =7.3 Hz, 2H), 1.54-1.50 (m, 2H), 1.49-1.31 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (125 MHz, DMSO-d6) δ 172.13, 74.80, 74.45, 73.00, 69.73, 33.65, 26.65, 21.54, 13.67. Elemental analysis: calcd for $C_{11}H_{20}O_7$: C, 49.99; H, 7.63%. Found: C, 49.83; H, 7.51%.

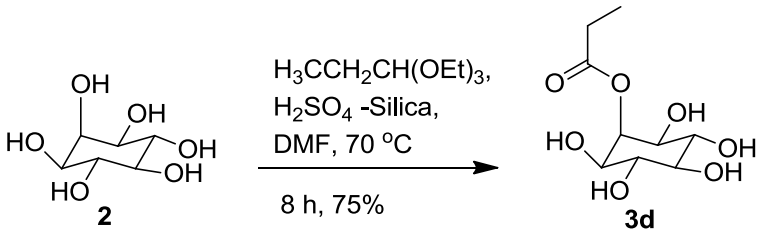
2-*O*-butanoyl-*myo*-inositol (3c)



A mixture of *myo*-inositol (0.36 g, 2 mmol) and triethylorthobutyrate (0.52 mL, 2.4 mmol) in dry DMF (10 mL) was stirred in presence of H_2SO_4-silica (50 mg) at 70 °C for 9 h under argon atmosphere. The resulting solution was neutralized by adding solid $NaHCO_3$, filtered and washed with DMF. The DMF was evaporated under reduced pressure. The residue thus obtained was chromatographed using isopropanol and ethyl acetate (1:9 v/v) to yield compound **3c** (0.363 g, 73%) as a white solid.

mp: 158-160 °C. ^{1}H NMR (500 MHz, DMSO-d6) δ 5.19 (t, *J* = 2.5 Hz, H-2), 4,70 (bs, -OH), 3.33-3.25 (m, 4H, Ins-H), 2.96 (t, *J* = 8.6 Hz, H-5), 2.24 (t, *J* = 7.2 Hz, 2H), 1.56-1.52 (m, 2H), 0.90 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, DMSO-d6) δ 171.99, 74.83, 74.43, 73.03, 69.77, 35.89, 18.05, 13.47. Elemental analysis: calcd for $C_{10}H_{18}O_7$: C, 48.00; H, 7.25%. Found: C, 48.24; H, 7.43%.

2-O-propanoyl-*myo*-inositol (3d)

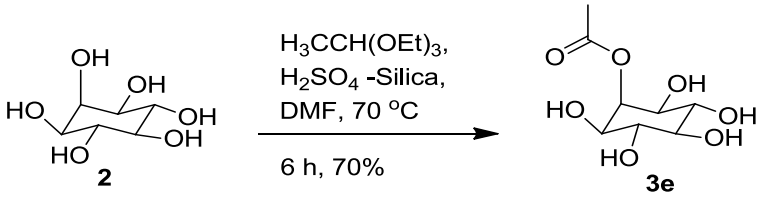


A mixture of *myo*-inositol (0.36 g, 2 mmol) and triethylorthopropionate (0.48 mL, 2.4 mmol) in dry DMF (10 mL) was stirred in presence of H_2SO_4-silica (50 mg) at 70 °C for 8 h under argon atmosphere. The resulting solution was neutralized by adding solid $NaHCO_3$, filtered and washed with DMF. The DMF was evaporated under reduced

pressure. The residue thus obtained was chromatographed using isopropanol and ethyl acetate (1:9 v/v) to yield compound **3d** (0.349 g, 75%) as a white solid.

mp: 164-166 ^{o}C. ^{1}H NMR (500 MHz, DMSO-d6) δ 5.19 (t, *J* = 2.6 Hz, H-2), 3.54 (bs, -OH), 3.33-3.27 (m, 4H, Ins-H), 2.97 (t, *J* = 8.5 Hz, H-5), 2.30 (q, *J* = 7.5 Hz, 2H), 1.03 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, DMSO-d6) δ 173.00, 74.61, 74.46, 72.81, 69.61, 27.14, 9.06. Elemental analysis: calcd for $C_9H_{16}O_7$: C, 45.76; H, 6.83%. Found: C, 45.48; H, 6.91%.

2-*O*-acetyl-*myo*-inositol (3e)



A mixture of *myo*-inositol (0.36 g, 2 mmol) and triethylorthoacetate (0.37 mL, 2.4 mmol) in dry DMF (10 mL) was stirred in presence of H_2SO_4-silica (50 mg) at 70 ^{o}C for 6 h under argon atmosphere. The resulting solution was neutralized by adding solid $NaHCO_3$, filtered and washed with DMF. The DMF was evaporated under reduced pressure. The residue obtained was chromatographed using isopropanol and ethyl acetate (1:9 v/v) to yield compound **3e** (0.31 g, 70%) as a white solid.

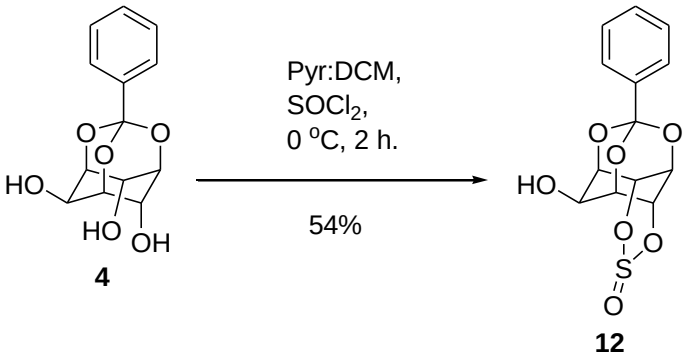
mp: 170-172 ^{o}C. ^{1}H NMR (500 MHz, DMSO-d6) δ 5.18 (t, *J* = 2.5 Hz, H-2), 4.70 (bs, -OH), 3.33-3.27 (m, 4H, Ins-H), 2.97 (t, *J* = 8.5 Hz, H-5), 1.99 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d6) δ 169.69, 74.83, 74.68, 72.99, 69.76, 21.05. Elemental analysis: calcd for $C_8H_{14}O_7$: C, 43.24; H, 6.35%. Found: C, 43.11; H, 6.17%.

2-*O*-formyl-*myo*-inositol (3f)

A mixture of *myo*-inositol (0.36 g, 2 mmol) and triethylorthoformate (0.42 mL, 2.4 mmol) in dry DMF (10 mL) was stirred in presence of H_2SO_4-silica (50 mg) at 70 ^{o}C for 6 h under argon atmosphere. A major spot (Rf 0.15, isopronaol: ethylacetate 1:9 v/v) was observed just above the *myo*-inositol. The resulting solution was neutralized by adding solid $NaHCO_3$ and the filtrate was evaporated. However, during the filtration and chromatography the labile formate ester got hydrolyzed to the inositol.[1]

Supplementary Information

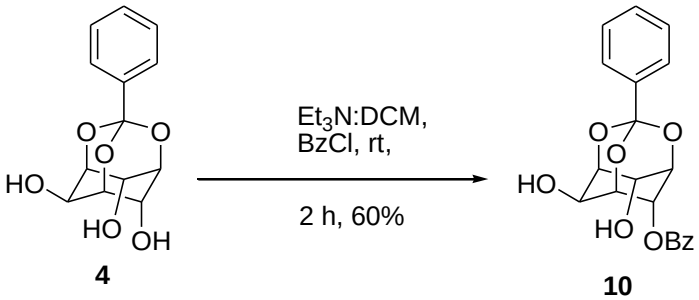
Myo-inositol-4,6-_O_-sulfite-1,3,5-orthobenzoate (12)



To a solution of *myo*-inositol-1,3,5-orthobenzoate[2] (0.11 g, 0.41 mmol) in a mixture of pyridine and dichloromethane (1:4, v/v, 10 mL) was added thionylchloride (0.04 mL, 0.49 mmol) drop wise at 0 ^{o}C. The reaction was stirred at 0 ^{o}C for 2 h. After the completion of starting material (checked by TLC), the reaction mixture was concentrated under reduced pressure. The residue was co-evaporated with toluene to dryness. The resulting residue was adsorbed on silica by dissolving it into dichloromethane. The crude product was purified by column chromatography using ethyl acetate and petroleum ether (1:4, v/v) to yield **12** (0.08 g, 54 %) as a white solid.

mp: 115-117 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56-7.54 (m, 2H, Ar-*H*), 7.36-7.31 (m, 3H, Ar-*H*), 5.84 (t, *J* = 4.2 Hz, 1H, H-5), 5.22 (t, *J* = 4.5 Hz, 2H, H-4 & H-6), 4.46 (dd, *J* = 2.85 and 1.60 Hz, 2H, H-1 & H-3), 4.14 (bs, 1H, H-2); ^{13}C NMR (125 MHz, $CDCl_3$) δ 135.51, 130.22, 128.25, 125.22, 107.31, 72.52, 68.55, 60.70, 60.27. Elemental analysis: calcd for $C_{13}H_{12}O_7S$: C, 50.00; H, 3.87; S, 10.27%. Found: C, 50.18; H, 3.79; S, 10.42%.

4-_O_-benzoyl-_myo_-inositol 1,3,5-othobenzoate (10)



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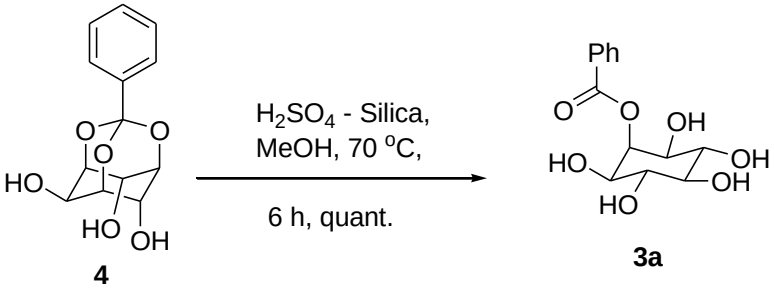
To a solution of *myo*-inositol-1,3,5-orthobenzoate (0.180 g, 0.67 mmol) in a mixture of triethylamine and dichloromethane (1:4, v/v, 10 mL) benzoyl chloride (0.09 mL, 0.74 mmol) was added drop wise at 0 ^{o}C. The reaction was allowed to stir at rt for 2 h. After the completion of starting material (checked by TLC), the reaction mixture was concentrated under reduced pressure. The residue was dissolved in dichloromethane (20 mL) and washed successively with water (3 x 10 mL) and brine (10 mL). The organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by column chromatography using ethyl acetate and petroleum ether (1:4, v/v) to yield **10** (0.145 g, 60 %) as a white solid.

mp: 135-137 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94-7.92 (m, 2H, Ar-*H*), 7.61-7.59 (m, 2H, Ar-*H*), 7.54 (t, *J* = 7.5 Hz, 1H, Ar-*H*), 7.39 (t, *J* = 7.5 Hz, 2H, Ar-*H*), 7.33-7.31 (m, 3H, Ar-*H*), 5.87-5.86 (m, 1H, H-4), 4.73 (bs, 1H, H-6), 4.58-4.57 (m, 1H, H-5), 4.50 (dd, *J* = 3.90 and 1.80 Hz, 1H, H-3), 4.39 (dd, *J* = 3.90 and 1.90 Hz, 1H, H-1), 4.22 (bs, 1H, H-2); ^{13}C NMR (125 MHz, $CDCl_3$**)** δ 165.03, 136.40, 133.83, 129.88, 129.81, 128.91, 128.74, 128.18, 125.30, 107.80, 75.62, 73.31, 69.05, 68.59, 67.35, 60.29. Elemental analysis: calcd for $C_{20}H_{18}O_7$: C, 64.86; H, 4.90%. Found: C, 64.63; H, 4.87%.

Hydrolysis of *myo*-inositol 1,3,5-orthobenzoate (4) in wet methanol



A mixture of *myo*-inositol-1,3,5-orthobenzoate (0.05 g, 0.18 mmol) and H_2SO_4-silica (20 mg) in methanol (5 mL) was stirred at 70 ^{o}C. The reaction was monitored by TLC. When the reaction was complete (6 h), the reaction mixture was neutralized by adding solid $NaHCO_3$, filtered and washed with MeOH (10 mL). The MeOH was evaporated under reduced pressure. The residue thus obtained was chromatographed using isopropanol and ethyl acetate (1:9, v/v) to yield compound **3a** (0.045 g, quant) as a white solid.

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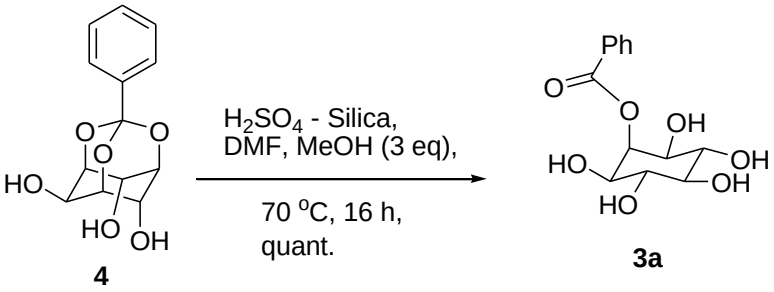
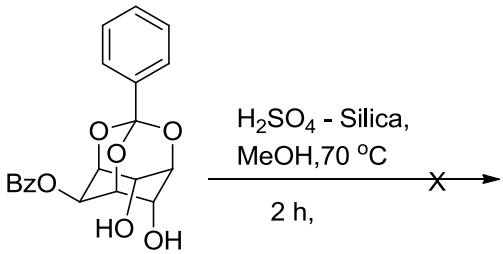
Hydrolysis of *myo*-inositol 1,3,5-orthobenzoate (4) in dry DMF and 3 equiv of methanol



4 **3a**

A mixture of *myo*-inositol-1,3,5-orthobenzoate (0.05 g, 0.18 mmol), dry methanol (22 μL, 0.54 mmol, 3 eq) and H_2SO_4-silica (20 mg) in dry DMF (5 mL) was stirred at 70 °C under argon atmosphere. The reaction was monitored by TLC. When the reaction was complete (16 h), the reaction mixture was neutralized by adding solid $NaHCO_3$, filtered and washed with MeOH (10 mL). The solvents were evaporated under reduced pressure. The residue thus obtained was chromatographed using isopropanol and ethyl acetate (1:9, v/v) to yield compound **3a** (0.045 g, quant) as a white solid.

Hydrolysis of 2-*O*-benzoyl-*myo*-inositol 1,3,5-orthobenzoate (7)



7

To a solution of 2-*O*-benzoyl-*myo*-inositol1,3,5-orthobenzoate[3] (0.05 g, 0.13 mmol) in methanol (5 mL) was added H_2SO_4-silica (20 mg) and stirred the reaction at 70 °C for 2 h. There was no change in starting material as judged by TLC. The reaction was further heated to 80 °C for 12 h. There was no change in reaction mixture and the starting material could be recovered.

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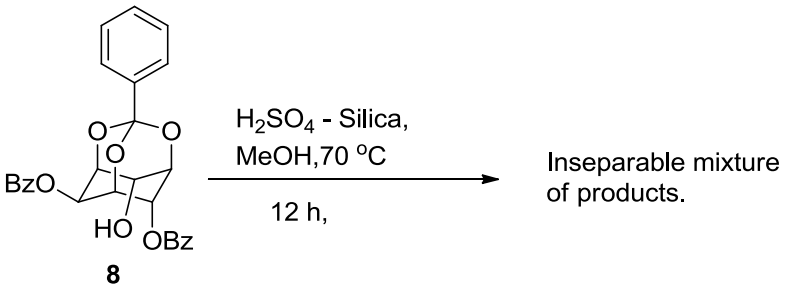
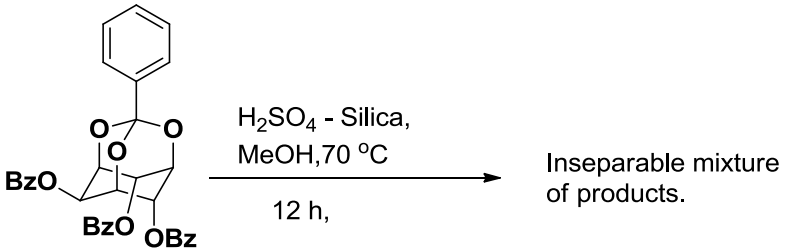
Hydrolysis of 2,4-di-*O*-benzoyl-*myo*-inositol1,3,5-orthobenzoate (8)



8

To a solution of 2,4-di-*O*-benzoyl-*myo*-inositol 1,3,5-orthobenzoate[3] (0.05 g, 0.10 mmol) in methanol (5 mL), H_2SO_4-silica (20 mg) was added and the mixture was stirred at 70 °C for 12 h. There was no reaction as judged by TLC. However, an inseparable mixture of high polar products was obtained when the reaction was heated to 80 °C for 60 h.

Hydrolysis of 2,4,6-tri-*O*-benzoyl-*myo*-inositol 1,3,5-orthobenzoate (9)



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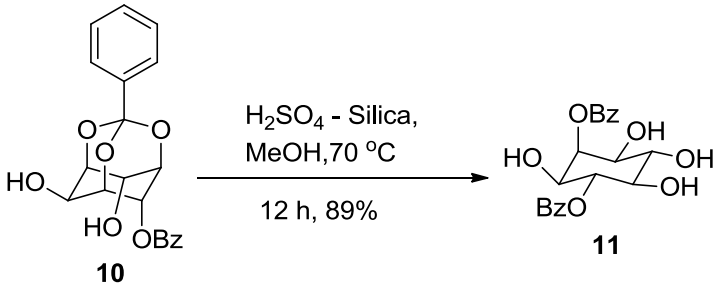
To a solution of 2,4,6-tri-*O*-benzoyl-*myo*-inositol 1,3,5-orthobenzoate[3] (0.05 g, 0.086 mmol) in methanol (5 mL), H_2SO_4-silica (20 mg) was added and the mixture was stirred at 70 °C for 12 h. There was no change in starting material as judged by TLC. As in the case of dibenzoate, an inseparable mixture of products was obtained when the reaction was done at higher temperature (80 °C) for several four days.

Hydrolysis of 4-*O*-benzoyl-*myo*-inositol1,3,5-othobenzoate (10)



10 **11**

Supplementary Information

To a solution of 4-*O*-benzoyl-*myo*-inositol 1,3,5-othobenzoate (0.15 g, 0.4 mmol) in methanol (10 mL), H_2SO_4-silica (50 mg) was added and the mixture was stirred at 70 ^oC. The reaction was monitored by TLC. When the reaction was complete (12 h), it was quenched by adding solid $NaHCO_3$. The resulting mixture was filtered and washed with methanol. The solvent was evaporated under reduced pressure. The crude product was purified by flash chromatography using ethyl acetate and petroleum ether (9:1, v/v) as the eluent to get the known[4] compound **11** (0.14 g, 89%) as a white solid. The ^1H NMR of **11** was reported in CD_3OD. We have compared the NMR data of our sample in CD_3OD with the reported data and found that they are identical. However, we observed that dibenzoate **11** underwent some transesterification in CD_3OD to give minor amounts of impurities (See ^1H NMR spectrum on page No. S62). But dibenzoate **11** was stable in DMSO-d6 solution. So we have recorded the ^1H NMR spectrum of **11** in DMSO-d6 to establish its purity. Also a COSY NMR was done to assign the peaks.

Reported data [4]

mp: 193-195 °C, ^1H NMR (500 MHz, CD_3OD) δ 8.14-7.43 (m, 10H, 2Ph), 5.75 (app. t, *J* = 2.8 Hz, 1H, H-2), 5.53 (app. t, *J* = 9.9 Hz, 1H, H-4), 4.01 (dd, *J* = 10.1 Hz & 2.8 Hz, 1H, H-3), 3.85 (app. t, *J* = 9.5 Hz, 1H, H-6), 3.73 (dd, *J* = 9.8 Hz & 2.7 Hz, 1H, H-1), 3.60 (app. t, *J* = 9.4 Hz, 1H, H-5).

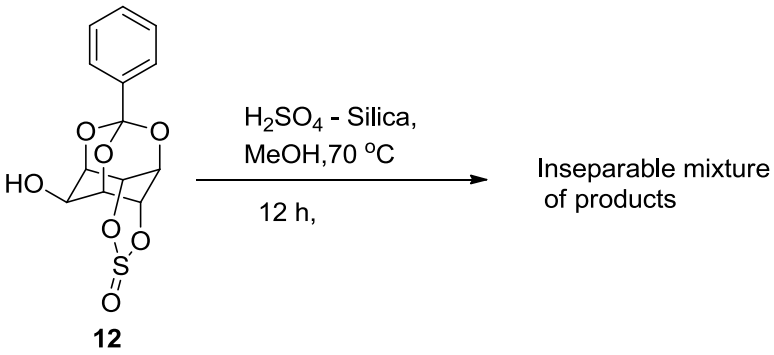
Data obtained

mp: 194 °C, ^1H NMR (500 MHz, CD_3OD) δ 8.03 (d, *J* = 7.3 Hz, 2H, Ar-H), 7.98 (d, *J* = 7.3 Hz, 2H, Ar-H), 7.54-7.47 (m, 2H, Ar-H), 7.43-7.35 (m, 4H, Ar-H), 5.65 (t, *J* = 2.8 Hz, 1H, H-2), 5.44 (t, *J* = 9.9 Hz, 1H, H-4), 3.92 (dd, *J* = 10.1 Hz & 2.8 Hz, 1H, H-3), 3.76 (t, *J* = 9.6 Hz, 1H, H-6), 3.64 (dd, *J* = 9.8 Hz & 2.8 Hz, 1H, H-1), 3.51 (t, *J* = 9.4 Hz, 1H, H-5).

^1H NMR (500 MHz, DMSO-d6) δ 8.03-7.98 (m, 4H, Ar-H), 7.71-7.52 (m, 6H, Ar-H), 5.53 (t, *J* = 2.5 Hz, 1H, H-2), 5.32-5.28 (m, 2H, H-4, 3-OH), 5.16 (d, *J* = 5.6 Hz , 5-OH) , 5.09-5.04 (m, 2H, 1-OH, 6- OH), 3.92-3.88 (m, 1H, H-3), 3.63-3.58 (m, 2H, H-1, H-6), 3.46-3.43 (m, 1H, H-5).

Hydrolysis of *myo*-inositol-4,6-*O*-sulfite-1,3,5-orthobenzoate (12)

Supplementary Information



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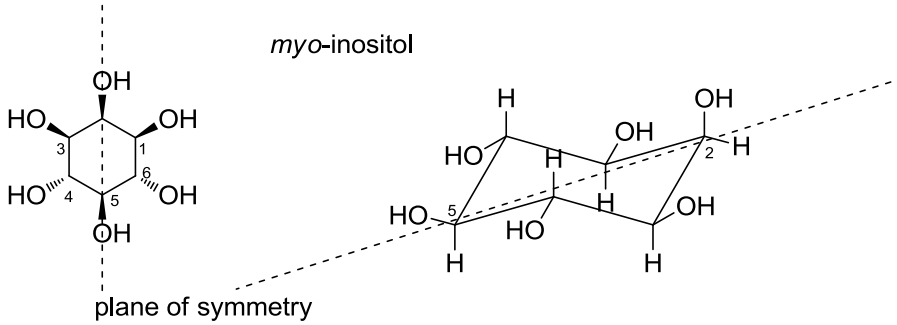
A mixture of *myo*-inositol-4,6-*O*-sulfite-1,3,5-orthobenzoate (0.05 g, 0.16 mmol) and H_2SO_4-silica (20 mg) in methanol (5 mL) was stirred at 70 ^oC for 12 h. There was no change in starting material as judged by TLC. But prolonged (4 days) heating at 80 ^oC resulted in the formation of an inseparable mixture of products.

Structural Assignment of 2-*O*-esters

Myo-inositol being a meso compound with a plane of symmetry through C2 and C5 (see figure below), esterification of C2-OH or C5-OH gives a meso product and hence a symmetrical NMR spectrum with four signals in the ratio 1:2:2:1 and monoesterification at any other hydroxyl will result in an unsymmetrical product (hence unsymmetrical spectra). ^{13}C NMR of all the monoesters obtained from H_2SO_4-silica catalyzed reaction between *myo*-inositol and trialkylorthoester revealed that they are symmetrical compounds. Since H-2 is an equatorially oriented hydrogen flanked by two axially oriented hydrogens on neighboring carbons (dihedral angle ϕ around 60°), its $^3J_{HH}$ coupling constants will be small. As H-5 is an axial hydrogen flanked by two axial hydrogens (dihedral angle ϕ around 180°), its $^3J_{HH}$ coupling constants will be higher. H of –CH(OH)- motif will shift downfield on esterification of the OH and hence it is easy to determine the position of esterification. The most downfield shifted hydrogen (δ >5.2 ppm; as a result of esterification) in all these monoesters (**3a-e**), showed a smaller coupling constant of ≈2.6 Hz suggesting that 2-OH is acylated and the other lone proton (upfield) showed a large coupling constant of ≈8.6 Hz (H-5) confirming that 5-OH is not acylated. These signal assignments were further confirmed by 2D NMR.

Supplementary Information



Reactivity comparison of H_2SO_4-silica with other acid catalysts

Treatment of *myo*-inositol with trialkylorthoesters in presence of acid catalysts such as PTSA,[5] Camphorsulfonic acid,[6] Amberlyst[7] and triflic acid[8] have been reported to yield *myo*-inositol 1,3 5-orthoesters in good yields. We have reproduced some of these results in our own lab several times and in all the cases, we got only the corresponding orthoesters but not the 2-O-ester. Thus H_2SO_4-silica is a special case wherein the corresponding 2-O-ester is formed regioselectively instead of the orthoesters.

Establishment of regioselectivity

The ^{13}C NMR of the crude reaction mixture showed minor amounts of inositol along with the major 2-O-esters. In order to prove that the minor impurity was not any other isomeric ester but just inositol, we have added *myo*-inositol to a pure sample of **3a** (approximately 1:1 ratio) and recorded its ^{13}C NMR. A comparison of this mixed NMR spectrum with ^{13}C NMR spectrum of the crude reaction mixture established that the minor impurity in the crude mixture is just inositol.

References

[1] S. W. Garret, C. Liu, A. M. Riley, B. V. L. Potter, *J. Chem. Soc. Perkin Trans. 1*, **1998**, 1367-1368.

[2] G. Bhosekar, C. Murali, R. G. Gonnade, M. S. Shashidhar, M. M. Bhadbhade, *Crystal Growth & Design* **2005**, *5*, 1977-1982.

[3] C. Murali, R. G. Gonnade, M. S. Shashidhar, M. M. Bhadbhade, *Eur. J. Org. Chem.* **2007**, 1153–1159.

[4] S-K. Chung, *et al*. *J. Org. Chem.* **2002**, *67*, 5626-5637.

[5] H. W. Lee and Y. Kishi. *J. Org. Chem.* **1985**, *50*, 4402-4404.

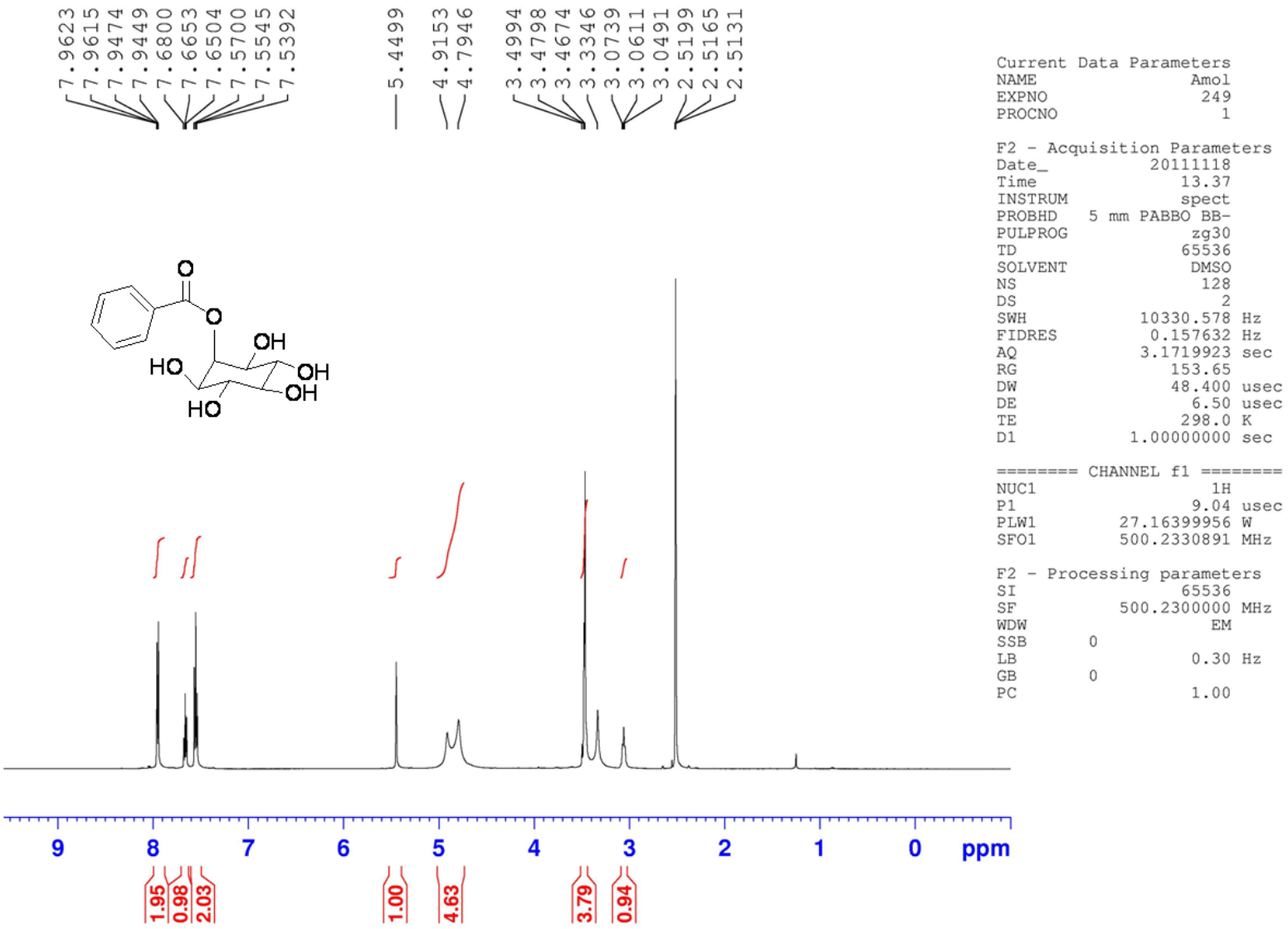
[6] M. A. Biamonte and A. Vasella, *Helv. Chem. Acta*. **1998**, *81*, 4, 688-694.

[7] A. Krief, W. Dumont, D. Billen, J. J. Leteson, P. Lestrate, P. J. Murphy and D. Lacroix *Tetrahedron Lett.* **2004**, *45*, 1461-1463.

[8] P. Andersch and M. P. Schneider, *Tetrahedron Asym.* **1993**, *4*, 2135-2138.

Supplementary Information

^{1}H NMR of **3a** in DMSO- d_6

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EXPNO                  249
PROCNO                   1

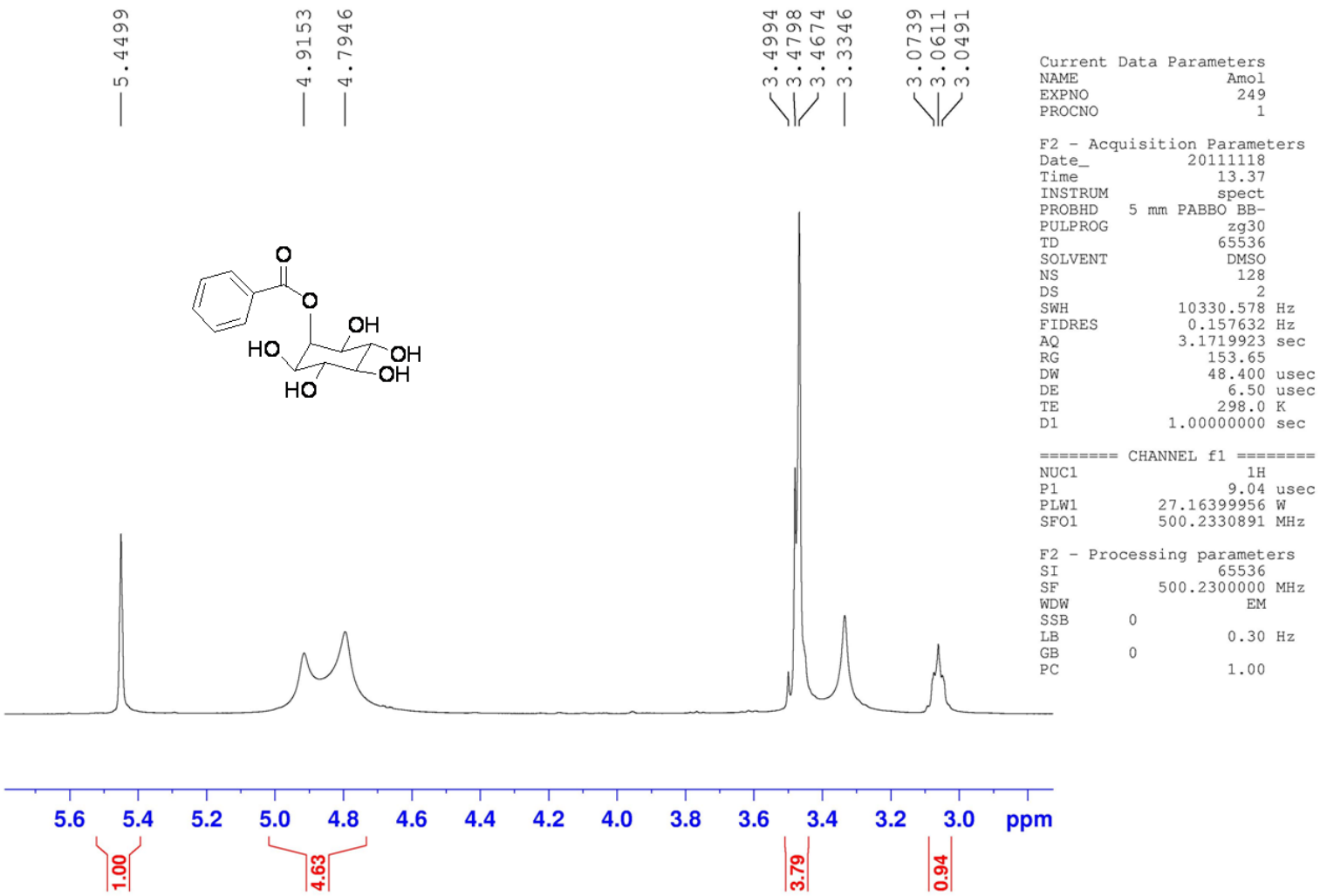
F2 - Acquisition Parameters
Date_           20111118
Time               13.37
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                   128
DS                     2
SWH            10330.578 Hz
FIDRES          0.157632 Hz
AQ             3.1719923 sec
RG                153.65
DW                48.400 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.04 usec
PLW1         27.16399956 W
SFO1         500.2330891 MHz

F2 - Processing parameters
SI                 65536
SF          500.2300000 MHz
WDW                  EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Supplementary Information

^{1}H NMR of **3a** (zoom)

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Current Data Parameters
NAME                  Amol
EXPNO                  249
PROCNO                   1

F2 - Acquisition Parameters
Date_              20111118
Time                 13.37
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT               DMSO
NS                     128
DS                       2
SWH              10330.578 Hz
FIDRES            0.157632 Hz
AQ               3.1719923 sec
RG                  153.65
DW                 48.400 usec
DE                  6.50 usec
TE                 298.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.04 usec
PLW1          27.16399956 W
SFO1          500.2330891 MHz

