Electronic Supplementary Material (ESI) for ChemComm.

Supplementary Information For

Transition from Low Molecular Weight Non-gelating Oligo(amide-triazoles)s to a Restorable, Halide-Responsive Poly(amide-triazole) Supramolecular Gel

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Contents

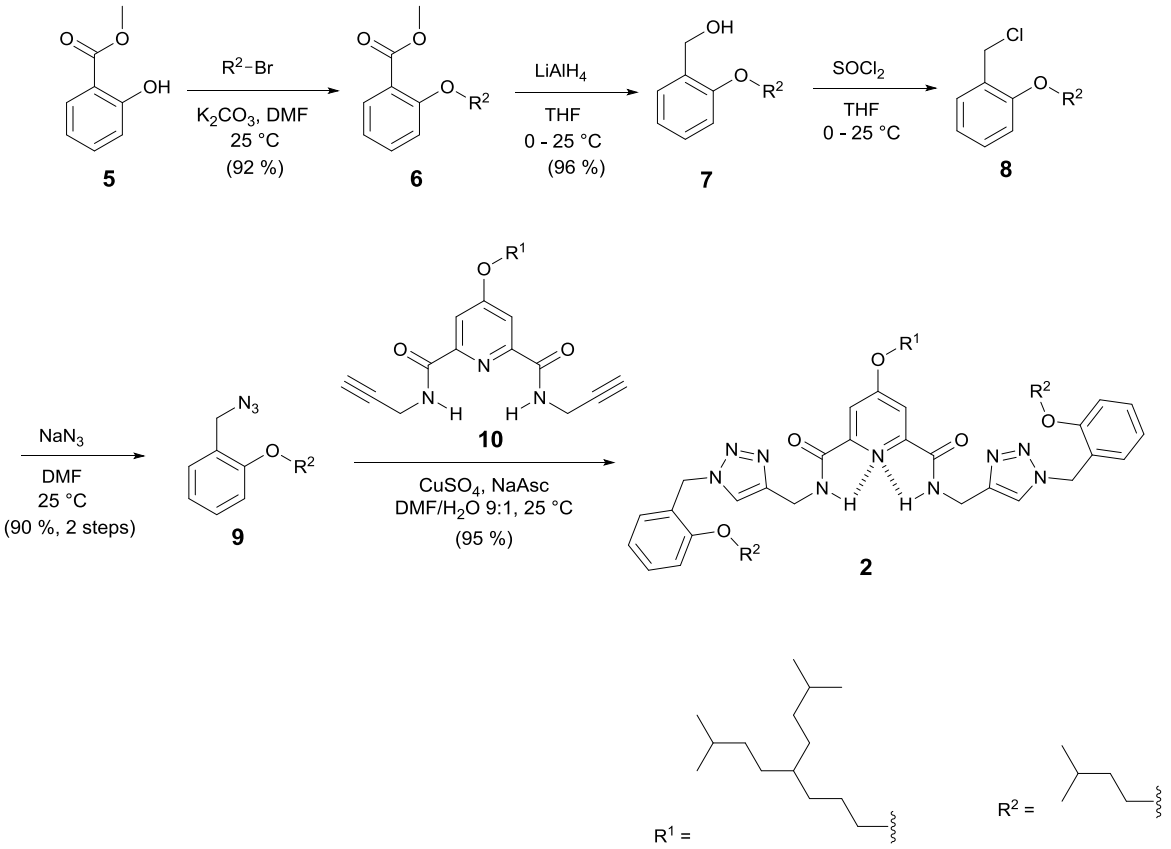
1. Synthesis

1.1 General

All reagents were purchased from commercial suppliers and used without further purification unless otherwise specified. Dichloromethane was freshly distilled from calcium hydride under nitrogen. Tetrahydrofuran (THF) was freshly distilled from sodium/benzophenone ketyl under nitrogen prior to use. *N*,*N*-dimethylformamide (DMF) was freshly distilled from magnesium sulphate under vacuum prior to use. For all copper catalyzed azide-alkyne cycloaddition (CuAAC) reactions, the solvents were used directly without any purification prior to use. All reactions were carried out at 298 K under nitrogen unless otherwise specified. The progress of reaction were monitored by thin layer chromatography (TLC) performed on Merck pre-coated silica gel $60F_{254}$ plates, and the compounds were visualized under UV and/or using a spray solution of 5% (wt/v) dodecamolybdophosphoric acid in ethanol followed by heating. Flash column chromatography was carried out on columns of Merck Kwiselgel 60 (230–400 mesh).

^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were obtained on a Brüker Avance DPX 400 spectrometer. Unless otherwise specified, all spectra were obtained in $CDCl_3$ at 298 K. The deuterated solvent residual signals were used as the internal standard. The Chemical shift (δ) values were reported as parts per million (ppm) in δ scale. The coupling constants (*J*) were reported in hertz. Melting points were measured on an Electrothermal® 9100 digital melting point apparatus. High resolution electrospray ionization (ESI) mass spectrometry were performed on a Finnigan MAT 95XL Mass Spectrometer using electrospray ionization (ESI) technique. The reported mass to charge ratio (*m/z*) were mono-isotopic.

*1.2 Synthesis of Oligomer **2***



Scheme S1. Synthesis of oligomer **2**.

6: 1-bromo-3-methylbutane (5.0 mL, 41.7 mmol) and K_2CO_3 (6.80 g, 49.2 mmol) were added to a stirred solution of methyl salicylate **5** (5.00 g, 32.9 mmol) in DMF (40 mL). The reaction mixture was allowed to stir at 25 °C for 24 h. Water (80 mL) and Et_2O (80 mL) was added to quench the reaction. The organic layer was separated and the aqueous layer was extracted with Et_2O (2 × 80 mL). The combined organic extracts were washed with saturated NaCl, dried ($MgSO_4$) and concentrated to give a pale yellow liquid. It was then purified by flash column chromatography (hexane/EtOAc = 10:1 gradient to 3:1) to afford the target compound **6** (6.73 g, 30.3 mmol, 92 %) as a colorless oil. R_f: 0.23 (hexane/EtOAc = 9:1). ^{1}H NMR: δ 0.96 (6 H, d, J = 6.8, CH_3), 1.74 (2 H, q, J = 6.4, CH_2CH_2CH), 1.81–1.95 (1 H, m, $CHMe_2$), 3.87 (3 H, s, $COOCH_3$), 4.05 (2 H, t, J = 6.4, OCH_2), 6.88–7.00 (2 H, m, ArH), 7.39–7.46 (1 H, m, ArH), 7.74–7.79 (1 H, m, ArH).

^{13}C NMR: δ 22.5, 24.9, 37.8, 51.7, 67.2, 113.0, 119.9, 120.4, 131.5, 133.2, 158.5, 166.8. *m/z* (ESI) 245 (M + Na^+, 100 %). HRMS (ESI) Calcd for $C_{13}H_{18}O_3$ + Na^+: 245.1148, found 245.1150.

7: A solution of compound **6** (2.01 g, 9.04 mmol) in THF (10 mL) was added dropwise to a stirring solution of lithium aluminum hydride (LAH) (350 mg, 9.4 mmol) in THF (10 mL) at 0 °C. The reaction temperature was raised to 25 °C after 30 min. Upon stirring for 4 h, the reaction mixture was poured into water (10 mL) carefully. Aqueous HCl (30 mL 1.2 M) was added to neutralize the mixture and the organic layer was separated. The aqueous layer was then extracted with Et_2O (2 × 20 mL). The combined organic layers were washed with saturated NaCl, dried ($MgSO_4$), and concentrated to give the alcohol **7** (1.67 g, 8.67 mmol, 96 %) as a colorless oil. The oil was used in the next step without further purification. R_f: 0.21 (hexane/EtOAc = 2:1). ^{1}H NMR: δ 0.98 (6 H, d, *J* = 6.4, CH_3), 1.72 (2 H, q, *J* = 6.4, CH_2CH_2CH), 1.77–1.90 (2 H, m, $CHMe_2$), 4.06 (2 H, t, *J* = 6.8, OCH_2), 4.69 (2 H, s, $ArCH_2OH$), 6.85–6.96 (2 H, m, ArH), 7.20–7.30 (2 H, m, ArH). ^{13}C NMR: δ 22.6, 25.2, 38.0, 62.0, 66.3, 110.9, 120.5, 128.5, 128.8, 129.3, 156.9. *m/z* (ESI) 194 (M^+, 20 %). HRMS (ESI) Calcd for $C_{12}H_{18}O_2^+$: 194.1301, found 194.1302.

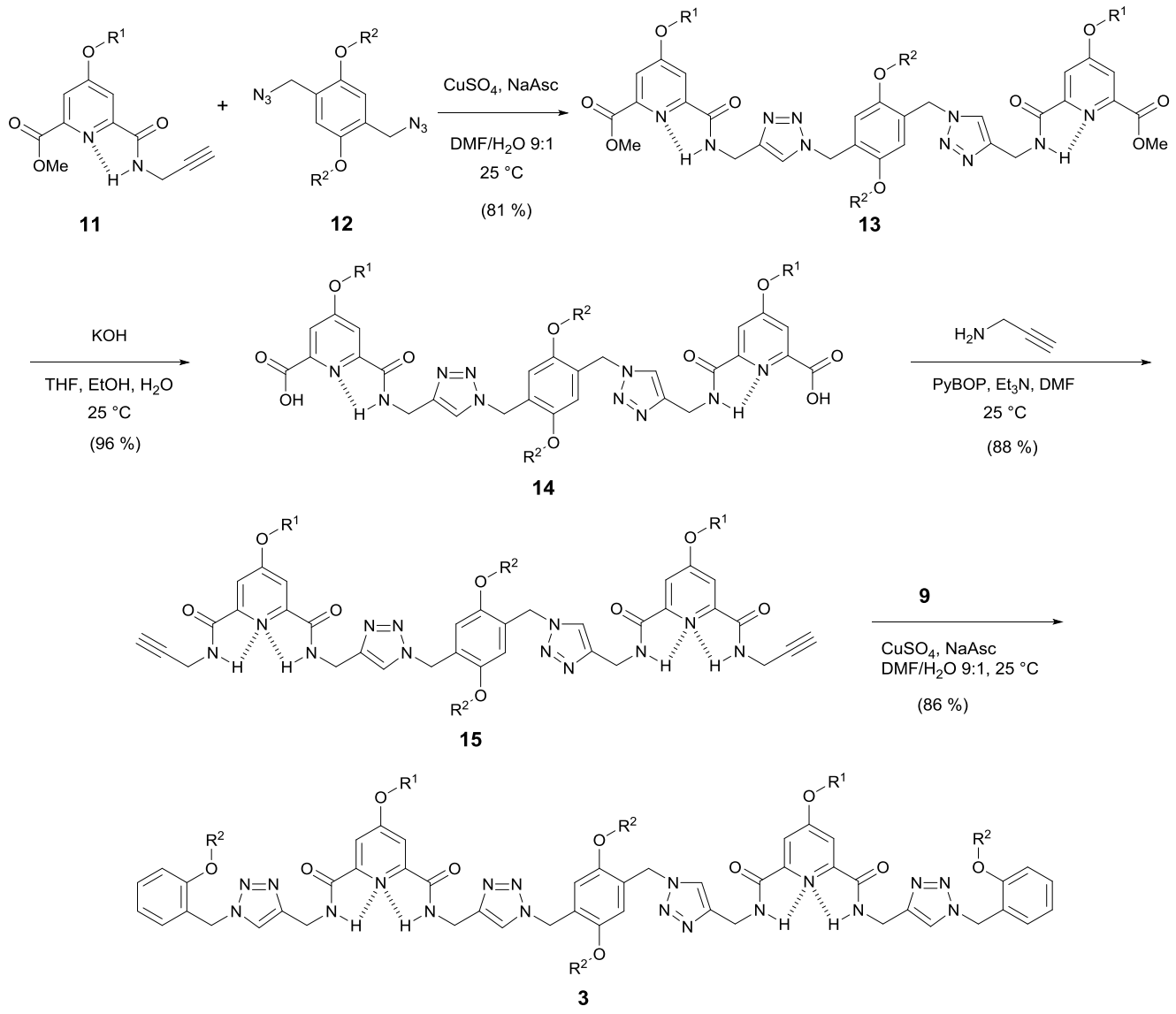
9: A solution of alcohol **7** (1.00 g, 5.15 mmol) in THF (5 mL) was added dropwise to a solution of $SOCl_2$ (5 mL) in THF (10 mL) at 0 °C. The reaction mixture was stirred for 2 h and then quenched by pouring into iced water (20 mL). The mixture was then extracted with Et_2O (3 × 10 mL) and the combined organic extracts were washed with saturated Na_2CO_3, saturated NaCl, dried ($MgSO_4$), and concentrated to give the chloride **8** as a pale yellow oil. R_f: 0.68 (hexane/EtOAc = 9:1) **8** was used directly without further purification. Sodium azide (1.40 g, 21.5 mmol) was added to a stirring solution of **8** (0.67 g, 10.0 mmol) in DMF (10 mL). The reaction mixture was stirred at 25 °C for 48 h. Water (20 mL) was added to quench the reaction. The solution mixture was then extracted with Et_2O (3 × 15 mL). The combined organic extracts were washed with saturated NaCl, dried ($MgSO_4$) and then concentrated to give a yellow liquid. The liquid was purified by flash column chromatography (hexane/EtOAc = 15:1 gradient to 9:1) to afford the desired compound **9** (2.08 g, 5.77 mmol, 91 %) as a colorless liquid. R_f: 0.73 (hexane/EtOAc = 9:1); ^{1}H NMR: δ 0.98

(6 H, d, J = 6.4, CH_3), 1.72 (2 H, q, J = 6.4, CH_2CH_2CH), 1.80–1.92 (1 H, m, $CHMe_2$), 4.03 (2 H, t, J = 6.8, OCH_2), 4.36 (2 H, s, $ArCH_2N_3$), 6.87–6.97 (2 H, m, ArH), 7.22–7.33 (2 H, m, ArH). ^{13}C NMR: δ 22.7, 25.2, 38.0, 50.3, 66.5, 111.3, 120.4, 124.0, 129.8, 130.1, 157.2. *m/z* (ESI) 219 (M^+, 35 %). HRMS (ESI) Calcd for $C_{12}H_{17}N_3O^+$: 219.1366, found 219.1370.

2: An aqueous $CuSO_4$ (0.20 mL, 0.056 M) solution was added to a stirred solution of **10**[1] (120 mg, 0.26 mmol), **9** (125 mg, 0.58 mmol) and sodium ascorbate (10 mg, 0.05 mmol) in DMF (1.8 mL) at 25 °C. After 12 h, warm DMF was added to the solution mixture until a clear solution was obtained, and then the mixture was poured into water (15 mL). The solid was then filtered, washed with a mixture of Et_2O/EtOH and then purified by column chromatography (EtOAc) to afford **2** (224 mg, 0.25 mmol, 95 %) as a white solid. M.p. 42–45 °C; R_f: 0.53 (EtOAc); ^{1}H NMR: ([D_8]-THF) δ 0.87–0.95 (24 H, m, CCH_3), 1.14–1.90 (21 H, m), 4.01 (4 H, t, J = 6.8, OCH_2), 4.16 (2 H, t, J = 6.8, OCH_2), 4.54 (4 H, d, J = 6.0, NCH_2Triaz), 5.47 (4 H, s, $ArCH_2$Triaz), 6.83–6.88 (2 H, m, ArH), 6.93–6.97 (2 H, m, ArH), 7.09–7.14 (2 H, m, ArH), 7.22–7.27 (2 H, m, ArH), 7.66 (2 H, s, TriazH), 7.76 (2 H, s, ArH), 9.29 (2 H, t, J = 6.0, NH). ^{13}C NMR: ([D_8]-THF) δ 22.7, 22.9, 26.8, 29.2, 30.4, 31.9, 35.5, 36.6, 38.4, 38.6, 49.3, 69.7, 111.0, 112.1, 121.0, 123.2, 124.7, 130.4, 130.6, 145.9, 152.0, 157.5, 163.9, 168.3. *m/z* (ESI) 915 ($M + Na^+$, 100 %). HRMS (ESI) Calcd for $C_{51}H_{73}N_9O_5 + Na^+$: 914.5627, found 914.5632.

[1] S.-L. Yim, H.-F. Chow, M.-C. Chan, C.-M. Che and K.-H. Low, *Chem. Eur. J.*, 2013, **19**, 2478.

*1.3 Synthesis of Oligomer **3***



Scheme S2. Synthesis of oligomer **3**.

12: Aqueous $CuSO_4$ (0.40 mL, 0.056 M) was added to a stirred solution of **11**[1] (273 mg, 0.63 mmol), **12**[1] (108 mg, 0.30 mmol) and sodium ascorbate (20 mg, 0.1 mmol) in DMF (3.6 mL) at 25 °C. After 12 h, water (10 mL) and ethyl acetate (10 mL) were added to quench the reaction. The organic layer was separated and the aqueous layer was extracted with ethyl acetate (2 × 10 mL). The combined organic extracts were washed with saturated NaCl, dried (Na_2SO_4) and concentrated to give a yellow solid. The solid was then purified by

flash column chromatography (hexane/EtOAc = 2:1 gradient to 1:2) to obtain **13** (627 mg, 0.51 mmol, 81 %) as a white solid. M.p. 55–50 °C; R_f: 0.50 (Hexane/EtOAc = 1:2); ^{1}H NMR: δ 0.76–0.90 (36 H, m, CCH_3), 1.05–1.80 (36 H, m), 3.85 (4 H, t, *J* = 6.4, OCH_2), 3.90 (6 H, s, $COOCH_3$), 4.05 (4 H, t, *J* = 6.4, OCH_2), 4.66 (4 H, d, *J* = 6.0, NCH_2Triaz), 5.41 (4 H, s, $ArCH_2$Triaz), 6.73 (2 H, s, ArH), 7.52 (2 H, s, TriazH), 7.64 (2 H, d, *J* = 2.4, ArH), 7.77 (2 H, d, *J* = 2.4, ArH), 8.58 (2 H, t, *J* = 6, NH). ^{13}C NMR: δ 22.5, 22.7, 25.0, 25.9, 28.4, 29.6, 31.0, 34.9, 35.8, 37.5, 37.9, 48.9, 52.8, 67.2, 69.4, 110.9, 113.9, 114.5, 122.6, 124.2, 144.6, 148.1, 150.4, 151.6, 163.6, 165.0, 167.4. *m/z* (ESI) 1244 (M + Na^+, 100 %). HRMS (ESI) Calcd for $C_{68}H_{104}N_{10}O_{10}$ + Na^+: 1243.7829, found 1243.7825.