F2 - Processing parameters
SI                   65536
SF            500.2300000 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```
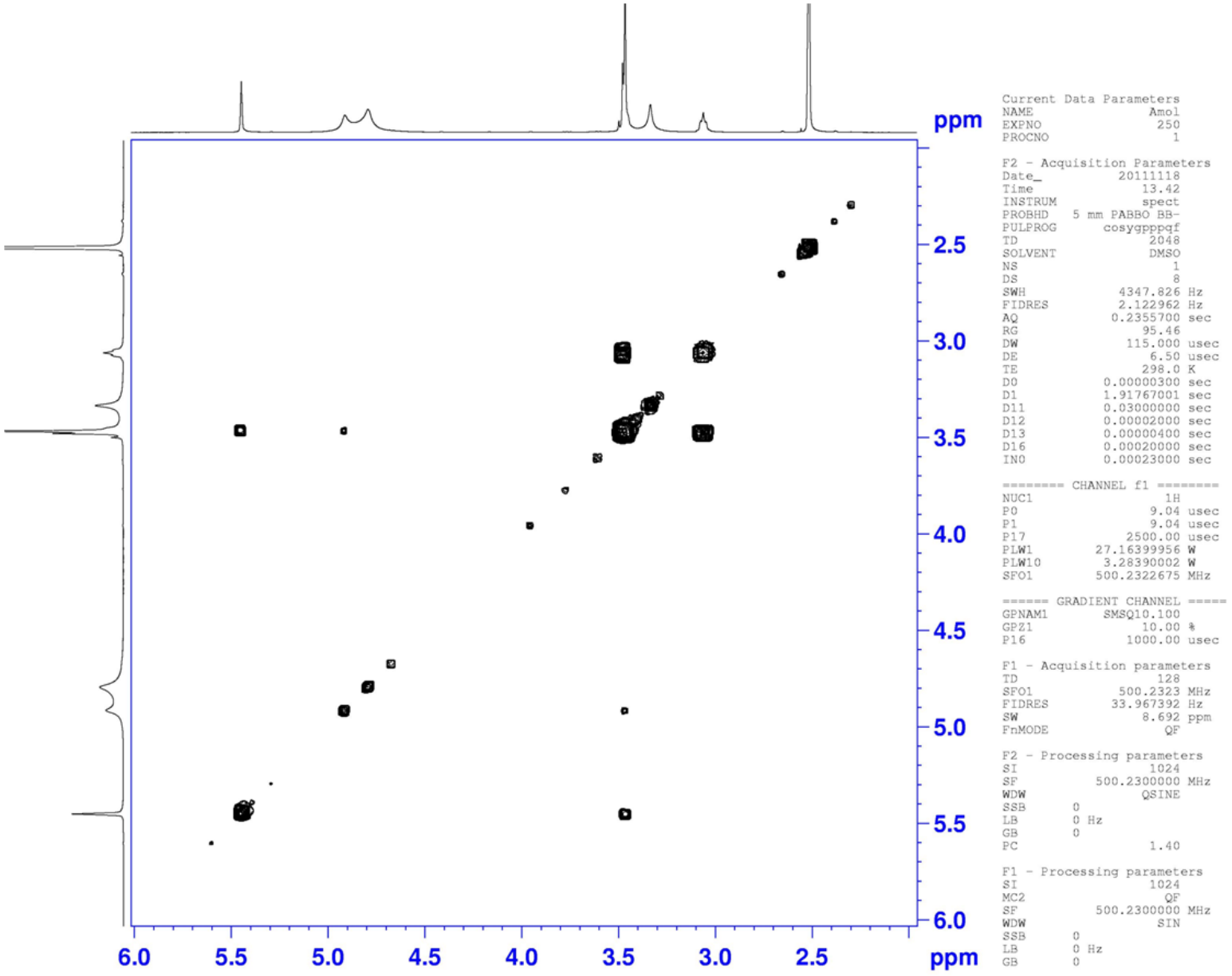

Supplementary Information

COSY of **3a**

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EXPNO                  250
PROCNO                   1

F2 - Acquisition Parameters
Date_              20111118
Time                 13.42
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG       cosygpppqf
TD                    2048
SOLVENT               DMSO
NS                       1
DS                       8
SWH               4347.826 Hz
FIDRES            2.122962 Hz
AQ                0.2355700 sec
RG                    95.46
DW                  115.000 usec
DE                    6.50 usec
TE                   298.0 K
D0                0.00000300 sec
D1                1.91767001 sec
D11               0.03000000 sec
D12               0.00002000 sec
D13               0.00000400 sec
D16               0.00020000 sec
IN0               0.00023000 sec

======== CHANNEL f1 ========
NUC1                     1H
P0                    9.04 usec
P1                    9.04 usec
P17                2500.00 usec
PLW1              27.16399956 W
PLW10              3.28390002 W
SFO1             500.2322675 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SMSQ10.100
GPZ1                 10.00 %
P16                1000.00 usec

F1 - Acquisition parameters
TD                       128
SFO1              500.2323 MHz
FIDRES           33.967392 Hz
SW                    8.692 ppm
FnMODE                   QF

F2 - Processing parameters
SI                      1024
SF             500.2300000 MHz
WDW                   QSINE
SSB          0
LB           0 Hz
GB           0
PC                    1.40

F1 - Processing parameters
SI                      1024
MC2                       QF
SF             500.2300000 MHz
WDW                     SIN
SSB          0
LB           0 Hz
GB           0
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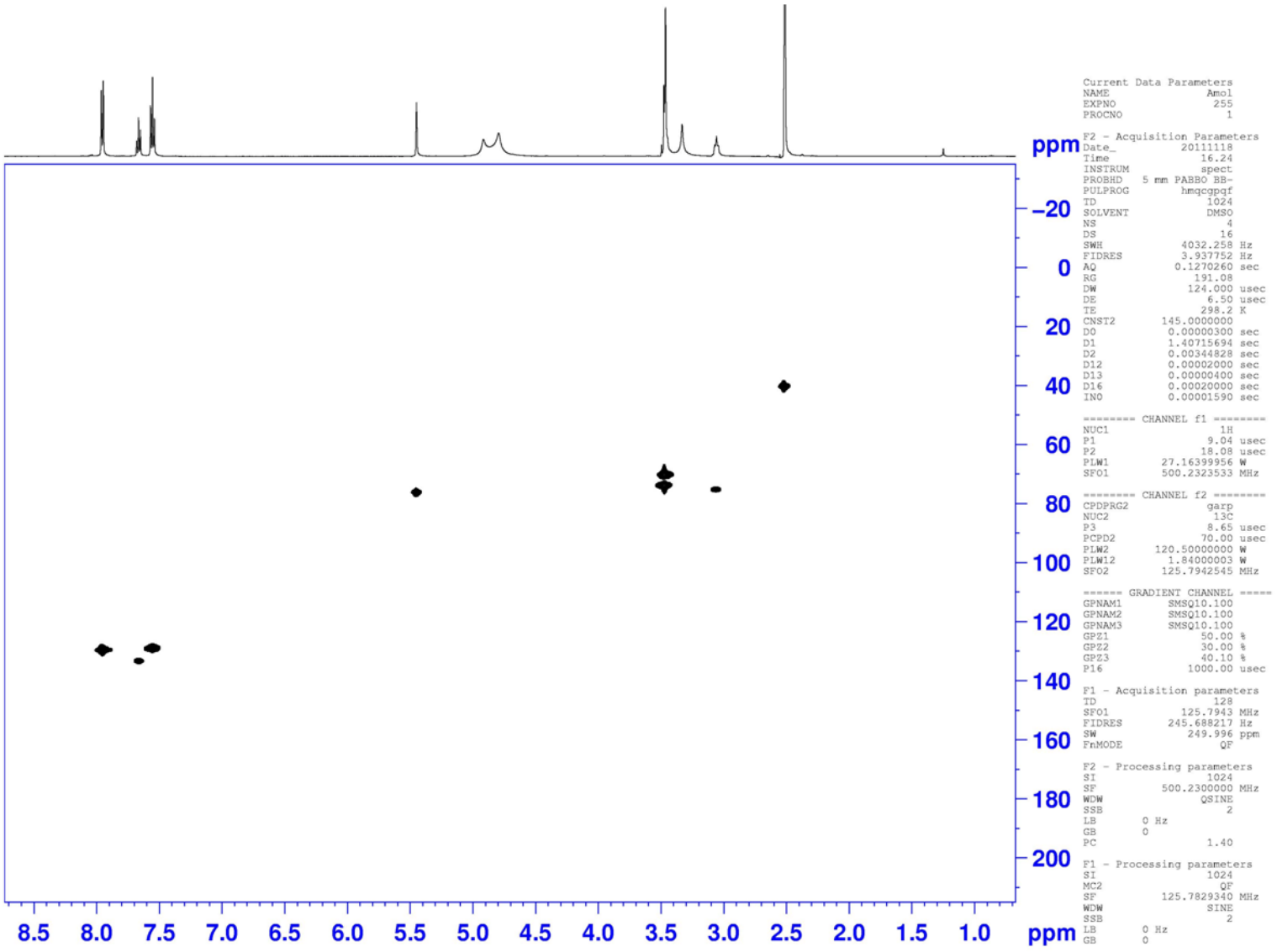
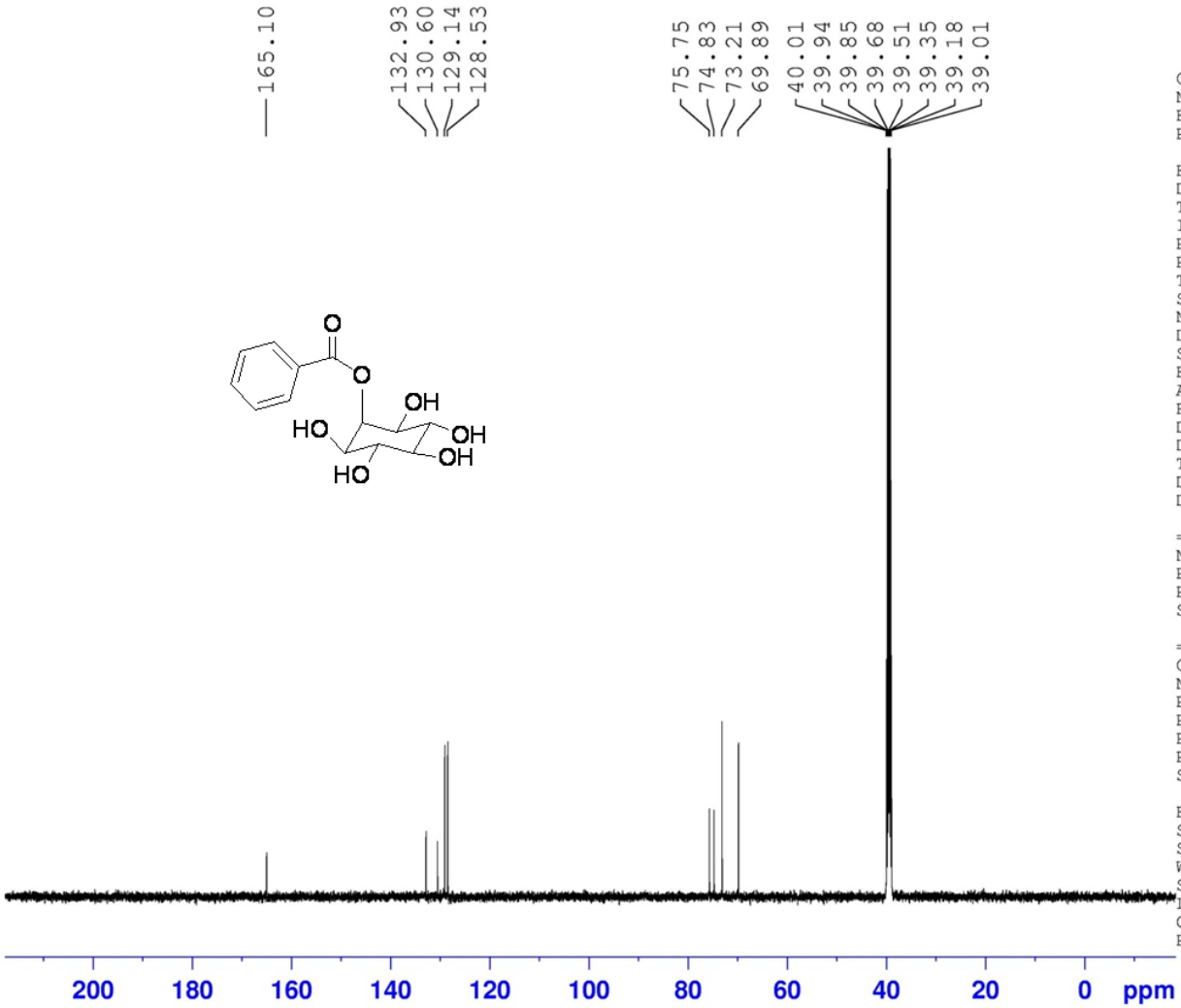
Supplementary Information

HMQC of **3a**



Supplementary Information

^{13}C NMR of **3a** in DMSO-d_6

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Current Data Parameters
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EXPNO                  240
PROCNO                   1

F2 - Acquisition Parameters
Date_              20111117
Time                 16.50
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT                DMSO
NS                       512
DS                         4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ                1.1010548 sec
RG                   191.08
DW                  16.800 usec
DE                   6.50 usec
TE                  300.1 K
D1               2.00000000 sec
D11              0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                     8.65 usec
PLW1          120.50000000 W
SFO1           125.7955112 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PLW2          27.16399956 W
PLW12          0.34685999 W
PLW13          0.22199000 W
SFO2          500.2320009 MHz

F2 - Processing parameters
SI                      32768
SF           125.7829969 MHz
WDW                        EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
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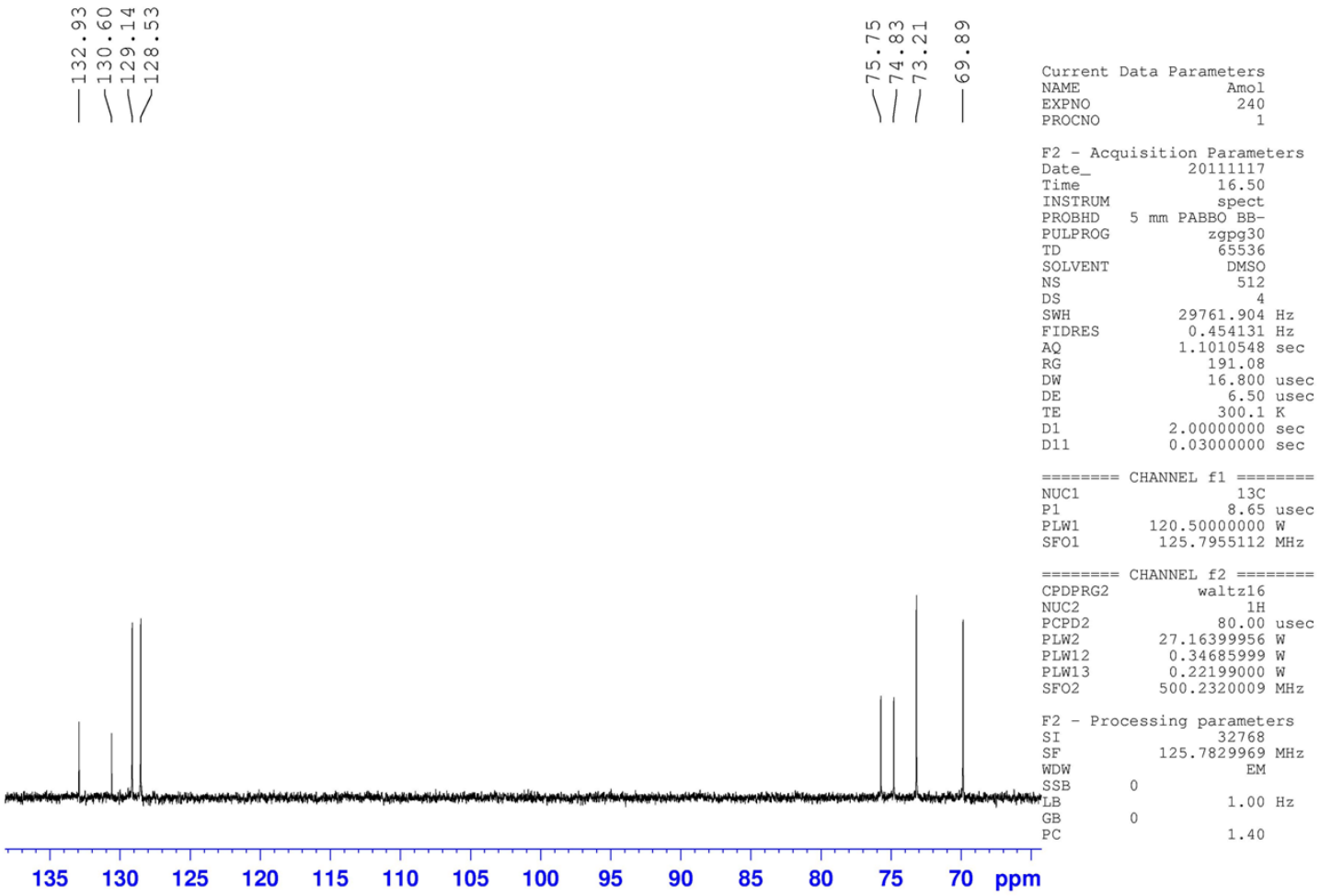
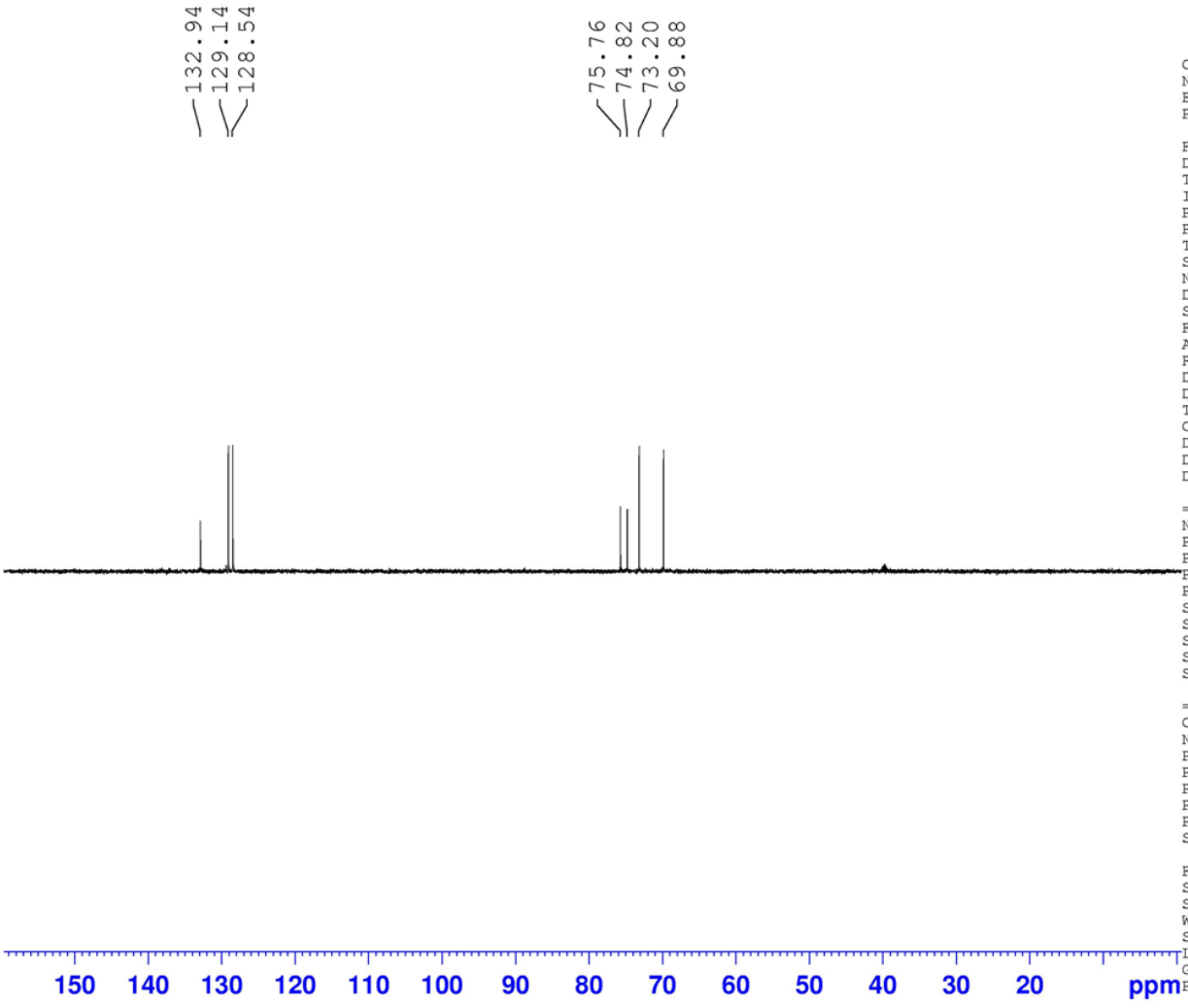
Supplementary Information

^{13}C NMR of **3a** (zoom)



Supplementary Information

DEPT of **3a**

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Current Data Parameters
NAME                  Amol
EXPNO                  227
PROCNO                   1

F2 - Acquisition Parameters
Date_           20111117
Time                9.47
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG        deptsp135
TD                 65536
SOLVENT             DMSO
NS                    256
DS                      4
SWH            20161.291 Hz
FIDRES          0.307637 Hz
AQ             1.6253428 sec
RG                191.08
DW                24.800 usec
DE                 6.50 usec
TE                298.0 K
CNST2        145.0000000
D1             2.00000000 sec
D2             0.00344828 sec
D12            0.00002000 sec

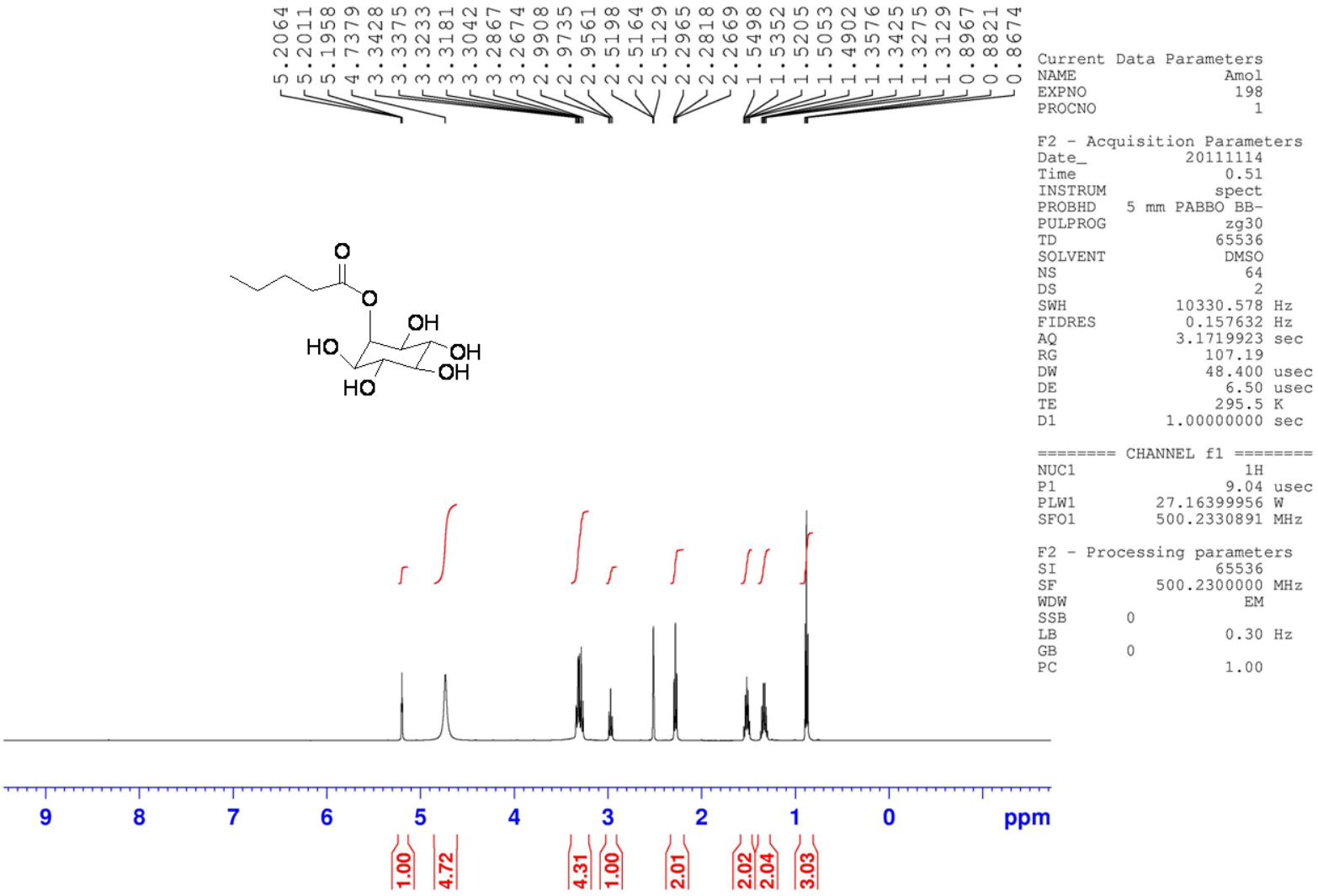
======== CHANNEL f1 ========
NUC1                 13C
P1                  8.65 usec
P13              2000.00 usec
PLW0      0 W
PLW1        120.50000000 W
SFO1         125.7929956 MHz
SPNAM5       Crp60comp.4
SPOAL5             0.500
SPOFFS5   0 Hz
SPW5          13.77600002 W

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                 1H
P3                  9.03 usec
P4                 18.06 usec
PCPD2              80.00 usec
PLW2         27.16399956 W
PLW12         0.34685999 W
SFO2         500.2315998 MHz

F2 - Processing parameters
SI                 32768
SF           125.7829969 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Supplementary Information

^{1}H NMR of **3b** in DMSO-d_6

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Current Data Parameters
NAME                  Amol
EXPNO                  198
PROCNO                   1

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Date_              20111114
Time                  0.51
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT               DMSO
NS                       64
DS                        2
SWH              10330.578 Hz
FIDRES            0.157632 Hz
AQ               3.1719923 sec
RG                  107.19
DW                  48.400 usec
DE                   6.50 usec
TE                  295.5 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.04 usec
PLW1          27.16399956 W
SFO1          500.2330891 MHz

F2 - Processing parameters
SI                   65536
SF            500.2300000 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
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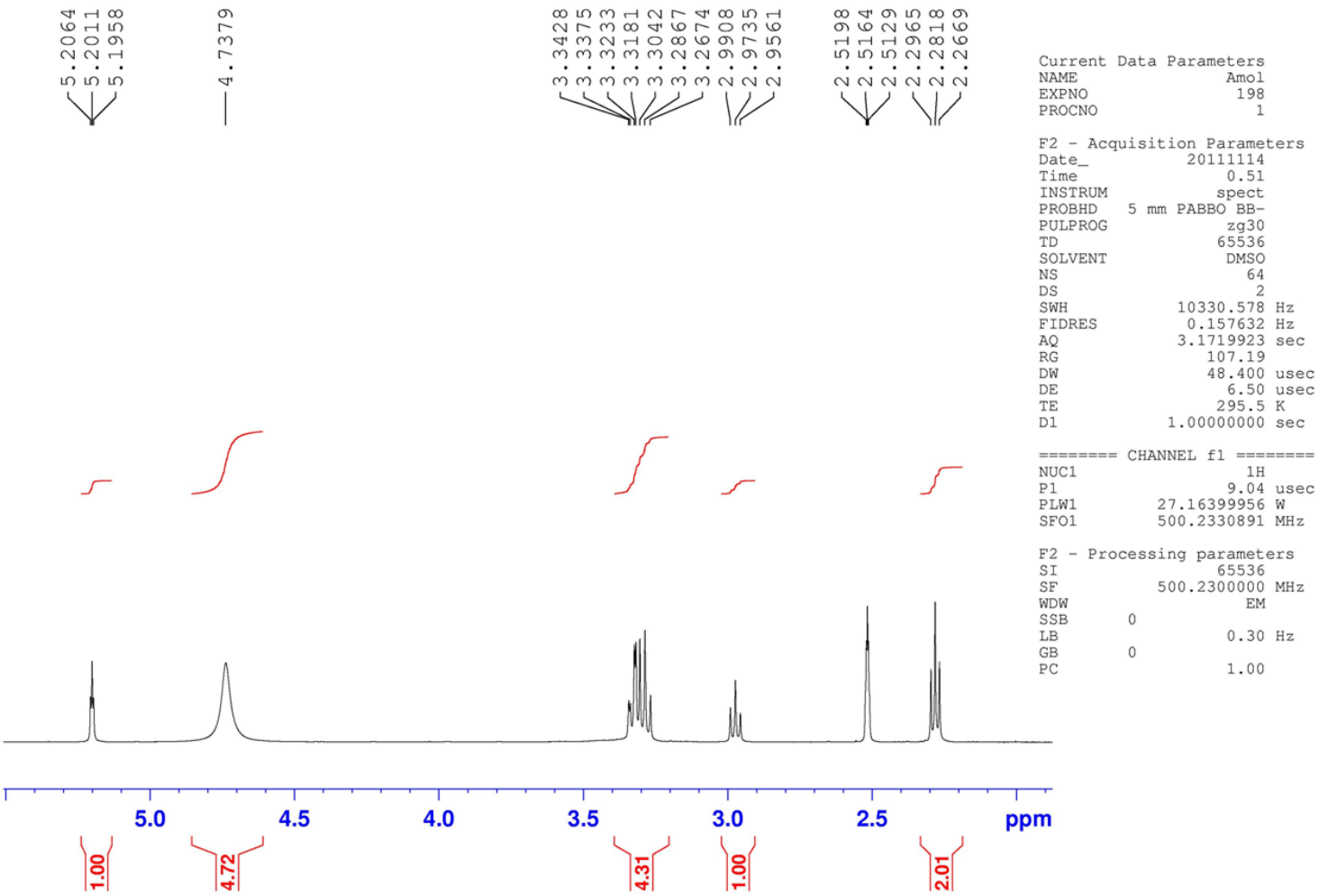
Supplementary Information