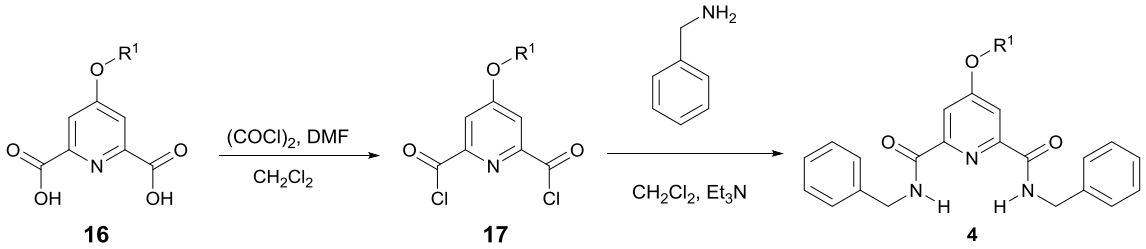
14: An aqueous KOH (0.5 mL, 2.5 M) solution was added to a stirred solution of compound **13** (281 mg, 0.23 mmol) in ethanol and THF (~1:1, 4 mL) at 25 °C. After 12 h, the mixture was acidified with aqueous HCl (10 mL, 0.3 M). The organic layer was obtained, and the aqueous layer was further extracted with EtOAc (2 × 10 mL). The combined organic extracts were washed with saturated NaCl, dried (Na_2SO_4), and concentrated to obtain **14** (262 mg, 0.22 mmol, 96 %) as a white solid. M.p. 106–112 °C; R_f: 0.31 (CH_2Cl_2/MeOH = 9:1); ^{1}H NMR: ([D_6]-DMSO, COOH not observed) δ 0.79 (12 H, d, *J* = 6.8, CCH_3), 0.83 (24 H, d, *J* = 6.4, CCH_3), 1.07–1.77 (36 H, m), 3.84 (4 H, t, *J* = 6.4, OCH_2), 4.20 (4 H, t, *J* = 6.4, OCH_2), 4.55 (4 H, d, *J* = 6.0, NCH_2Triaz), 5.44 (4 H, s, $ArCH_2$Triaz), 6.93 (2 H, s, ArH), 7.62–7.70 (4 H, m, ArH), 7.81 (2 H, s, TriazH), 9.55–9.63 (2 H, m, NH). *m/z* (ESI) 1216 (M + Na^+, 100 %). The solubility of the compound was too poor to obtain the ^{13}C NMR spectrum even in [D_6]-DMSO. HRMS (ESI) Calcd for $C_{66}H_{100}N_{10}O_{10}$ + Na^+: 1215.7516, found 1215.7514.

15: Benzotriazol-1-yl-oxytripyrrolidinophosphonium hexafluorophosphate (PyBOP) (180 mg, 0.37 mmol) was added to a stirred solution of **14** (200 mg, 0.17 mmol) and propargylamine (0.1 mL, 1.56 mmol) in DMSO (5 mL). Triethylamine (0.1 mL) was then added to the solution mixture. The reaction was allowed to stir for 24 h at 25°C. EtOAc (15 mL) and H_2O (15 mL) was then added to quench the reaction. The organic layer was separated and the aqueous layer was extracted with EtOAc (2 × 15 mL). The combined organic

extracts were washed with saturated NaCl, dried (Na_2SO_4), concentrated and purified by flash column chromatography (hexane/EtOAc = 1:2 gradient to 1:4) to afford the dialkyne **15** (189 mg, 0.15 mmol, 88 %) as a white solid. M.p. 88–93 °C; R_f. 0.38 (hexane/EtOAc = 1:2). ^{1}H NMR: δ 0.82–0.94 (36 H, m, CH_3), 1.09–1.84 (36 H, m), 2.21–2.26 (2 H, m, C≡CH), 3.90 (4 H, t, J = 6.4, OCH_2), 4.09 (4 H, t, J = 6.4, OCH_2), 4.12–4.22 (4 H, m, $CH_2C{\equiv}C$), 4.65 (4 H, d, J = 6, NCH_2Triaz), 5.46 (4 H, s, NCH_2Triaz), 6.81 (2 H, s, ArH), 7.62 (2 H, s, TriazH), 7.72–7.86 (4 H, m, ArH), 8.11 – 8.20 (2 H, m, NH), 8.60 – 8.70 (2 H, m, NH). ^{13}C NMR: δ 22.6, 22.8, 25.1, 26.1, 28.4, 28.9, 29.7, 31.1, 34.8, 35.9, 37.6, 37.9, 49.2, 67.4, 69.5, 71.2, 77.5, 79.6, 111.2, 111.4, 114.2, 123.1, 124.2, 145.3, 150.4, 150.51, 150.54, 163.4, 163.9, 167.9. *m/z* (ESI) 1290 (M + Na^+, 100 %). HRMS (ESI) Calcd for $C_{72}H_{106}N_{12}O_8$ + Na^+: 1289.8149, found 1289.8148.

3: Aqueous $CuSO_4$ (0.20 mL, 0.03 M) was added to a stirred solution of **15** (180 mg, 0.14 mmol), **6** (80 mg, 0.36 mmol) and sodium ascorbate (10 mg, 0.05 mmol) in DMF (1.8 mL) at 25 °C. After 12 h, water (10 mL) and ethyl acetate (10 mL) were added to quench the reaction. The organic layer was separated and the aqueous layer was extracted with ethyl acetate (2 × 10 mL). The combined organic extracts were washed with saturated NaCl, dried (Na_2SO_4) and concentrated to give a colorless liquid. It was then purified by flash column chromatography (CH_2Cl_2/EtOAc = 2:1 gradient to 1:3) to obtain **3** (213 mg, 0.12 mmol, 86 %) as a white solid. M.p. 108–115 °C; R_f: 0.10 (CH_2Cl_2/EtOAc = 2:1); ^{1}H NMR: ([D_8]-THF) δ 0.68–1.10 (48 H, m, CCH_3), 1.15–1.90 (42 H, m), 3.89 (4 H, t, J = 6.4, OCH_2), 4.01 (4 H, t, J = 6.4, OCH_2), 4.15 (4 H, t, J = 6.4, OCH_2), 4.48–4.58 (8 H, m, NCH_2Triaz), 5.39–5.50 (8 H, m, $ArCH_2$Triaz), 6.80–6.91 (4 H, m, ArH), 6.92–7.00 (2 H, m, ArH), 7.10–7.18 (2 H, m, ArH), 7.20–7.28 (2 H, m, ArH), 7.68 (2 H, s, TriazH), 7.71 (2 H, s, TriazH), 7.72–7.80 (4 H, m, ArH), 9.28–9.40 (4 H, m, NH). ^{13}C NMR: ([D_8]-THF) δ 22.7, 22.9, 25.65, 25.74, 25.8, 26.8, 29.2, 30.4, 31.9, 35.5, 36.6, 38.5, 38.63, 38.67, 48.8, 49.1, 49.3, 69.8, 111.0, 112.1, 113.7, 114.7, 121.0, 123.3, 124.7, 125.7, 126.7, 130.0, 130.4, 130.6, 145.9, 146.0, 151.3, 152.0, 156.3, 157.5, 163.9, 168.4. *m/z* (ESI) 1729 (M + Na^+, 55 %). HRMS (ESI) Calcd for $C_{96}H_{140}N_{18}O_{10}$ + Na^+: 1729.0922, found 1729.0952.

*1.4 Synthesis of Compound **4***



Scheme S3. Synthesis of control compound **4**.

4: Oxalyl chloride (0.75 mL, 8.64 mmol) in CH_2Cl_2 (3 mL) was added dropwise to a stirring solution of the diacid **16** (1.09 g, 2.87 mmol) in CH_2Cl_2 (10 mL) at 0 °C. One drop of DMF in CH_2Cl_2 (1 mL) was then added into the solution mixture. The reaction mixture was allowed to stir for 4 h from 0 °C to 25 °C, and then concentrated under reduced pressure to give the diacyl chloride **17** as a yellow solid, which was used directly without purification by mixing with triethylamine (5 mL) in CH_2Cl_2 (10 mL) at 0 °C. After stirring for about 10 min, a solution of propargylamine (0.50 mL, 7.81 mmol) in CH_2Cl_2 (3 mL) was added dropwise at 0 °C. The reaction mixture was allowed to stir for 10 h at 25 °C, and then quenched by the addition of aqueous HCl (30 mL, 1.2 M). The two layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 × 30 mL). The combined organic extracts were washed with saturated NaCl solution, dried (Na_2SO_4), filtered and concentrated to give a brown oil. The oil was purified by flash column chromatography (hexane/EtOAc = 3:1 gradient to 1:1) to generate **4** (1.52 g, 2.73 mmol, 95 %) as a colorless oil. R_f: 0.64 (hexane/EtOAc = 2:1); ^{1}H NMR: δ 0.88 (12 H, d, J = 6.8, CH_3), 1.10–1.55 (13 H, m, CH_2 and CH_2CHCH_2), 1.75–1.85 (2 H, m, $CHMe_2$), 4.12 (2 H, t, J = 6.4, OCH_2), 4.60–4.70 (4 H, d, $J \approx 7$, $ArCH_2$), 7.26–7.37 (10 H, m, ArH), 7.87 (2 H, s, ArH), 7.98–8.10 (2 H, t, J ≈ 7, NH). ^{13}C NMR: δ 22.8, 26.1, 28.5, 29.7, 31.1, 35.9, 37.6, 43.4, 69.4, 111.4, 127.4, 127.6, 128.6, 138.2, 150.6, 163.9, 167.9. *m/z* (ESI) 581 (M + Na^+, 100 %). HRMS (ESI) Calcd for $C_{35}H_{47}N_3O_3 + Na^+$: 580.3510, found 580.3516.

2. Job Plot Analysis – Determination of Binding Stoichiometry

Job plot analyses were performed to determine the binding stoichiometry between the oligomers (**2** and **3**) and various halide anions (Cl^-, Br^- and I^-). The data point for Job plot analyses were obtained from the ^1H NMR (400 MHz) spectra recorded on a Brüker Avance DPX 400 spectrometer at 298 K. For each set of binding experiment, [D_8]-THF solutions of the host compound and TBAX were separately prepared with the same concentration. Then they were mixed in ten different NMR tubes with 10:0 (v/v), 9:1, 8:2, 7:3, 6:4, 5:5, 4:6, 3:7, 2:8 and 1:9, respectively.

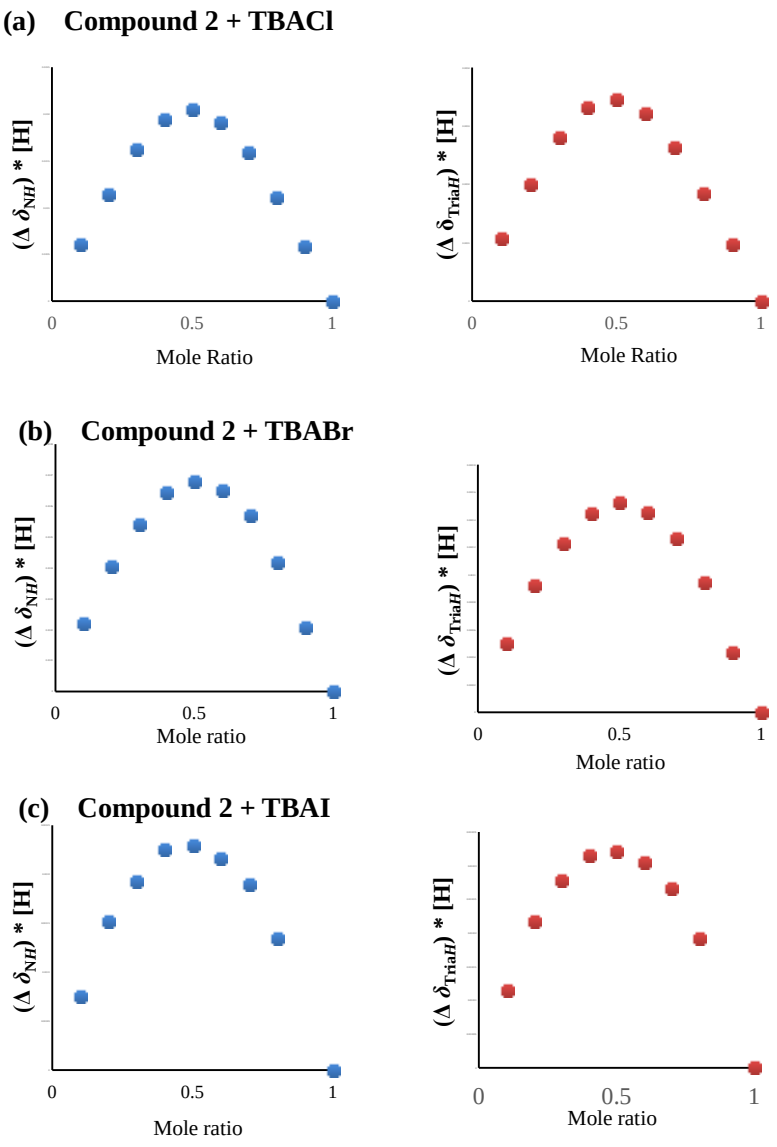


Figure S1. Job Plot analyses of the binding between **2** and (a) TBACl, (b) TBABr and (c) TBAI. Data points were obtained from amide NHs (blue) and triazole CHs (red) resonance signals in ^1H NMR spectra.

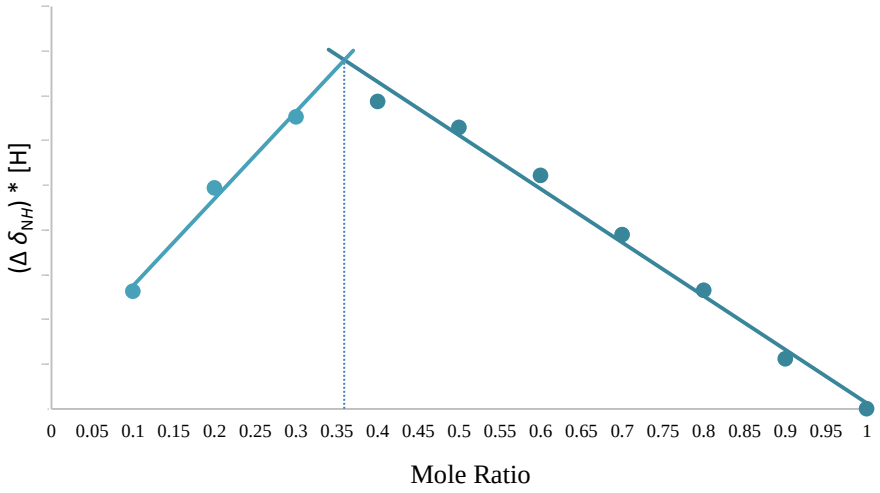


Figure S2. Job Plot analyses of the binding between **3** and TBACl. Data points were obtained from amide NHs (blue) resonance signals in ^{1}H NMR spectra.

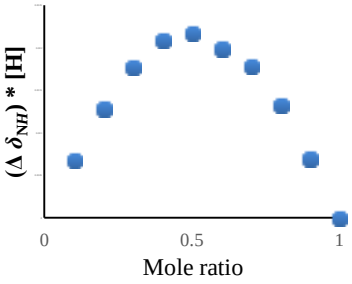


Figure S3. Job Plot analyses of the binding between control **4** and TBACl. Data points were obtained from amide NHs resonance signals in ^{1}H NMR.

The Job plot results (Figure S1) indicated 1:1 binding between **2** and halides (Cl, Br and I), 1:2 binding between **3** and chloride (Figure S2), and 1:1 binding between compound **4** and chloride (Figure S3).