^{1}H NMR of **3b** (zoom)




```
Current Data Parameters
NAME                  Amol
EXPNO                  198
PROCNO                   1

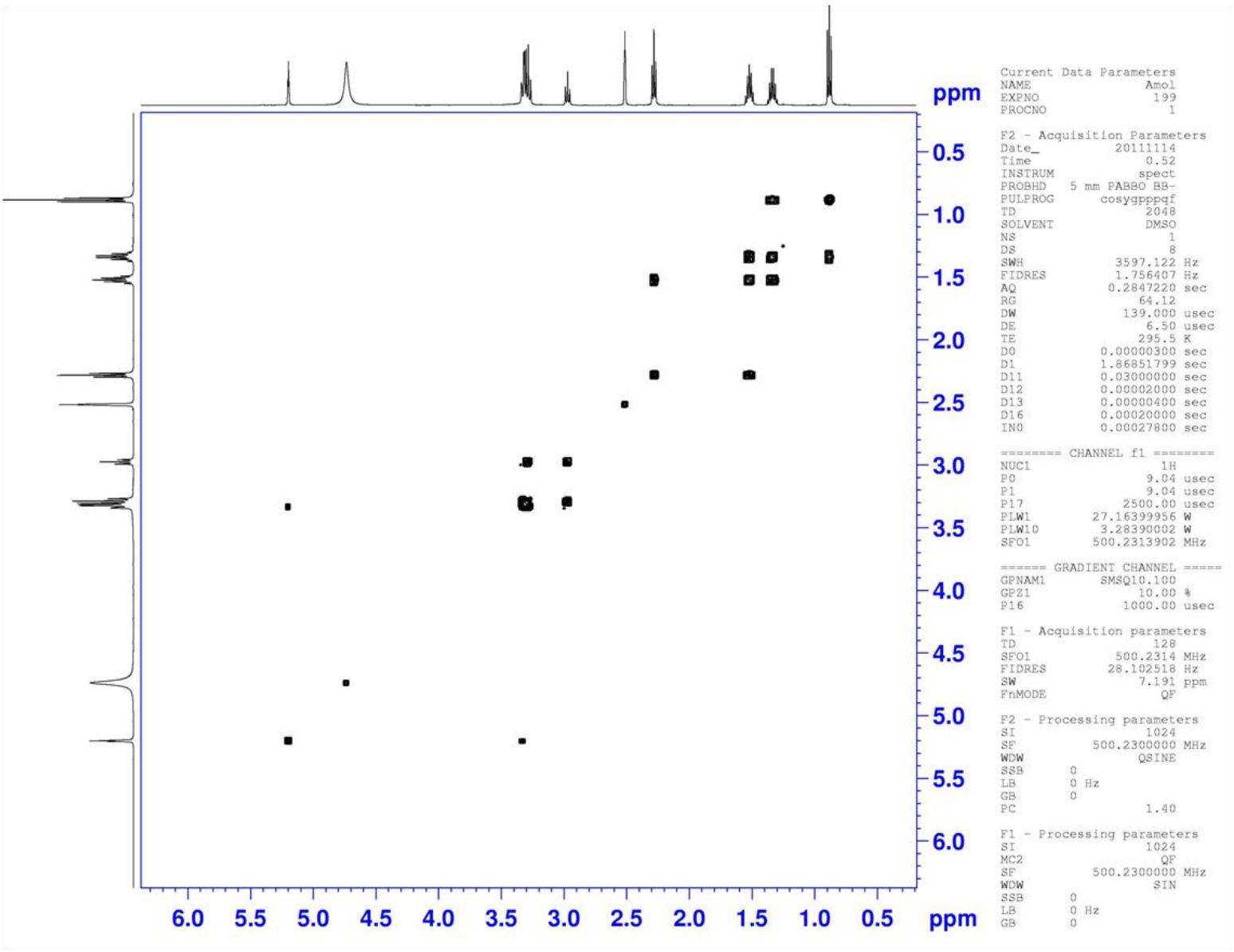
F2 - Acquisition Parameters
Date_           20111114
Time                0.51
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    64
DS                     2
SWH            10330.578 Hz
FIDRES          0.157632 Hz
AQ             3.1719923 sec
RG                107.19
DW                48.400 usec
DE                 6.50 usec
TE                295.5 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.04 usec
PLW1        27.16399956 W
SFO1        500.2330891 MHz

F2 - Processing parameters
SI                 65536
SF          500.2300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

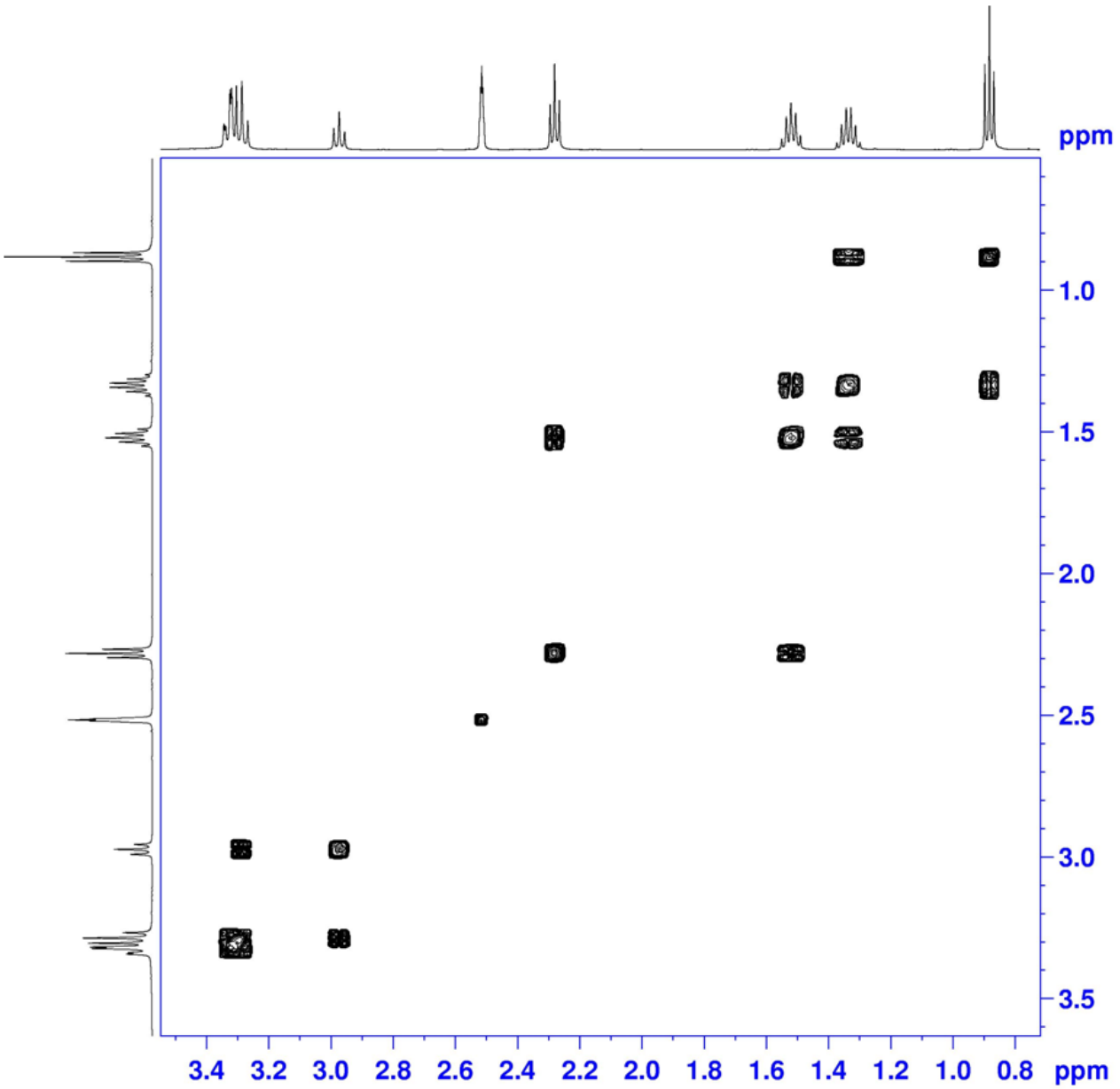
Supplementary Information

COSY of **3b**



Supplementary Information

COSY of **3b** (zoom)

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EXPNO                  199
PROCNO                   1

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Date_              20111114
Time                   0.52
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG       cosygpppqf
TD                     2048
SOLVENT                DMSO
NS                        1
DS                        8
SWH                3597.122 Hz
FIDRES             1.756407 Hz
AQ                 0.2847220 sec
RG                     64.12
DW                   139.000 usec
DE                     6.50 usec
TE                   295.5 K
D0                 0.00000300 sec
D1                 1.86851799 sec
D11                0.03000000 sec
D12                0.00002000 sec
D13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00027800 sec

======== CHANNEL f1 ========
NUC1                      1H
P0                      9.04 usec
P1                      9.04 usec
P17                  2500.00 usec
PLW1              27.16399956 W
PLW10              3.28390002 W
SFO1             500.2313902 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SMSQ10.100
GPZ1                 10.00 %
P16                1000.00 usec

F1 - Acquisition parameters
TD                       128
SFO1               500.2314 MHz
FIDRES            28.102518 Hz
SW                     7.191 ppm
FnMODE                   QF

F2 - Processing parameters
SI                       1024
SF              500.2300000 MHz
WDW                    QSINE
SSB          0
LB           0 Hz
GB           0
PC                     1.40

F1 - Processing parameters
SI                       1024
MC2                      QF
SF              500.2300000 MHz
WDW                     SIN
SSB          0
LB           0 Hz
GB           0
```
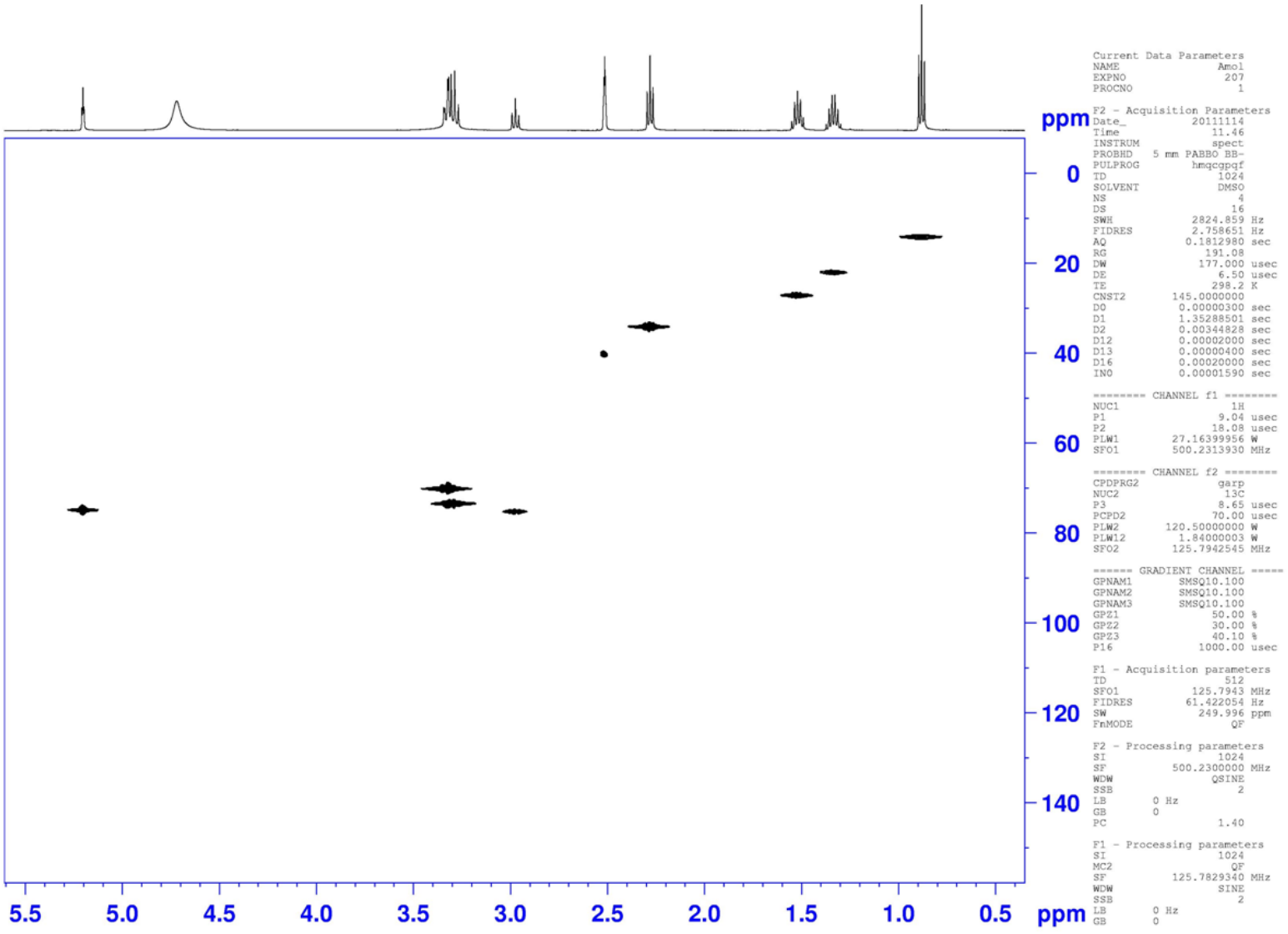
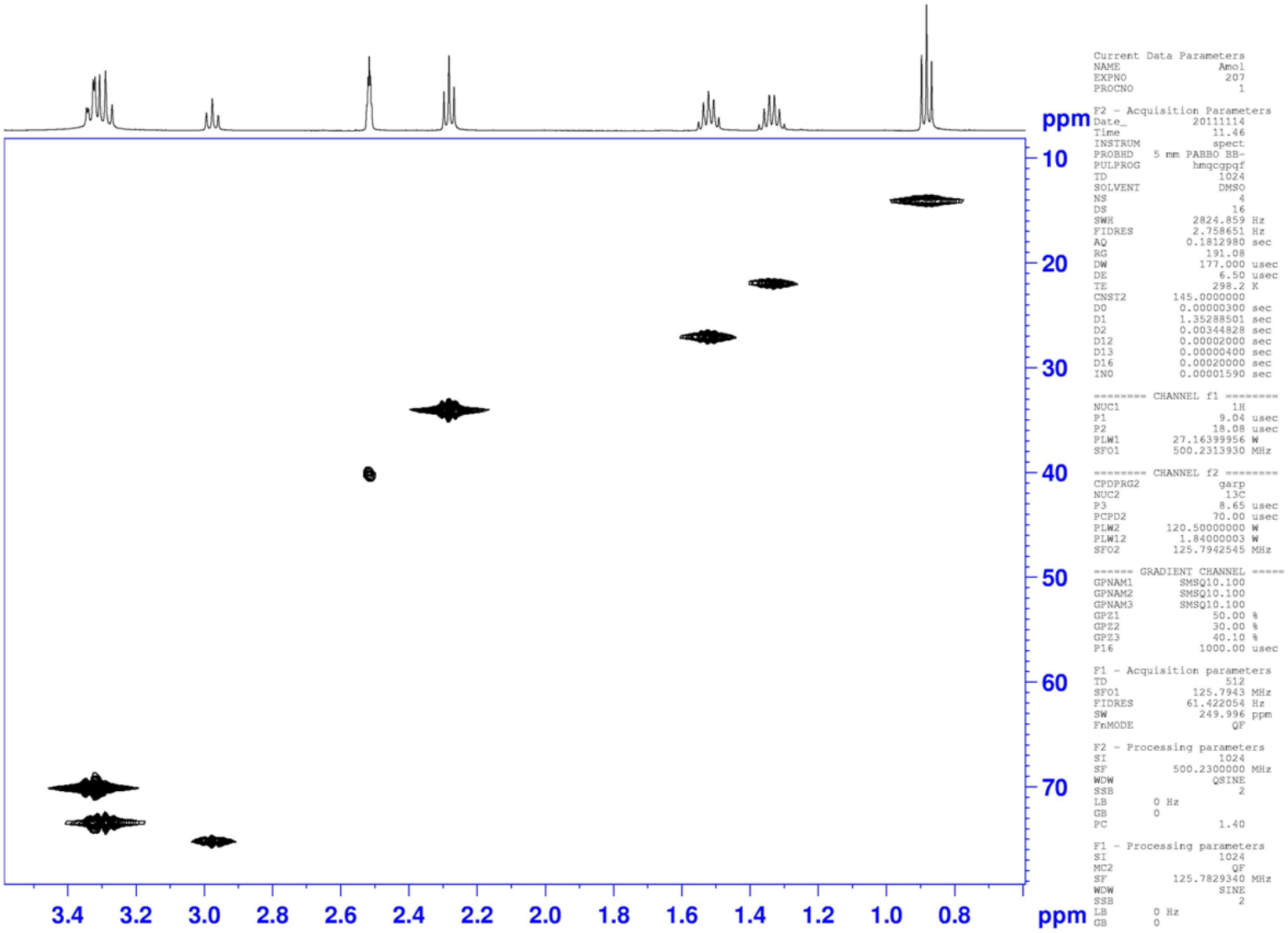

Supplementary Information

HMQC of **3b**



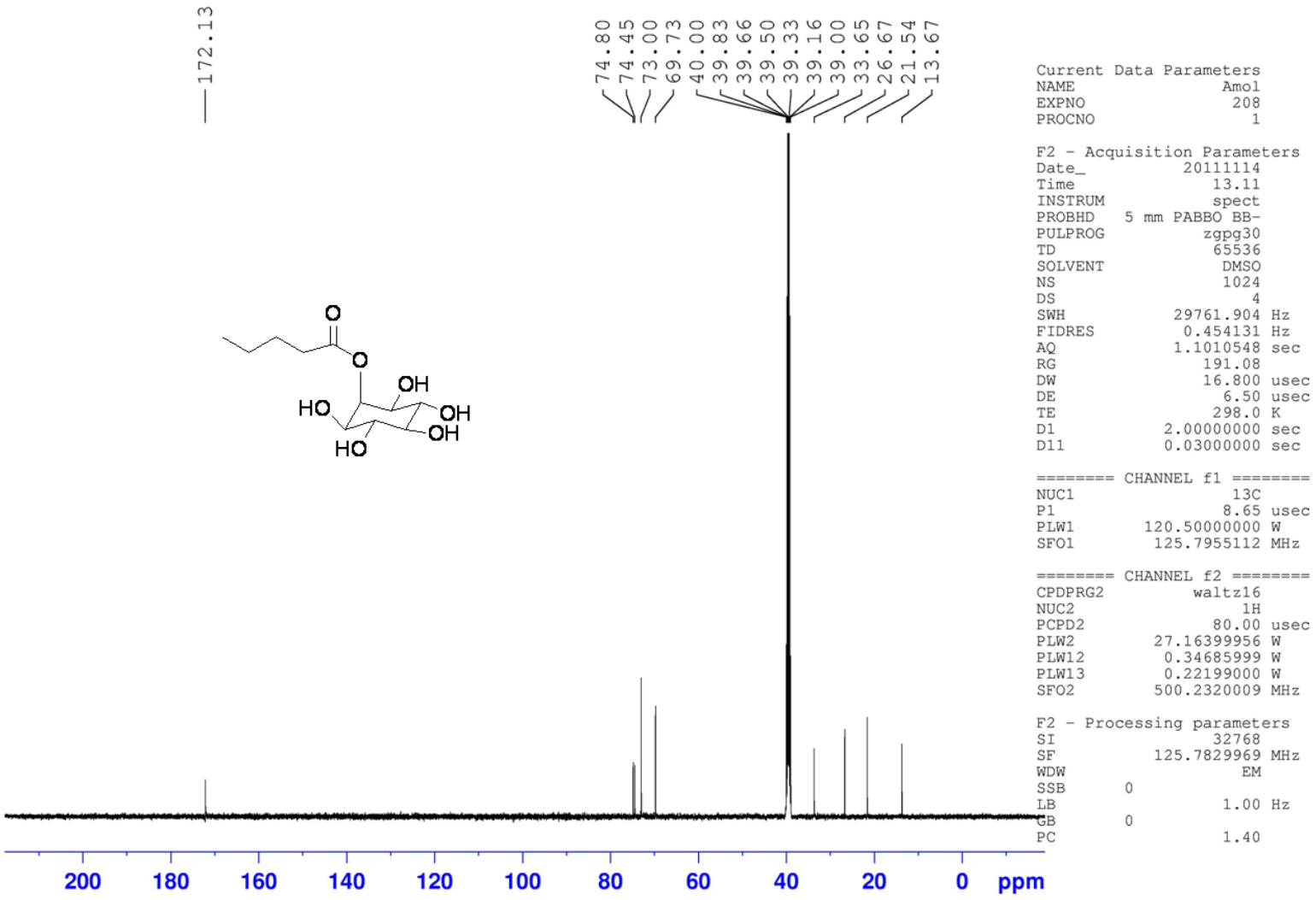
Supplementary Information

HMQC of **3b** (zoom)



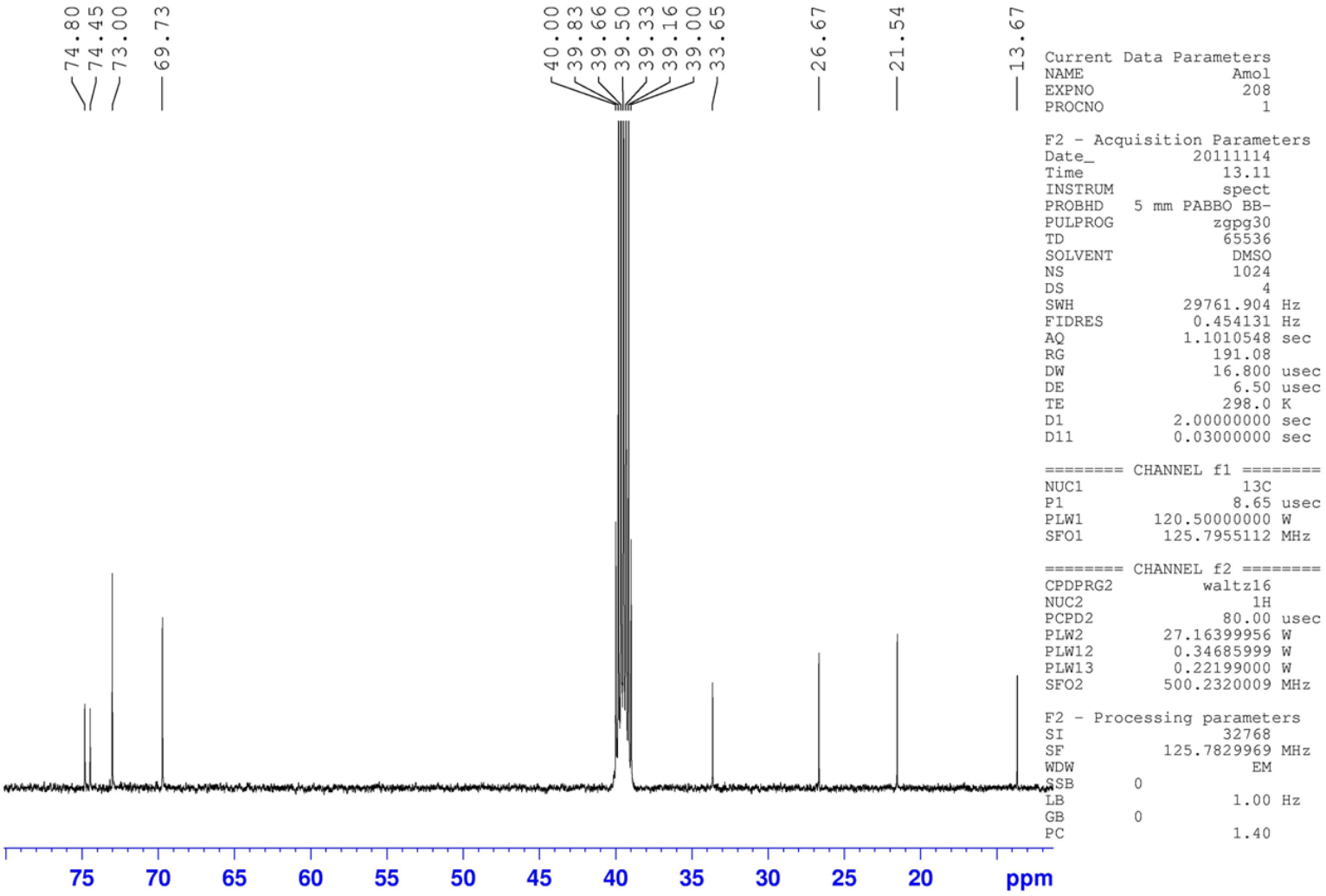
Supplementary Information

^{13}C NMR of **3b** in DMSO-d_6



Supplementary Information

^{13}C NMR of **3b** (zoom)

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Current Data Parameters
NAME                  Amol
EXPNO                  208
PROCNO                   1

F2 - Acquisition Parameters
Date_              20111114
Time                 13.11
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT               DMSO
NS                    1024
DS                       4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ               1.1010548 sec
RG                  191.08
DW                  16.800 usec
DE                   6.50 usec
TE                  298.0 K
D1               2.00000000 sec
D11              0.03000000 sec

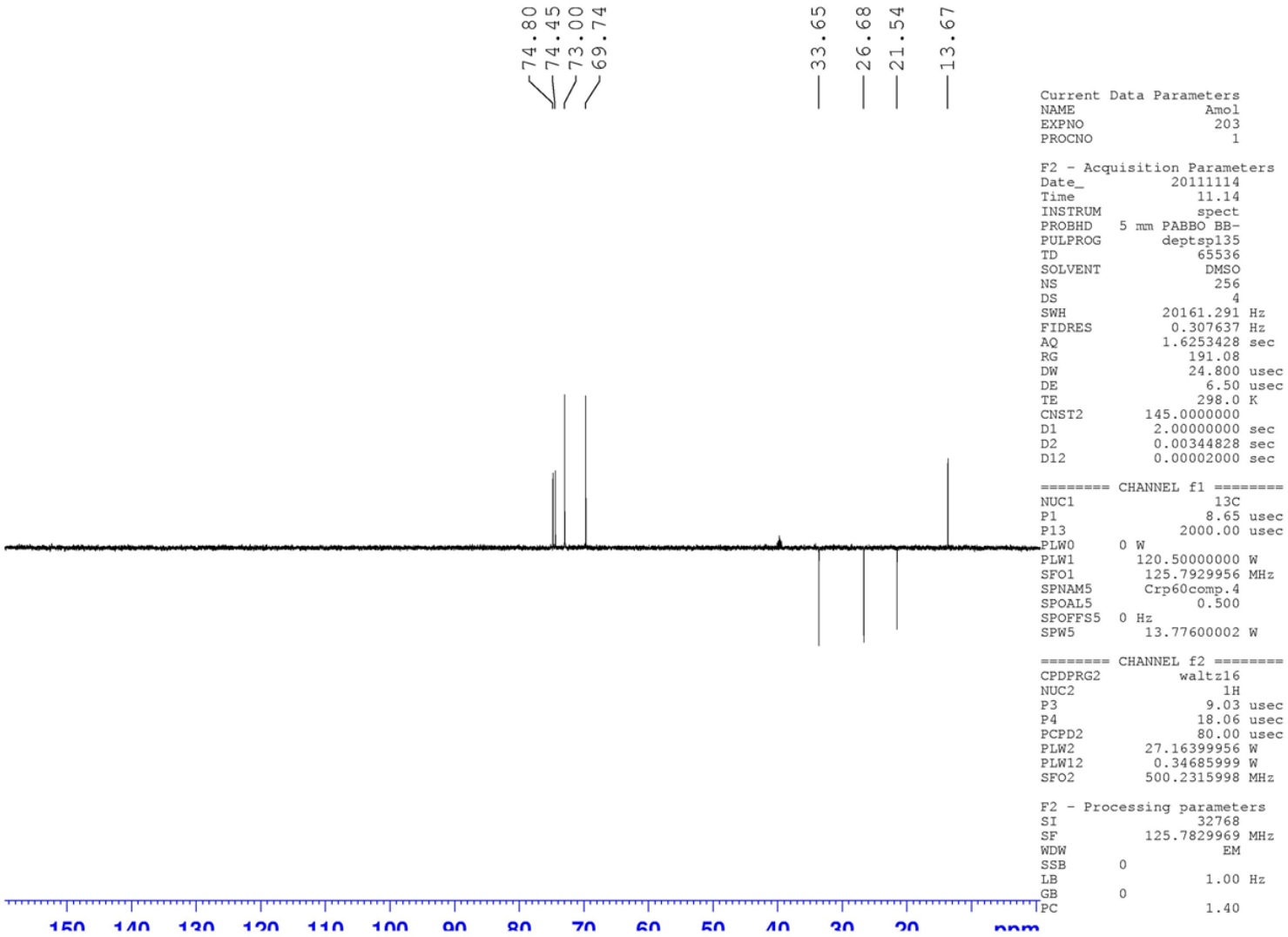
======== CHANNEL f1 ========
NUC1                   13C
P1                    8.65 usec
PLW1          120.50000000 W
SFO1           125.7955112 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2                80.00 usec
PLW2           27.16399956 W
PLW12           0.34685999 W
PLW13           0.22199000 W
SFO2           500.2320009 MHz

F2 - Processing parameters
SI                    32768
SF             125.7829969 MHz
WDW                     EM
SSB         0
LB                    1.00 Hz
GB          0
PC                    1.40
```

Supplementary Information

DEPT of **3b**

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Current Data Parameters
NAME                  Amol
EXPNO                  203
PROCNO                   1

F2 - Acquisition Parameters
Date_           20111114
Time               11.14
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG        deptsp135
TD                 65536
SOLVENT             DMSO
NS                   256
DS                     4
SWH            20161.291 Hz
FIDRES         0.307637 Hz
AQ             1.6253428 sec
RG                191.08
DW                24.800 usec
DE                 6.50 usec
TE                298.0 K
CNST2        145.0000000
D1            2.00000000 sec
D2            0.00344828 sec
D12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.65 usec
P13              2000.00 usec
PLW0      0 W
PLW1       120.50000000 W
SFO1        125.7929956 MHz
SPNAM5      Crp60comp.4
SPOAL5            0.500
SPOFFS5   0 Hz
SPW5         13.77600002 W

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
P3                  9.03 usec
P4                 18.06 usec
PCPD2              80.00 usec
PLW2         27.16399956 W
PLW12         0.34685999 W
SFO2         500.2315998 MHz

F2 - Processing parameters
SI                 32768
SF           125.7829969 MHz
WDW                  EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```
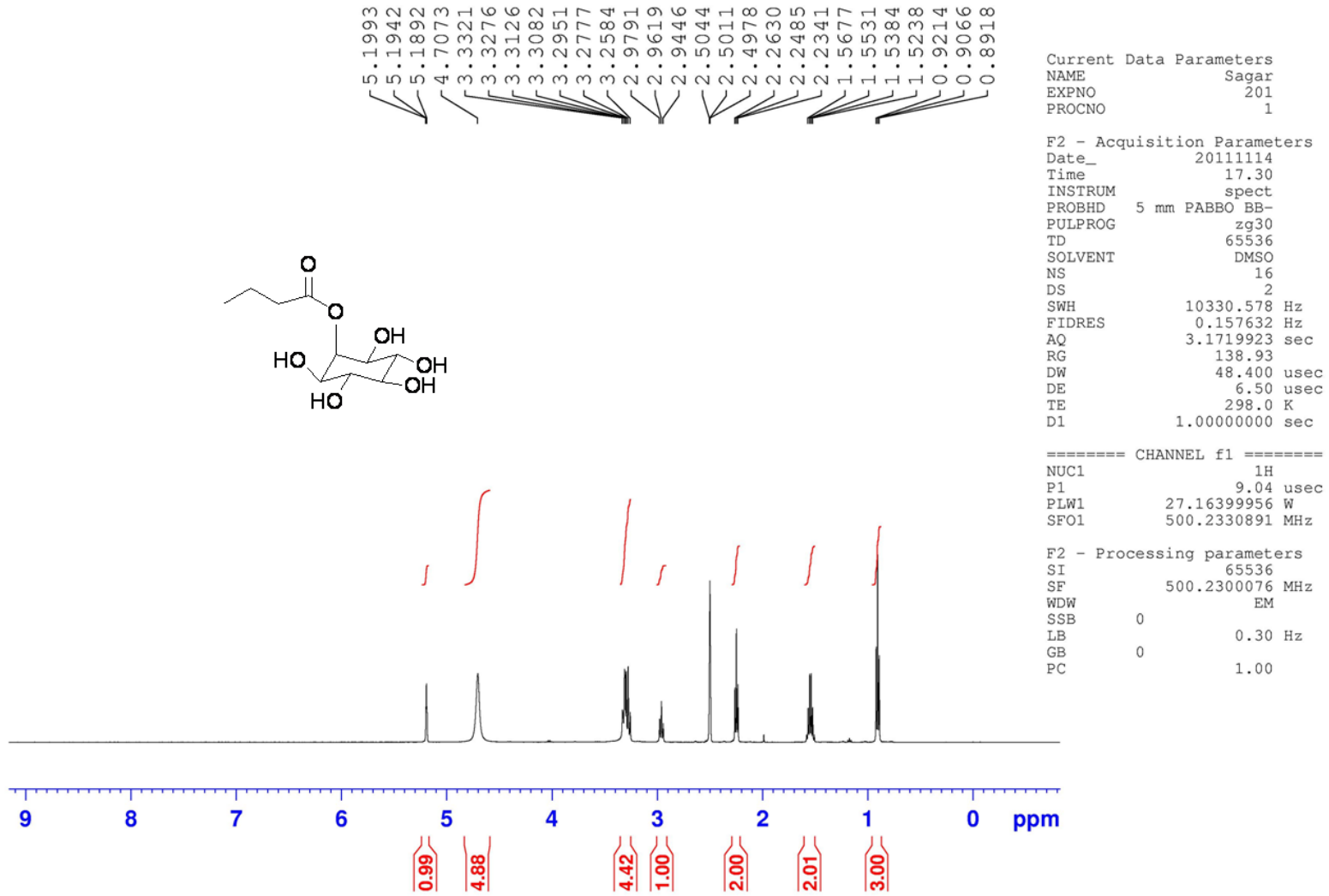

Supplementary Information

^{1}H NMR of **3c** in DMSO-d_6

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Current Data Parameters
NAME                  Sagar
EXPNO                   201
PROCNO                    1

F2 - Acquisition Parameters
Date_           20111114
Time                17.30
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                      16
DS                       2
SWH             10330.578 Hz
FIDRES           0.157632 Hz
AQ              3.1719923 sec
RG                  138.93
DW                  48.400 usec
DE                   6.50 usec
TE                  298.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.04 usec
PLW1          27.16399956 W
SFO1          500.2330891 MHz

F2 - Processing parameters
SI                  65536
SF            500.2300076 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
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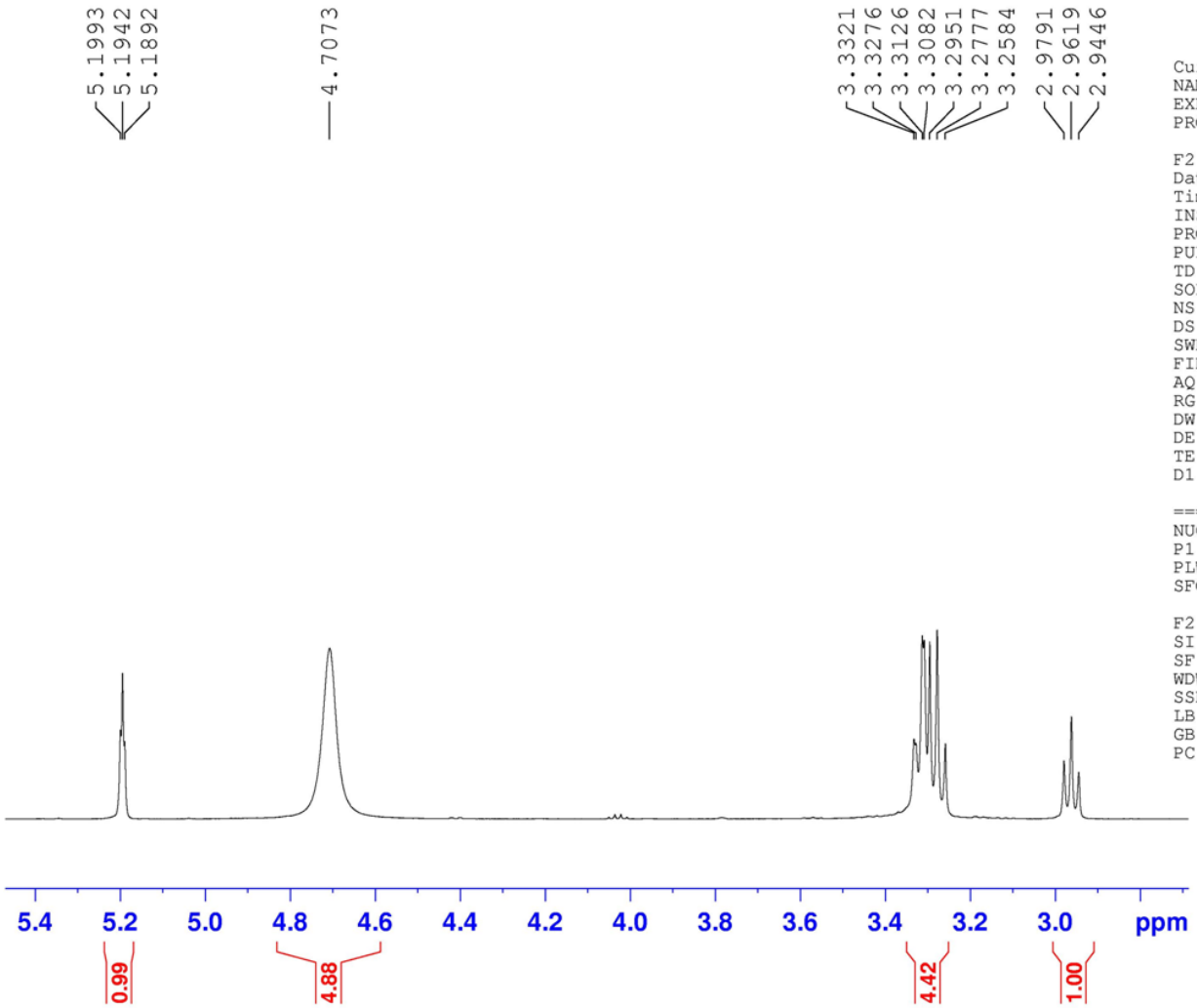
Supplementary Information

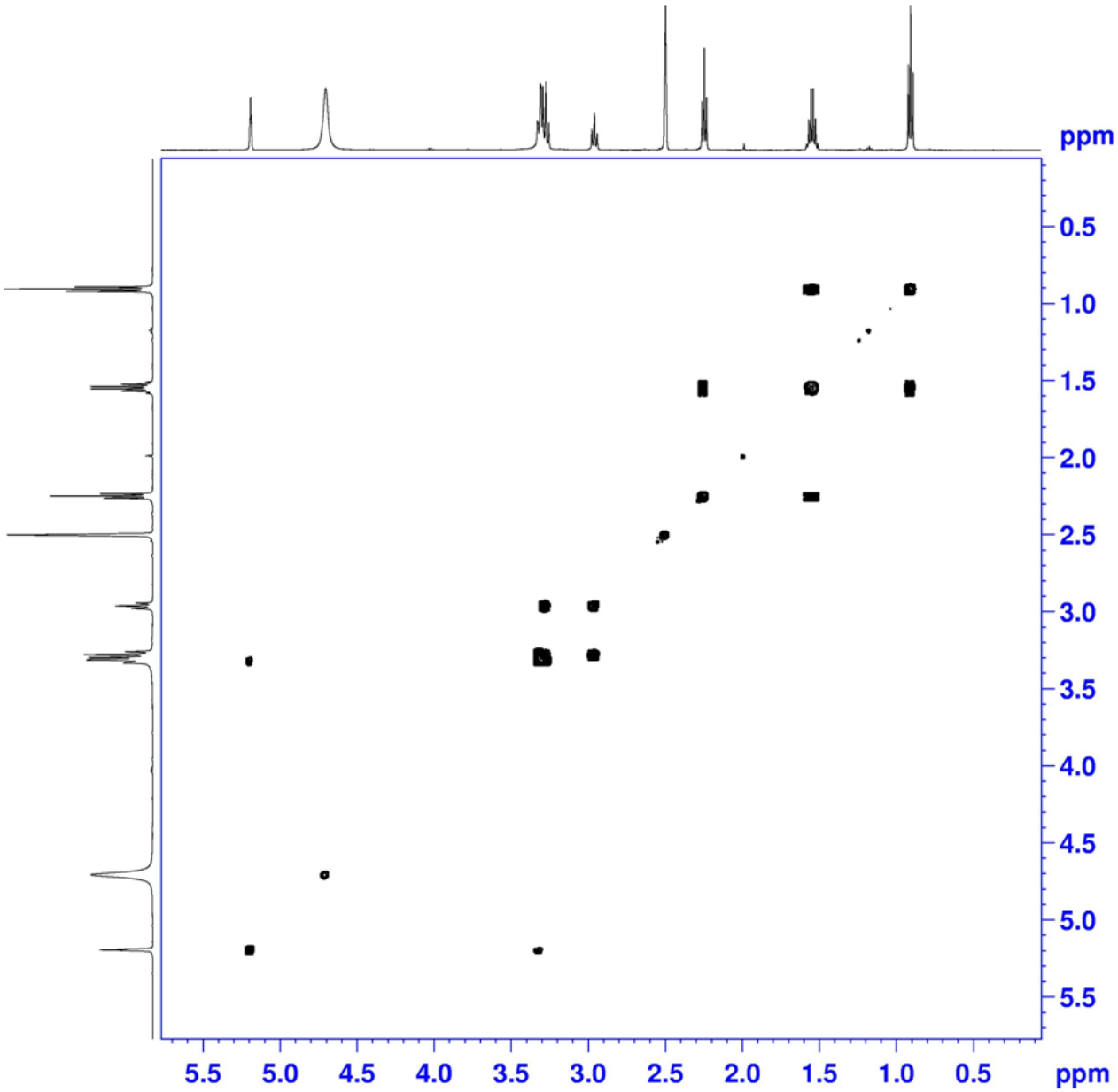
^{1}H NMR of **3c** (zoom)




```
Current Data Parameters
NAME                 Sagar
EXPNO                  201
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111114
Time                 17.30
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     16
DS                      2
SWH              10330.578 Hz
FIDRES            0.157632 Hz
AQ                3.1719923 sec
RG                   138.93
DW                   48.400 usec
DE                    6.50 usec
TE                   298.0 K
D1                1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.04 usec
PLW1          27.16399956 W
SFO1          500.2330891 MHz

F2 - Processing parameters
SI                   65536
SF            500.2300076 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```

Supplementary Information

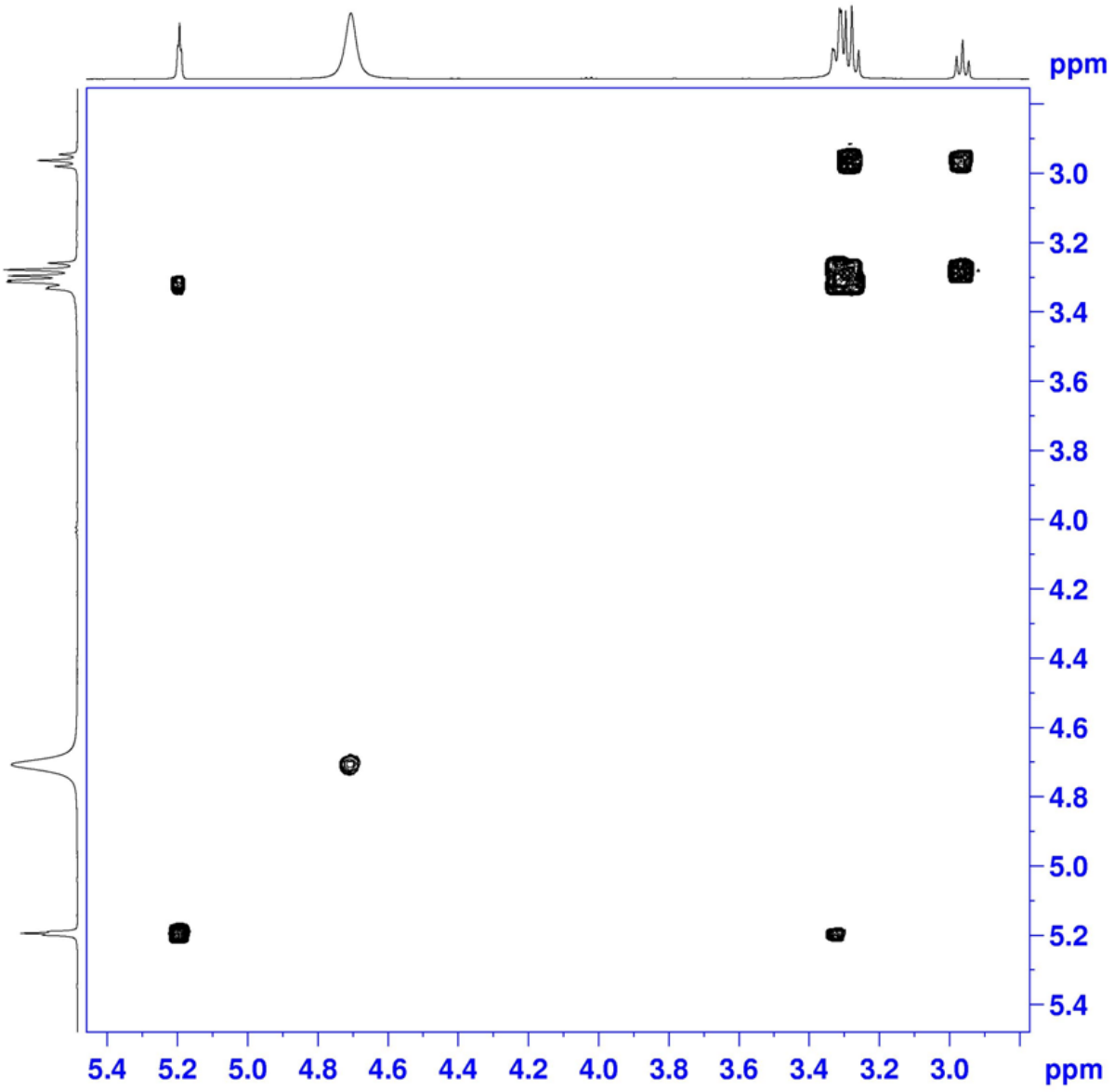
COSY of **3c**




```
Current Data Parameters
NAME                  Sagar
EXPNO                   202
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111114
Time                  17.32
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG        cosygpppqf
TD                     2048
SOLVENT                DMSO
NS                        1
DS                        8
SWH                2857.143 Hz
FIDRES             1.395089 Hz
AQ                0.3584500 sec
RG                    64.12
DW                  175.000 usec
DE                    6.50 usec
TE                   298.0 K
D0                0.00000300 sec
D1                1.79479003 sec
D11               0.03000000 sec
D12               0.00002000 sec
D13               0.00000400 sec
D16               0.00020000 sec
IN0               0.00035000 sec

======== CHANNEL f1 ========
NUC1                      1H
P0                      9.04 usec
P1                      9.04 usec
P17                  2500.00 usec
PLW1              27.16399956 W
PLW10              3.28390002 W
SFO1             500.2314661 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SMSQ10.100
GPZ1                 10.00 %
P16                1000.00 usec

F1 - Acquisition parameters
TD                       128
SFO1               500.2315 MHz
FIDRES            22.321428 Hz
SW                     5.712 ppm
FnMODE                    QF

F2 - Processing parameters
SI                       1024
SF              500.2300076 MHz
WDW                     QSINE
SSB          0
LB           0 Hz
GB           0
PC                     1.40

F1 - Processing parameters
SI                       1024
MC2                       QF
SF              500.2300076 MHz
WDW                      SIN
SSB          0
LB           0 Hz
GB           0
```

Supplementary Information

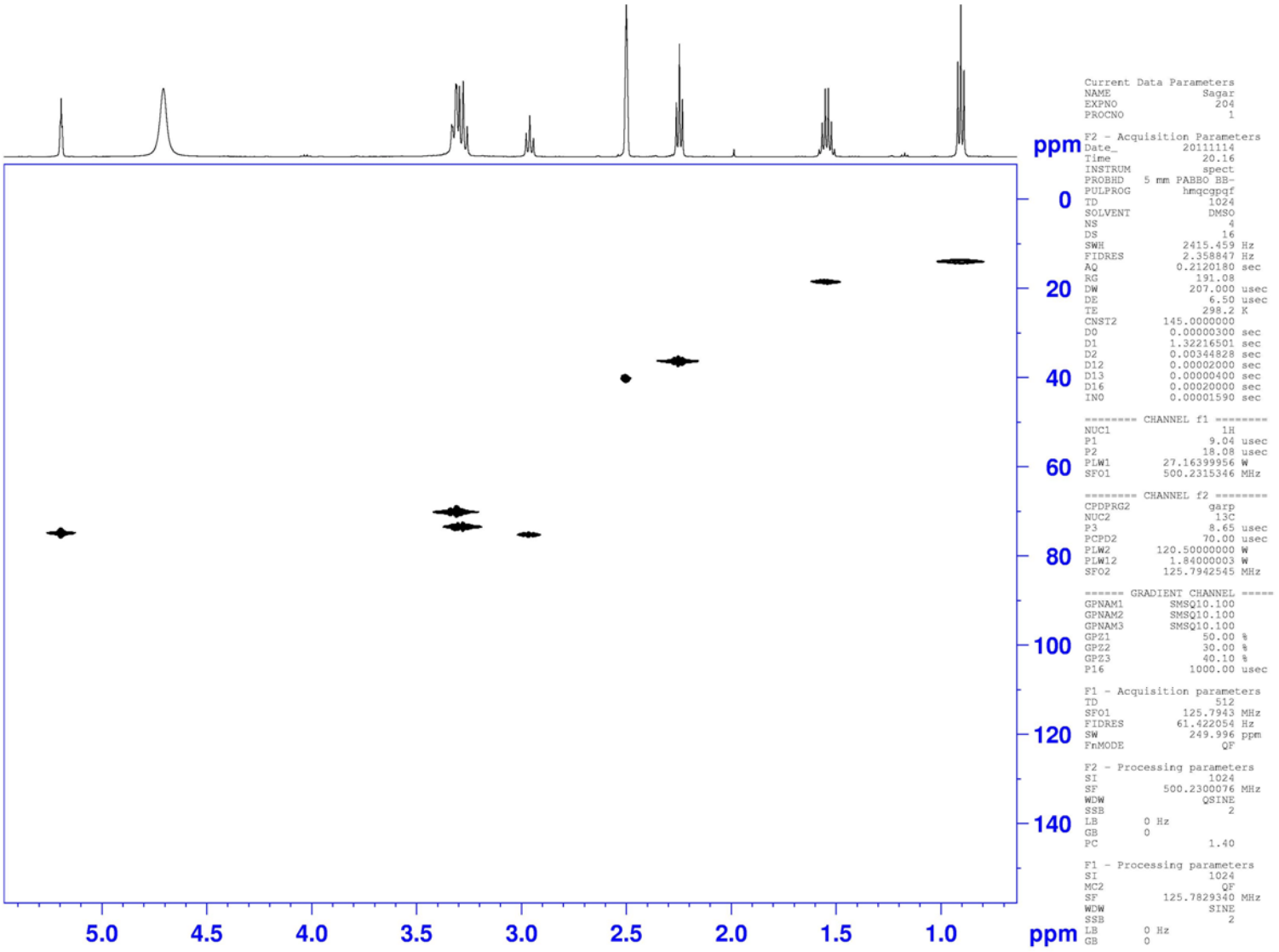
COSY of **3c** (zoom)




```
Current Data Parameters
NAME                  Sagar
EXPNO                   202
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111114
Time                  17.32
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG          cosygpppqf
TD                      2048
SOLVENT                 DMSO
NS                         1
DS                         8
SWH                2857.143 Hz
FIDRES             1.395089 Hz
AQ                 0.3584500 sec
RG                      64.12
DW                    175.000 usec
DE                      6.50 usec
TE                     298.0 K
D0                 0.00000300 sec
D1                 1.79479003 sec
D11                0.03000000 sec
D12                0.00002000 sec
D13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00035000 sec

======== CHANNEL f1 ========
NUC1                      1H
P0                      9.04 usec
P1                      9.04 usec
P17                  2500.00 usec
PLW1              27.16399956 W
PLW10              3.28390002 W
SFO1             500.2314661 MHz

====== GRADIENT CHANNEL =====
GPNAM1           SMSQ10.100
GPZ1                 10.00 %
P16                1000.00 usec

F1 - Acquisition parameters
TD                       128
SFO1               500.2315 MHz
FIDRES            22.321428 Hz
SW                     5.712 ppm
FnMODE                    QF

F2 - Processing parameters
SI                       1024
SF               500.2300076 MHz
WDW                     QSINE
SSB          0
LB           0 Hz
GB           0
PC                     1.40

F1 - Processing parameters
SI                       1024
MC2                        QF
SF               500.2300076 MHz
WDW                       SIN
SSB          0
LB           0 Hz
GB           0
```

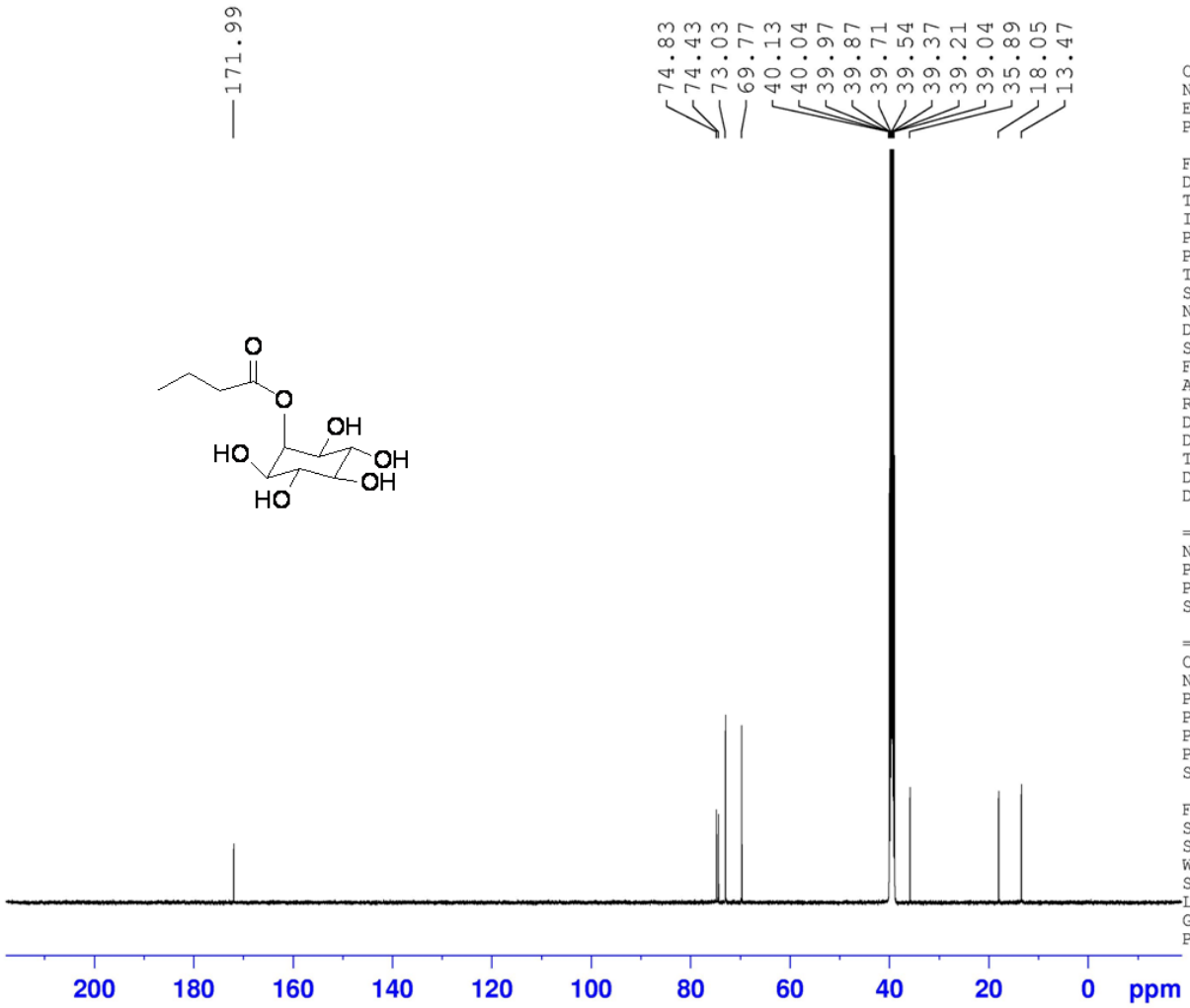
Supplementary Information

HMQC of **3c**



Supplementary Information

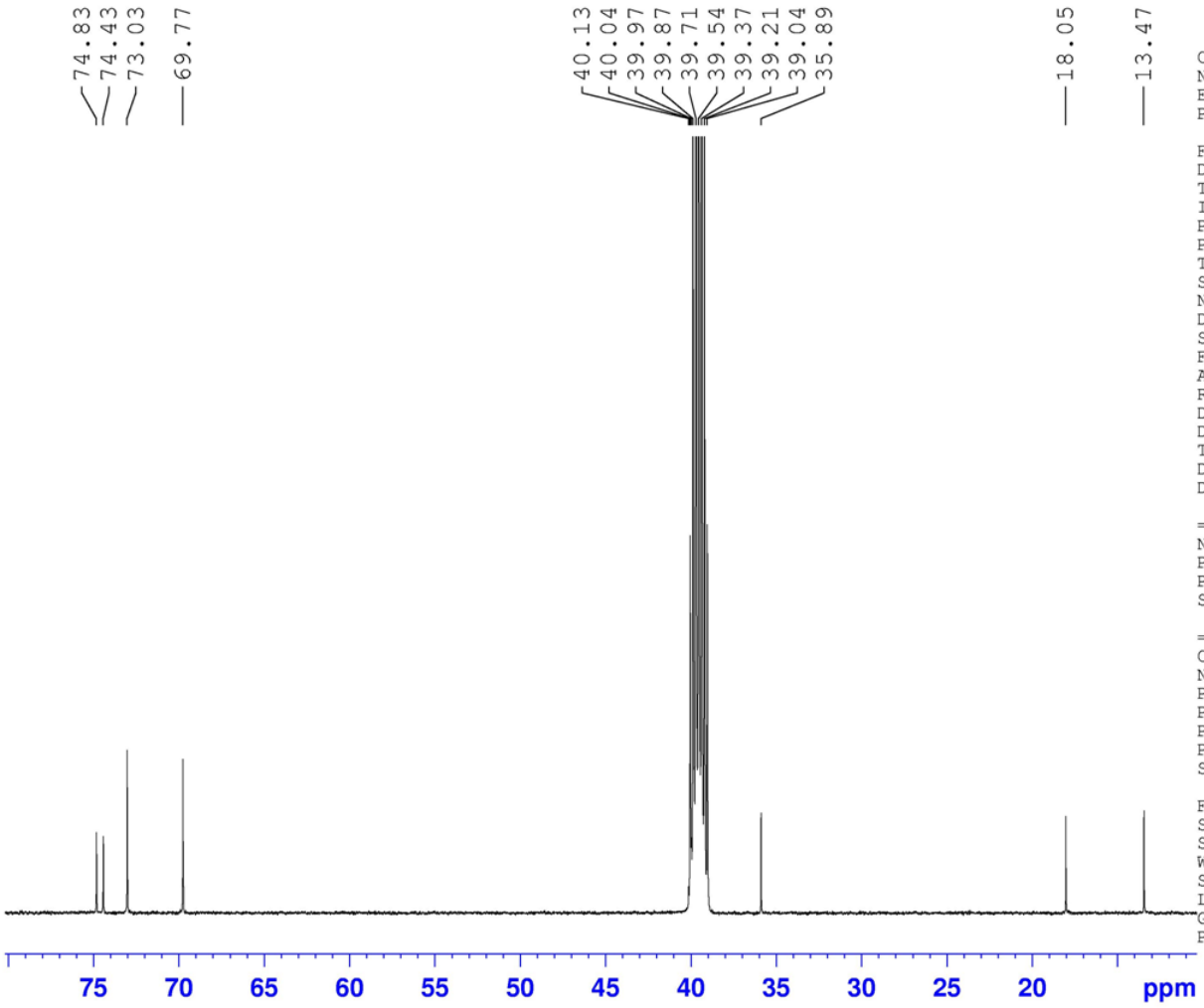
^{13}C NMR of **3c** in DMSO-d_6




```
Current Data Parameters
NAME                  Sagar
EXPNO                   206
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111115
Time                   7.27
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT                DMSO
NS                    10240
DS                        4
SWH               29761.904 Hz
FIDRES             0.454131 Hz
AQ                1.1010548 sec
RG                   191.08
DW                   16.800 usec
DE                    6.50 usec
TE                   298.8 K
D1              2.00000000 sec
D11             0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    8.65 usec
PLW1         120.50000000 W
SFO1          125.7955112 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PLW2          27.16399956 W
PLW12          0.34685999 W
PLW13          0.22199000 W
SFO2          500.2320009 MHz

F2 - Processing parameters
SI                    32768
SF             125.7829969 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

Supplementary Information

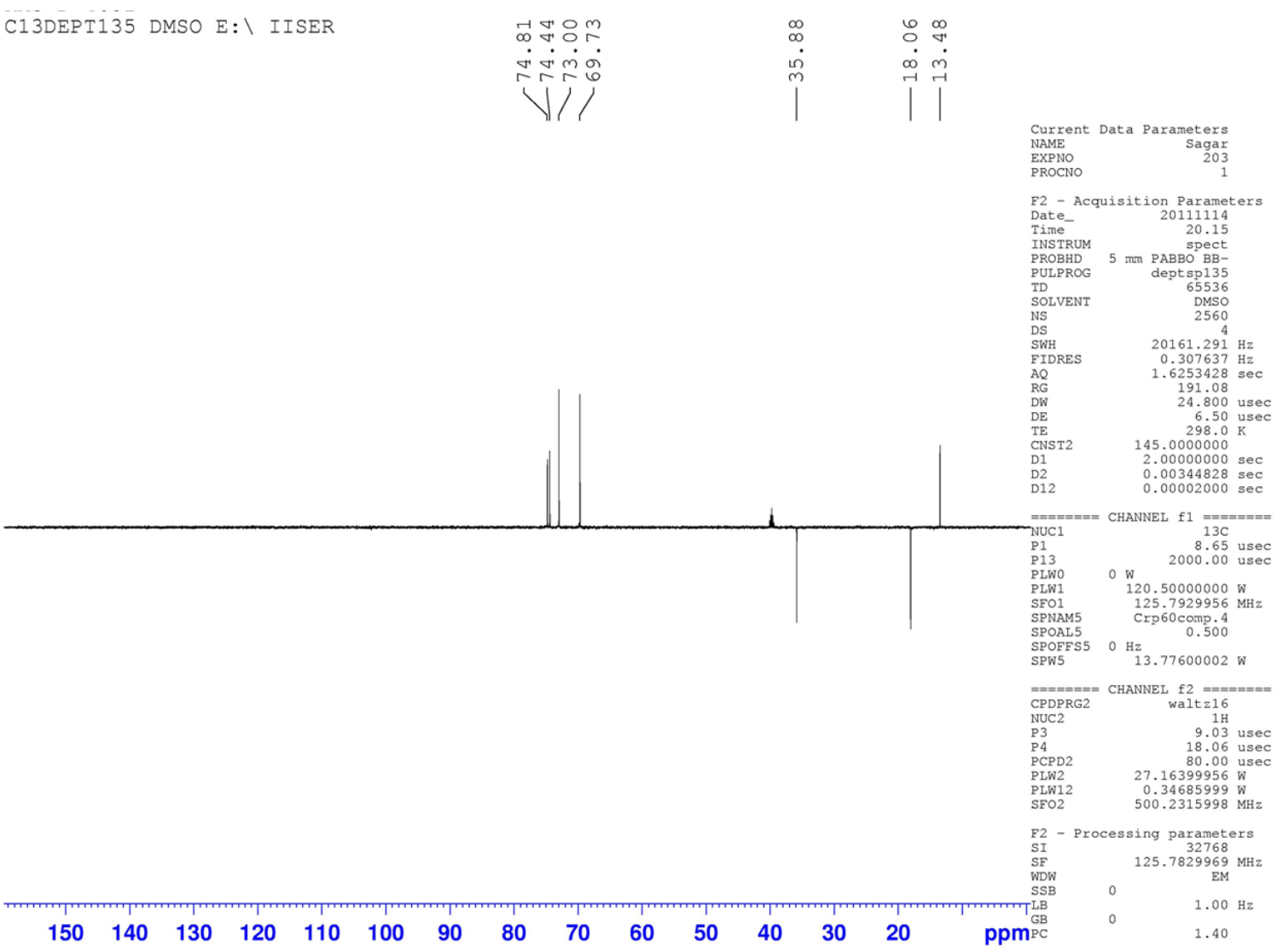
^{13}C NMR of **3c** (zoom)




```
Current Data Parameters
NAME                 Sagar
EXPNO                  206
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111115
Time                  7.27
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT               DMSO
NS                   10240
DS                       4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ                1.1010548 sec
RG                   191.08
DW                   16.800 usec
DE                    6.50 usec
TE                   298.8 K
D1               2.00000000 sec
D11              0.03000000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    8.65 usec
PLW1          120.50000000 W
SFO1           125.7955112 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2                80.00 usec
PLW2           27.16399956 W
PLW12           0.34685999 W
PLW13           0.22199000 W
SFO2          500.2320009 MHz

F2 - Processing parameters
SI                    32768
SF           125.7829969 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

Supplementary Information

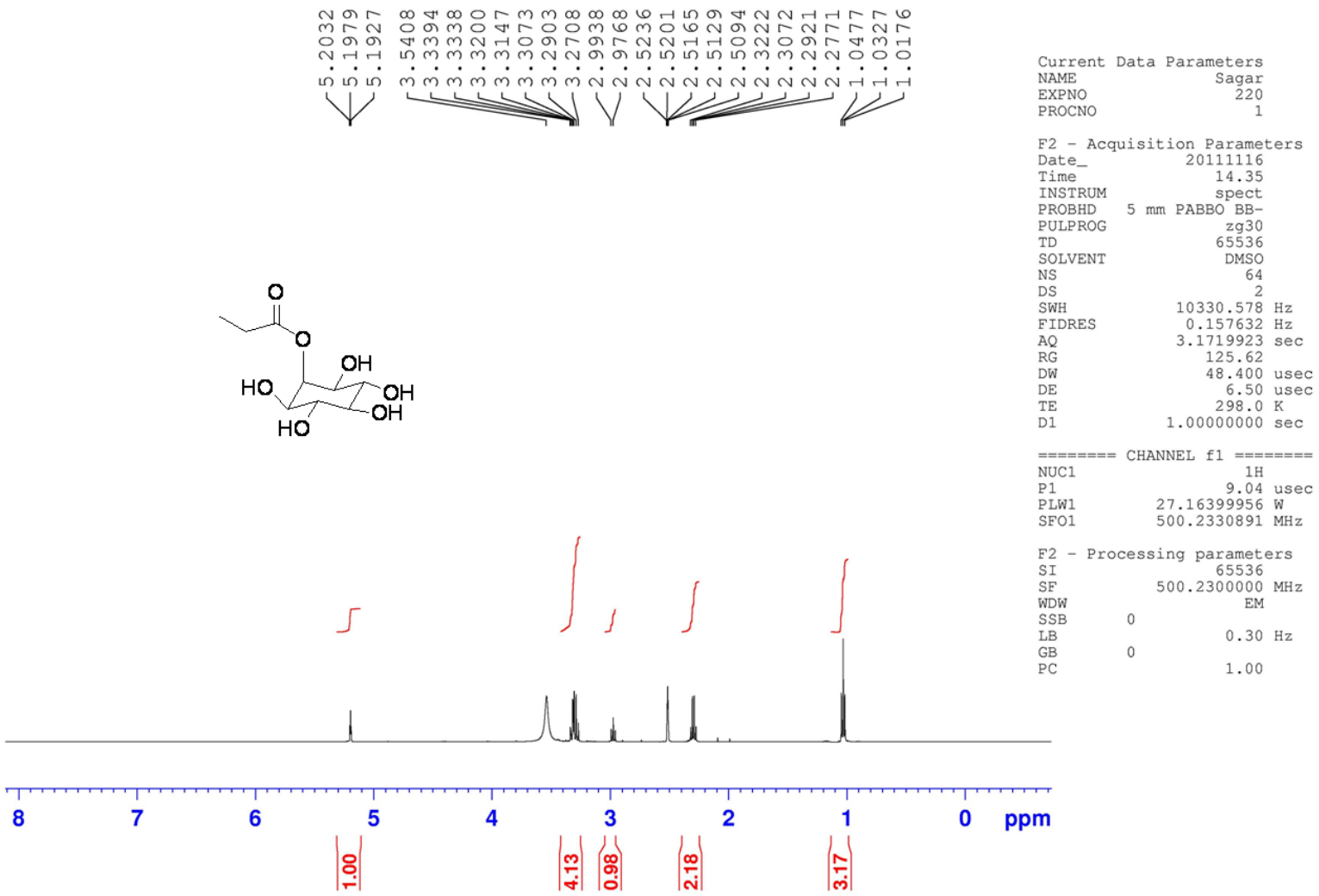
DEPT of **3c**




```
Current Data Parameters
NAME                  Sagar
EXPNO                   203
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111114
Time                  20.15
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG          deptsp135
TD                   65536
SOLVENT               DMSO
NS                    2560
DS                       4
SWH              20161.291 Hz
FIDRES            0.307637 Hz
AQ                1.6253428 sec
RG                   191.08
DW                  24.800 usec
DE                   6.50 usec
TE                  298.0 K
CNST2           145.0000000
D1                2.00000000 sec
D2                0.00344828 sec
D12               0.00002000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    8.65 usec
P13                2000.00 usec
PLW0       0 W
PLW1          120.50000000 W
SFO1           125.7929956 MHz
SPNAM5         Crp60comp.4
SPOAL5               0.500
SPOFFS5    0 Hz
SPW5           13.77600002 W

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
P3                    9.03 usec
P4                   18.06 usec
PCPD2                80.00 usec
PLW2           27.16399956 W
PLW12           0.34685999 W
SFO2           500.2315998 MHz

F2 - Processing parameters
SI                    32768
SF             125.7829969 MHz
WDW                      EM
SSB        0
LB                    1.00 Hz
GB         0
PC                    1.40
```

Supplementary Information

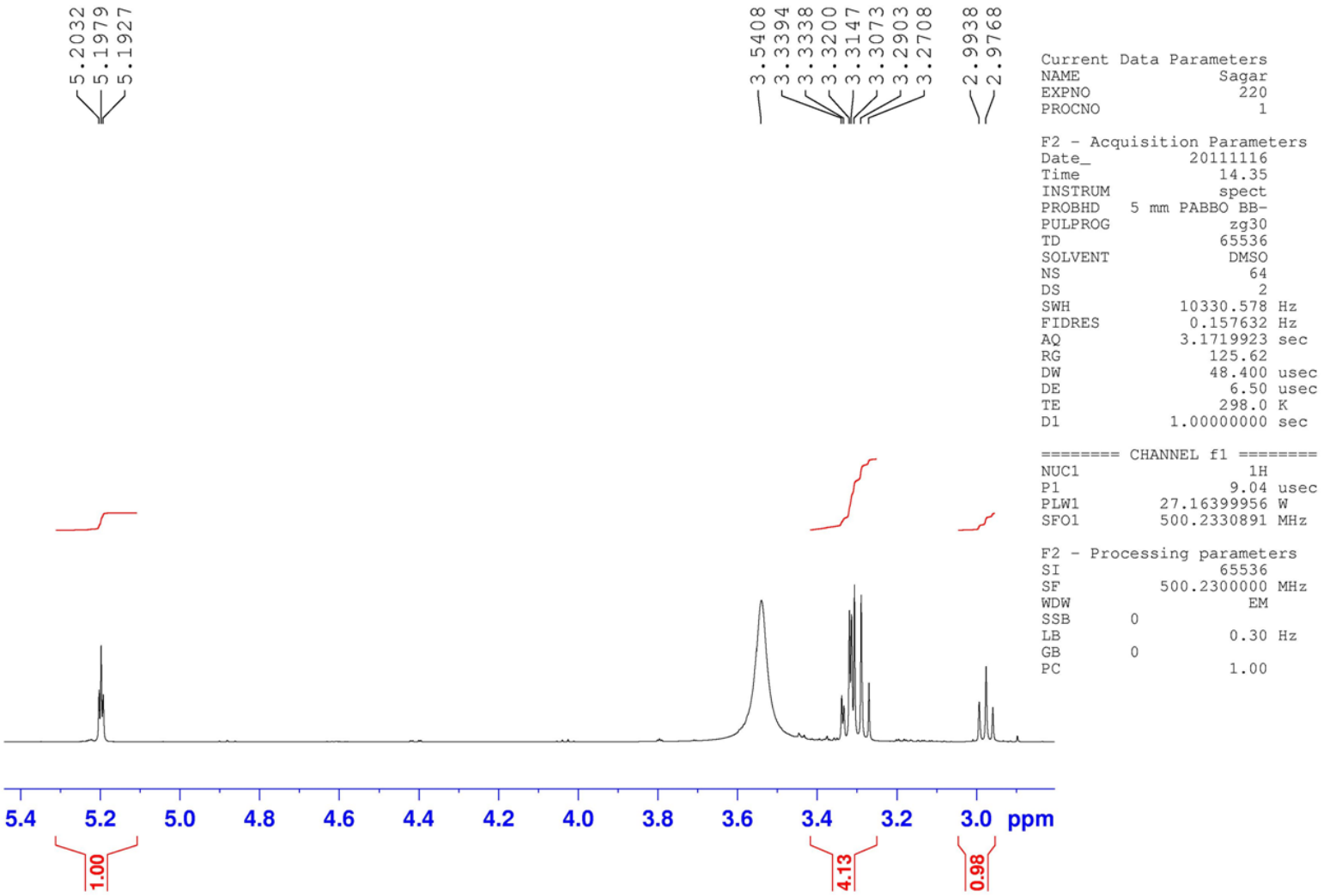
^{1}H NMR of **3d** in DMSO-d_6




```
Current Data Parameters
NAME                  Sagar
EXPNO                   220
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111116
Time                  14.35
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       64
DS                        2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                3.1719923 sec
RG                   125.62
DW                   48.400 usec
DE                    6.50 usec
TE                   298.0 K
D1                1.00000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    9.04 usec
PLW1           27.16399956 W
SFO1           500.2330891 MHz

F2 - Processing parameters
SI                    65536
SF             500.2300000 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

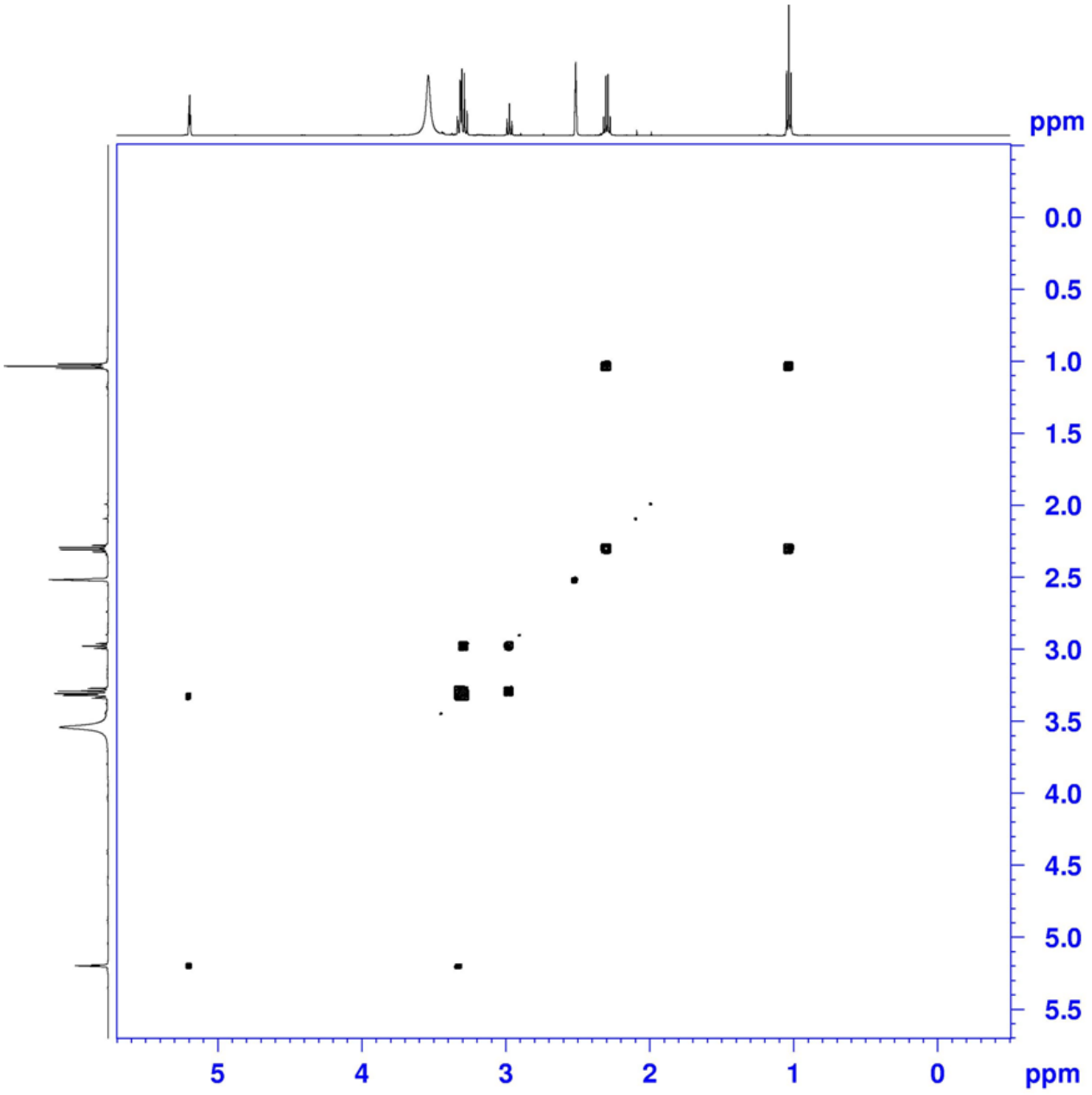
Supplementary Information

^{1}H NMR of **3d** (zoom)



Supplementary Information

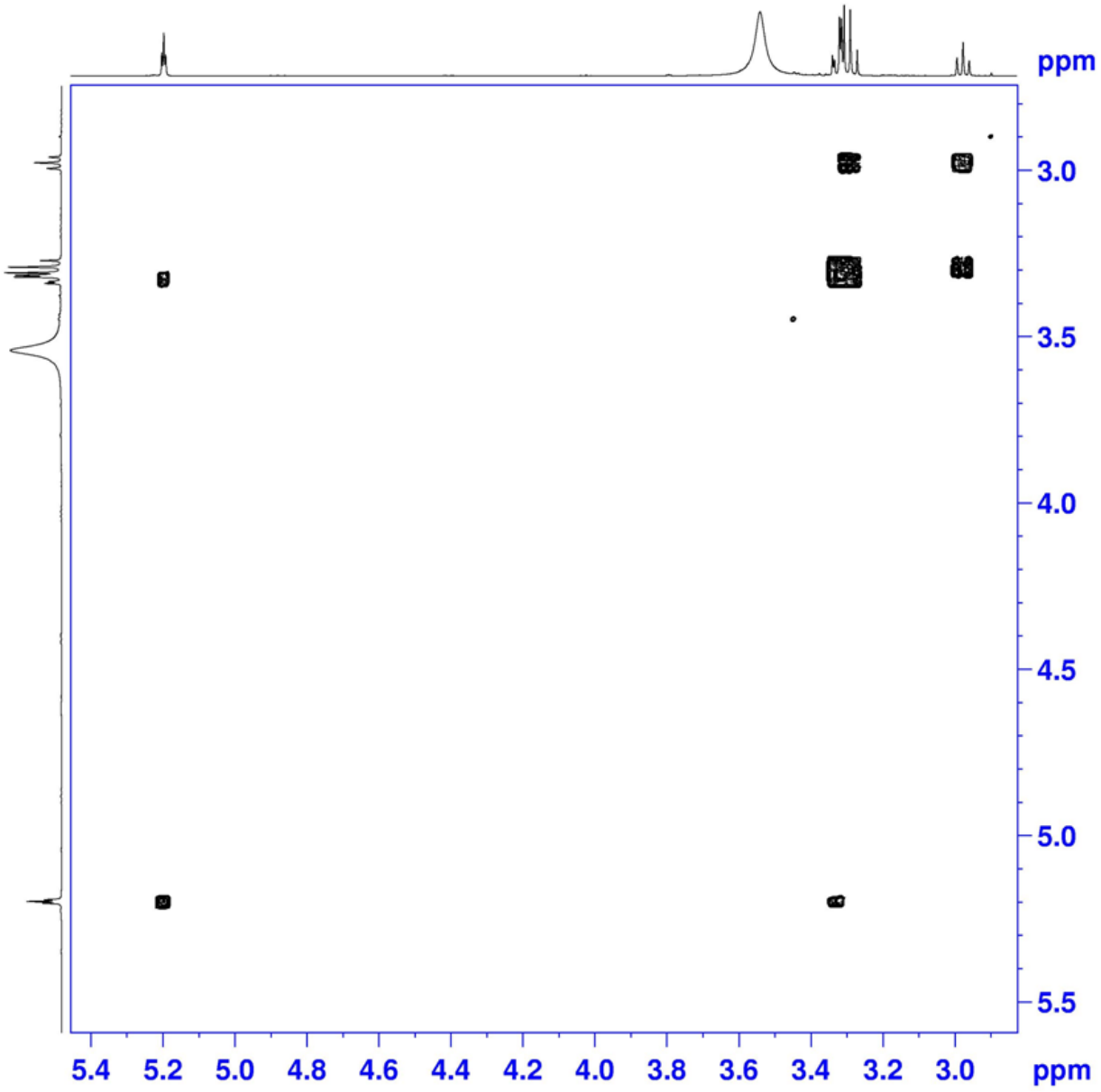
COSY of **3d**