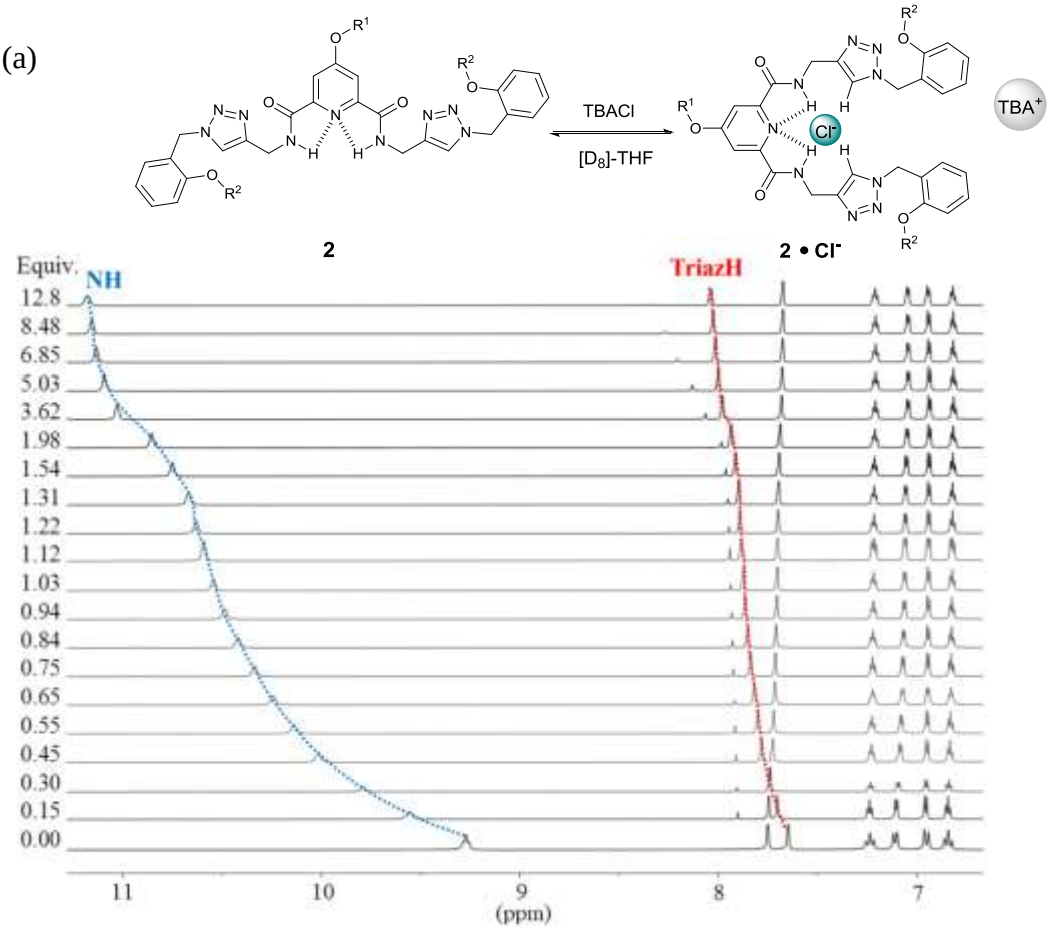
3. ^{1}H NMR Titration Experiment – Determination of Association Constant

For the titration experiment, ^{1}H NMR spectral data (700 MHz) were measured on a Brüker Avance III 700 spectrometer equipped with UltraShield Plus 700 MHz Magnets at 295 K in [D_8]-THF. Tetrabutylammonium halide (TBAX) were purchased from Sigma-Aldrich and pre-dried under vacuum for 1 day prior to use. To exclude the dilution effect on the host concentration, the added halide solution contained the host with the same concentration. Generally, a mixture of TABX (~50–80 mM) and the host (~2.5 mM) in [D_8]-THF was added successively to host (~2.5 mM), and the chemical shift values of amide NHs, triazole CHs and aromatic protons were recorded.

Curve fittings were performed using the commercial program OriginPro 8.1. As the concentration dependent change of chemical shift value is more pronounced on the amide NH and hence the chemical shift data from this proton are more accurate, the curve fitted binding constants were those derived from amide NH data. Nonetheless, the binding constants derived from the triazole CH data were also shown alongside. Generally the two binding constants agreed well with each other for the same host-guest pair.

3.1 ^{1}H NMR Titration Experiment of **2** vs TBACl

(a)



(b)

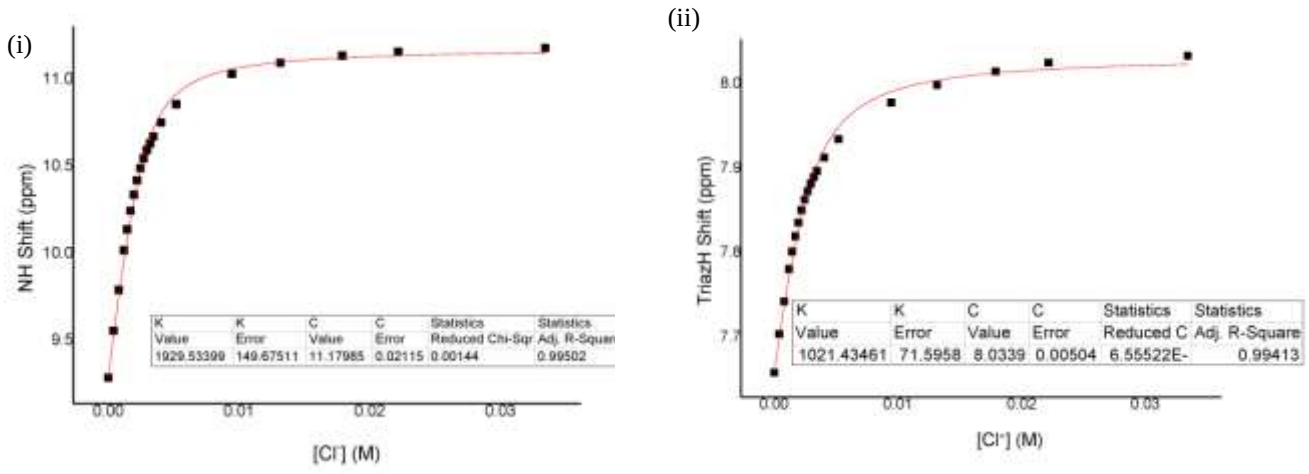


Figure S4. (a) Stacked ^{1}H NMR spectra ([D_8]-THF, 700 MHz, aromatic region) of **2** upon the addition of TBACl; (b) Non-linear curve fitting which use (i) NH and (ii) triazole CH to obtain the association constant.

Experimental Parameters:

(1) Concentration of **2** in [D_8]-THF: 2.62 mM; (2) Concentration of stock TBACl in [D_8]-THF: 67.11 mM.

*3.2 ^{1}H NMR Titration Experiment of **2** vs TBABr*

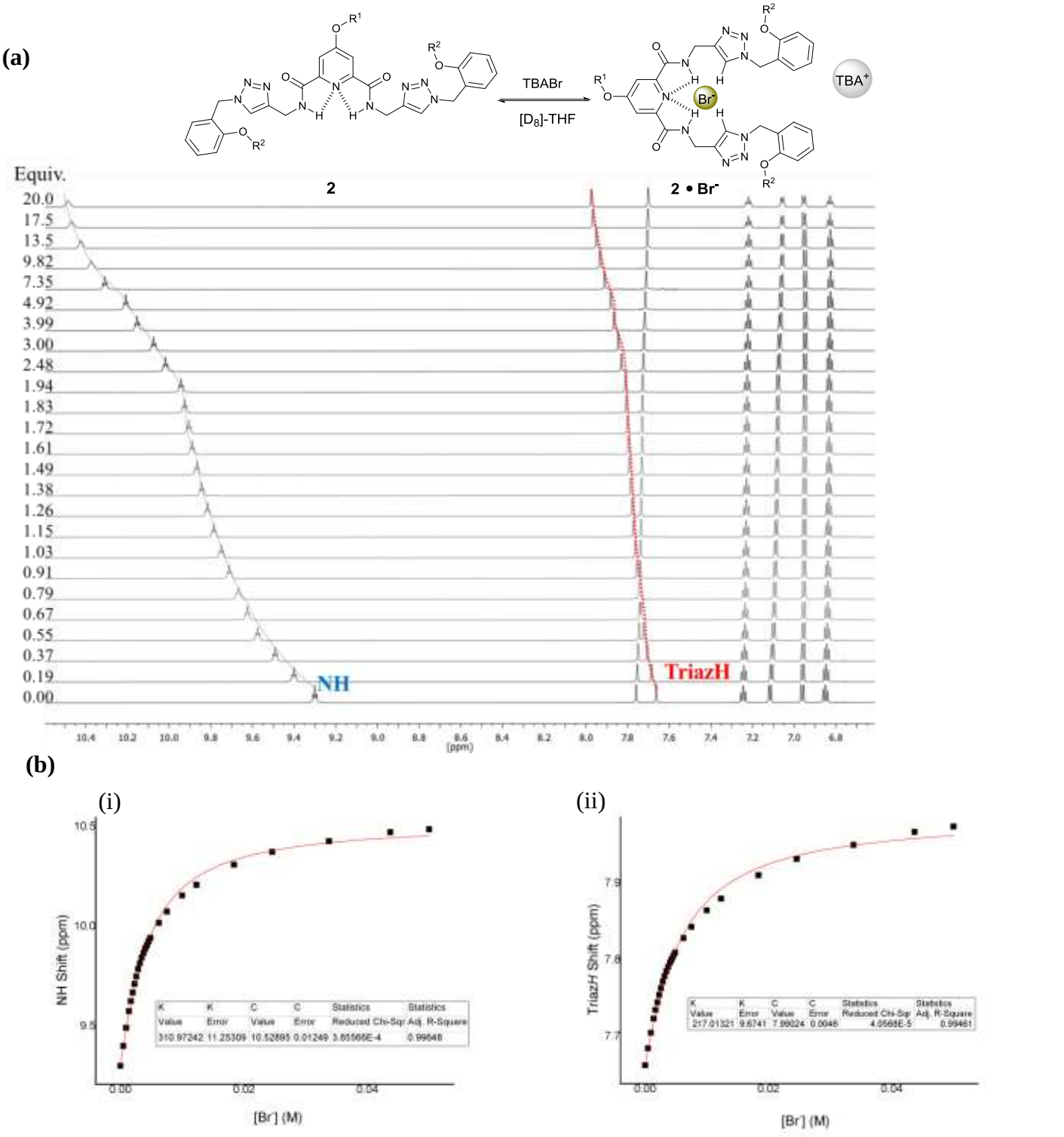


Figure S5. **(a)** Stacked ^{1}H NMR spectra ([D$_8$]-THF, 700 MHz, aromatic region) of **2** upon the addition of TBABr; **(b)** Non-linear curve fitting which use (i) amide NHs and (ii) triazole CHs to obtain the association constant.

Experimental Parameters:

(1). Concentration of **2** in [D$_8$]-THF : 2.50 mM; **(2)**. Concentration of stock TBABr in [D$_8$]-THF : 78.48 mM.

3.3 ^{1}H NMR Titration Experiment of **2** vs TBAI

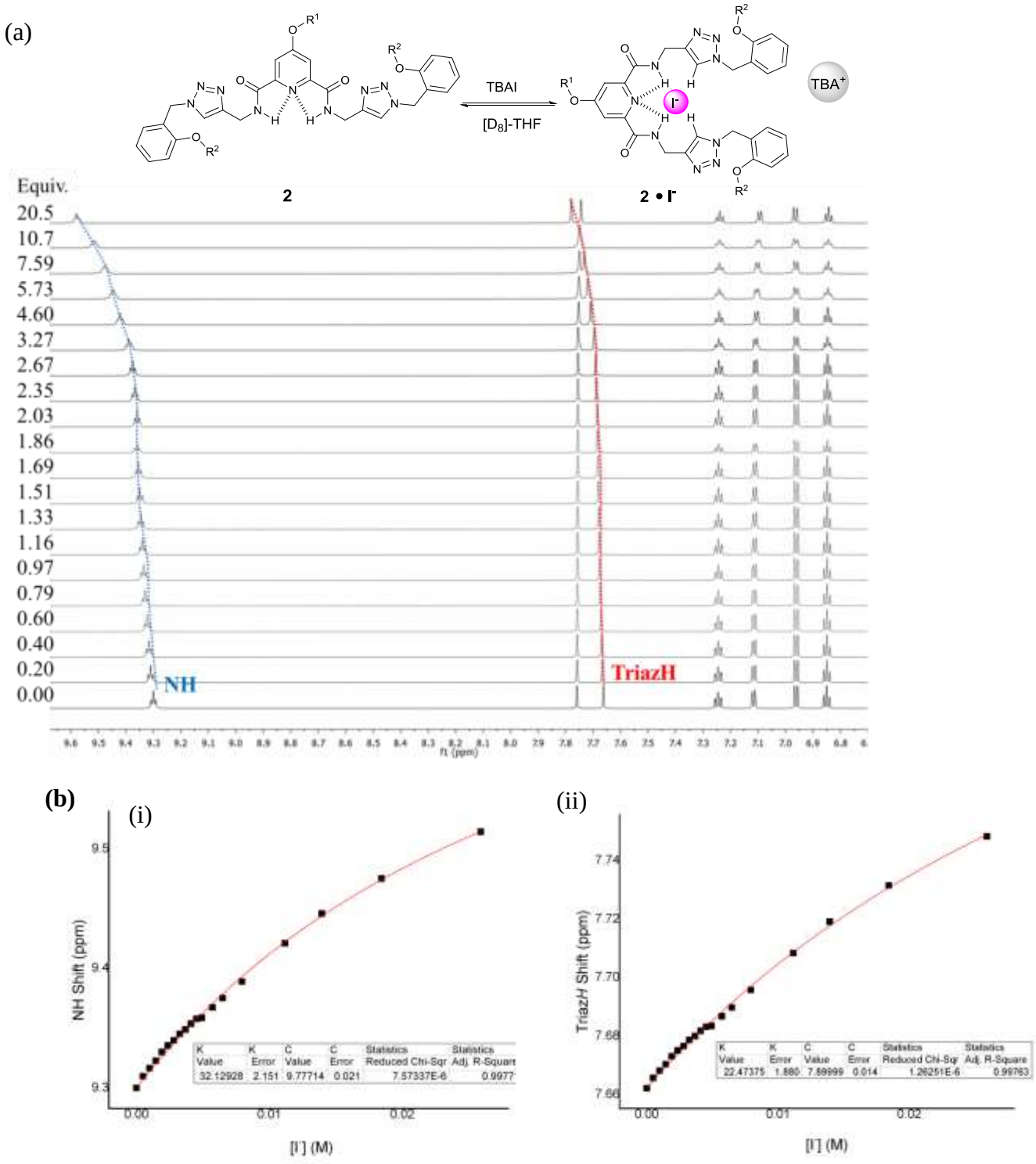


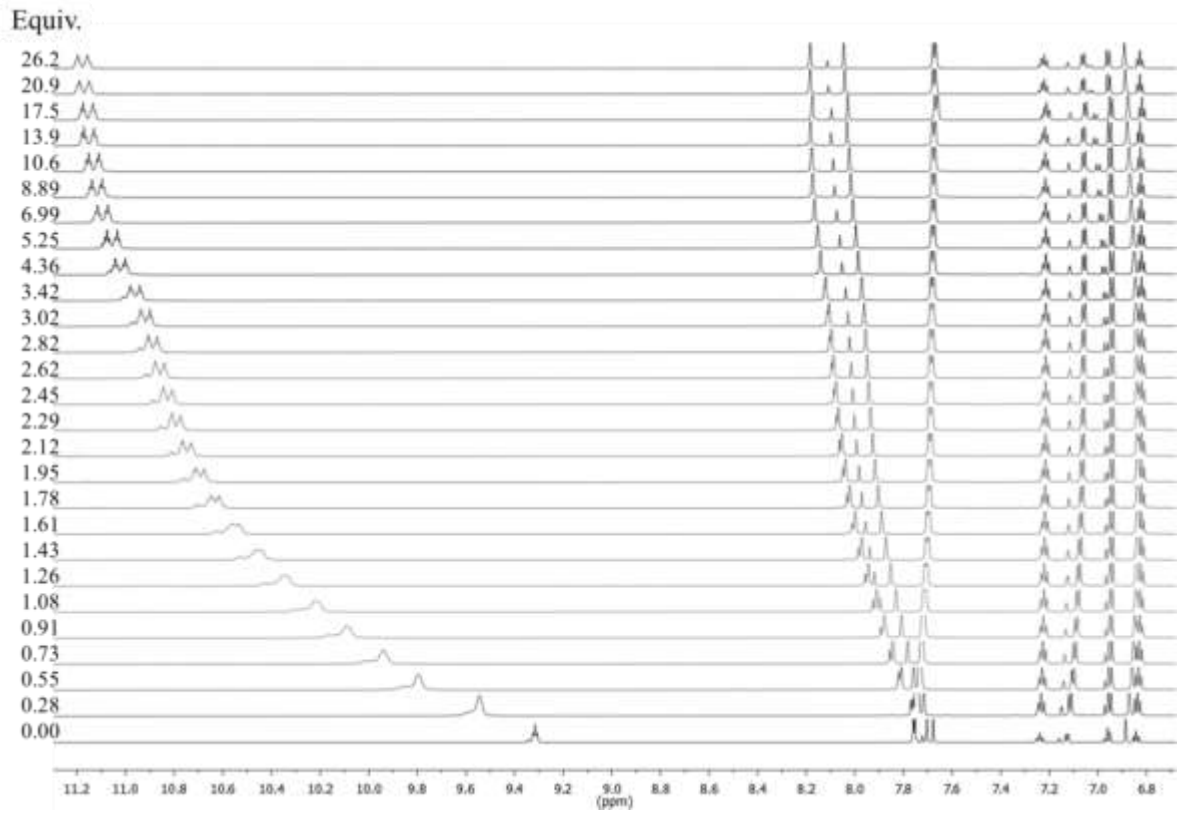
Figure S6. (a) Stacked ^{1}H NMR spectra (aromatic region) of **2** upon the addition of TBAI; (b) Non-linear curve fitting which use (i) amide NHs and (ii) triazole CHs to obtain the association constant.

Experimental Parameters:

(1) Concentration of **2** in [D_8]-THF: 2.43 mM; (2) Concentration of stock TBAI in [D_8]-THF: 49.68 mM.