```
Current Data Parameters
NAME                  Sagar
EXPNO                   221
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111116
Time                  14.36
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG       cosygpppqf
TD                      2048
SOLVENT                DMSO
NS                        1
DS                        8
SWH                3105.590 Hz
FIDRES             1.516401 Hz
AQ                 0.3297780 sec
RG                     64.12
DW                   161.000 usec
DE                     6.50 usec
TE                    298.0 K
D0                 0.00000300 sec
D1                 1.82346201 sec
D11                0.03000000 sec
D12                0.00002000 sec
D13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00032200 sec

======== CHANNEL f1 ========
NUC1                      1H
P0                     9.04 usec
P1                     9.04 usec
P17                 2500.00 usec
PLW1               27.16399956 W
PLW10               3.28390002 W
SFO1             500.2312995 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SMSQ10.100
GPZ1                 10.00 %
P16                1000.00 usec

F1 - Acquisition parameters
TD                       128
SFO1              500.2313 MHz
FIDRES           24.262423 Hz
SW                    6.208 ppm
FnMODE                   QF

F2 - Processing parameters
SI                      1024
SF               500.2300000 MHz
WDW                    QSINE
SSB          0
LB           0 Hz
GB           0
PC                     1.40

F1 - Processing parameters
SI                      1024
MC2                      QF
SF               500.2300000 MHz
WDW                     SIN
SSB          0
LB           0 Hz
GB           0
```

Supplementary Information

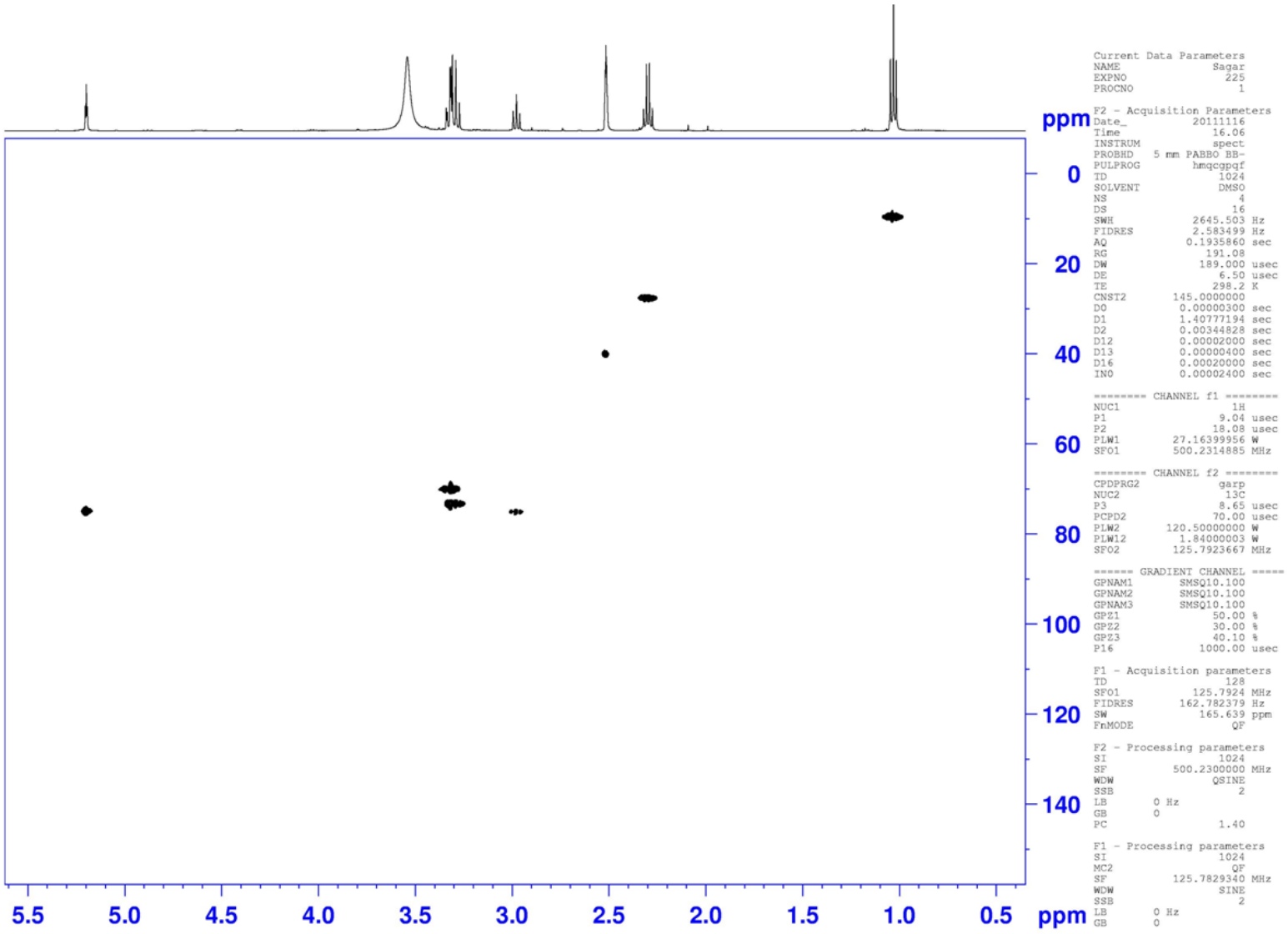
COSY of **3d** (zoom)




```
Current Data Parameters
NAME                  Sagar
EXPNO                   221
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111116
Time                  14.36
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG         cosygpppqf
TD                      2048
SOLVENT                 DMSO
NS                         1
DS                         8
SWH                3105.590 Hz
FIDRES                1.516401 Hz
AQ                 0.3297780 sec
RG                     64.12
DW                   161.000 usec
DE                     6.50 usec
TE                    298.0 K
D0                 0.00000300 sec
D1                 1.82346201 sec
D11                0.03000000 sec
D12                0.00002000 sec
D13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00032200 sec

======== CHANNEL f1 ========
NUC1                      1H
P0                      9.04 usec
P1                      9.04 usec
P17                  2500.00 usec
PLW1               27.16399956 W
PLW10               3.28390002 W
SFO1             500.2312995 MHz

====== GRADIENT CHANNEL =====
GPNAM1           SMSQ10.100
GPZ1                   10.00 %
P16                  1000.00 usec

F1 - Acquisition parameters
TD                       128
SFO1               500.2313 MHz
FIDRES              24.262423 Hz
SW                     6.208 ppm
FnMODE                   QF

F2 - Processing parameters
SI                      1024
SF               500.2300000 MHz
WDW                    QSINE
SSB          0
LB           0 Hz
GB           0
PC                     1.40

F1 - Processing parameters
SI                      1024
MC2                      QF
SF               500.2300000 MHz
WDW                     SIN
SSB          0
LB           0 Hz
GB           0
```

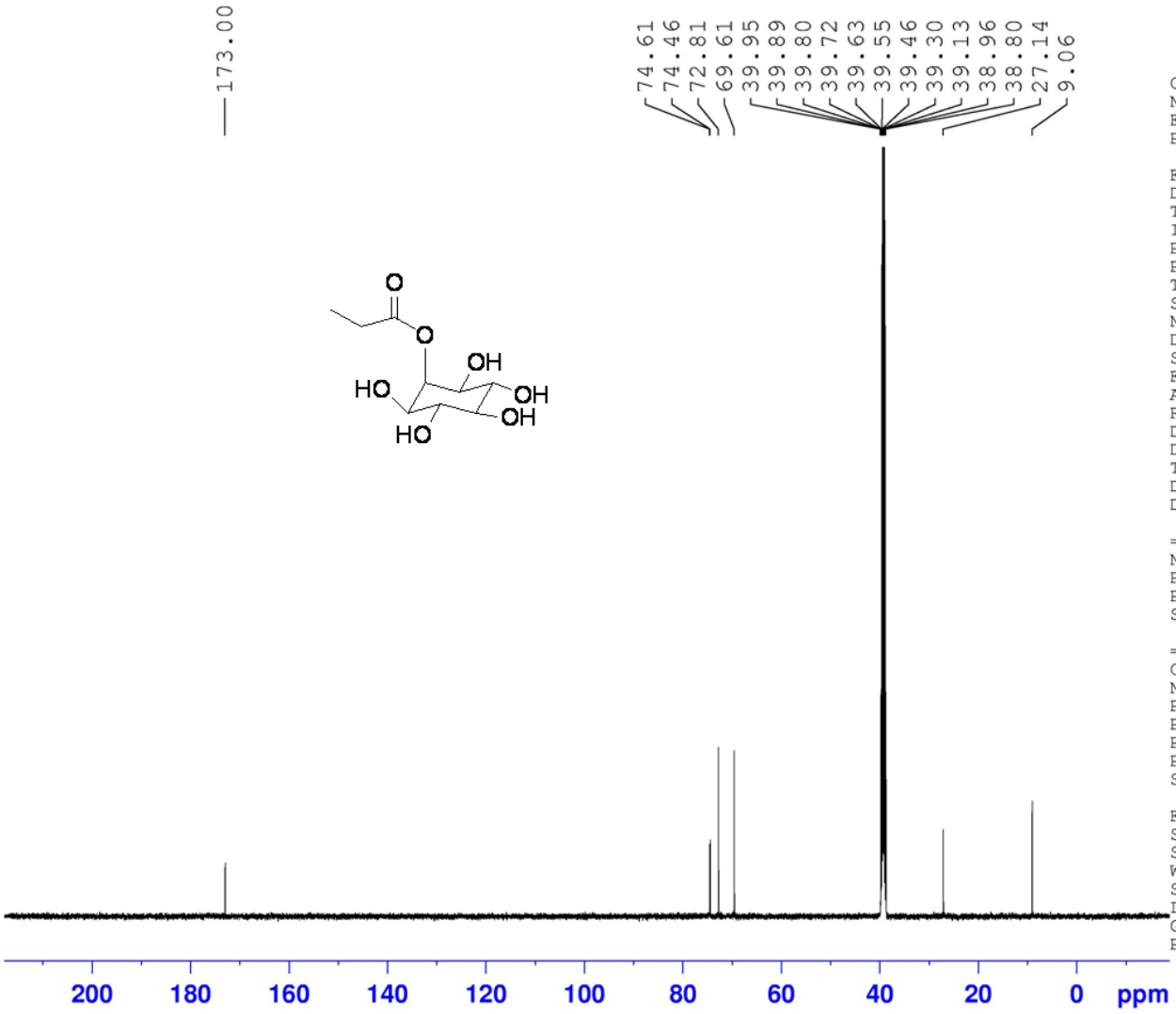
Supplementary Information

HMQC of **3d**



Supplementary Information

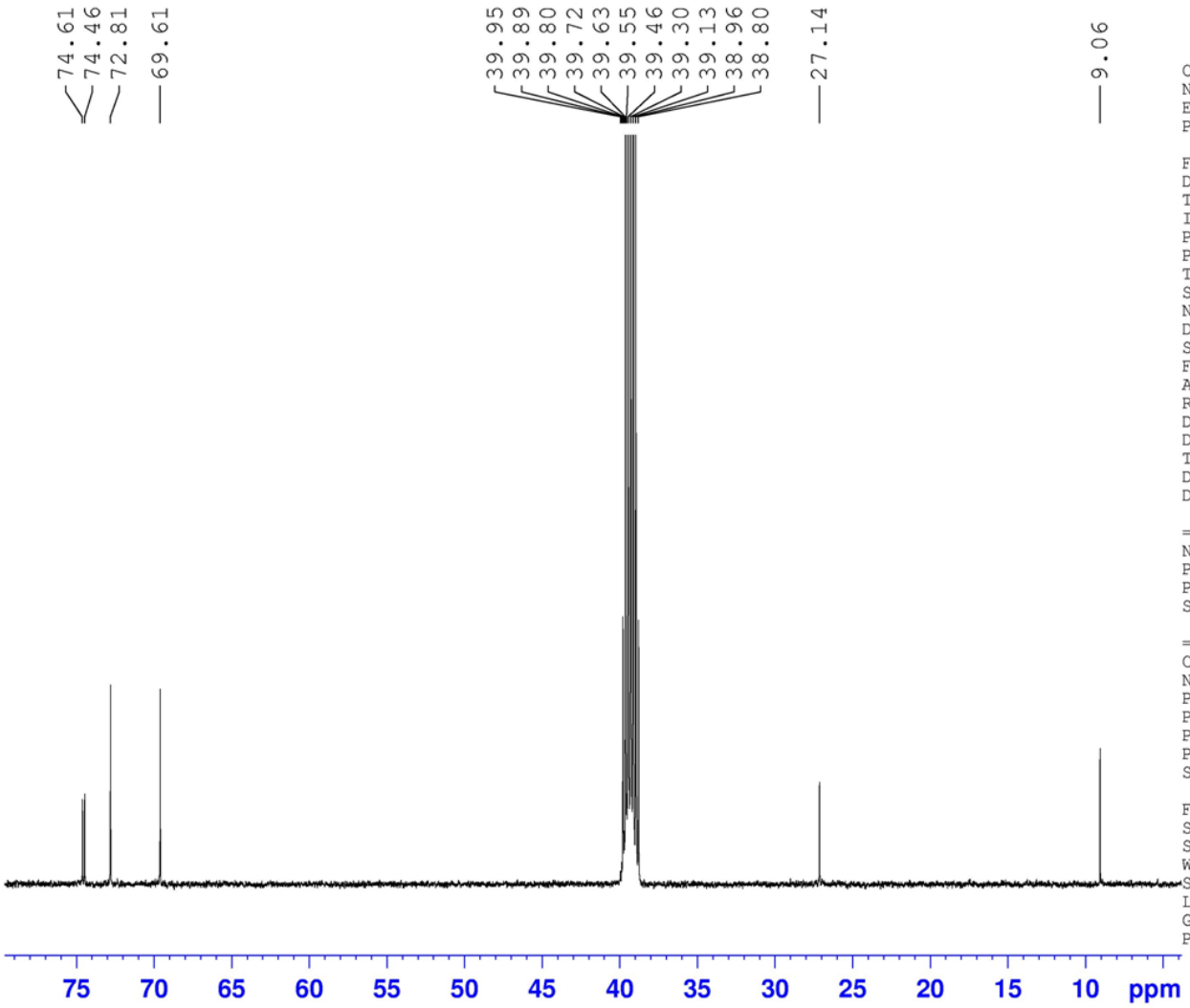
^{13}C NMR of **3d** in DMSO-d_6




```
Current Data Parameters
NAME                 Sagar
EXPNO                  223
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111116
Time                 16.03
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT               DMSO
NS                     1024
DS                        4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ                1.1010548 sec
RG                   191.08
DW                   16.800 usec
DE                    6.50 usec
TE                   298.0 K
D1               2.00000000 sec
D11              0.03000000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    8.65 usec
PLW1          120.50000000 W
SFO1           125.7955112 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2                80.00 usec
PLW2           27.16399956 W
PLW12           0.34685999 W
PLW13           0.22199000 W
SFO2           500.2320009 MHz

F2 - Processing parameters
SI                    32768
SF             125.7829969 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

Supplementary Information

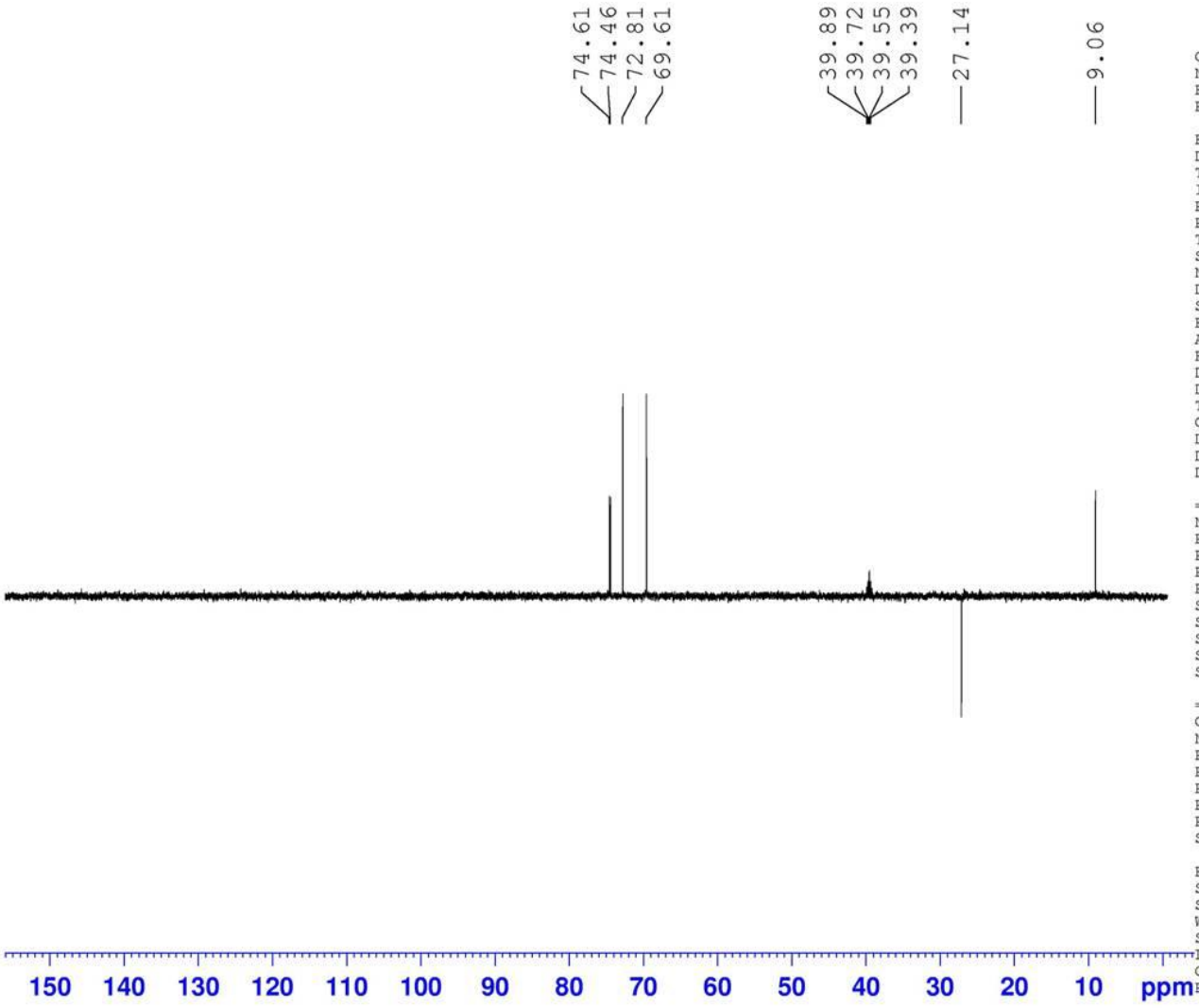
^{13}C NMR of **3d** (zoom)




```
Current Data Parameters
NAME                  Sagar
EXPNO                   223
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111116
Time                  16.03
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT                DMSO
NS                      1024
DS                         4
SWH               29761.904 Hz
FIDRES             0.454131 Hz
AQ                1.1010548 sec
RG                   191.08
DW                   16.800 usec
DE                    6.50 usec
TE                   298.0 K
D1                2.00000000 sec
D11               0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    8.65 usec
PLW1            120.50000000 W
SFO1             125.7955112 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                80.00 usec
PLW2            27.16399956 W
PLW12            0.34685999 W
PLW13            0.22199000 W
SFO2            500.2320009 MHz

F2 - Processing parameters
SI                      32768
SF               125.7829969 MHz
WDW                        EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
```

Supplementary Information

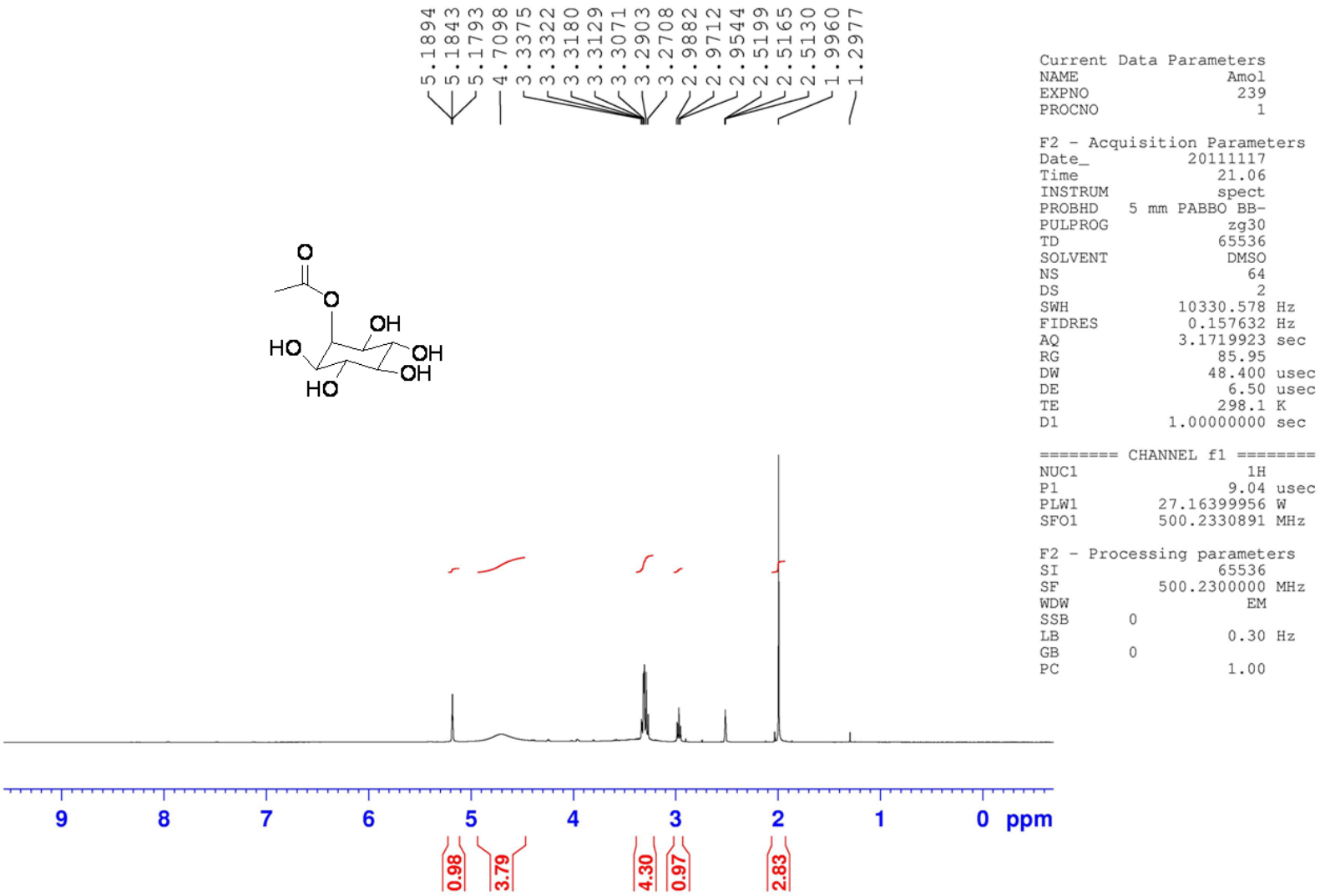
DEPT of **3d**




```
Current Data Parameters
NAME                  Sagar
EXPNO                   222
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111116
Time                  14.58
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG          deptsp135
TD                   65536
SOLVENT               DMSO
NS                      256
DS                        4
SWH              20161.291 Hz
FIDRES            0.307637 Hz
AQ                1.6253428 sec
RG                   191.08
DW                  24.800 usec
DE                   6.50 usec
TE                  298.0 K
CNST2           145.0000000
D1                2.00000000 sec
D2                0.00344828 sec
D12               0.00002000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    8.65 usec
P13                2000.00 usec
PLW0       0 W
PLW1          120.50000000 W
SFO1           125.7929956 MHz
SPNAM5         Crp60comp.4
SPOAL5               0.500
SPOFFS5    0 Hz
SPW5           13.77600002 W

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
P3                    9.03 usec
P4                   18.06 usec
PCPD2                80.00 usec
PLW2          27.16399956 W
PLW12          0.34685999 W
SFO2          500.2315998 MHz

F2 - Processing parameters
SI                    32768
SF             125.7829969 MHz
WDW                     EM
SSB        0
LB                    1.00 Hz
GB         0
PC                    1.40
```

Supplementary Information

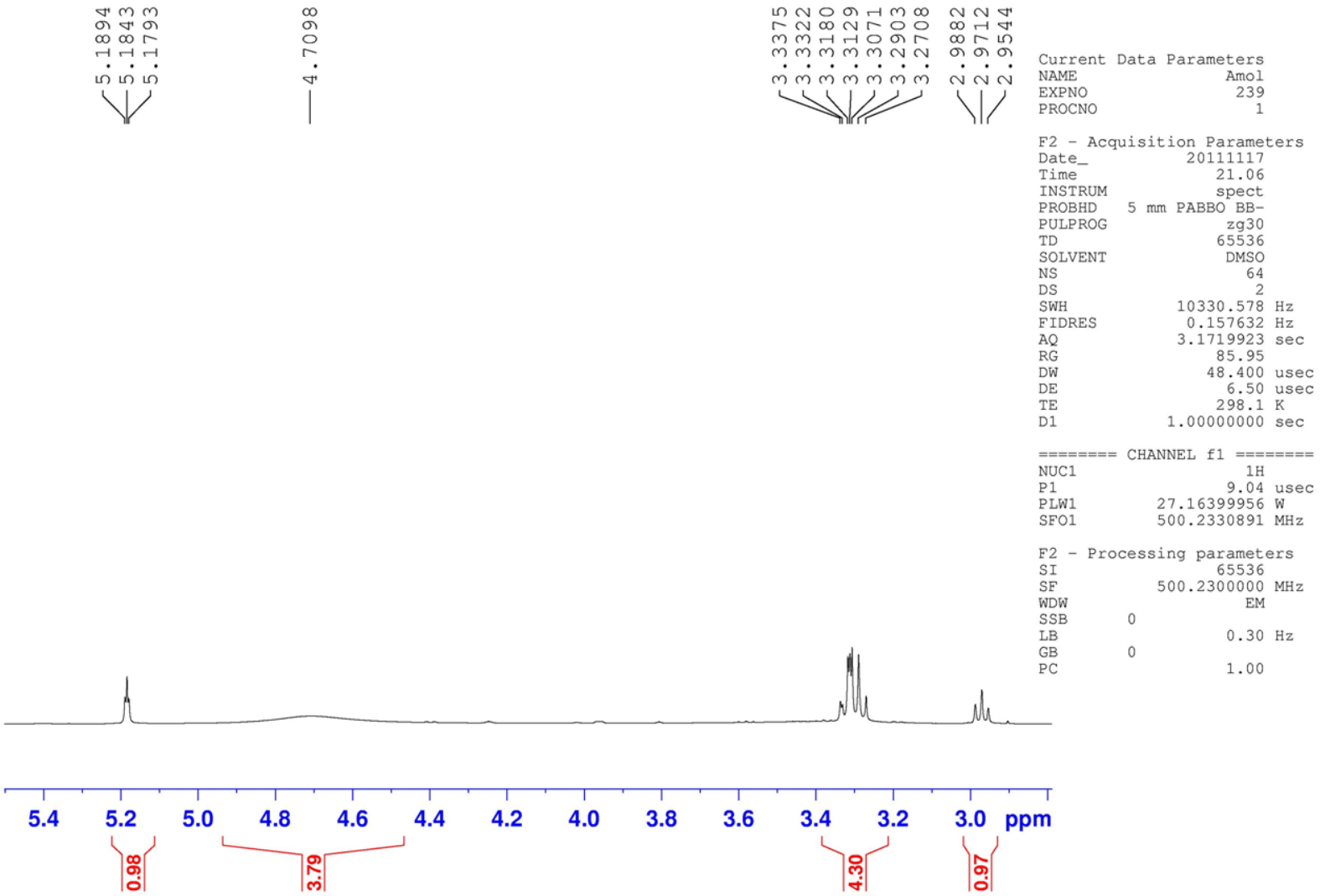
^{1}H NMR of **3e** in DMSO-d_6




```
Current Data Parameters
NAME                  Amol
EXPNO                  239
PROCNO                   1

F2 - Acquisition Parameters
Date_              20111117
Time                 21.06
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT               DMSO
NS                      64
DS                       2
SWH              10330.578 Hz
FIDRES            0.157632 Hz
AQ               3.1719923 sec
RG                   85.95
DW                  48.400 usec
DE                   6.50 usec
TE                  298.1 K
D1               1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.04 usec
PLW1          27.16399956 W
SFO1          500.2330891 MHz

F2 - Processing parameters
SI                   65536
SF            500.2300000 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```

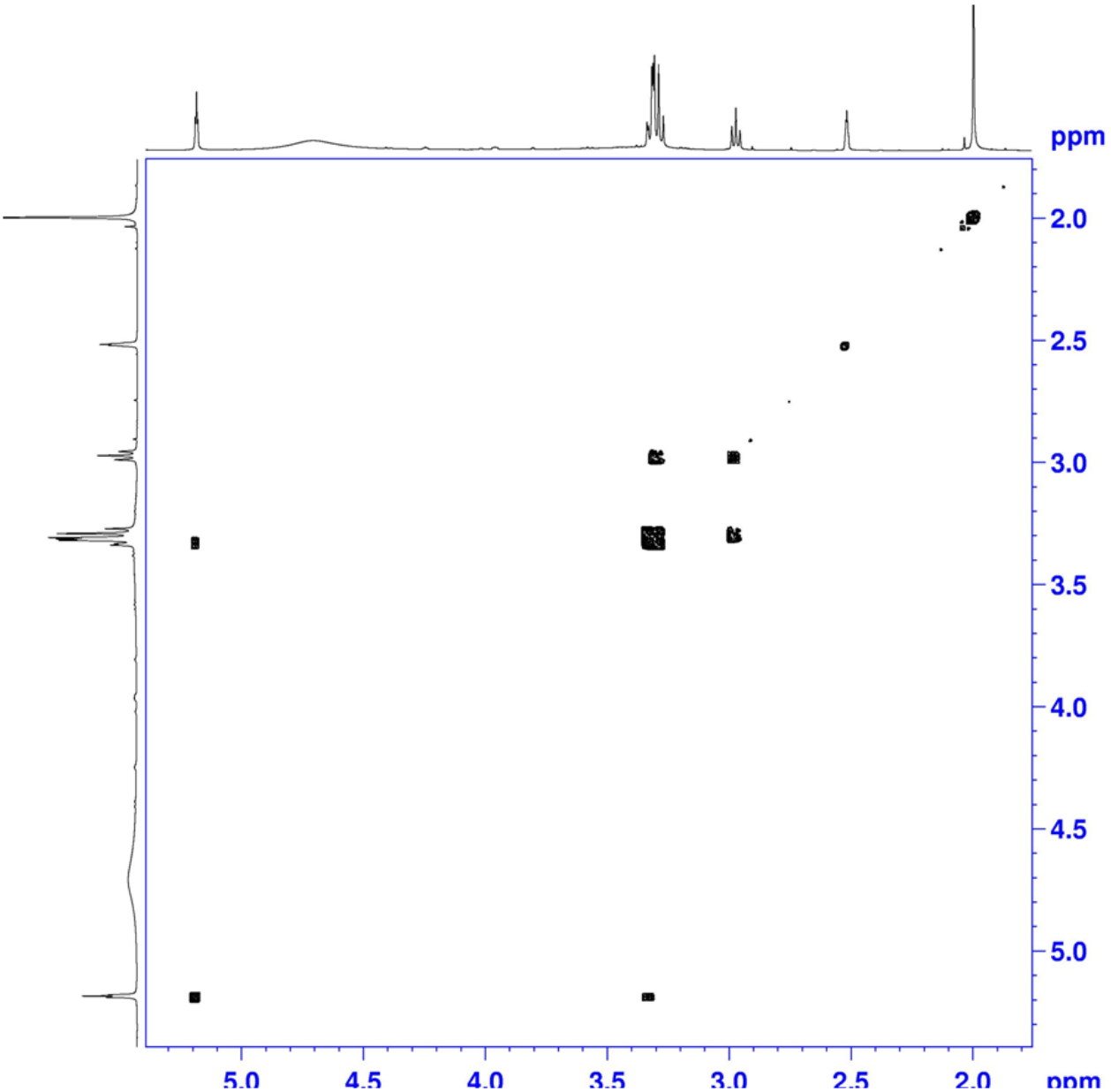
Supplementary Information

^{1}H NMR of **3e** (zoom)



Supplementary Information

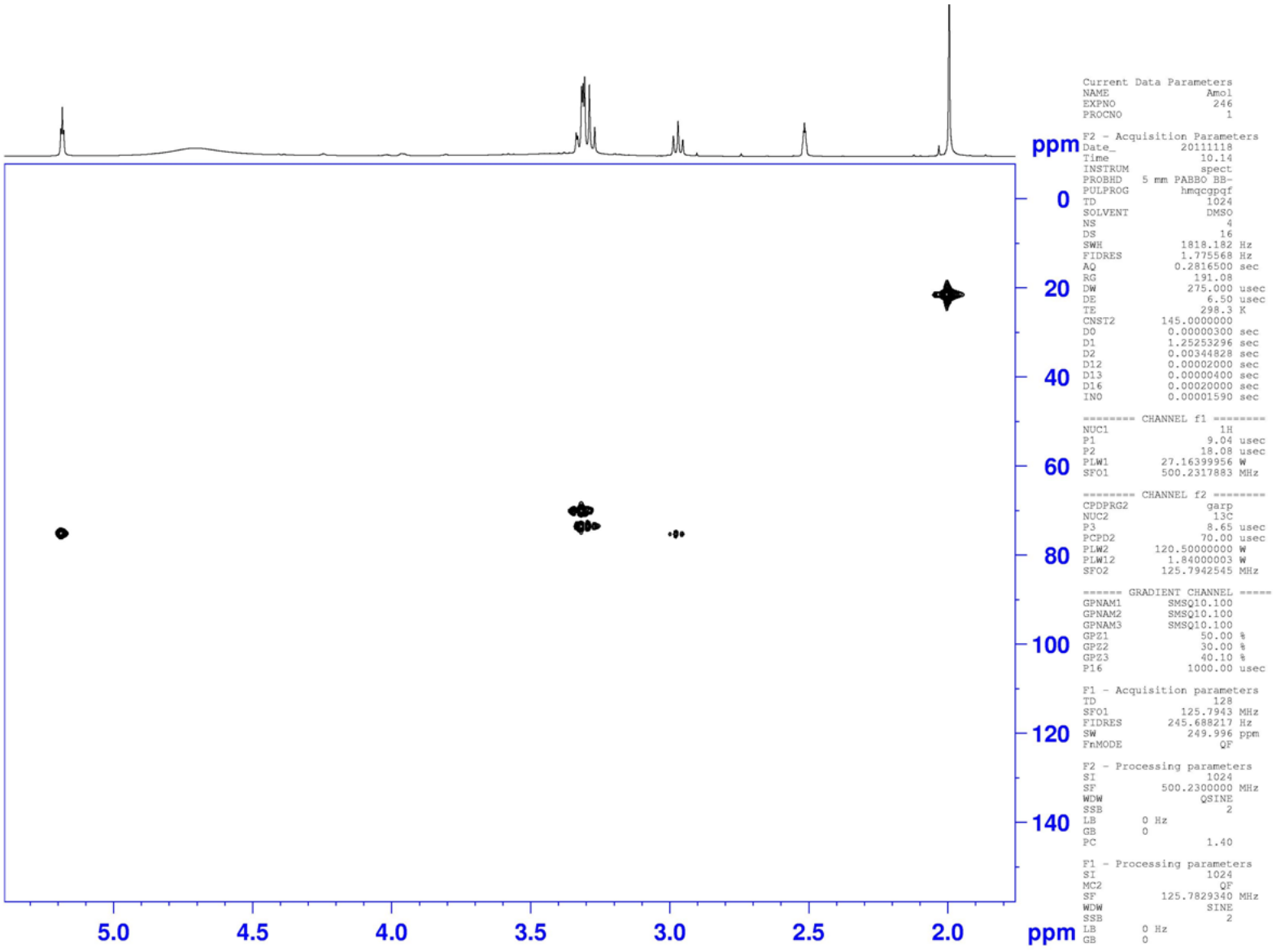
COSY of **3e**