*3.4 ^{1}H NMR Titration Experiment of **3** vs TBACl*

(a)



(b)

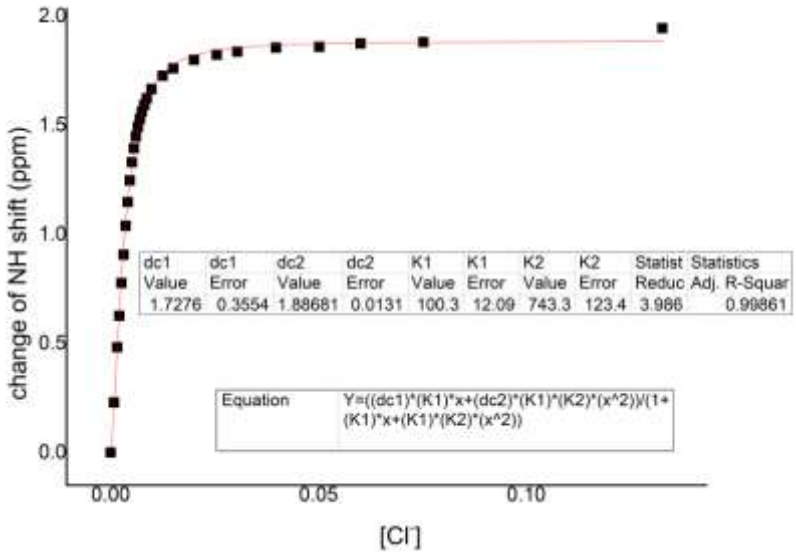


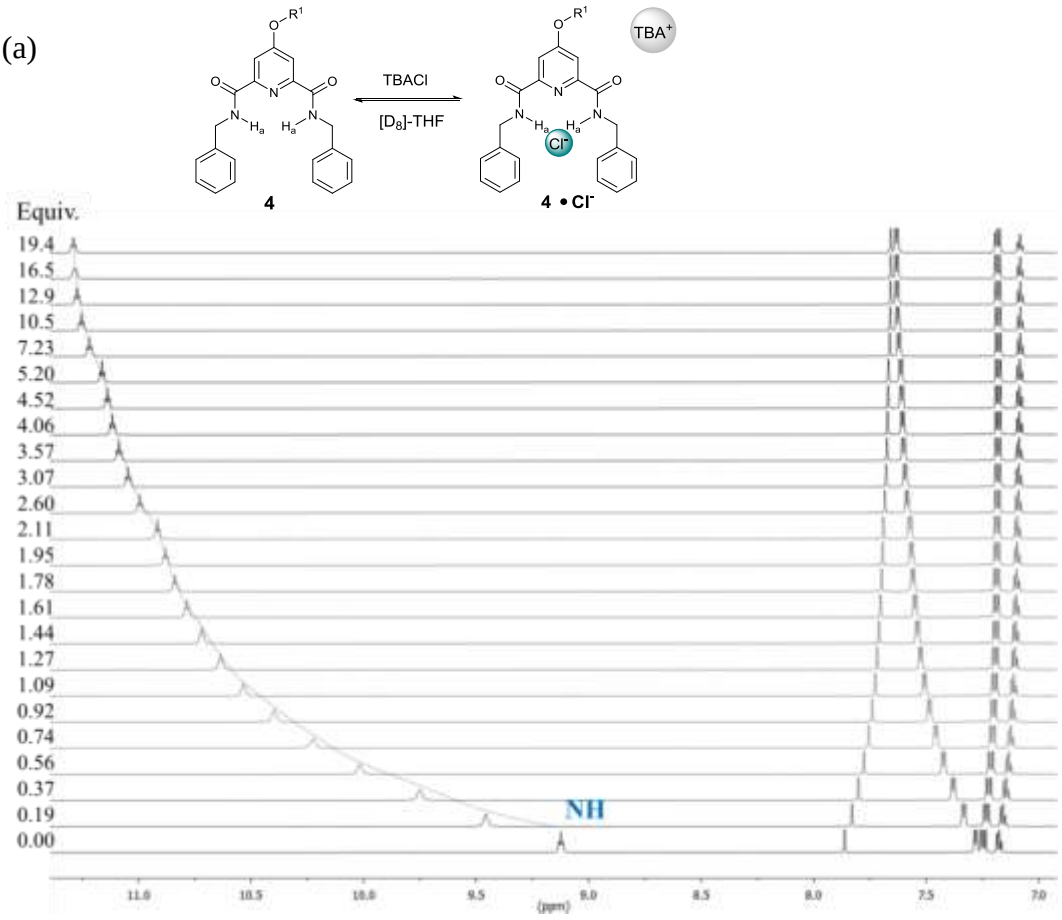
Figure S7. (a) Stacked ^{1}H NMR spectra ([D_8]-THF, 700 MHz, aromatic region) of **3** upon addition of TBACl; (b) Non-linear curve fitting which use NH to obtain the association constant.

Experimental Parameters:

(1) Concentration of **3** in [D_8]-THF: 2.87 mM; (2) Concentration of stock TBACl in [D_8]-THF: 132.8 mM.

*3.5 ^{1}H NMR Titration Experiment of Control **4** vs TBACl*

(a)



(b)

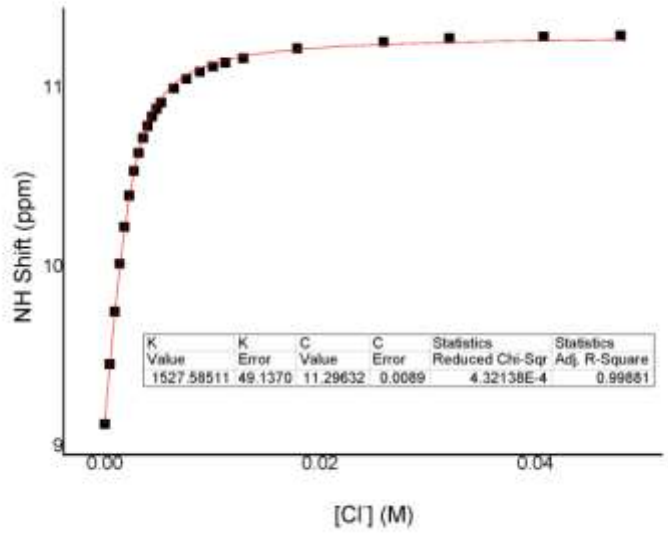


Figure S8. (a) Stacked ^{1}H NMR spectra (aromatic region) of compound **4** upon the addition of TBACl; (b) Non-linear curve fitting which use amide NHs to obtain the association constant.

Experimental Parameters:

(1). Concentration of **4** in [D_8]-THF: 2.47 mM; (2) Concentration of stock TBAI in [D_8]-THF: 77.90 mM.

4. Hill Plot of the Binding between Compound 3 and Cl^-

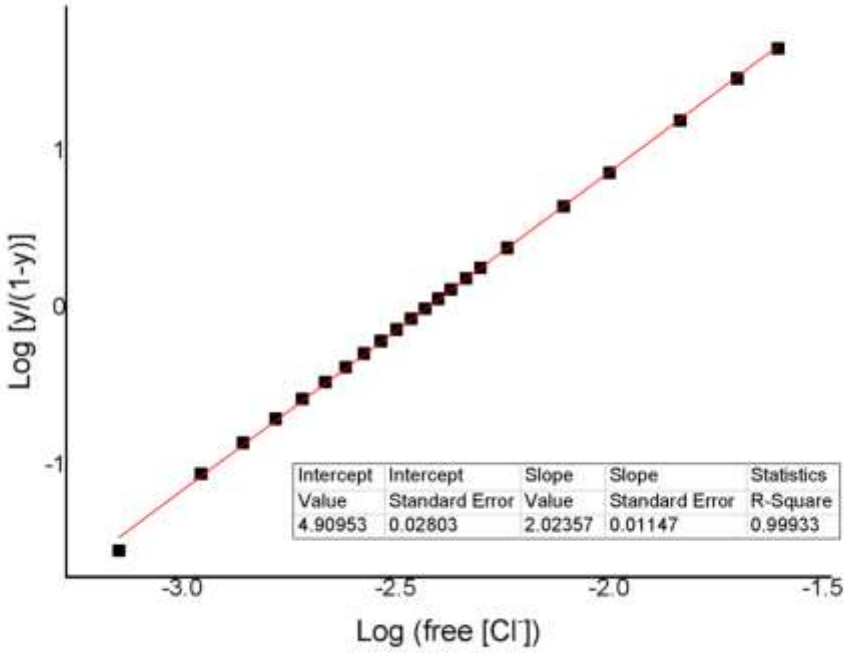


Figure S9. Hill plot of compound **3** with Cl^-. A slope of 2.0 was found, which indicated a positive cooperativity.

5. Fitting with Non-cooperative Model[2]

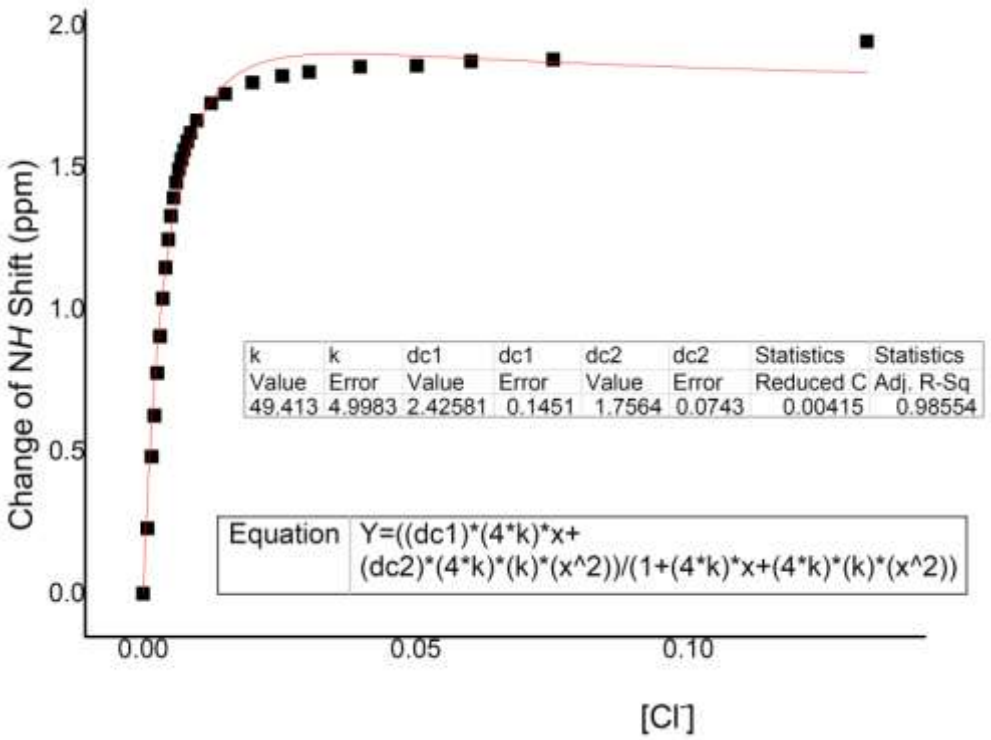


Figure S10. Non-linear curve fitting of **3** vs Cl^- base on a non-cooperative model, with K_1 fixed as 4 × K_2. As can be seen, fitting result was not satisfactory [r^2 = 0.9855 (non-cooperative model) vs r^2 = 0.9986 (independent model; Figure S7)].

[2] P. Thordarson, *Chem. Soc. Rev.* 2011, **40**, 1305.

6. FTIR Studies

Infra-red spectroscopic studies were performed on a Brüker Vertex 70 Fourier-transform spectrometer fitted with a globar source, a CaF_2 beam splitter, and a liquid nitrogen cooled HgCdTe detector. All the experiments were conducted using spectrophotometric grade THF at 298 K. All spectra were recorded at 2 cm^{-1} resolution. The sample cell was made of a 0.5 mm Teflon spacer sandwiched by two 4-mm thick CaF_2 disks, with 0.1 mm optical path. Build-in 'atmospheric compensation' routine from the spectrometer was used in order to minimize the background absorption.

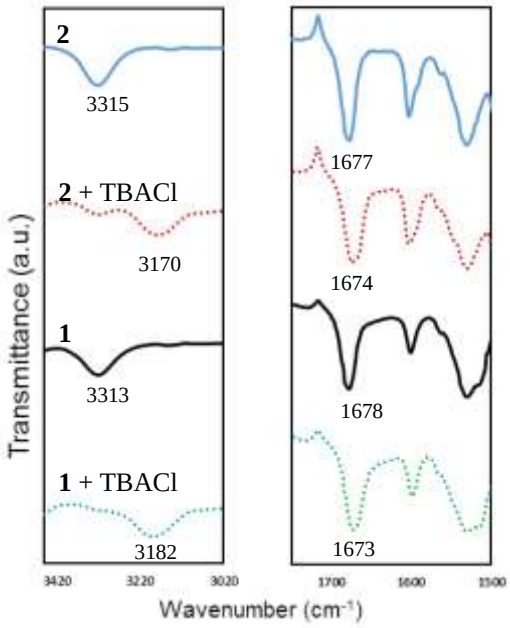


Figure S11. Stacked IR spectra of (from top to bottom) **2**, **2** + TBACl (1 equiv.), **1** and **1** + TBACl (1 equiv.) in THF.

7. Scanning Electron Microscopy (SEM) Analysis

Scanning electron microscopy (SEM) was performed on a Quanta 400F field emission scanning electron microscope. The samples analyzed were sputter-deposited with minute amount of gold to prevent charging. **1**-Cl was prepared by an air-dried solution mixture of 1:1 **1** and TBACl in toluene (binding site : TBACl = 1 : 1)

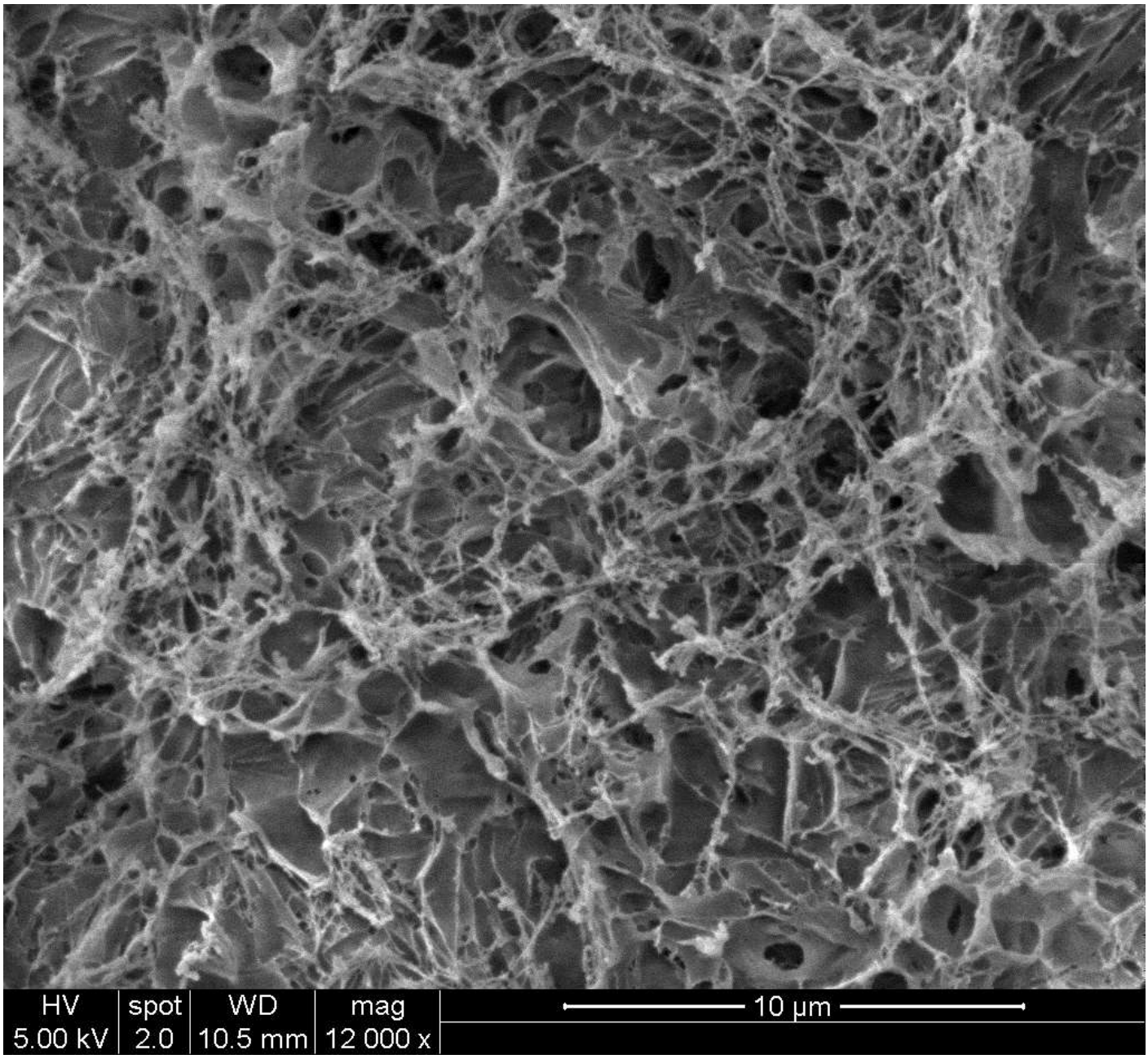


Figure S12. SEM images of freeze dried sample of **1** at 12 K magnification.