```
Current Data Parameters
NAME                  Amol
EXPNO                  244
PROCNO                   1

F2 - Acquisition Parameters
Date_              20111118
Time                  6.10
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG       cosygpppqf
TD                    2048
SOLVENT               DMSO
NS                      64
DS                       8
SWH              1818.182 Hz
FIDRES           0.887784 Hz
AQ               0.5632500 sec
RG                    64.12
DW                275.000 usec
DE                  6.50 usec
TE                 299.4 K
D0              0.00000300 sec
D1              1.58999002 sec
D11             0.03000000 sec
D12             0.00002000 sec
D13             0.00000400 sec
D16             0.00020000 sec
IN0             0.00055000 sec

======== CHANNEL f1 ========
NUC1                    1H
P0                    9.04 usec
P1                    9.04 usec
P17                2500.00 usec
PLW1         27.16399956 W
PLW10         3.28390002 W
SFO1          500.2317883 MHz

====== GRADIENT CHANNEL =====
GPNAM1         SMSQ10.100
GPZ1                10.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                      92
SFO1             500.2318 MHz
FIDRES          19.762846 Hz
SW                  3.635 ppm
FnMODE                 QF

F2 - Processing parameters
SI                    1024
SF            500.2300000 MHz
WDW                  QSINE
SSB       0
LB        0 Hz
GB        0
PC                    1.40

F1 - Processing parameters
SI                    1024
MC2                    QF
SF            500.2300000 MHz
WDW                   SIN
SSB       0
LB        0 Hz
GB        0
```

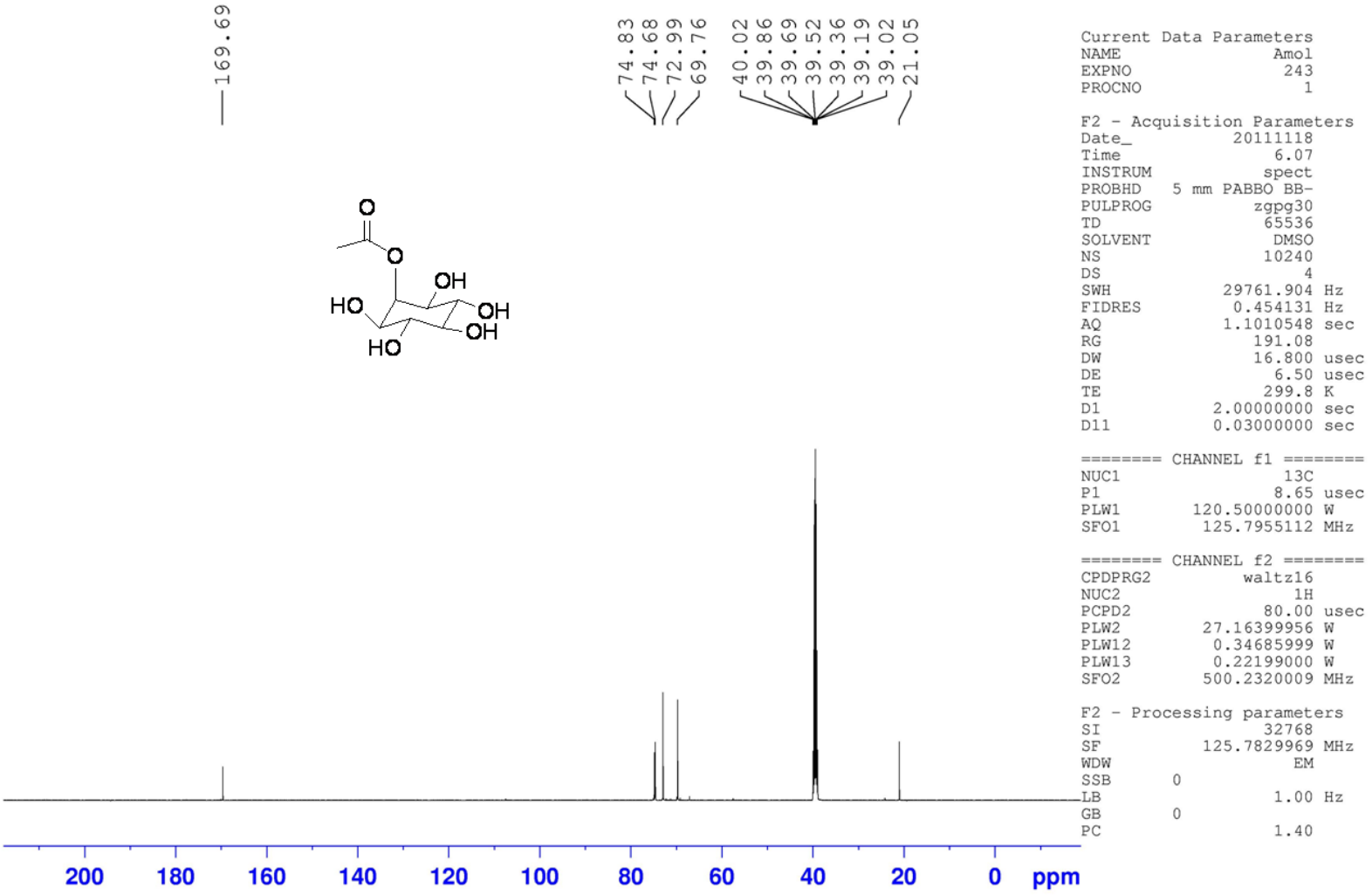
Supplementary Information

HMQC of **3e**



Supplementary Information

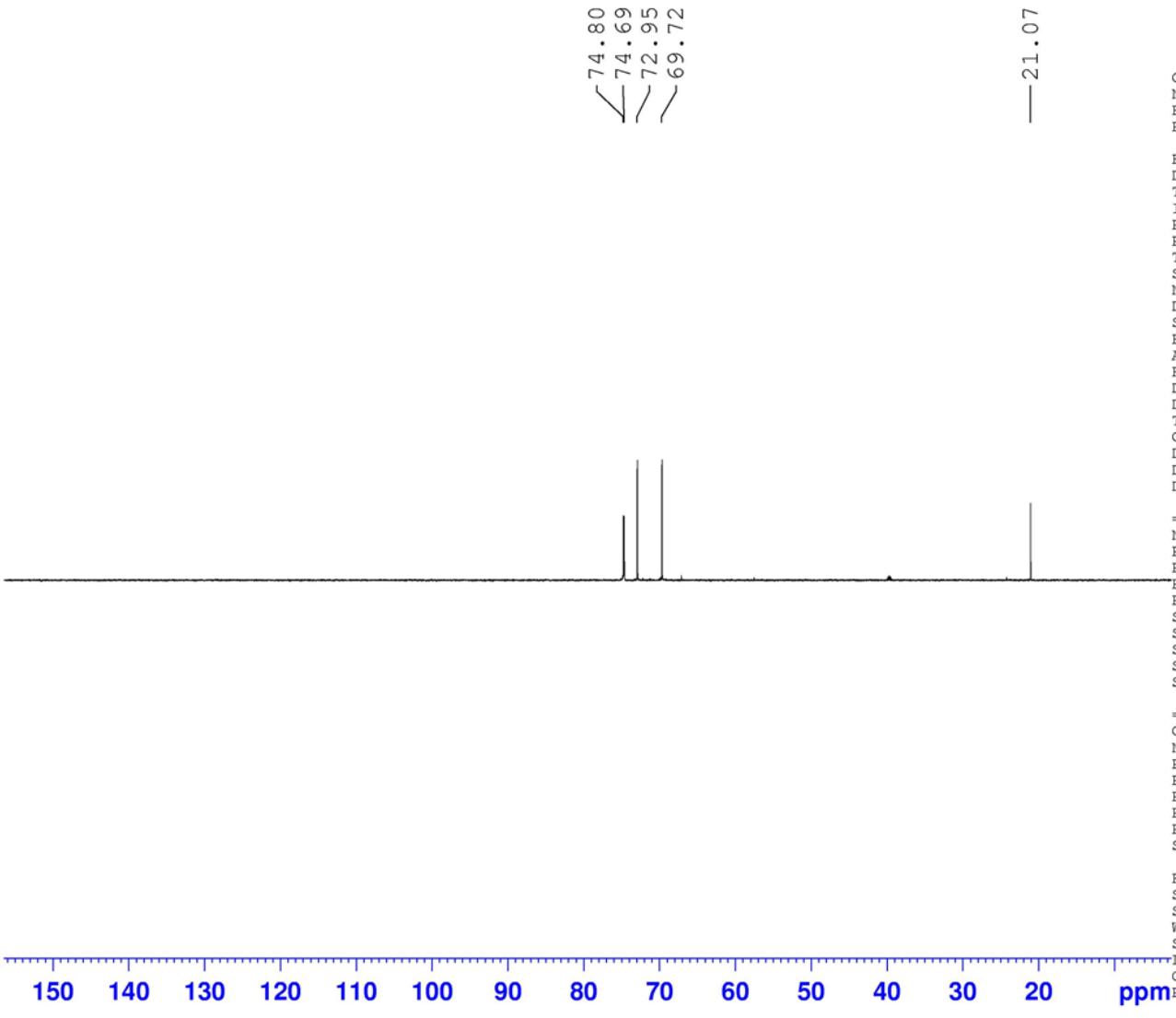
^{13}C NMR of **3e** in DMSO-d_6




```
Current Data Parameters
NAME                  Amol
EXPNO                  243
PROCNO                   1

F2 - Acquisition Parameters
Date_           20111118
Time                6.07
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                10240
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010548 sec
RG                191.08
DW                16.800 usec
DE                 6.50 usec
TE                299.8 K
D1            2.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 8.65 usec
PLW1       120.50000000 W
SFO1        125.7955112 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2        27.16399956 W
PLW12        0.34685999 W
PLW13        0.22199000 W
SFO2        500.2320009 MHz

F2 - Processing parameters
SI                 32768
SF          125.7829969 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

Supplementary Information

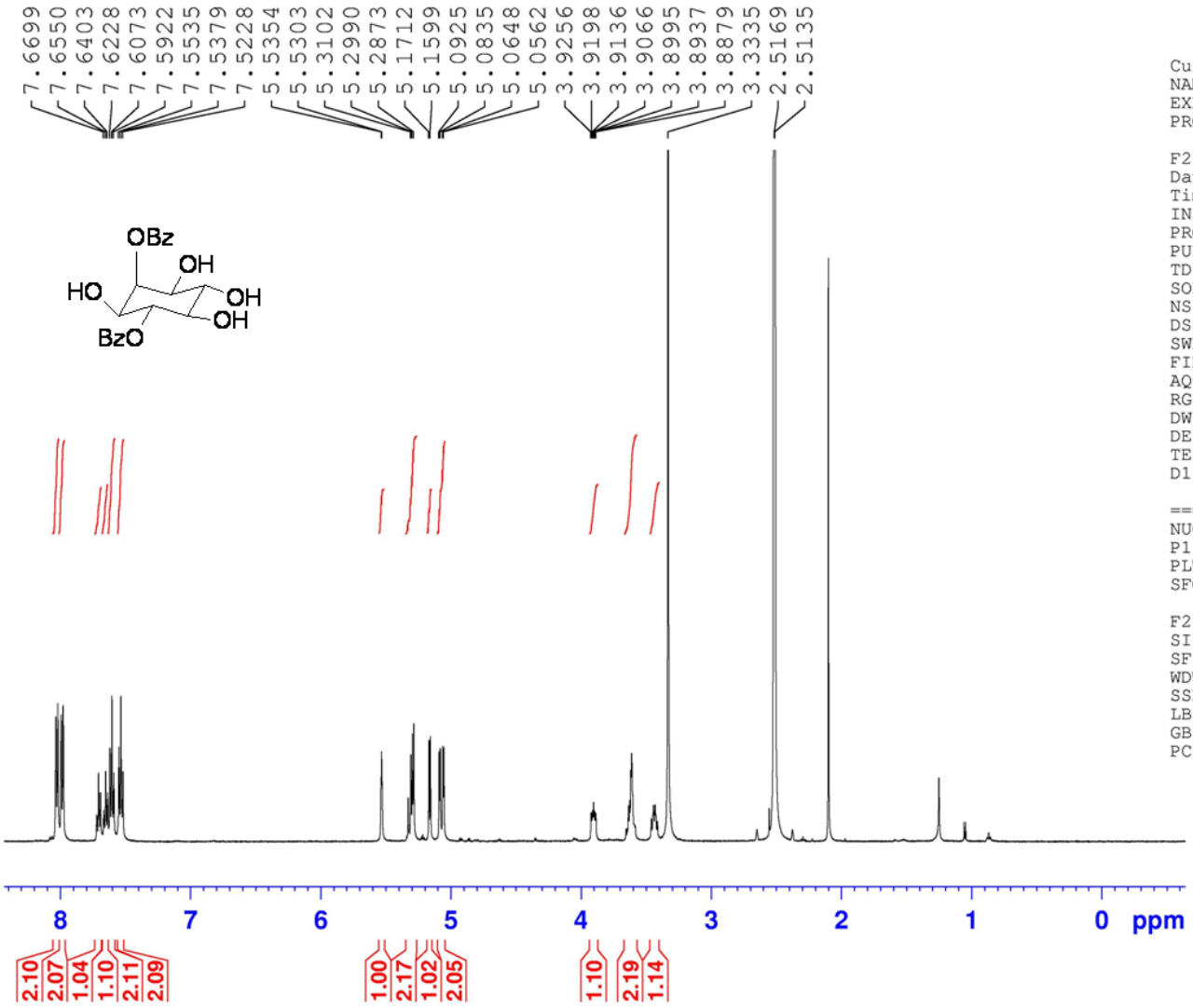
DEPT of **3e**




```
Current Data Parameters
NAME                  Amol
EXPNO                  245
PROCNO                   1

F2 - Acquisition Parameters
Date_              20111118
Time                  9.49
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG          deptsp135
TD                   65536
SOLVENT                DMSO
NS                       405
DS                         4
SWH               20161.291 Hz
FIDRES             0.307637 Hz
AQ                1.6253428 sec
RG                   191.08
DW                   24.800 usec
DE                    6.50 usec
TE                   297.1 K
CNST2           145.0000000
D1                2.00000000 sec
D2                0.00344828 sec
D12               0.00002000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                     8.65 usec
P13                 2000.00 usec
PLW0        0 W
PLW1          120.50000000 W
SFO1           125.7929956 MHz
SPNAM5         Crp60comp.4
SPOAL5                0.500
SPOFFS5     0 Hz
SPW5           13.77600002 W

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
P3                     9.03 usec
P4                    18.06 usec
PCPD2                 80.00 usec
PLW2            27.16399956 W
PLW12            0.34685999 W
SFO2           500.2315998 MHz

F2 - Processing parameters
SI                     32768
SF              125.7829969 MHz
WDW                       EM
SSB         0
LB                     1.00 Hz
GB          0
PC                     1.40
```

Supplementary Information

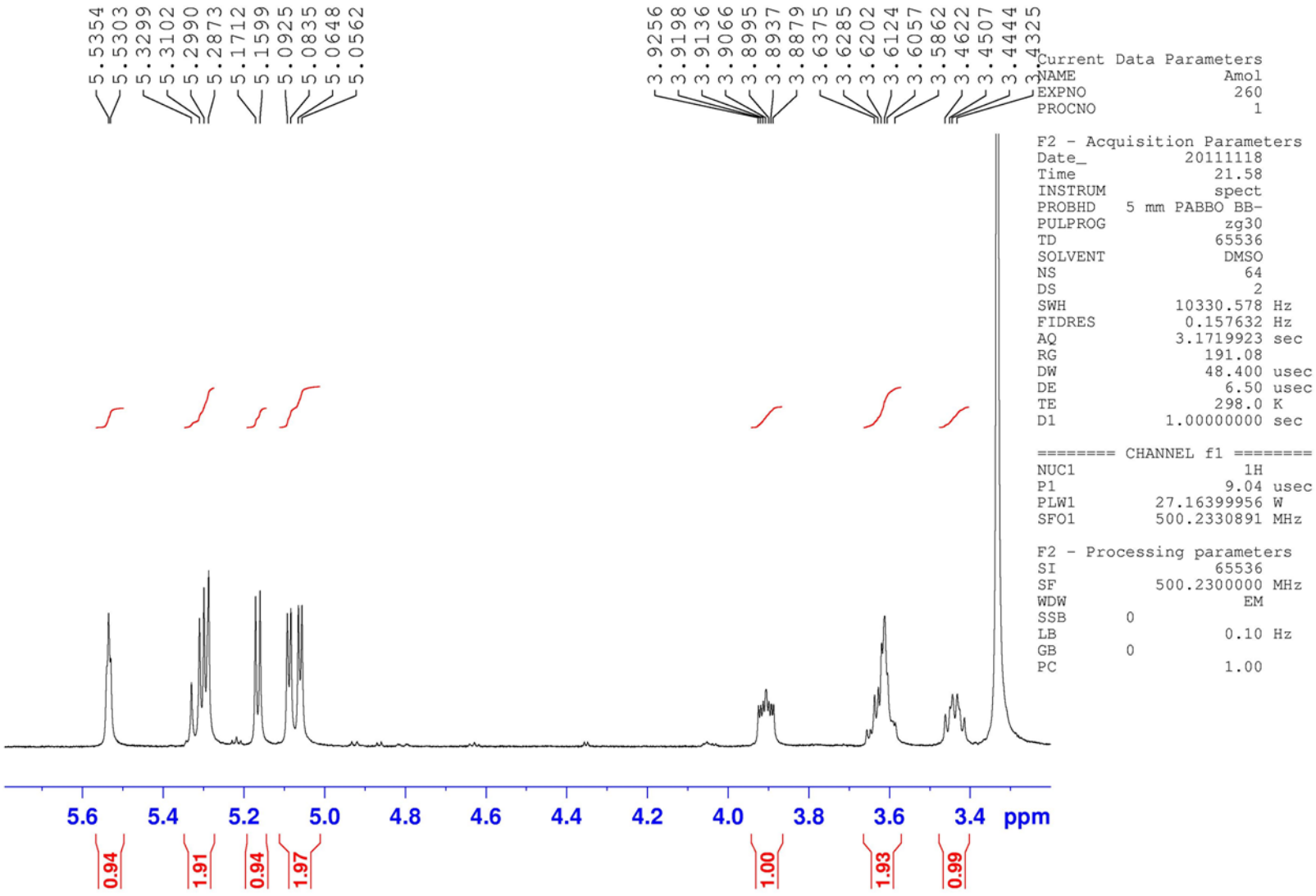
^{1}H NMR of **11** in DMSO-d_6




```
Current Data Parameters
NAME                  Amol
EXPNO                  260
PROCNO                   1

F2 - Acquisition Parameters
Date_           20111118
Time               21.58
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    64
DS                     2
SWH            10330.578 Hz
FIDRES          0.157632 Hz
AQ             3.1719923 sec
RG                191.08
DW                48.400 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.04 usec
PLW1         27.16399956 W
SFO1         500.2330891 MHz

F2 - Processing parameters
SI                 65536
SF           500.2300000 MHz
WDW                  EM
SSB       0
LB                 0.10 Hz
GB        0
PC                 1.00
```

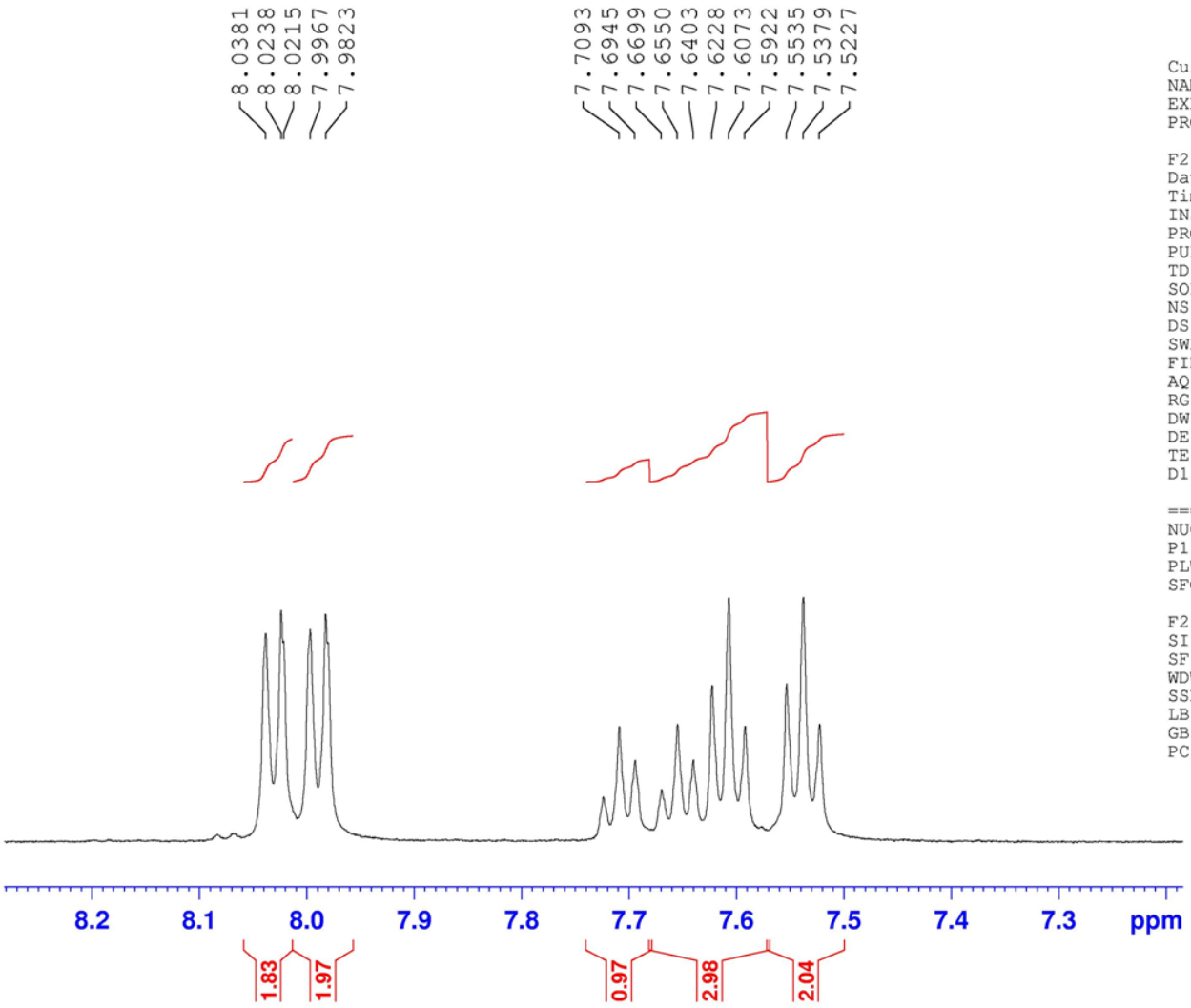
Supplementary Information

^{1}H NMR of **11** in DMSO-d_6 (zoom)



Supplementary Information

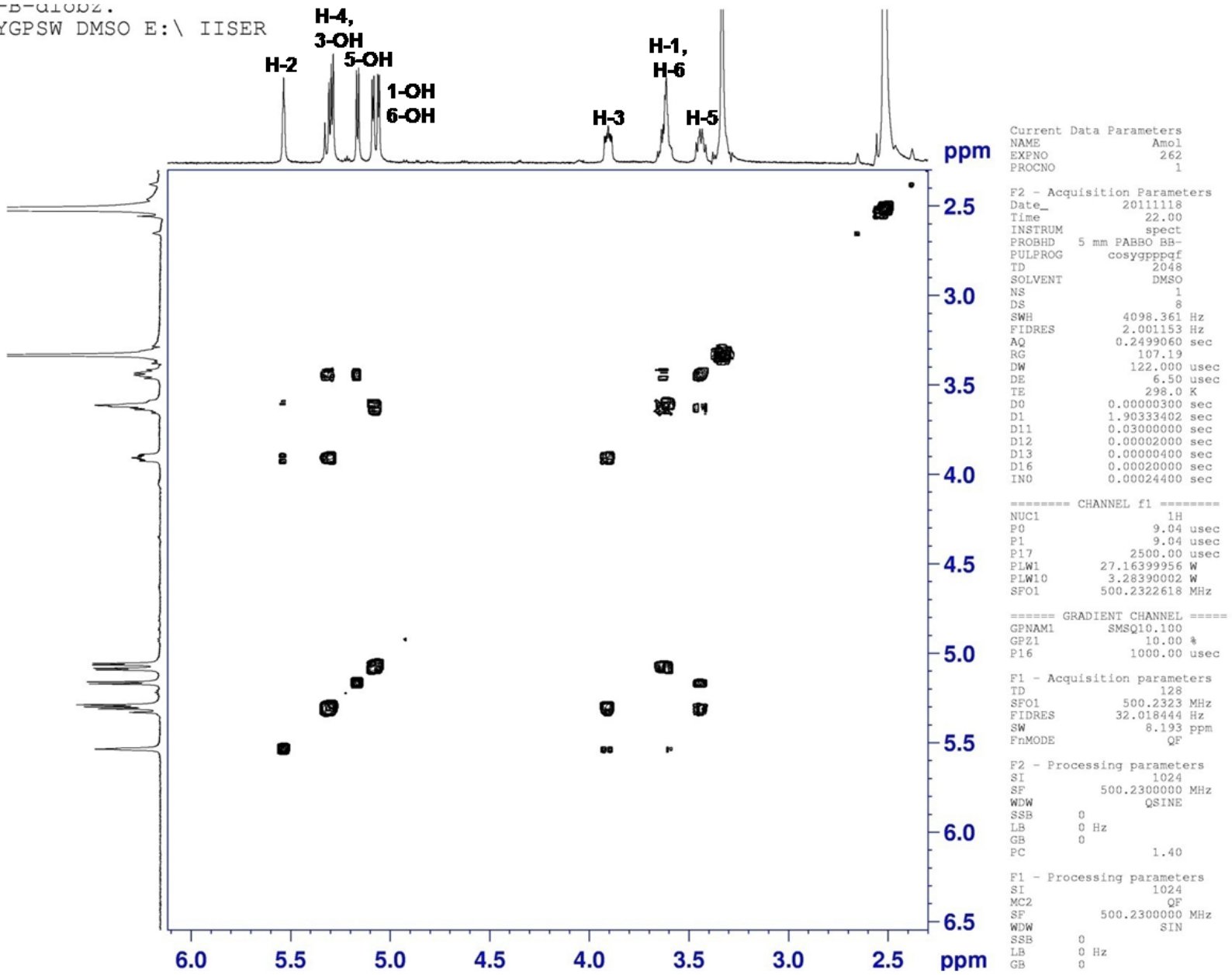
^{1}H NMR of **11** in DMSO-d_6 (zoom)




```
Current Data Parameters
NAME                  Amol
EXPNO                  260
PROCNO                   1

F2 - Acquisition Parameters
Date_              20111118
Time                 21.58
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT               DMSO
NS                      64
DS                       2
SWH             10330.578 Hz
FIDRES           0.157632 Hz
AQ               3.1719923 sec
RG                  191.08
DW                  48.400 usec
DE                   6.50 usec
TE                  298.0 K
D1               1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.04 usec
PLW1          27.16399956 W
SFO1          500.2330891 MHz

F2 - Processing parameters
SI                  65536
SF            500.2300000 MHz
WDW                    EM
SSB       0
LB                   0.10 Hz
GB        0
PC                   1.00
```

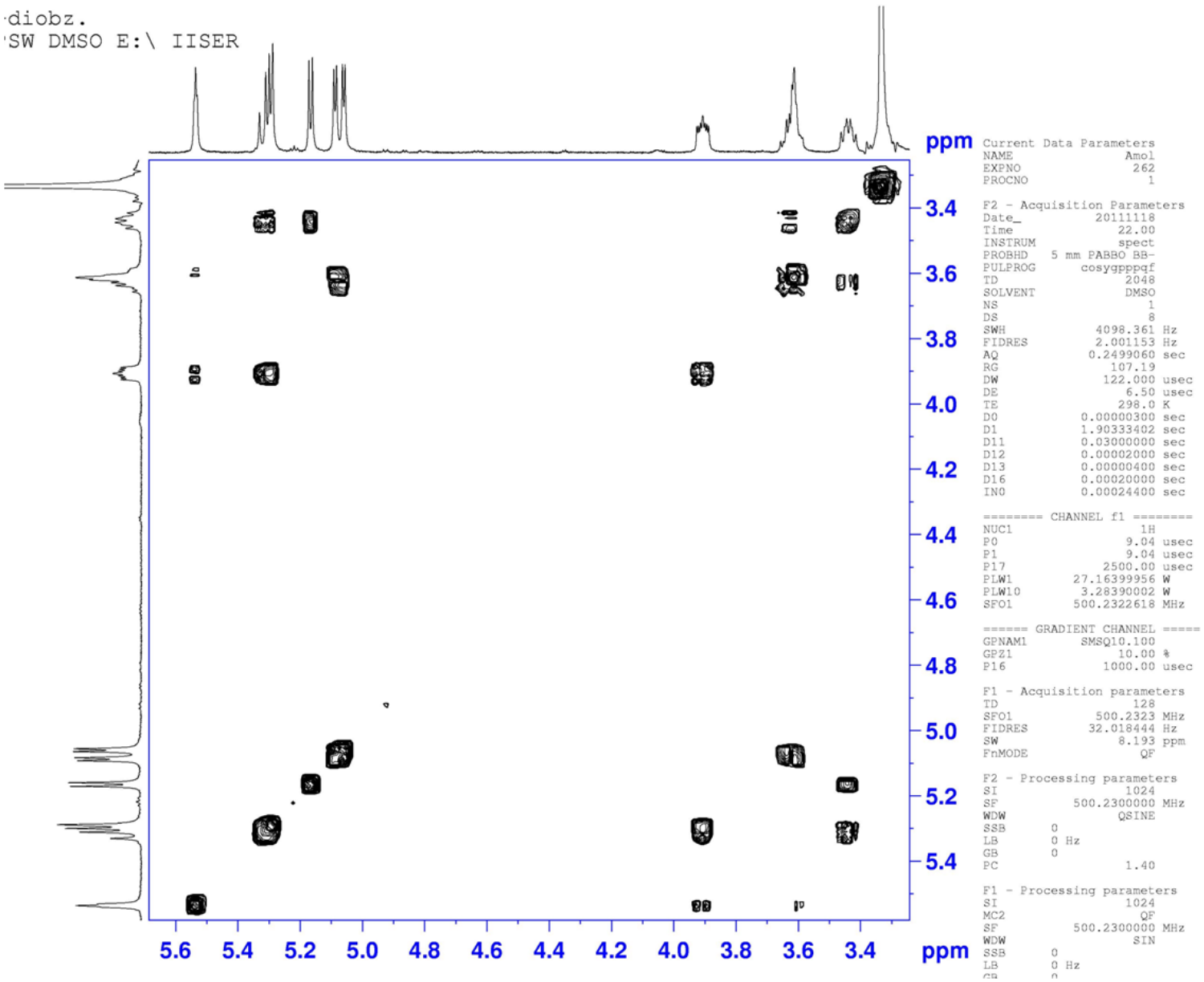
Supplementary Information

COSY of **11** in DMSO-d_6



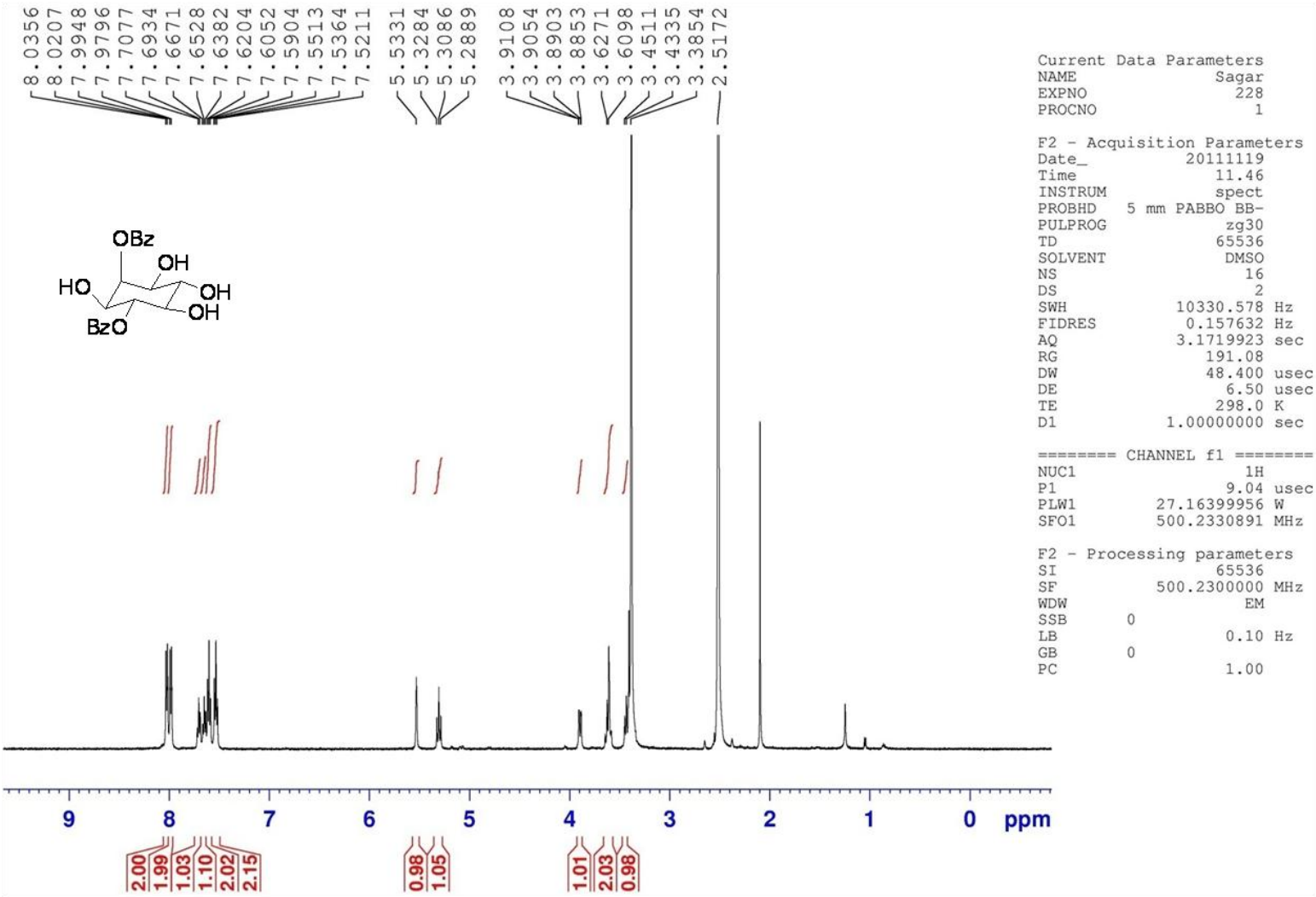
Supplementary Information

COSY of **11** in DMSO-d_6 (zoom)