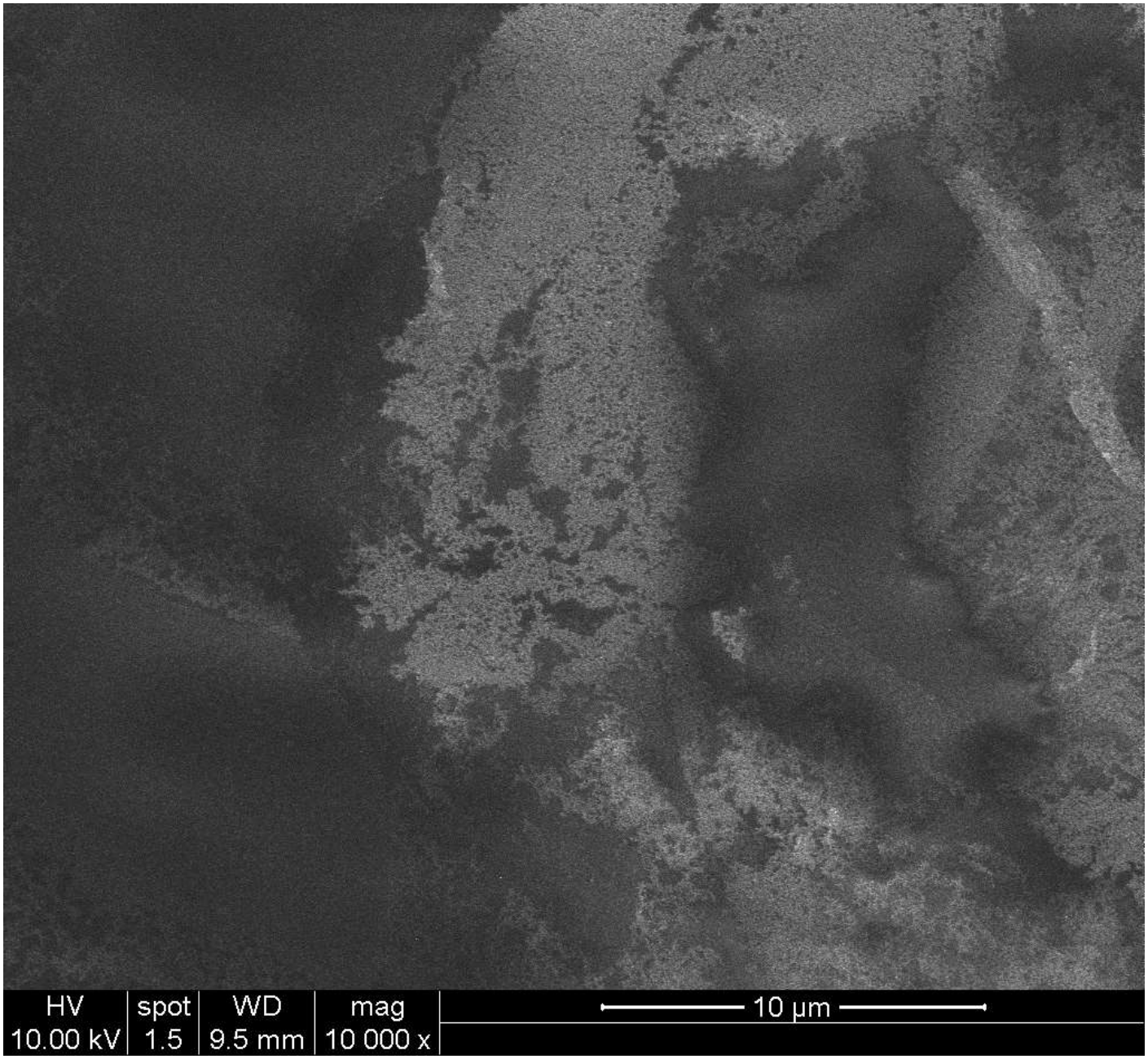


Figure S13. SEM images of **1**-Cl complex at 10 K magnification.

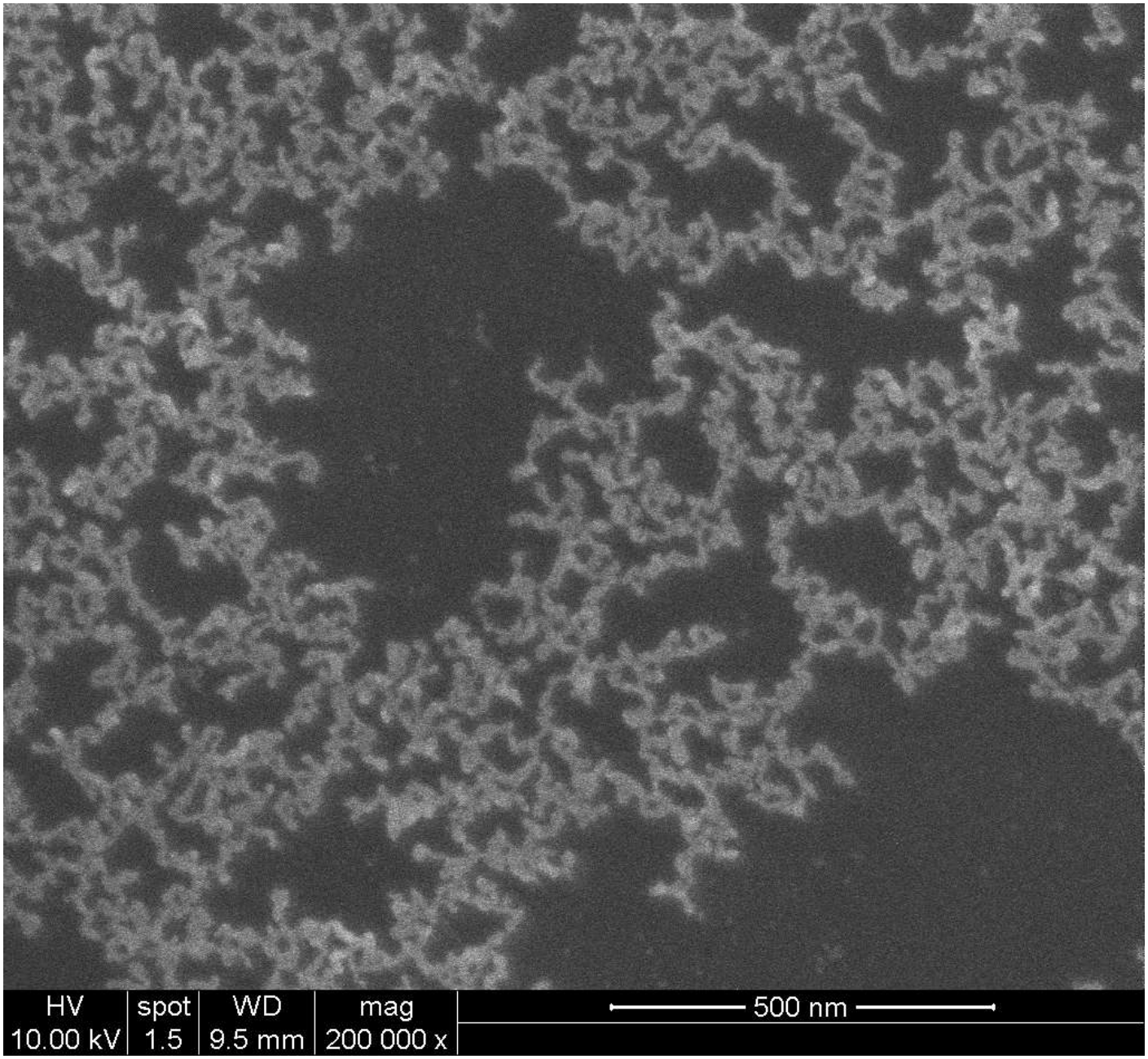


Figure S14. SEM images of **1**-Cl complex at 200 K magnification.

8. Mass Spectrum of 2•TBACl

Mass spectral measurement was conducted on an API QSTAR Pulsar I LC/MS/TOF mass spectrometer in ESI-MS negative ion mode using taurochloric acid as calibration standard.

Theoretical mass peak value for **2**-$^{35}Cl^-$ ($C_{51}H_{73}N_9O_5Cl^-$): 926.5428; Found: 926.5471.

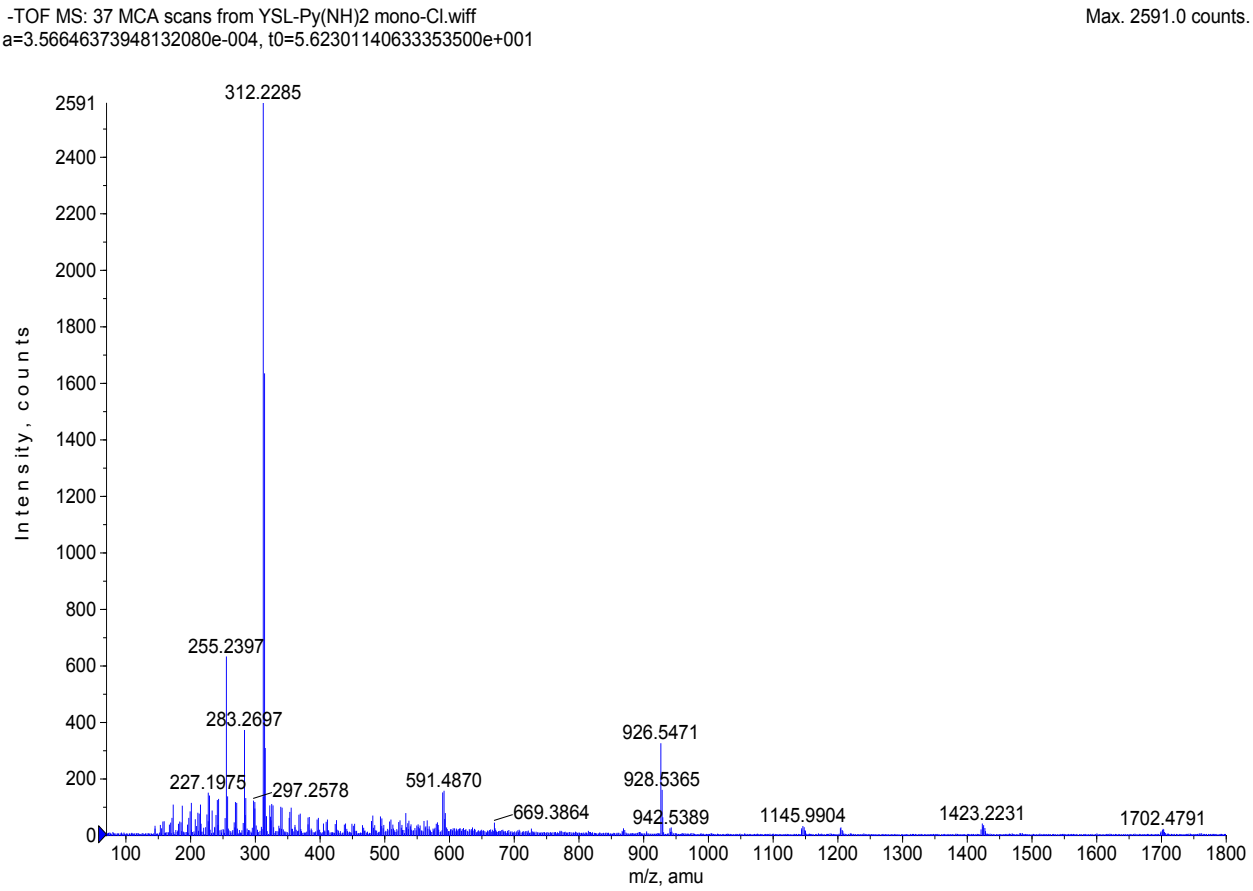
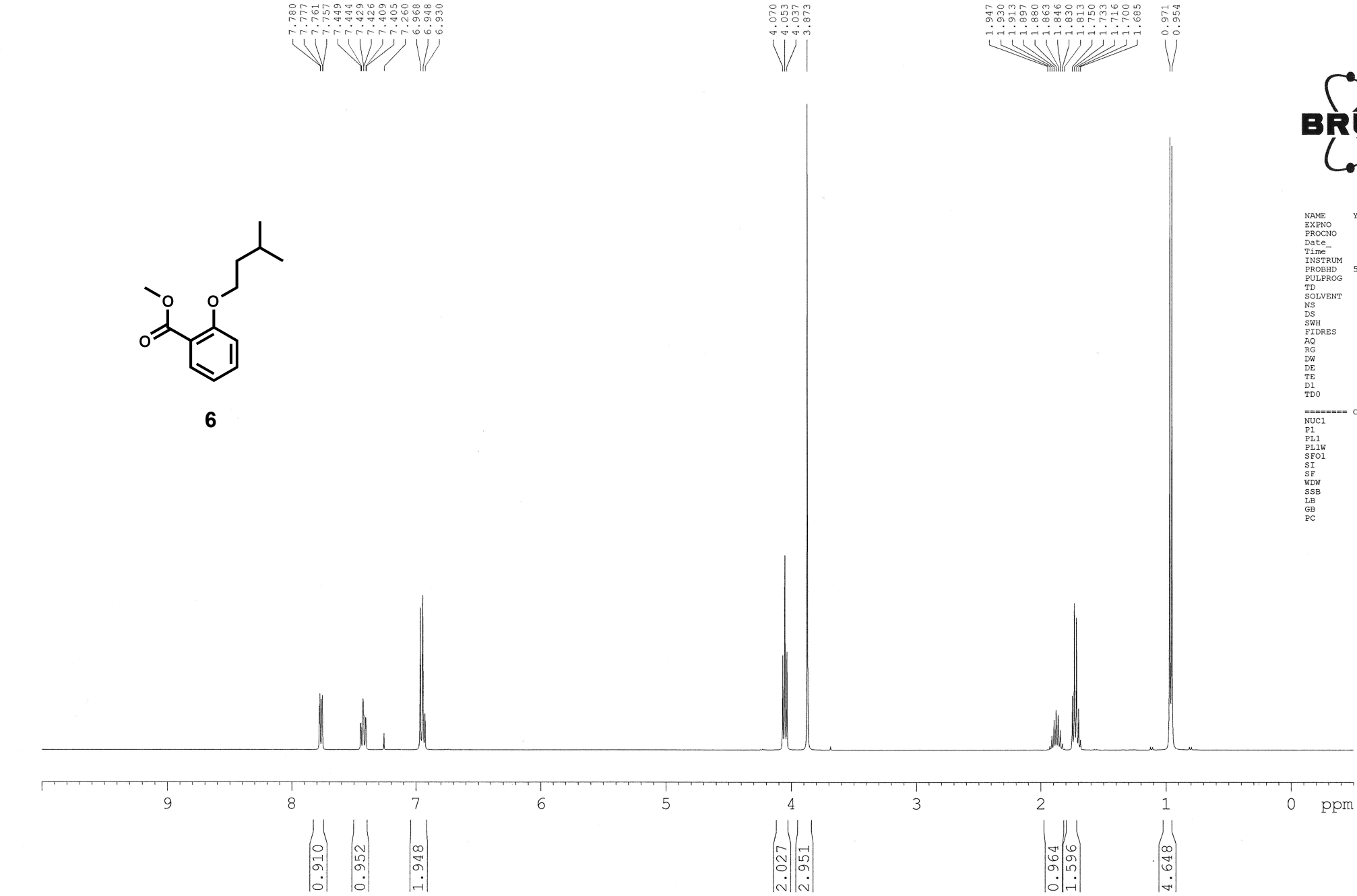


Figure S15. Mass spectrum of compound **2**-Cl^-.

9. List of Spectra



6

BRUKER

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PROCNO	1
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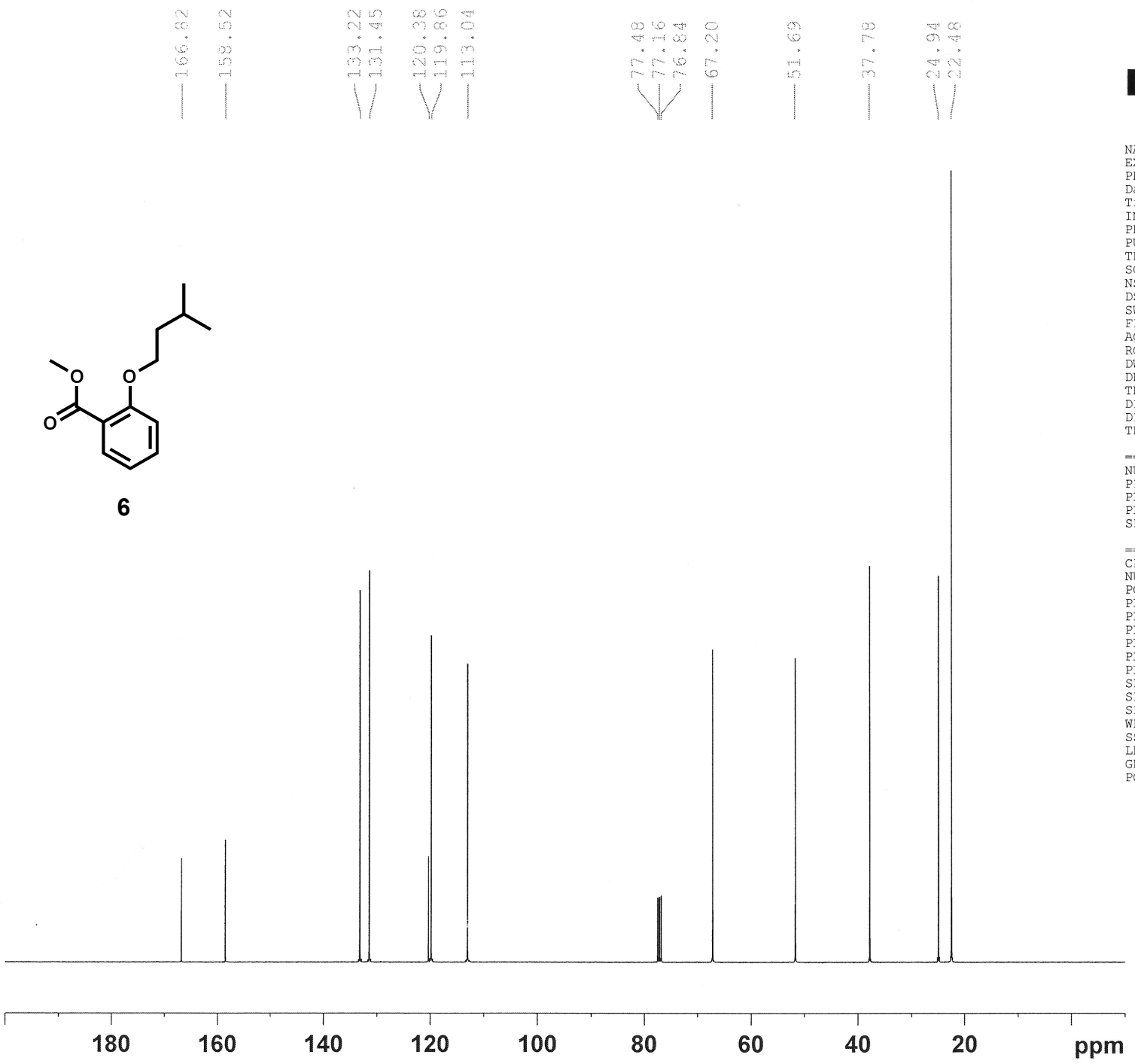
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	2
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	71.8
DW	60.800 usec
DE	6.50 usec
TE	294.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.69 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