Supplementary Information

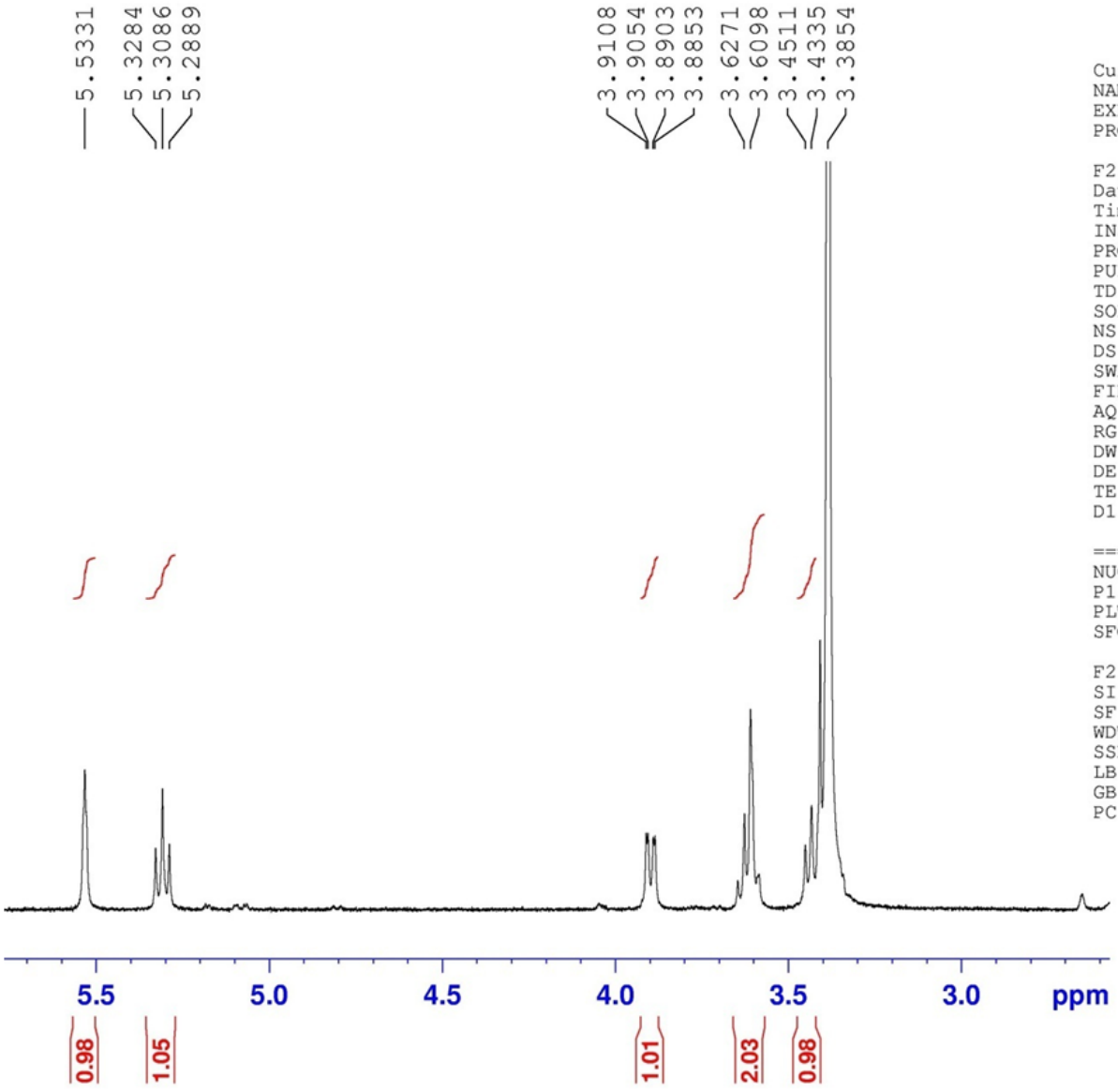
^{1}H NMR of **11** in DMSO-d_6 (D_2O Exchange)




```
Current Data Parameters
NAME                  Sagar
EXPNO                   228
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111119
Time                  11.46
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       16
DS                        2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                3.1719923 sec
RG                   191.08
DW                   48.400 usec
DE                    6.50 usec
TE                   298.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    9.04 usec
PLW1           27.16399956 W
SFO1           500.2330891 MHz

F2 - Processing parameters
SI                    65536
SF             500.2300000 MHz
WDW                      EM
SSB       0
LB                    0.10 Hz
GB        0
PC                    1.00
```

Supplementary Information

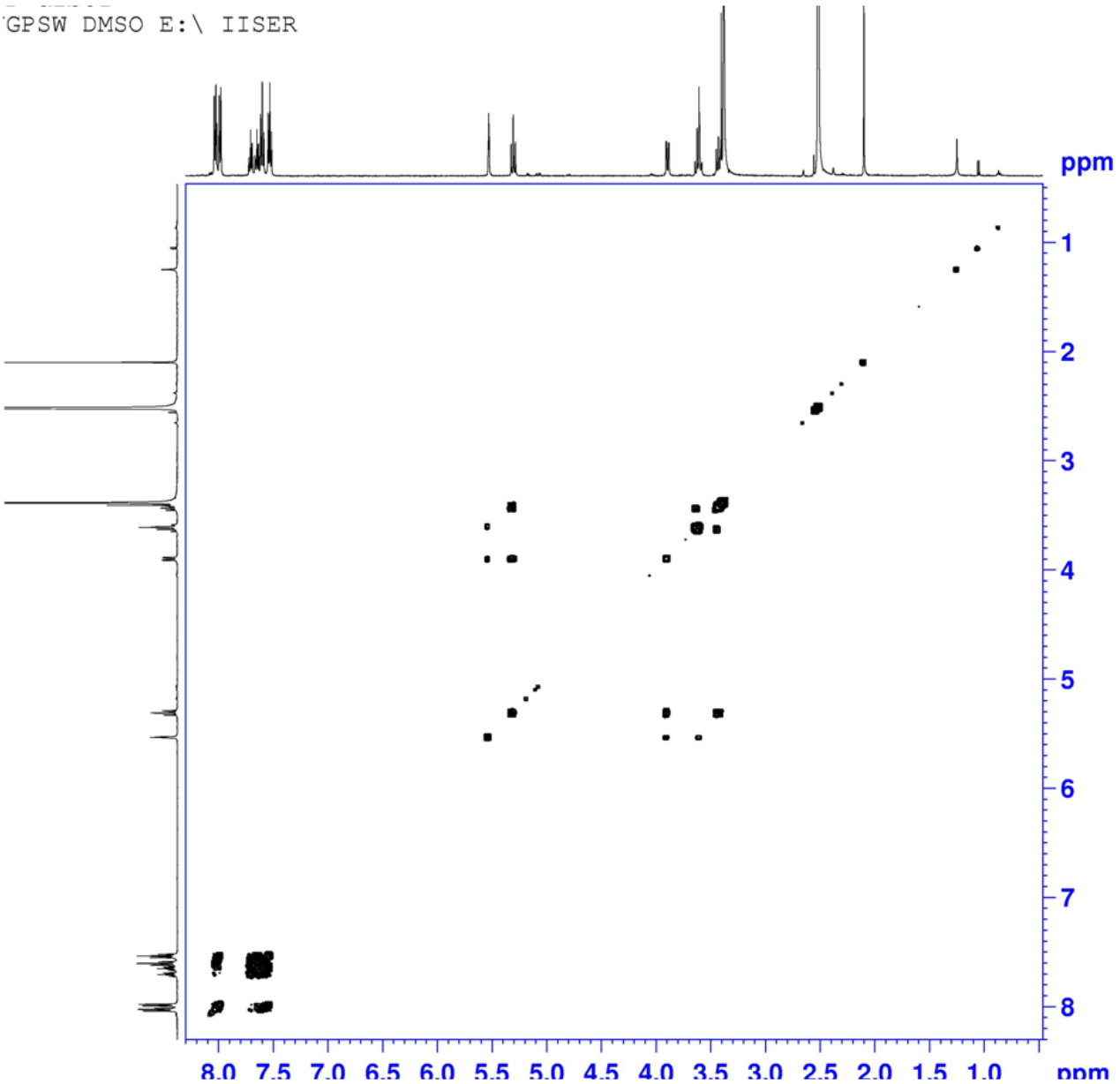
^{1}H NMR of **11** in DMSO-d_6 (D_2O Exchange) (zoom)




```
Current Data Parameters
NAME                  Sagar
EXPNO                   228
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111119
Time                  11.46
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       16
DS                        2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                3.1719923 sec
RG                   191.08
DW                   48.400 usec
DE                    6.50 usec
TE                   298.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    9.04 usec
PLW1          27.16399956 W
SFO1          500.2330891 MHz

F2 - Processing parameters
SI                    65536
SF            500.2300000 MHz
WDW                      EM
SSB       0
LB                    0.10 Hz
GB        0
PC                    1.00
```

Supplementary Information

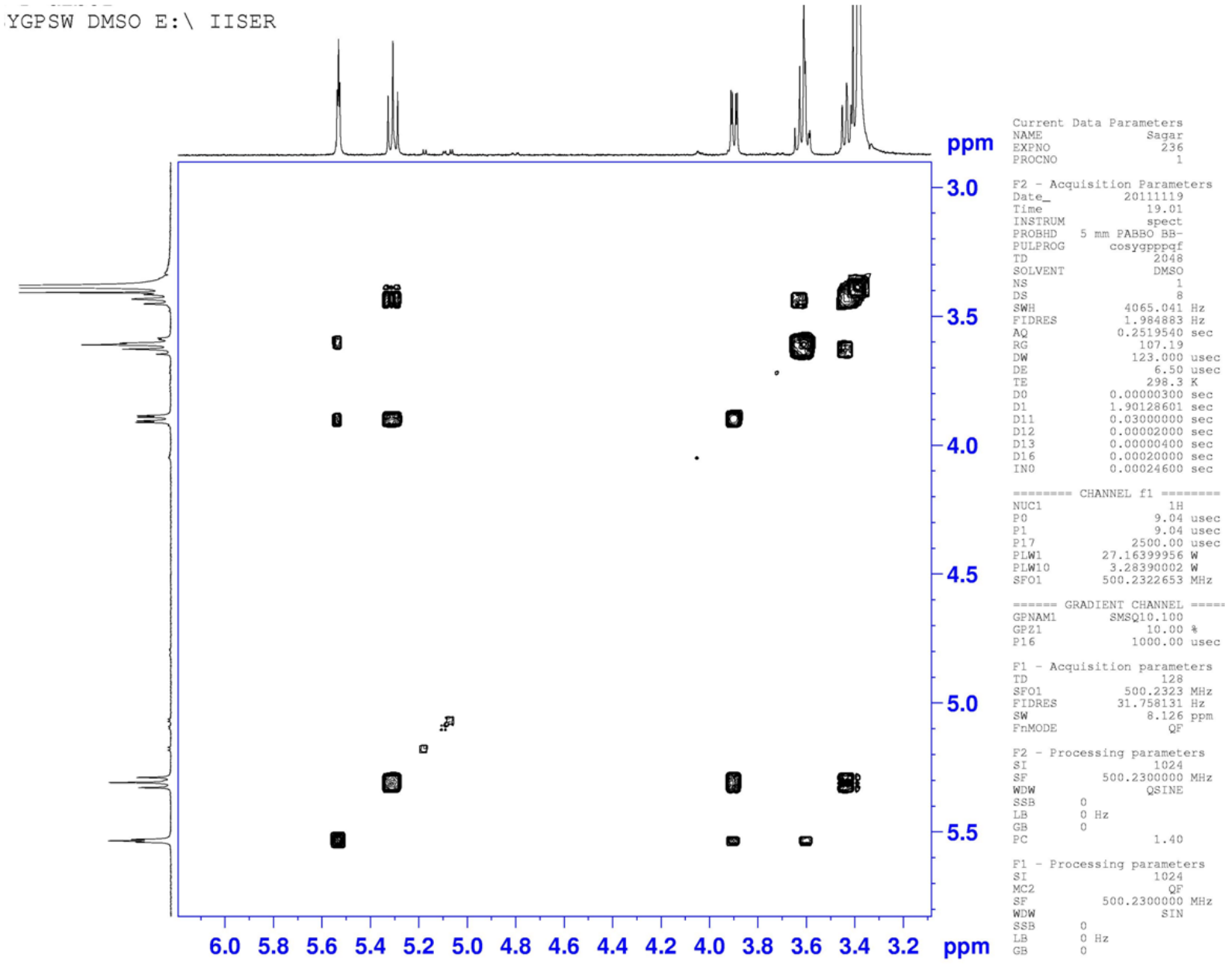
COSY of **11** in DMSO-d_6 (D_2O Exchange)




```
Current Data Parameters
NAME                  Sagar
EXPNO                   236
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111119
Time                  19.01
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG        cosygpppqf
TD                     2048
SOLVENT                DMSO
NS                        1
DS                        8
SWH                4065.041 Hz
FIDRES             1.984883 Hz
AQ                 0.2519540 sec
RG                   107.19
DW                   123.000 use
DE                     6.50 use
TE                   298.3 K
D0                 0.00000300 sec
D1                 1.90128601 sec
D11                0.03000000 sec
D12                0.00002000 sec
D13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00024600 sec

======== CHANNEL f1 =======
NUC1                      1H
P0                      9.04 use
P1                      9.04 use
P17                  2500.00 use
PLW1           27.16399956 W
PLW10           3.28390002 W
SFO1          500.2322653 MHz

====== GRADIENT CHANNEL ===
GPNAM1          SMSQ10.100
GPZ1                 10.00 %
P16                1000.00 use

F1 - Acquisition parameters
TD                       128
SFO1            500.2323 MHz
FIDRES          31.758131 Hz
SW                    8.126 ppm
FnMODE                   QF

F2 - Processing parameters
SI                      1024
SF            500.2300000 MHz
WDW                   QSINE
SSB         0
LB          0 Hz
GB          0
PC                    1.40

F1 - Processing parameters
SI                      1024
MC2                     QF
SF            500.2300000 MHz
WDW                   SIN
SSB         0
LB          0 Hz
GB          0
```

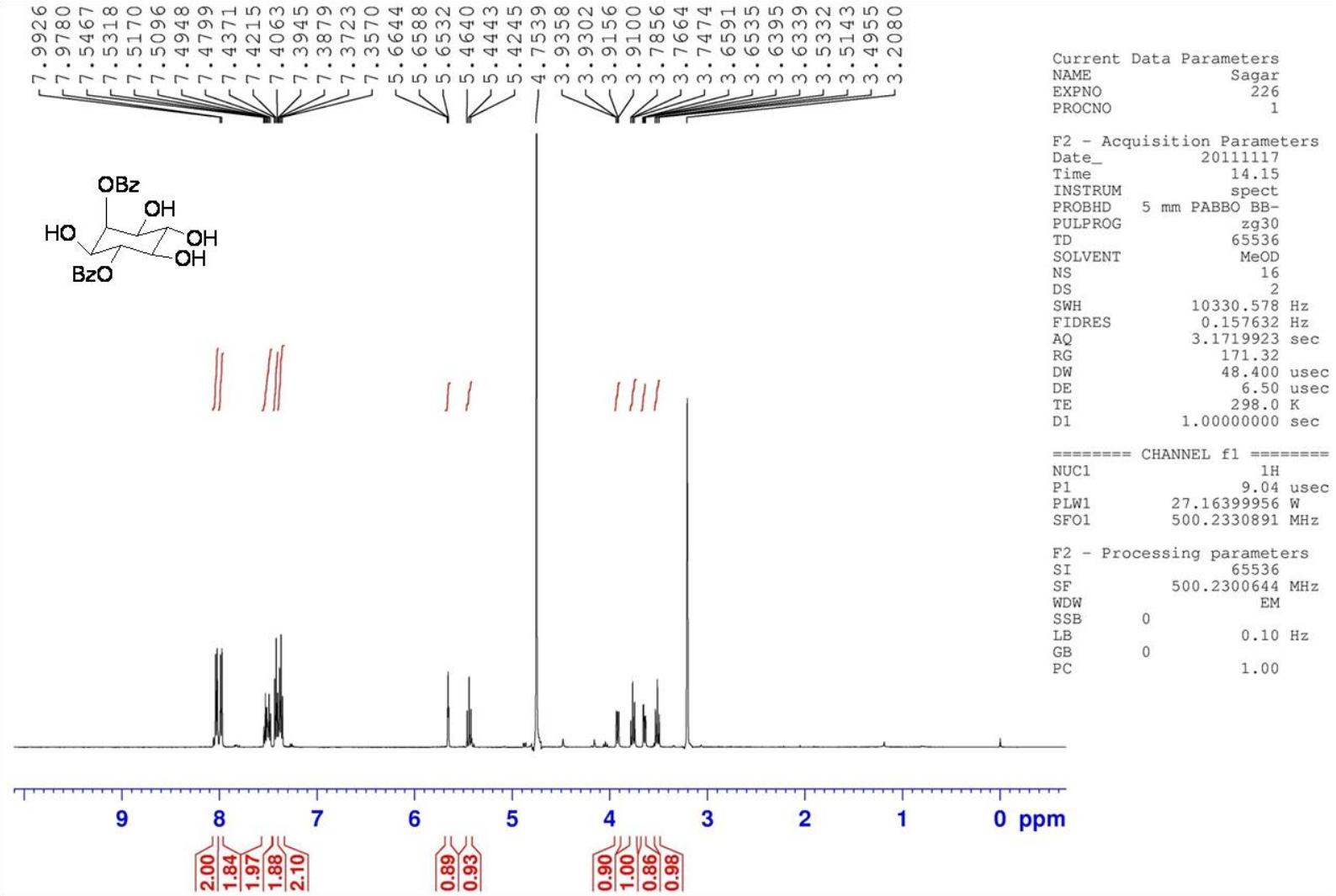
Supplementary Information

COSY of **11** in DMSO-d_6 (D_2O Exchange) (zoom)



Supplementary Information

^{1}H NMR of **11** in CD_3OD

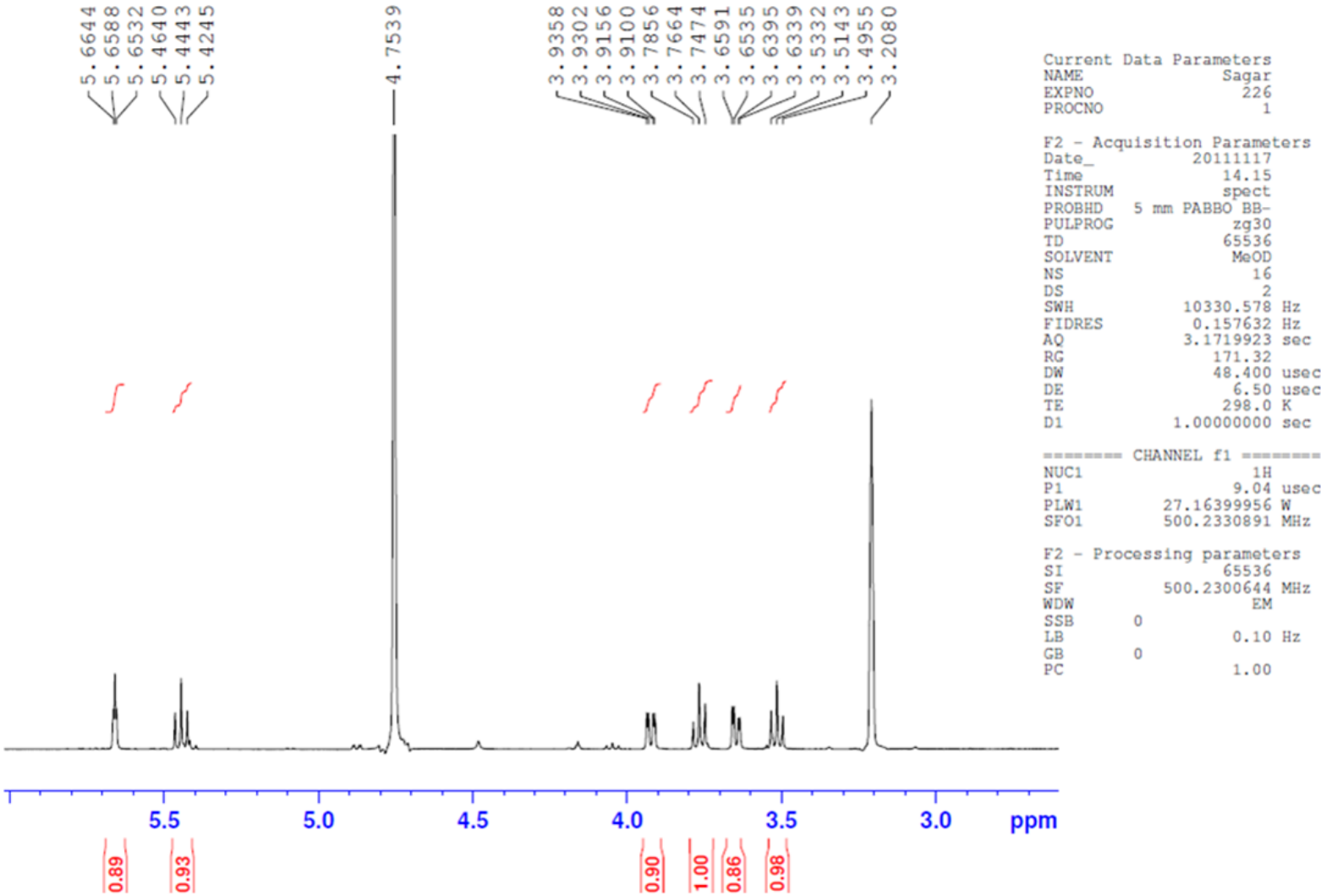



```
Current Data Parameters
NAME                  Sagar
EXPNO                   226
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111117
Time                  14.15
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                MeOD
NS                       16
DS                        2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                3.1719923 sec
RG                   171.32
DW                   48.400 usec
DE                    6.50 usec
TE                   298.0 K
D1                1.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.04 usec
PLW1            27.16399956 W
SFO1            500.2330891 MHz

F2 - Processing parameters
SI                        65536
SF              500.2300644 MHz
WDW                        EM
SSB       0
LB                     0.10 Hz
GB        0
PC                     1.00
```

The minor peaks in the spectrum are due to the transesterification of dibenzoate in the presence of methanol.

Supplementary Information

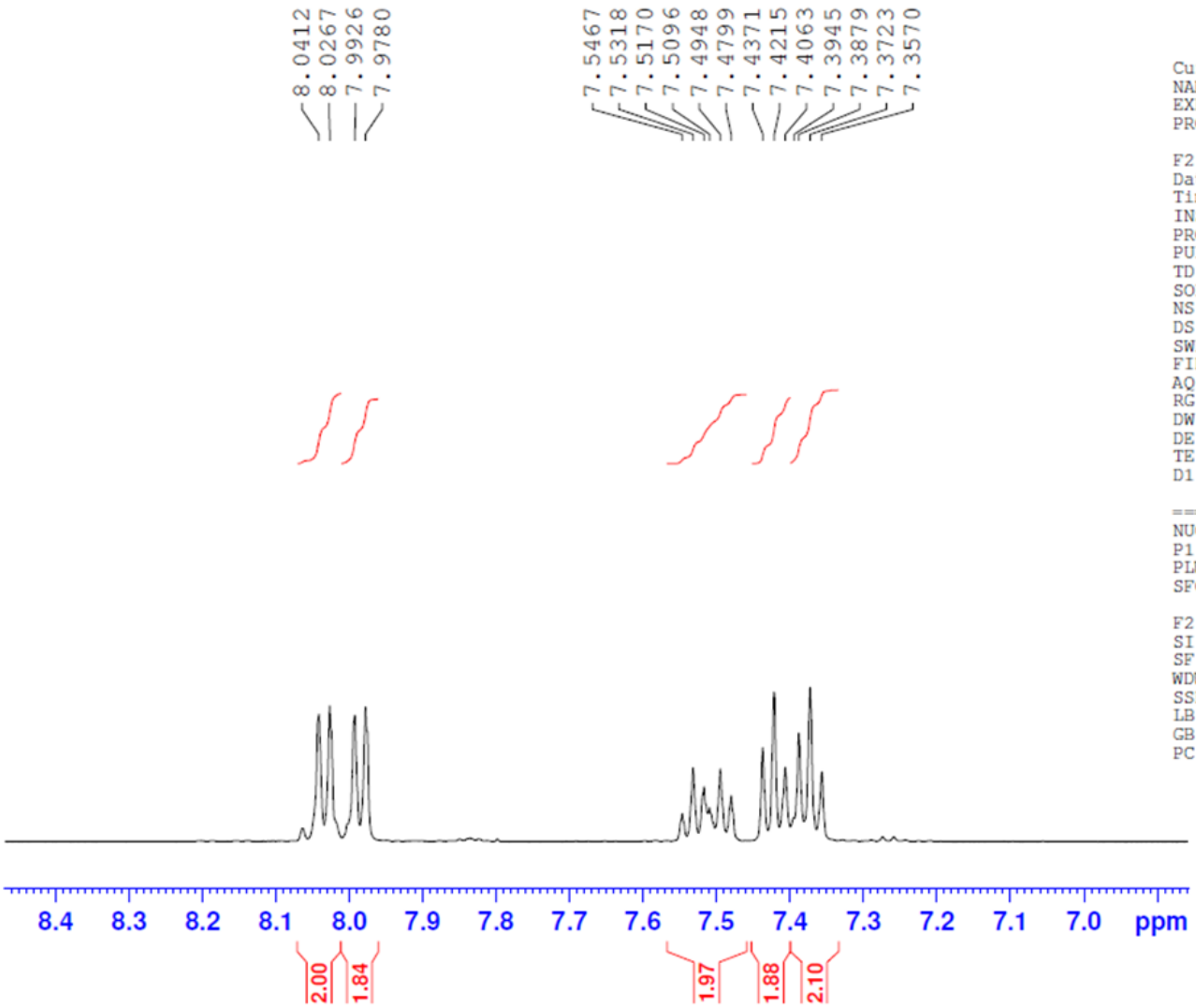
^{1}H NMR of **11** in in CD_3OD (zoom)




```
Current Data Parameters
NAME                  Sagar
EXPNO                   226
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111117
Time                  14.15
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                MeOD
NS                       16
DS                        2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                3.1719923 sec
RG                   171.32
DW                   48.400 usec
DE                    6.50 usec
TE                   298.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    9.04 usec
PLW1          27.16399956 W
SFO1          500.2330891 MHz

F2 - Processing parameters
SI                    65536
SF             500.2300644 MHz
WDW                      EM
SSB       0
LB                    0.10 Hz
GB        0
PC                    1.00
```

Supplementary Information

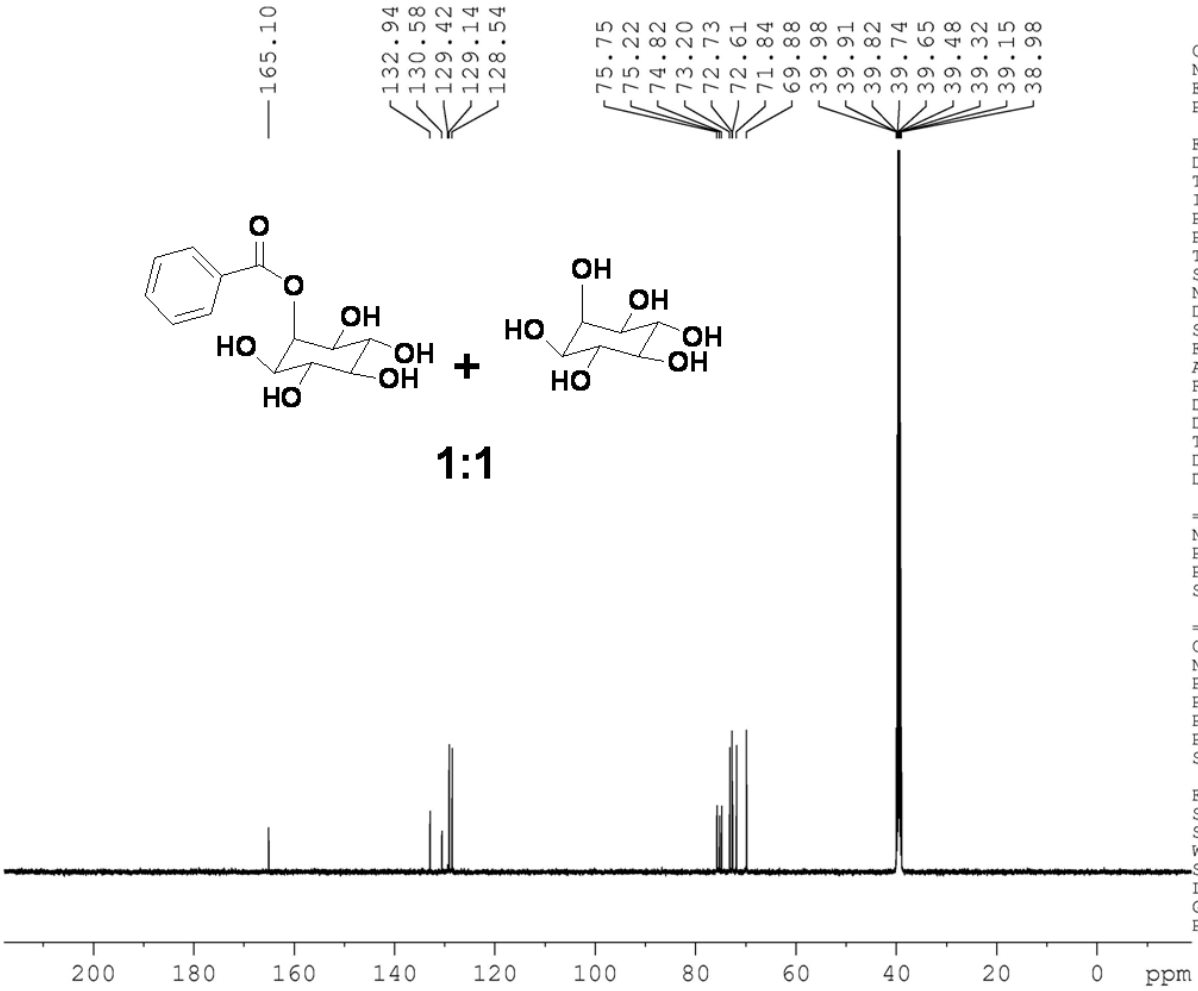
^{1}H NMR of **11** in in CD_3OD (zoom)




```
Current Data Parameters
NAME                  Sagar
EXPNO                   226
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111117
Time                  14.15
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                MeOD
NS                       16
DS                        2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1719923 sec
RG                     171.32
DW                     48.400 usec
DE                      6.50 usec
TE                     298.0 K
D1                1.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.04 usec
PLW1             27.16399956 W
SFO1            500.2330891 MHz

F2 - Processing parameters
SI                        65536
SF               500.2300644 MHz
WDW                        EM
SSB          0
LB                     0.10 Hz
GB           0
PC                     1.00
```

Supplementary Information

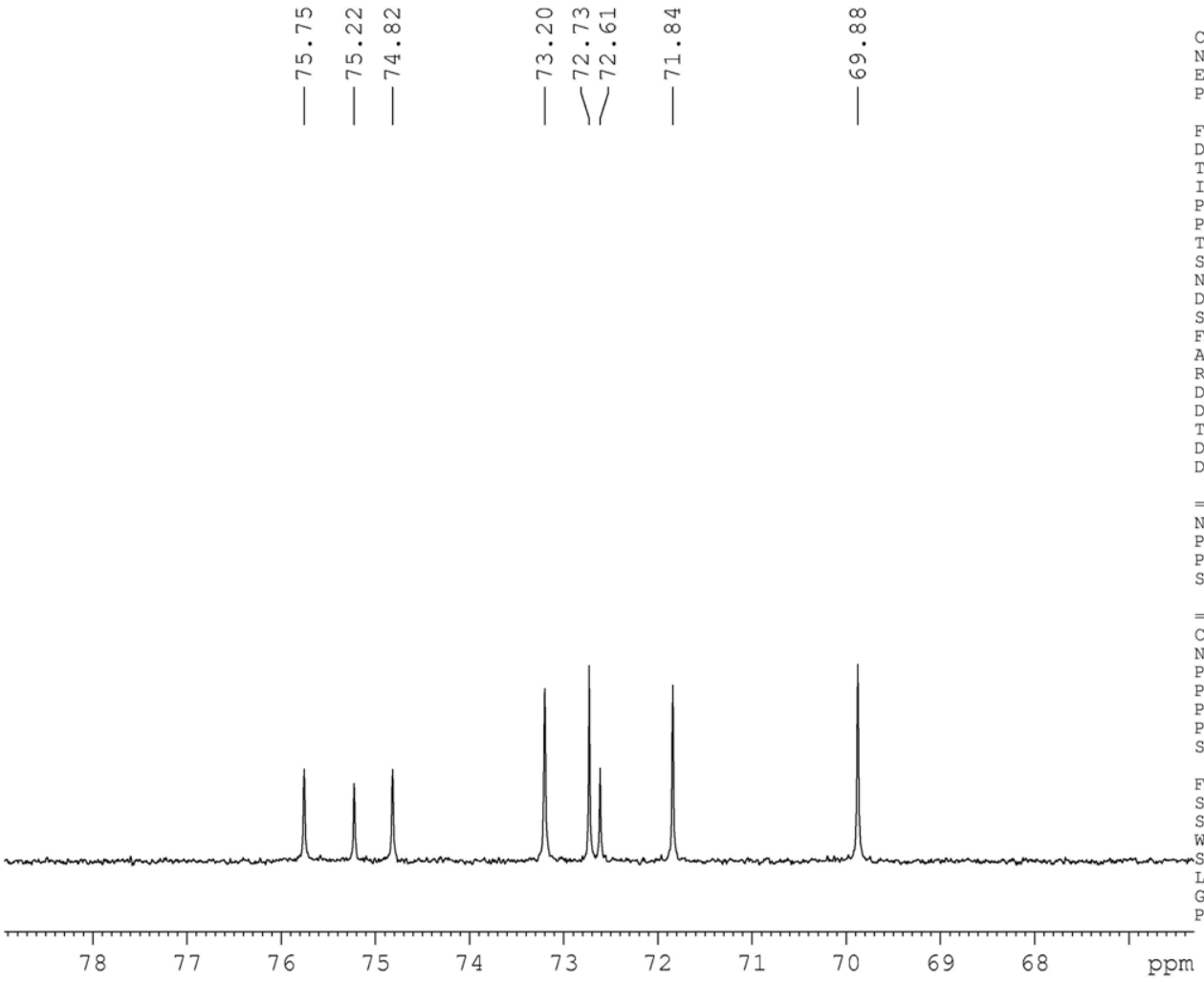
^{13}C NMR of **3a** + ***myo*-inositol (1:1)**




```
Current Data Parameters
NAME                  Amol
EXPNO                  237
PROCNO                   1

F2 - Acquisition Parameters
Date_           20111118
Time                11.30
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT              DMSO
NS                     651
DS                       4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010548 sec
RG                191.08
DW                16.800 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.65 usec
PLW1        120.50000000 W
SFO1         125.7955112 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PLW2         27.16399956 W
PLW12         0.34685999 W
PLW13         0.22199000 W
SFO2         500.2320009 MHz

F2 - Processing parameters
SI                  32768
SF           125.7829969 MHz
WDW                  EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

Supplementary Information

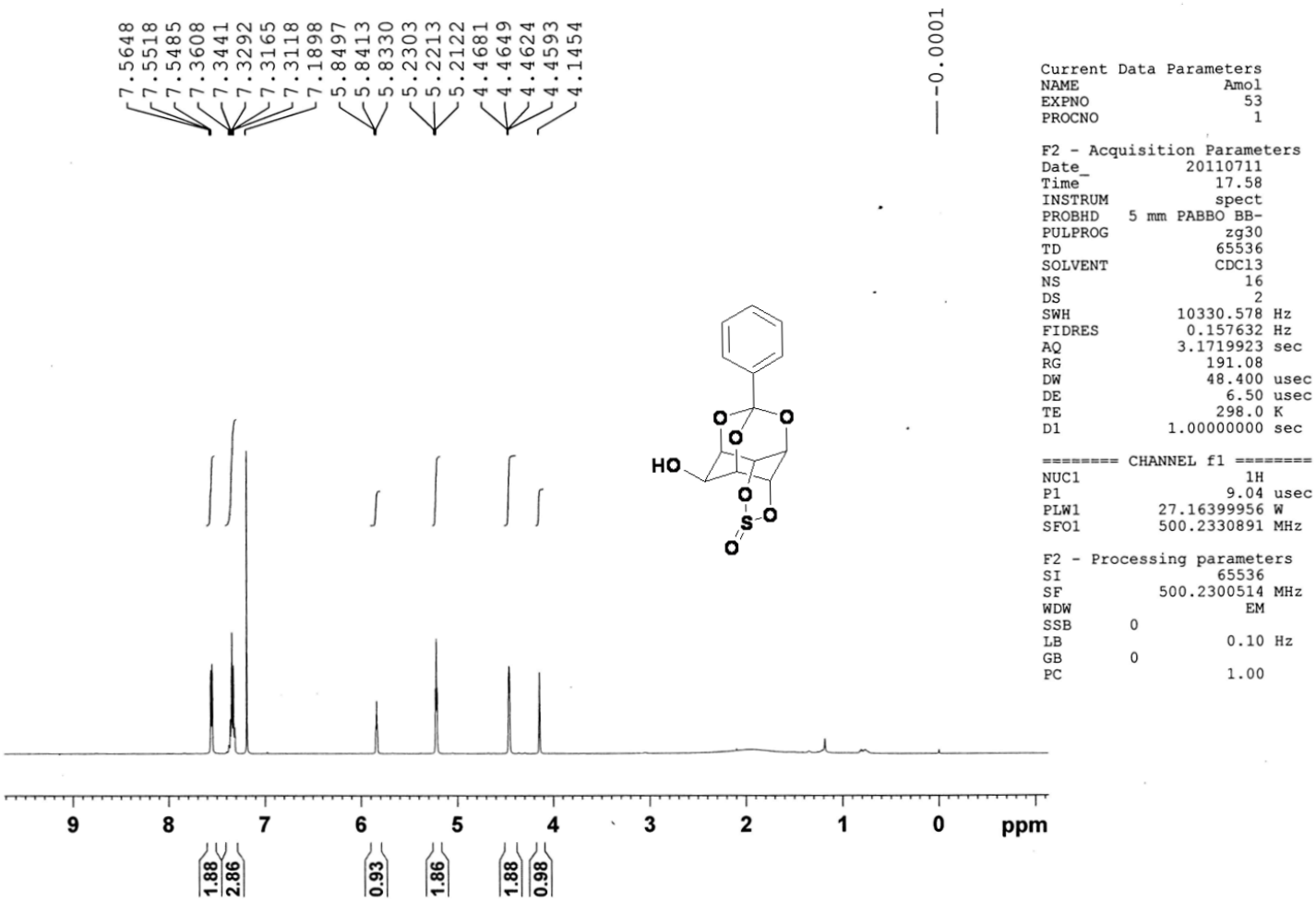
^{13}C NMR of **3a** + ***myo*-inositol (1:1)** (zoom)