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PROCNO	1
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PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	800
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	295.3 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.17 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.25282964 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127748 MHz
WDW	EM
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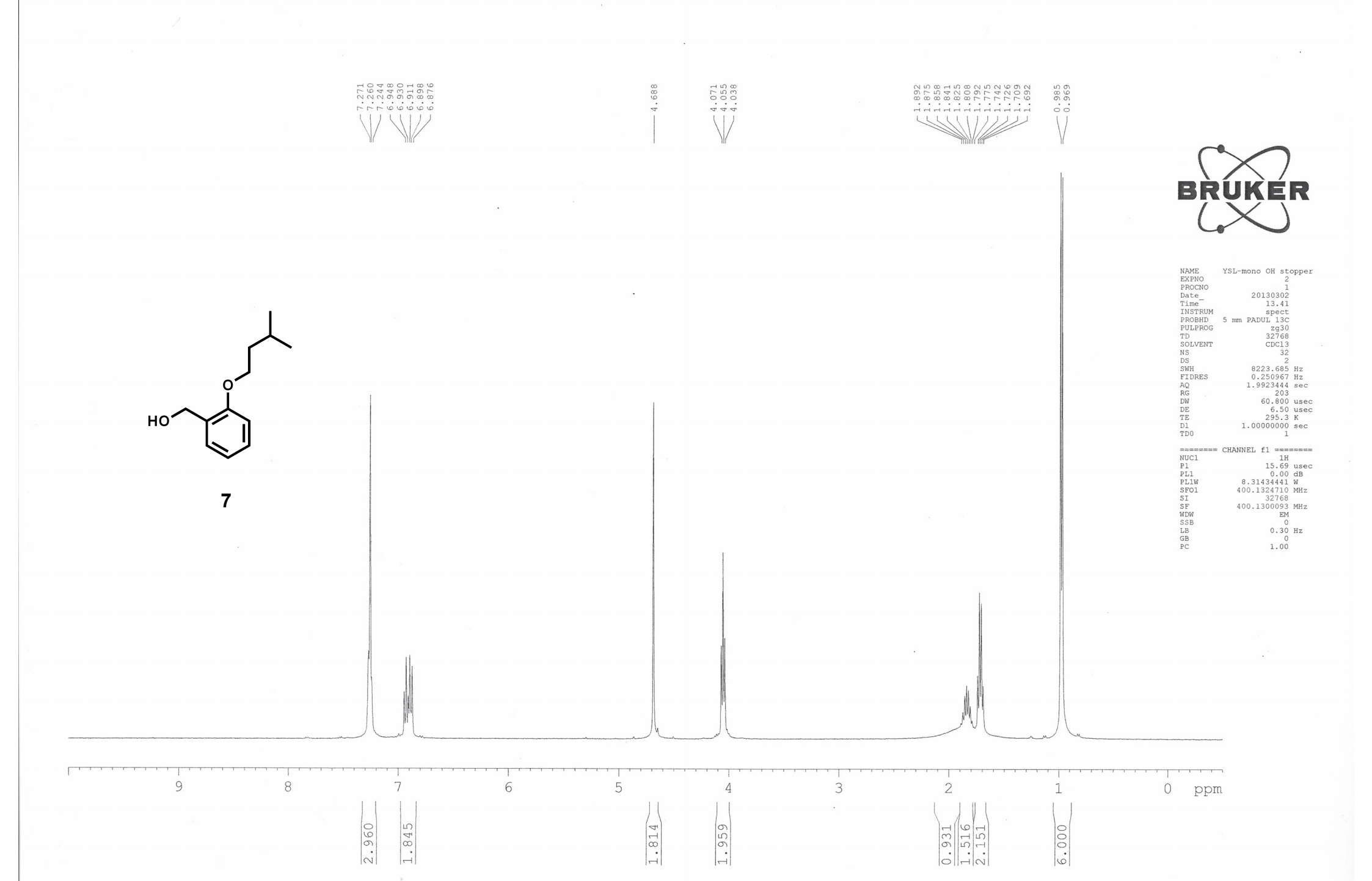
GB	0
PC	1.40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6.911
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1.875
1.858
1.841
1.825
1.808
1.792
1.775
1.742
1.726
1.709
1.692
0.985
0.969
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O
7
BRUKER
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EXPNO 2
PROCNO 1
Date_ 20130302
Time 13.41
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.69 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
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1.814
1.959
0.931
1.516
2.151
6.000

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EXPNO	1
PROCNO	1
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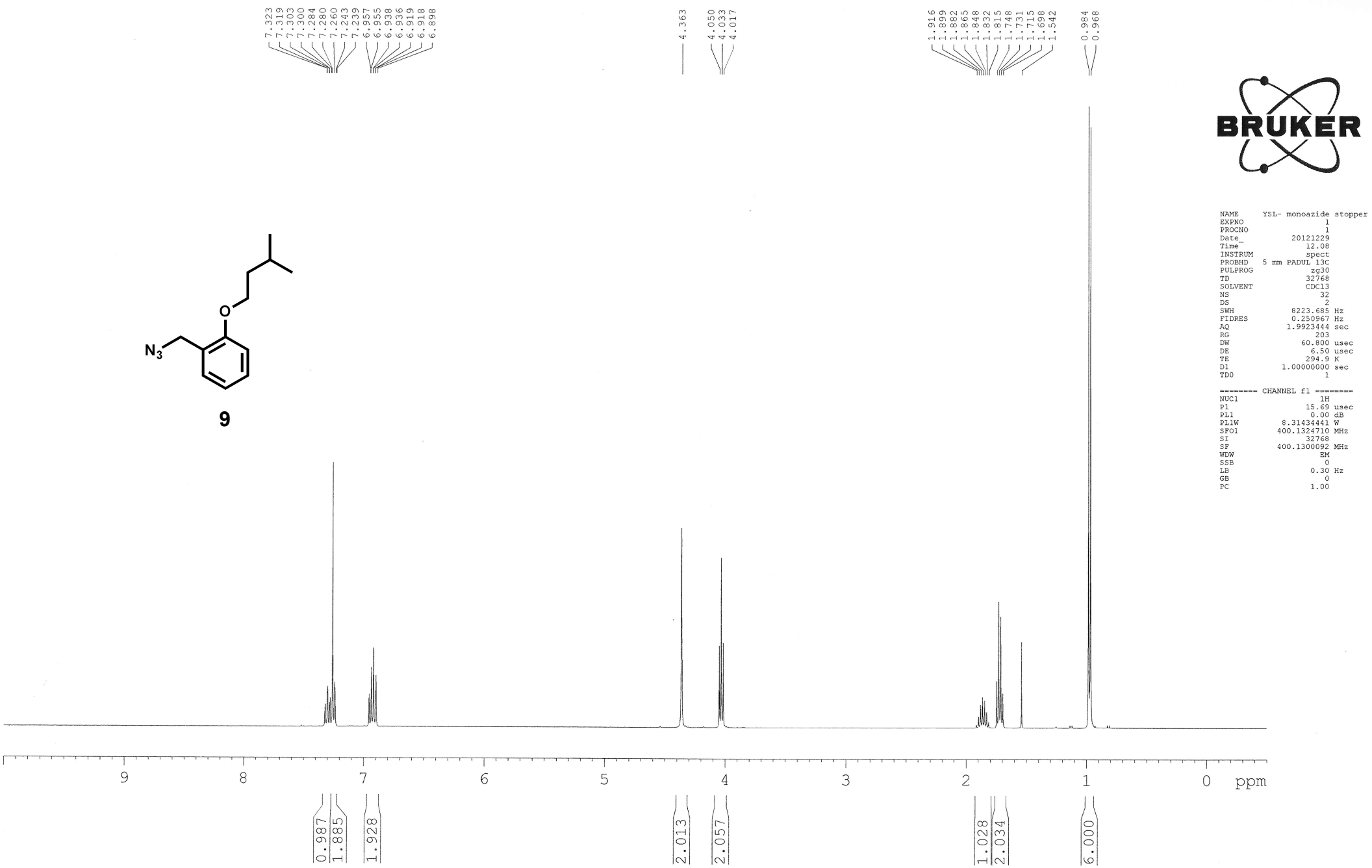
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	403
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	295.4 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.17 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.25282964 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127699 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	1.40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6.918
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4.033
4.017
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0.968
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PROCNO 1
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1
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PL1 0.00 dB
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SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
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PC 1.00
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8
7
6
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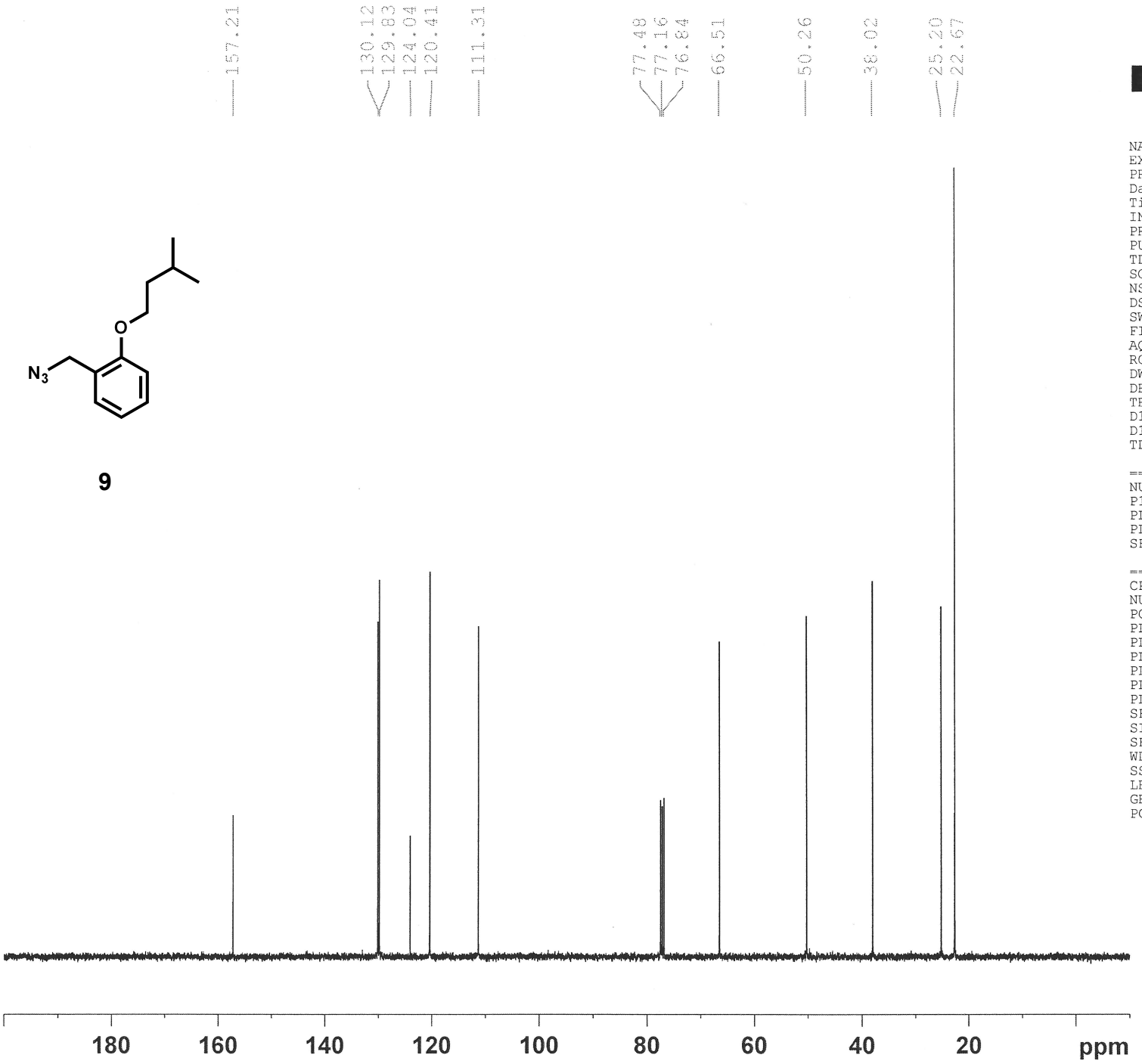