```
Current Data Parameters
NAME                  Amol
EXPNO                  237
PROCNO                   1

F2 - Acquisition Parameters
Date_              20111118
Time                  11.30
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT                DMSO
NS                       651
DS                         4
SWH               29761.904 Hz
FIDRES             0.454131 Hz
AQ                1.1010548 sec
RG                   191.08
DW                   16.800 usec
DE                    6.50 usec
TE                   298.0 K
D1                2.00000000 sec
D11               0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                     8.65 usec
PLW1         120.50000000 W
SFO1          125.7955112 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PLW2           27.16399956 W
PLW12           0.34685999 W
PLW13           0.22199000 W
SFO2          500.2320009 MHz

F2 - Processing parameters
SI                     32768
SF            125.7829969 MHz
WDW                       EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

Supplementary Information

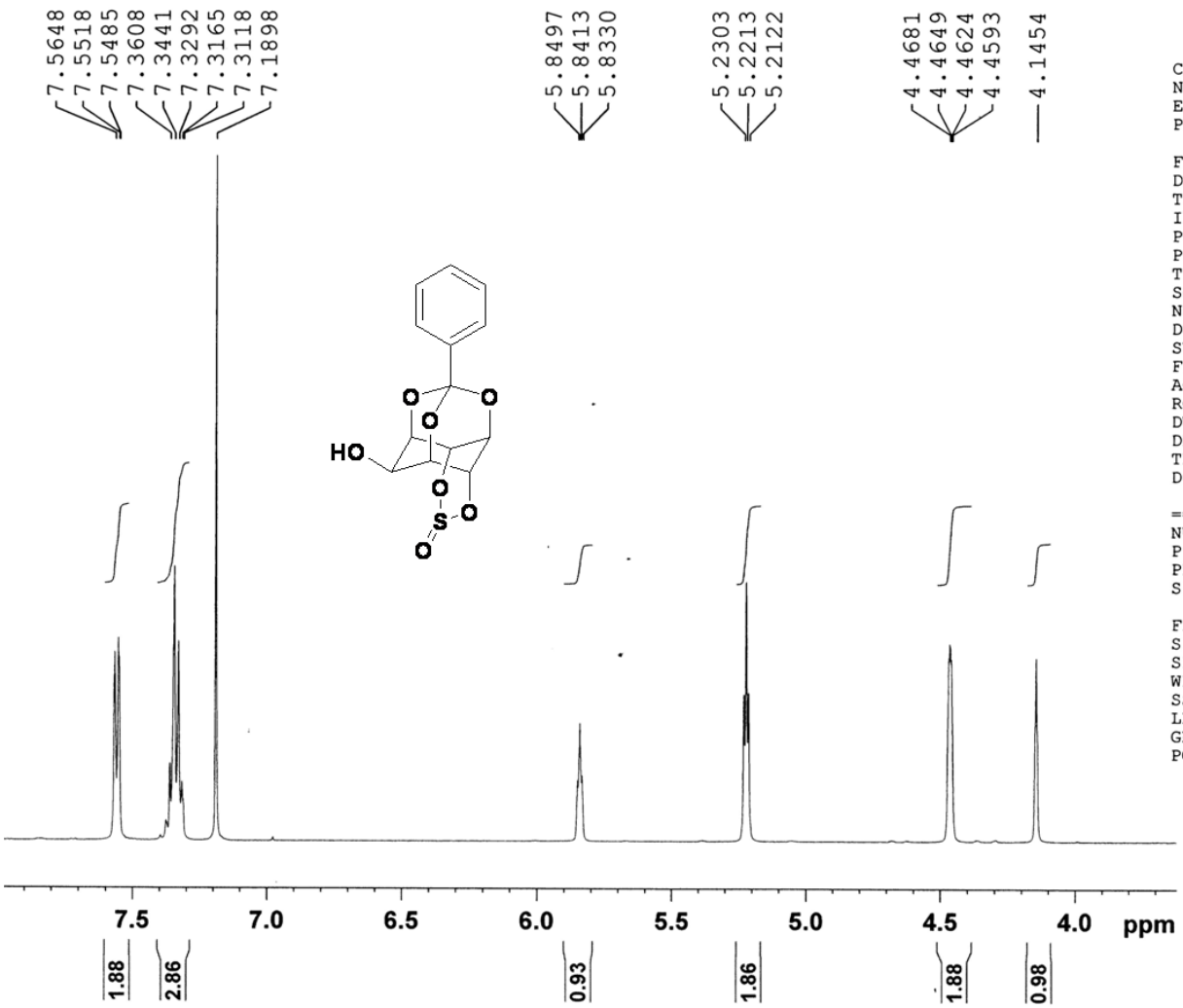
^{1}H NMR of **12**




```
Current Data Parameters
NAME                  Amol
EXPNO                   53
PROCNO                   1

F2 - Acquisition Parameters
Date_              20110711
Time                 17.58
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             10330.578 Hz
FIDRES           0.157632 Hz
AQ               3.1719923 sec
RG                  191.08
DW                  48.400 usec
DE                   6.50 usec
TE                  298.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.04 usec
PLW1          27.16399956 W
SFO1          500.2330891 MHz

F2 - Processing parameters
SI                  65536
SF            500.2300514 MHz
WDW                    EM
SSB       0
LB                   0.10 Hz
GB        0
PC                   1.00
```

Supplementary Information

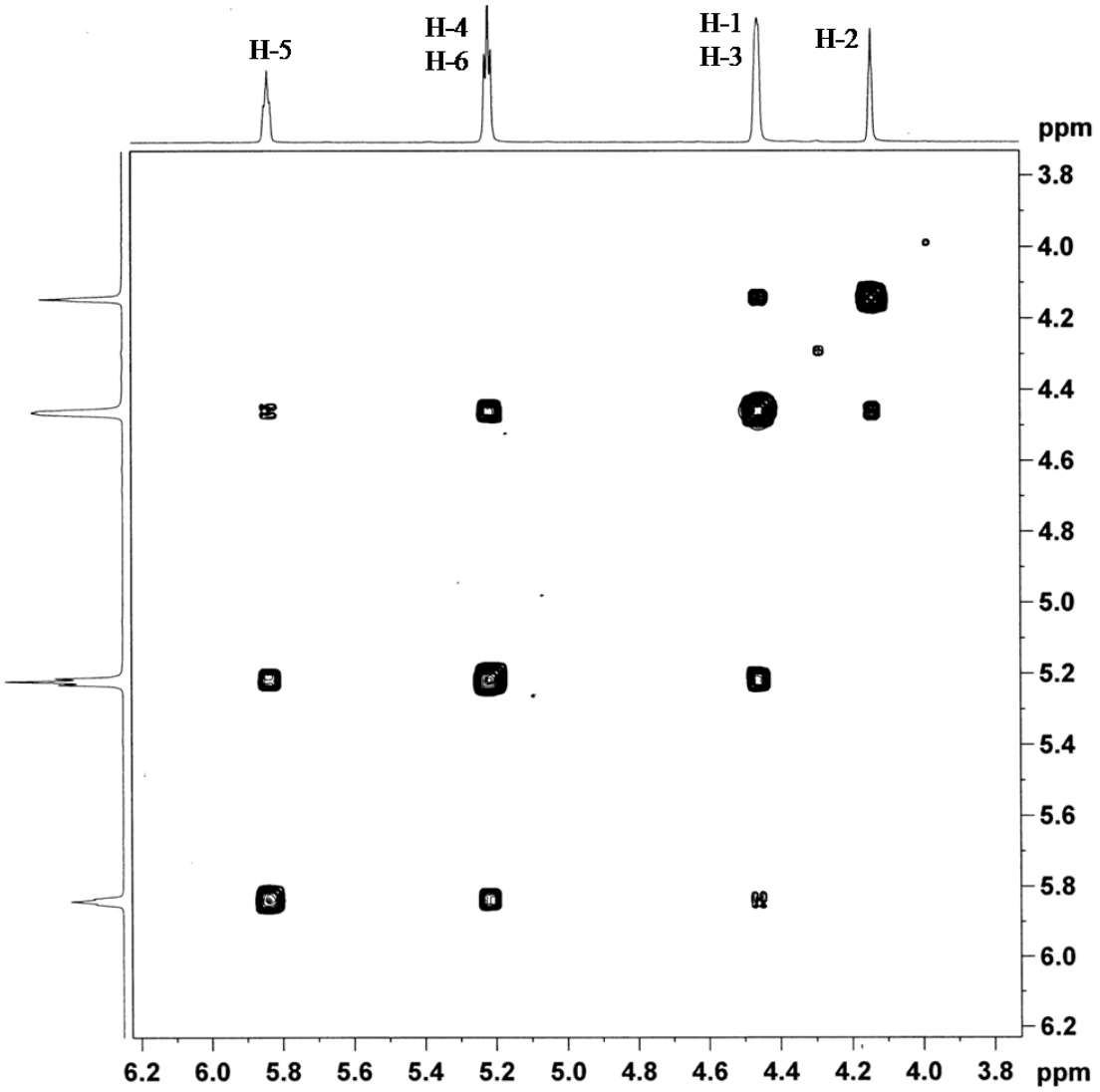
^{1}H NMR of **12** (zoom)




```
Current Data Parameters
NAME                  Amol
EXPNO                   53
PROCNO                   1

F2 - Acquisition Parameters
Date_              20110711
Time                 17.58
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH            10330.578 Hz
FIDRES          0.157632 Hz
AQ             3.1719923 sec
RG                 191.08
DW                 48.400 usec
DE                  6.50 usec
TE                 298.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.04 usec
PLW1         27.16399956 W
SFO1         500.2330891 MHz

F2 - Processing parameters
SI                  65536
SF           500.2300514 MHz
WDW                    EM
SSB       0
LB                  0.10 Hz
GB        0
PC                  1.00
```

Supplementary Information

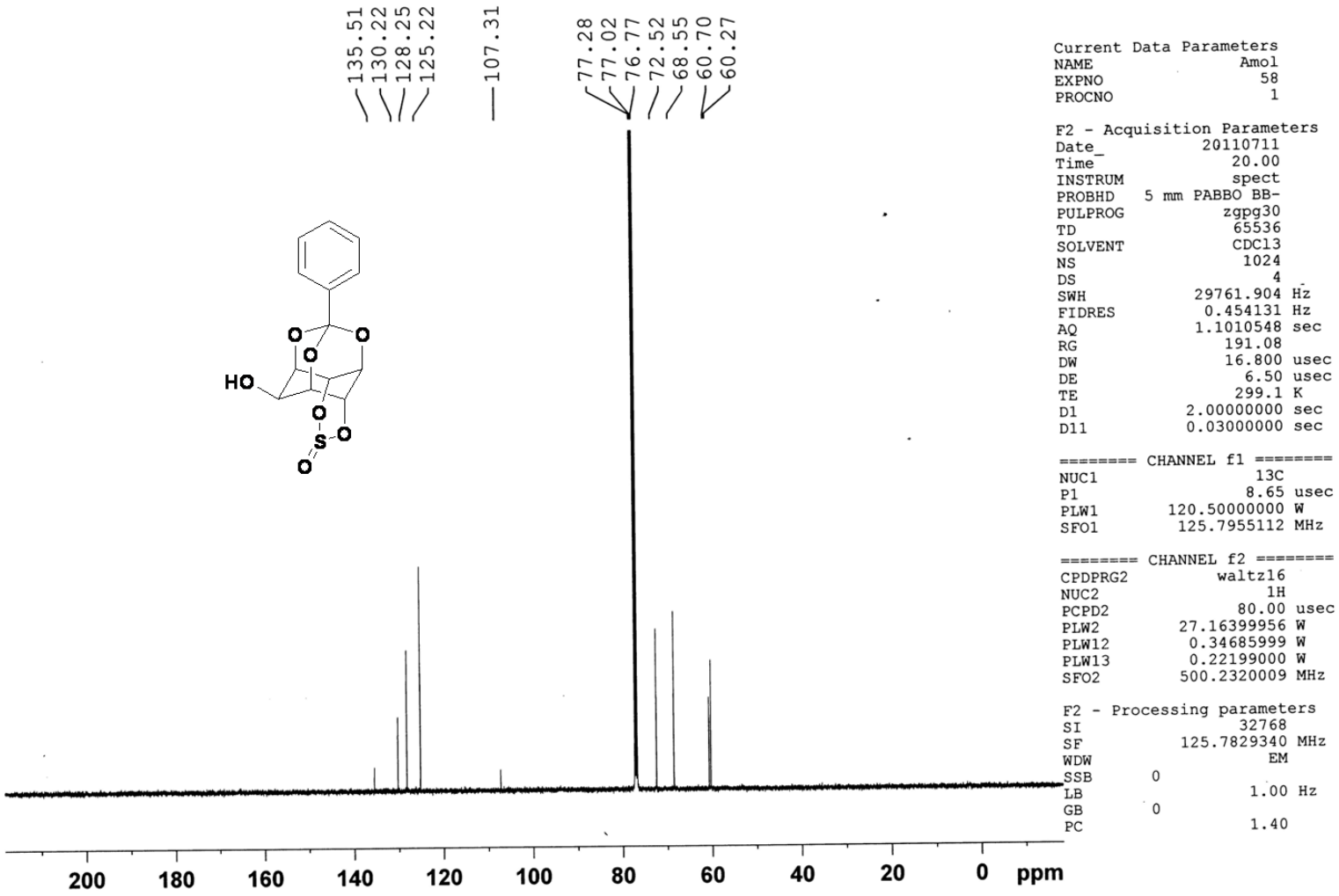
COSY of **12**




```
Current Data Parameters
NAME                  Amol
EXPNO                   55
PROCNO                   1

F2 - Acquisition Parameters
Date_              20110711
Time                  18.30
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG       cosygpppqf
TD                    2048
SOLVENT             CDCl3
NS                       1
DS                       8
SWH                4587.156 Hz
FIDRES             2.239822 Hz
AQ                 0.2232820 sec
RG                    64.12
DW                  109.000 usec
DE                    6.50 usec
TE                   298.0 K
D0                 0.00000300 sec
D1                 1.92995799 sec
D11                0.03000000 sec
D12                0.00002000 sec
D13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00021800 sec

======== CHANNEL f1 ========
NUC1                     1H
P0                    9.04 usec
P1                    9.04 usec
P17                2500.00 usec
PLW1              27.16399956 W
PLW10              3.28390002 W
SFO1             500.2319800 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SMSQ10.100
GPZ1                 10.00 %
P16                1000.00 usec

F1 - Acquisition parameters
TD                      128
SFO1                500.232 MHz
FIDRES             35.837154 Hz
SW                     9.170 ppm
FnMODE                  QF

F2 - Processing parameters
SI                      1024
SF               500.2300515 MHz
WDW                   QSINE
SSB          0
LB           0 Hz
GB           0
PC                     1.40

F1 - Processing parameters
SI                      1024
MC2                     QF
SF               500.2300515 MHz
WDW                   SIN
SSB          0
LB           0 Hz
GB           0
```

Supplementary Information

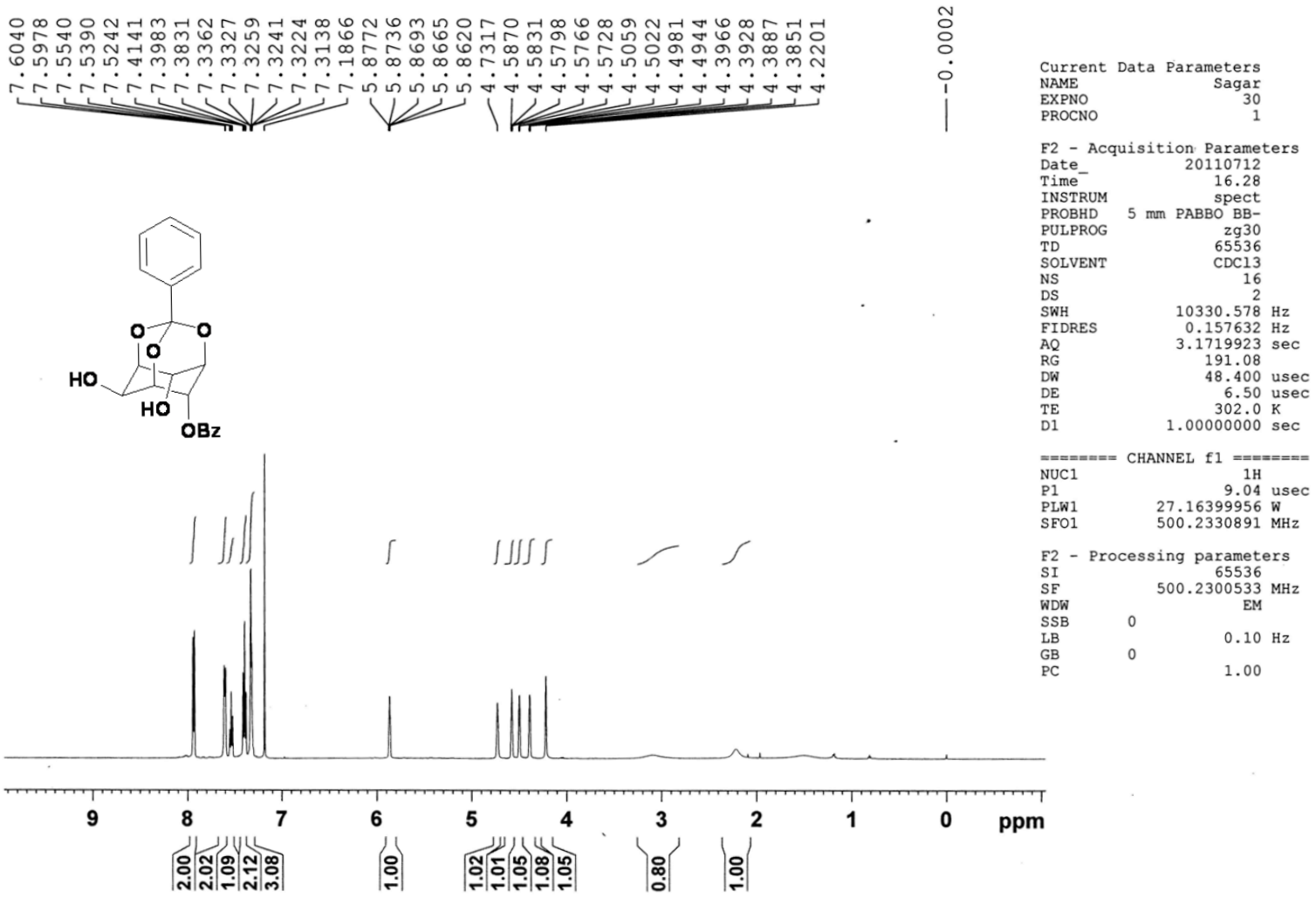
^{13}C NMR of **12**




```
Current Data Parameters
NAME                  Amol
EXPNO                   58
PROCNO                   1

F2 - Acquisition Parameters
Date_              20110711
Time                 20.00
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ                1.1010548 sec
RG                   191.08
DW                   16.800 usec
DE                    6.50 usec
TE                   299.1 K
D1                2.00000000 sec
D11               0.03000000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    8.65 usec
PLW1         120.50000000 W
SFO1          125.7955112 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2                80.00 usec
PLW2           27.16399956 W
PLW12           0.34685999 W
PLW13           0.22199000 W
SFO2           500.2320009 MHz

F2 - Processing parameters
SI                    32768
SF           125.7829340 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

Supplementary Information

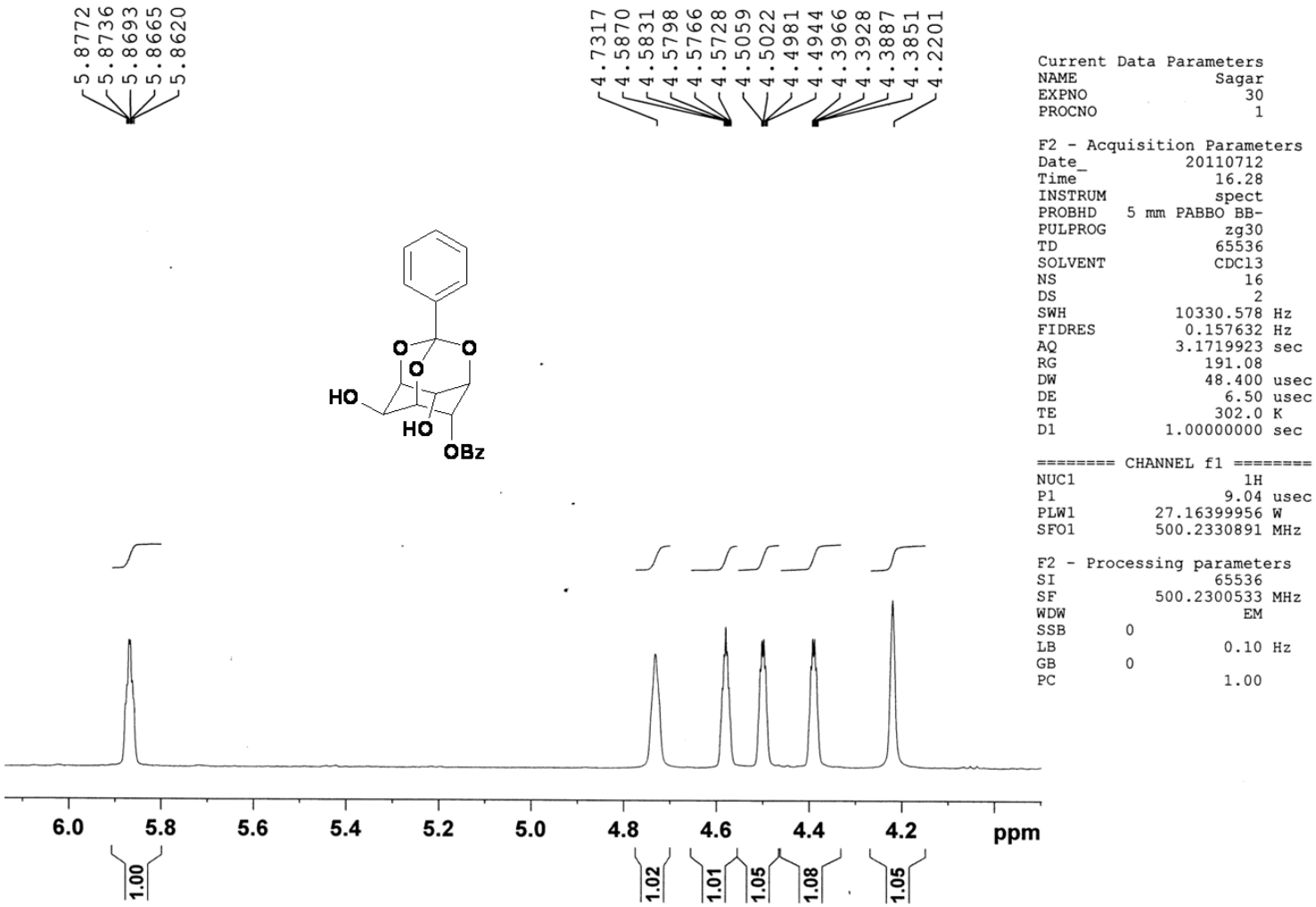
^{1}H NMR of **10**




```
Current Data Parameters
NAME                  Sagar
EXPNO                    30
PROCNO                    1

F2 - Acquisition Parameters
Date_              20110712
Time                  16.28
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                3.1719923 sec
RG                   191.08
DW                   48.400 usec
DE                    6.50 usec
TE                   302.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    9.04 usec
PLW1           27.16399956 W
SFO1          500.2330891 MHz

F2 - Processing parameters
SI                    65536
SF              500.2300533 MHz
WDW                      EM
SSB       0
LB                    0.10 Hz
GB        0
PC                    1.00
```

Supplementary Information

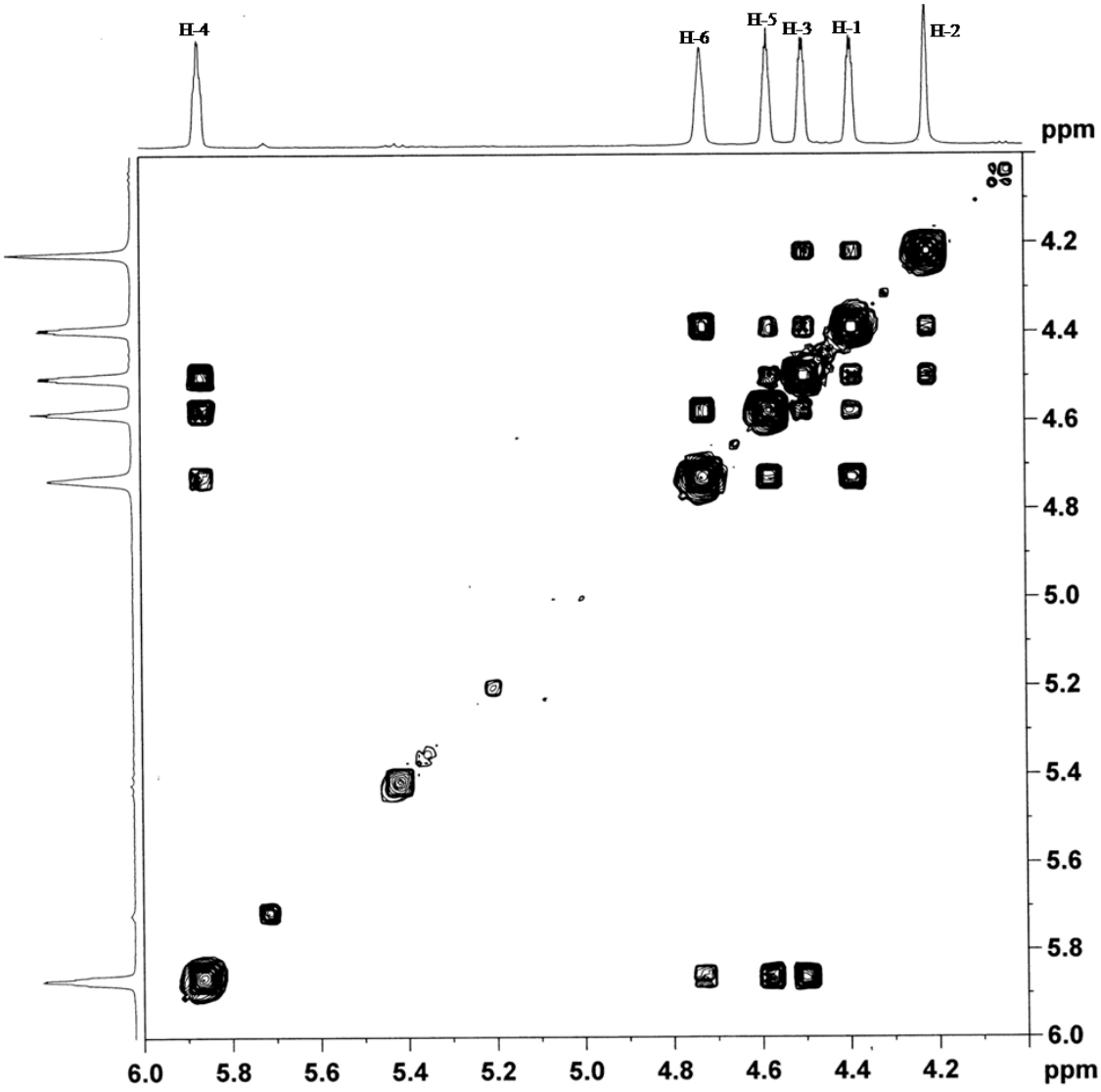
^{1}H NMR of **10** (zoom)




```
Current Data Parameters
NAME                  Sagar
EXPNO                    30
PROCNO                    1

F2 - Acquisition Parameters
Date_              20110712
Time                 16.28
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH             10330.578 Hz
FIDRES           0.157632 Hz
AQ               3.1719923 sec
RG                  191.08
DW                  48.400 usec
DE                    6.50 usec
TE                  302.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.04 usec
PLW1          27.16399956 W
SFO1          500.2330891 MHz

F2 - Processing parameters
SI                   65536
SF            500.2300533 MHz
WDW                    EM
SSB       0
LB                   0.10 Hz
GB        0
PC                   1.00
```

Supplementary Information

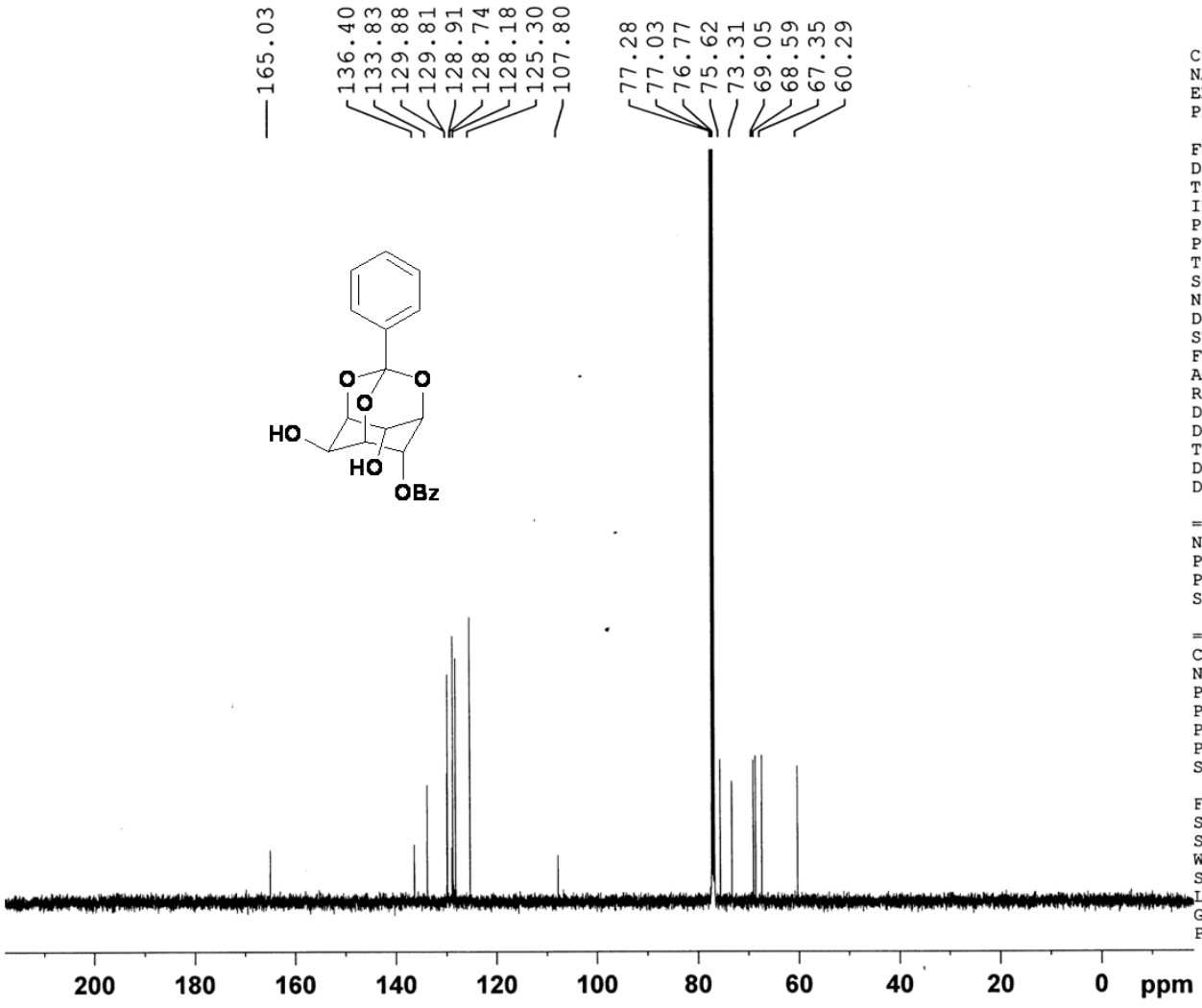
COSY of **10**




```
Current Data Parameters
NAME                   Sagar
EXPNO                     33
PROCNO                     1

F2 - Acquisition Parameters
Date_               20110712
Time                   16.58
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG          cosygpppqf
TD                      2048
SOLVENT                CDCl3
NS                         1
DS                         8
SWH                 4273.504 Hz
FIDRES              2.086672 Hz
AQ                  0.2396660 sec
RG                     64.12
DW                   117.000 usec
DE                     6.50 usec
TE                     298.7 K
D0                0.00000300 sec
D1                1.91357398 sec
D11               0.03000000 sec
D12               0.00002000 sec
D13               0.00000400 sec
D16               0.00020000 sec
IN0               0.00023400 sec

======== CHANNEL f1 ========
NUC1                       1H
P0                       9.04 usec
P1                       9.04 usec
P17                   2500.00 usec
PLW1             27.16399956 W
PLW10             3.28390002 W
SFO1            500.2322881 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SMSQ10.100
GPZ1                 10.00 %
P16                1000.00 usec

F1 - Acquisition parameters
TD                       128
SFO1              500.2323 MHz
FIDRES           33.386753 Hz
SW                    8.543 ppm
FnMODE                   QF

F2 - Processing parameters
SI                      1024
SF             500.2300527 MHz
WDW                    QSINE
SSB          0
LB           0 Hz
GB           0
PC                     1.40

F1 - Processing parameters
SI                      1024
MC2                       QF
SF             500.2300527 MHz
WDW                     SIN
SSB          0
LB           0 Hz
GB           0
```

Supplementary Information

^{13}C NMR of **10**




```
Current Data Parameters
NAME                  Sagar
EXPNO                    35
PROCNO                    1

F2 - Acquisition Parameters
Date_              20110712
Time                  17.08
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG               zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      502
DS                        4
SWH               29761.904 Hz
FIDRES             0.454131 Hz
AQ                 1.1010548 sec
RG                    191.08
DW                    16.800 usec
DE                     6.50 usec
TE                    298.7 K
D1                 2.00000000 sec
D11                0.03000000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                      8.65 usec
PLW1          120.50000000 W
SFO1           125.7955112 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                 80.00 usec
PLW2           27.16399956 W
PLW12           0.34685999 W
PLW13           0.22199000 W
SFO2           500.2320009 MHz

F2 - Processing parameters
SI                    32768
SF            125.7829340 MHz
WDW                      EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```