BRUKER

NAME	YSL- monoazide stopper (C)
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PROCNO	1
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SOLVENT	CDCl3
NS	800
DS	4
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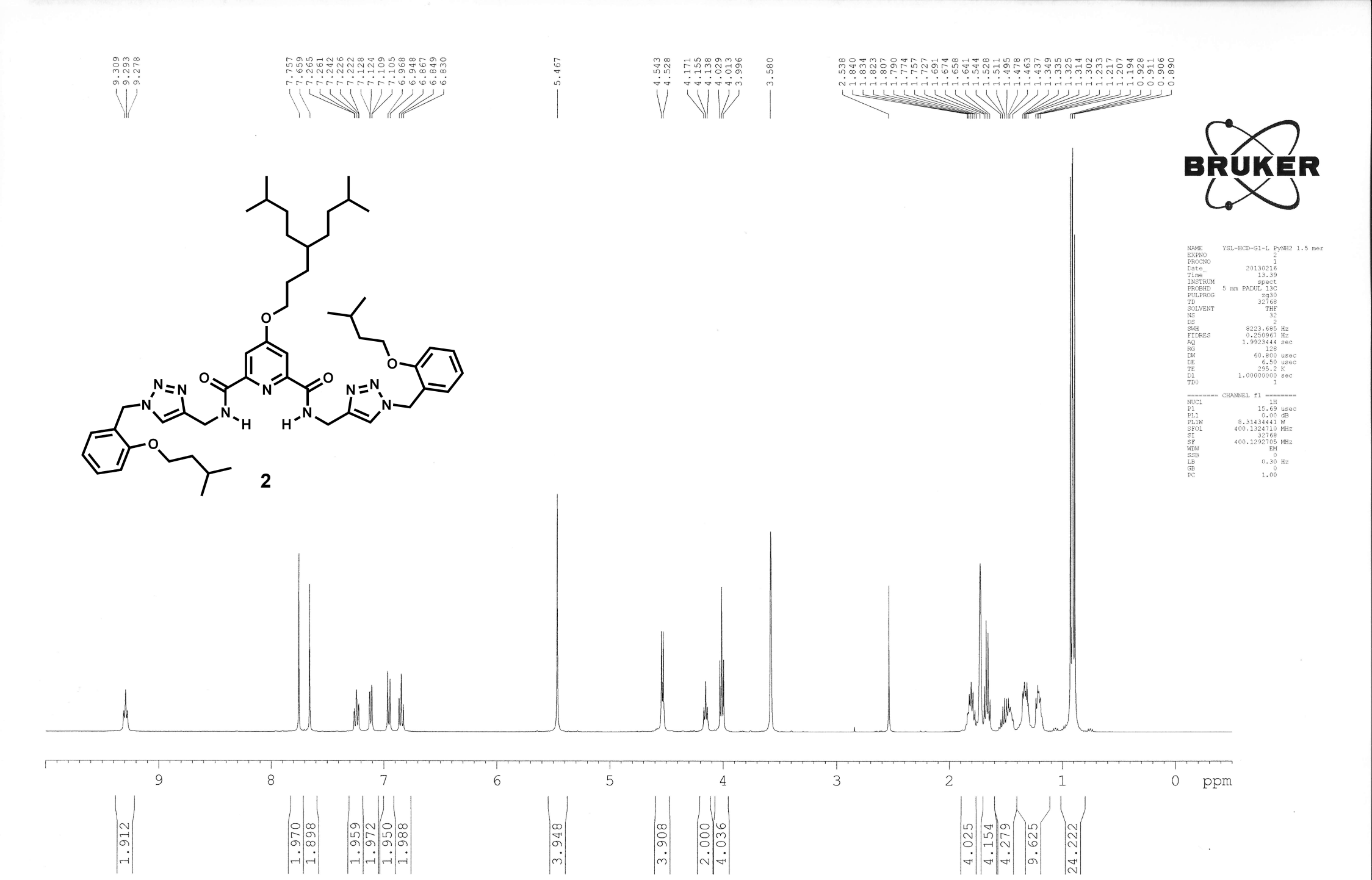
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	295.0 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.17 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.25282964 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127660 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40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6.867
6.849
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5.467
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4.528
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4.155
4.138
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1.217
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0.911
0.906
0.890
BRUKER
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NAME YSL-HCD-G1-L PyNH2 1.5 mer
EXPNO 2
PROCNO 1
Date_ 20130216
Time 13.39
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PULPROG zg30
TD 32768
SOLVENT THF
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FIDRES 0.250967 Hz
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RG 128
DW 60.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 15.69 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1292705 MHz
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SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
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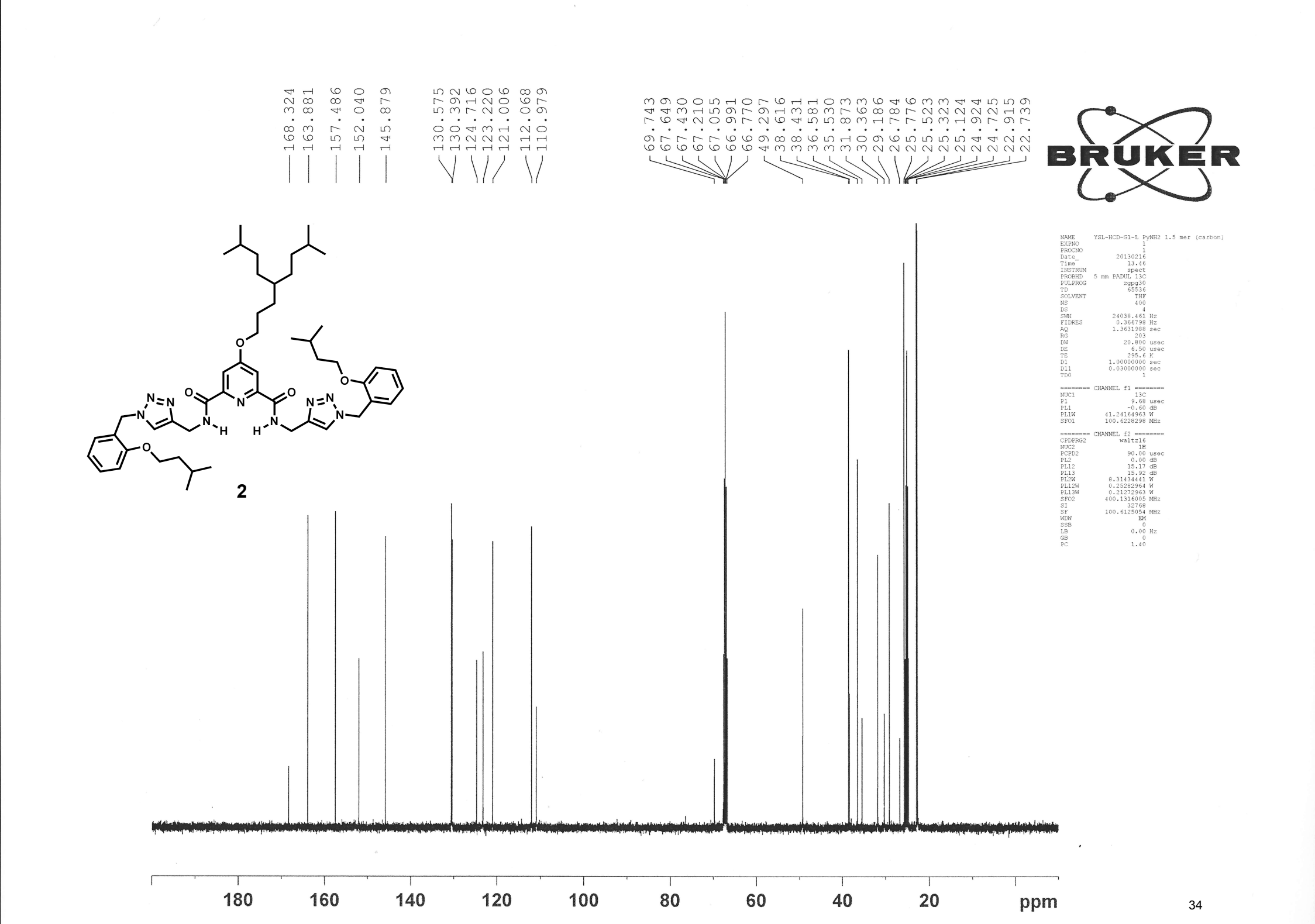
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31.873
30.363
29.186
26.784
25.776
25.523
25.323
25.124
24.924
24.725
22.915
22.739
BRUKER
2
NAME YSL-HCD-G1-L PyNH2 1.5 mer (carbon)
EXPNO 1
PROCNO 1
Date_ 20130216
Time 13.46
INSTRUM spect
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PULPROG zgpg30
TD 65536
SOLVENT THF
NS 400
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.68 usec
PL1 -0.60 dB
PL1W 41.24164963 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 15.17 dB
PL13 15.92 dB
PL2W 8.31434441 W
PL12W 0.25282964 W
PL13W 0.21272963 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6125054 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
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140
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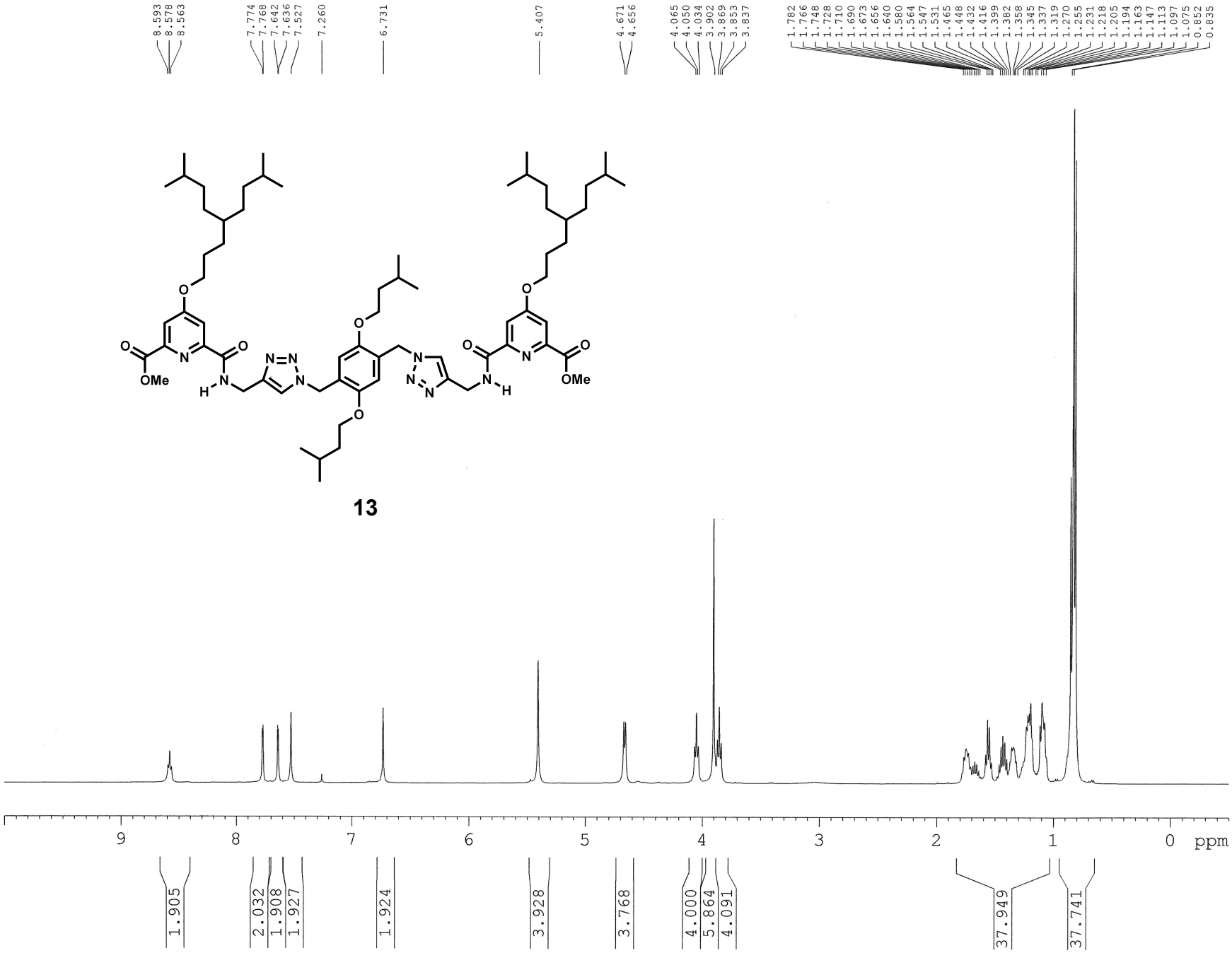
80
60
40
20
ppm



13

AV400Q NMR

BRUKER

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PROCNO	1
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PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	14.2
DW	60.800 usec
DE	6.50 usec
TE	295.7 K
D1	1.00000000 sec
TD0	1
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NUC1	1H
P1	14.00 usec
PL1	-1.00 dB
PL1W	13.56617069 W
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SI	32768
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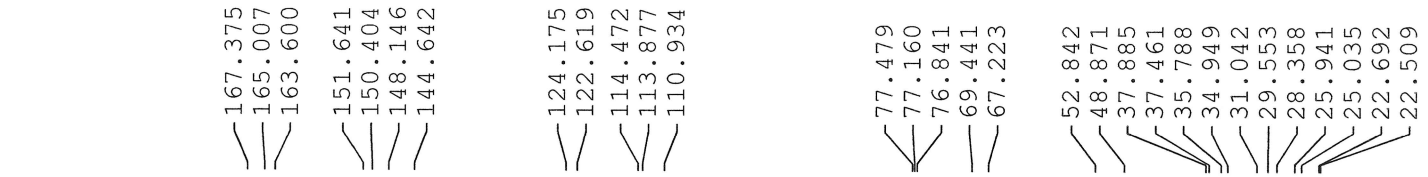
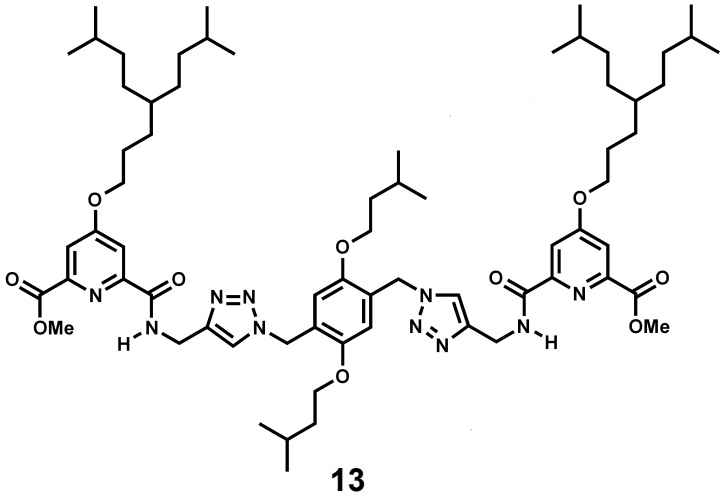
PC	1.00





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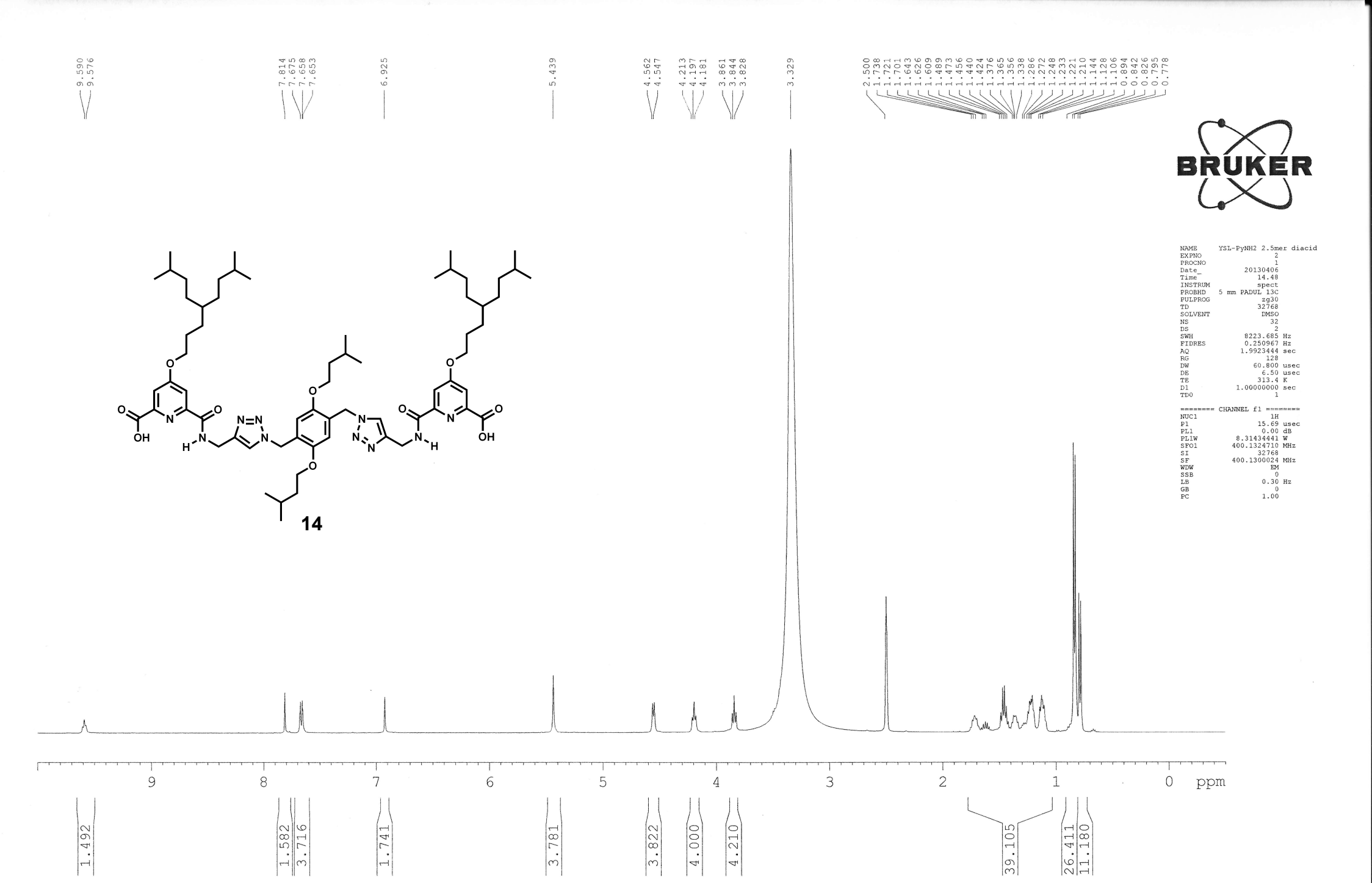
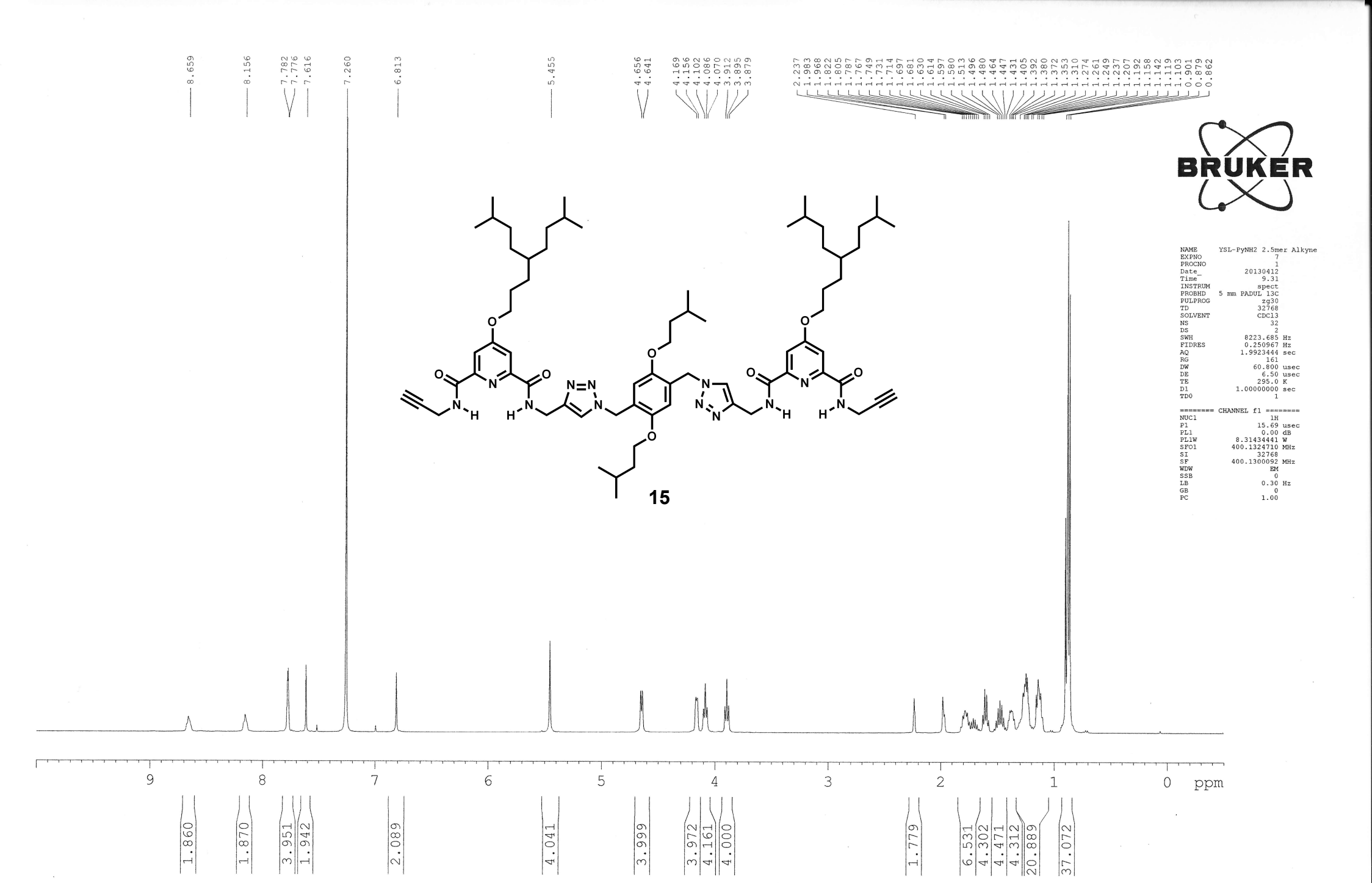
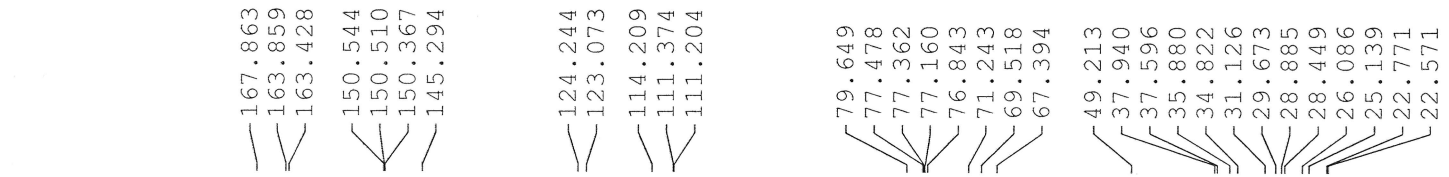
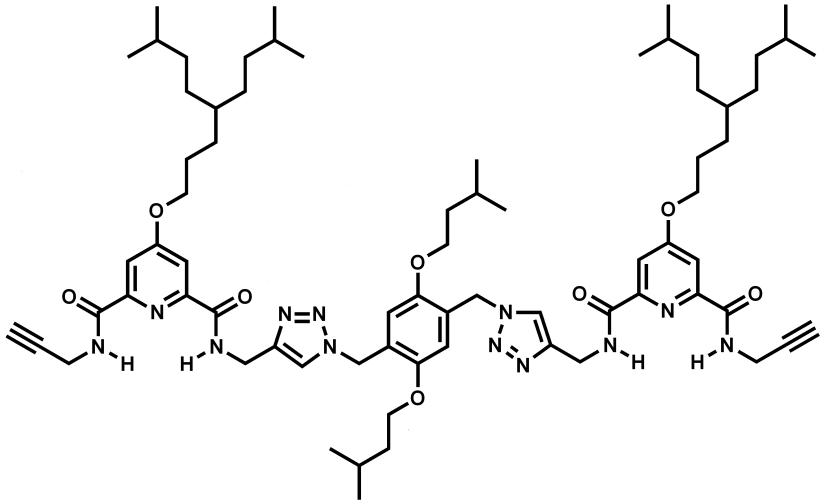
BRUKER

```
NAME          YSL-PyNH2 2.5mer OMe (carbon)
EXPNO                    1
PROCNO                   1
Date_             20130328
Time                18.16
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     400
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                     322
DW                  20.800 usec
DE                   6.50 usec
TE                  296.0 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.90 usec
PL1                  -2.00 dB
PL1W           55.33689499 W
SFO1           100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2                90.00 usec
PL2                  -1.00 dB
PL12                 15.16 dB
PL13                 18.62 dB
PL2W           13.56617069 W
PL12W           0.32844096 W
PL13W           0.14806664 W
SFO2           400.1916008 MHz
SI                   32768
SF             100.6278559 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```


9.590
9.576
7.814
7.675
7.658
7.653
6.925
5.439
4.562
4.547
4.213
4.197
4.181
3.861
3.844
3.828
3.329
2.500
1.738
1.721
1.701
1.643
1.626
1.609
1.489
1.473
1.456
1.440
1.424
1.376
1.365
1.356
1.338
1.286
1.272
1.248
1.233
1.221
1.210
1.144
1.128
1.106
0.894
0.842
0.826
0.795
0.778
BRUKER
NAME YSL-PyNH2 2.5mer diacid
EXPNO 2
PROCNO 1
Date_ 20130406
Time 14.48
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 313.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.69 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300024 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
14
9
8
7
6
5
4
3
2
1
0
ppm
1.492
1.582
3.716
1.741
3.781
3.822
4.000
4.210
39.105
26.411
11.180

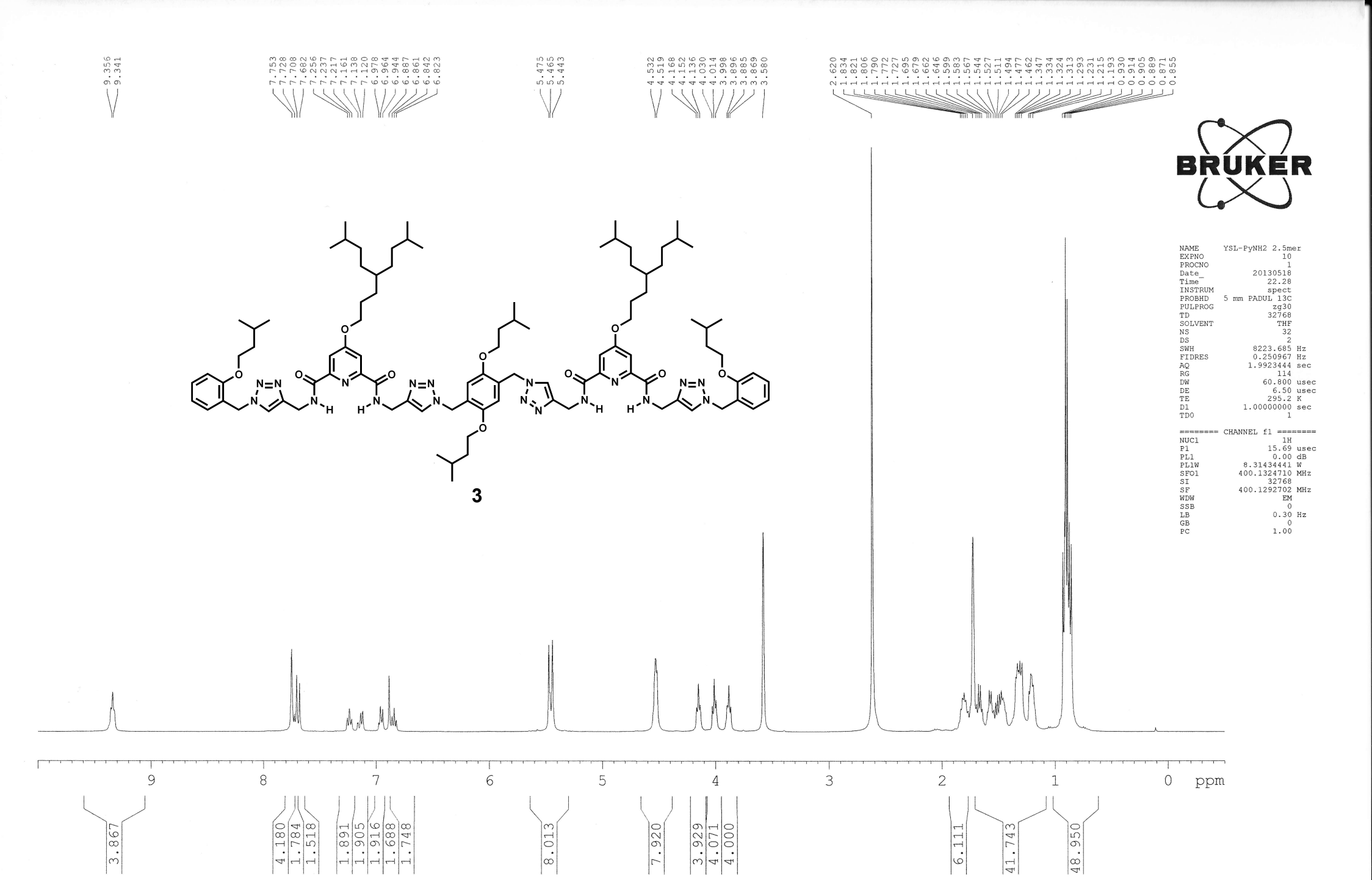

8.659
8.156
7.782
7.776
7.616
7.260
6.813
5.455
4.656
4.641
4.169
4.156
4.102
4.086
4.070
3.912
3.895
3.879
2.237
1.983
1.968
1.822
1.805
1.787
1.767
1.749
1.731
1.714
1.697
1.681
1.630
1.614
1.597
1.580
1.513
1.496
1.480
1.464
1.447
1.431
1.405
1.392
1.380
1.372
1.353
1.310
1.274
1.261
1.249
1.237
1.207
1.192
1.158
1.142
1.119
1.103
0.901
0.879
0.862
BRUKER
15
NAME YSL-PyNH2 2.5mer Alkyne
EXPNO 7
PROCNO 1
Date_ 20130412
Time_ 9.31
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.69 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.860
1.870
3.951
1.942
2.089
4.041
3.999
3.972
4.161
4.000
1.779
6.531
4.302
4.471
4.312
20.889
37.072

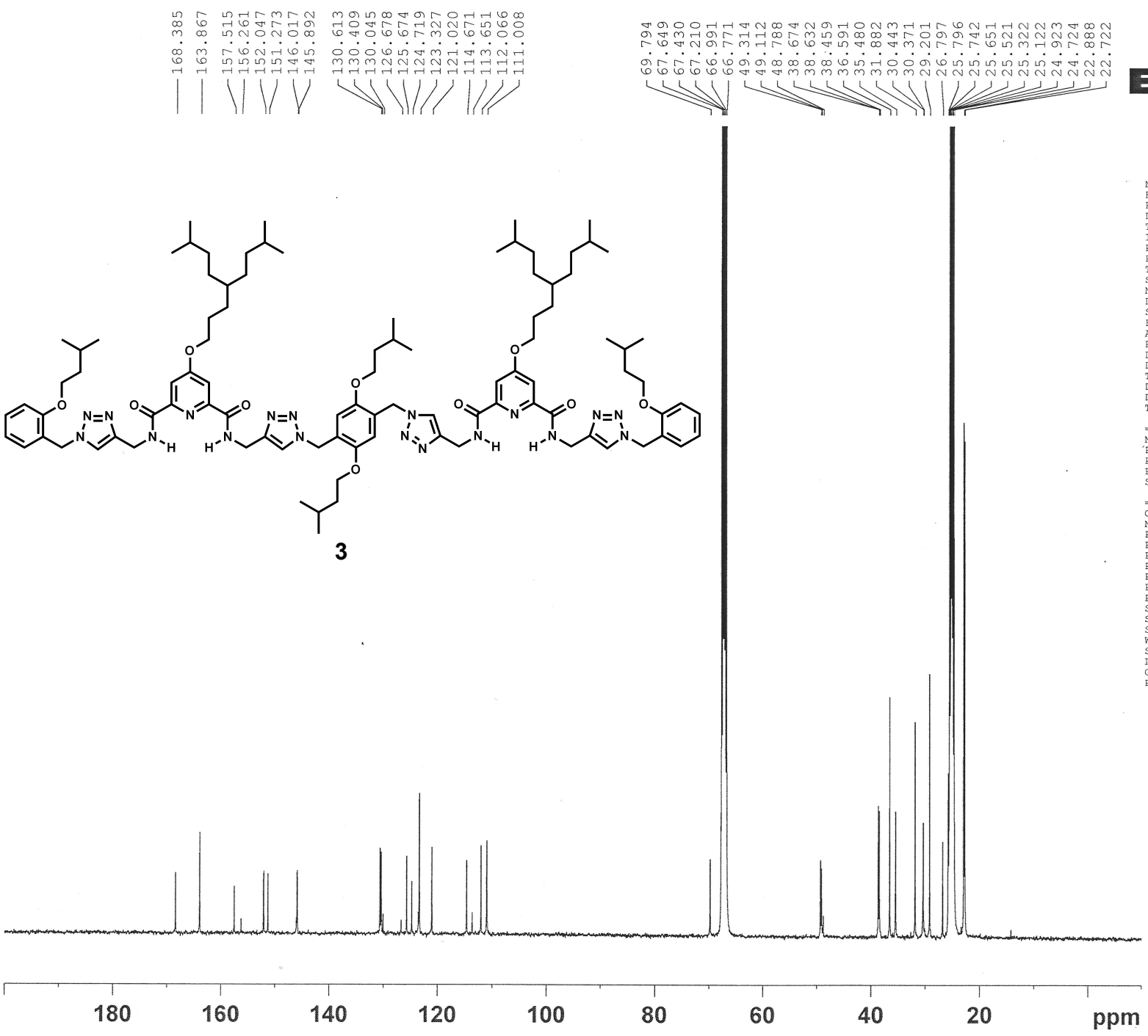




15

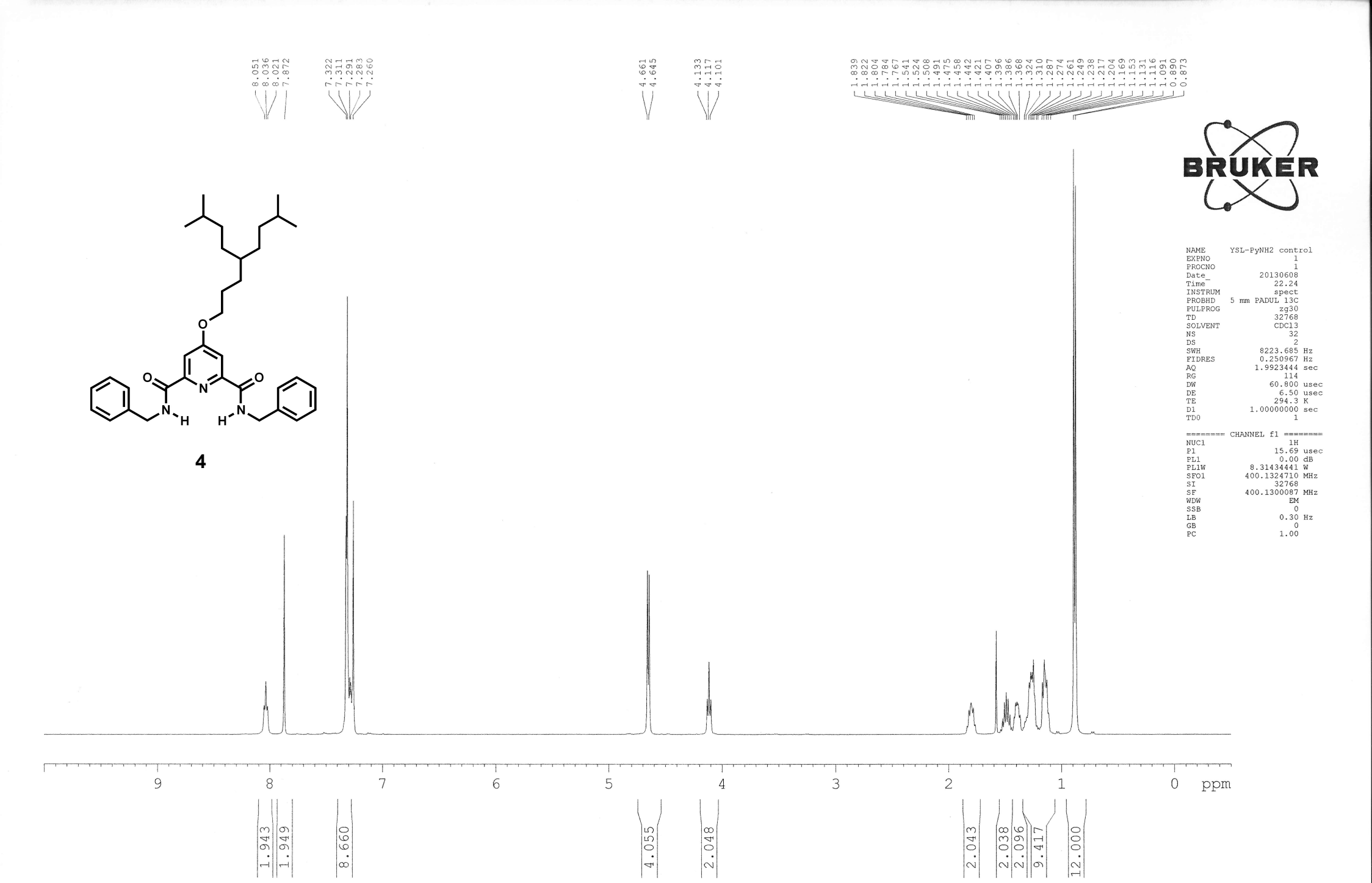

```
NAME          YSL-PyNH2 2.5mer alkyne (carbon)
EXPNO                    4
PROCNO                   1
Date_             20130412
Time                  9.45
INSTRUM              spect
PROBHD     5 mm PADUL 13C
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1000
DS                      4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                    203
DW                20.800 usec
DE                 6.50 usec
TE                295.6 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.68 usec
PL1                -0.60 dB
PL1W        41.24164963 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2                0.00 dB
PL12              15.17 dB
PL13              15.92 dB
PL2W         8.31434441 W
PL12W        0.25282964 W
PL13W        0.21272963 W
SFO2        400.1316005 MHz
SI                 32768
SF          100.6127613 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

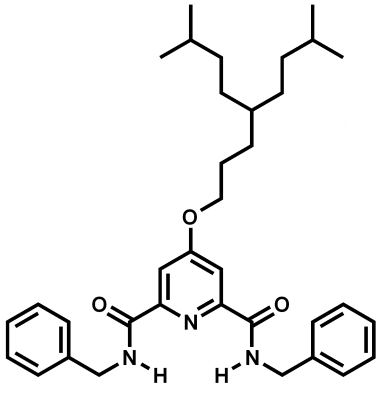

9.356
9.341
7.753
7.728
7.708
7.682
7.256
7.237
7.217
7.161
7.138
7.120
6.978
6.964
6.944
6.887
6.861
6.842
6.823
5.475
5.465
5.443
4.532
4.519
4.168
4.152
4.136
4.030
4.014
3.998
3.896
3.885
3.869
3.580
2.620
1.834
1.821
1.806
1.790
1.772
1.727
1.695
1.679
1.662
1.646
1.599
1.583
1.567
1.544
1.527
1.511
1.494
1.477
1.462
1.347
1.334
1.324
1.313
1.293
1.231
1.215
1.193
0.930
0.914
0.905
0.889
0.871
0.855
BRUKER
3
NAME YSL-PyNH2 2.5mer
EXPNO 10
PROCNO 1
Date_ 20130518
Time 22.28
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT THF
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 114
DW 60.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.69 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1292702 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
3.867
4.180
1.784
1.518
1.891
1.905
1.916
1.688
1.748
8.013
7.920
3.929
4.071
4.000
6.111
41.743
48.950



BRUKER

NAME	YSL-PyNH2 2.5mer (carbon)
EXPNO	5
PROCNO	1
Date_	20130518
Time	22.39
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	THF
NS	15000
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	295.3 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.17 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.25282964 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6125026 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40


8.051
8.036
8.021
7.872
7.322
7.311
7.291
7.283
7.260
4.661
4.645
4.133
4.117
4.101
1.839
1.822
1.804
1.784
1.767
1.541
1.524
1.508
1.491
1.475
1.458
1.442
1.421
1.407
1.396
1.386
1.368
1.324
1.310
1.287
1.274
1.261
1.249
1.238
1.217
1.204
1.169
1.153
1.131
1.116
1.091
0.890
0.873
BRUKER
4
NAME YSL-PyNH2 control
EXPNO 1
PROCNO 1
Date_ 20130608
Time 22.24
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 114
DW 60.800 usec
DE 6.50 usec
TE 294.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.69 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.943
1.949
8.660
4.055
2.048
2.043
2.038
2.096
9.417
12.000



4


```
NAME        YSL-PyNH2 control (carbon)
EXPNO                               1
PROCNO                              1
Date_                        20130608
Time                            22.30
INSTRUM                          spect
PROBHD     5 mm PADUL 13C
PULPROG                       zgpg30
TD                             65536
SOLVENT                        CDCl3
NS                              1000
DS                                 4
SWH                      24038.461 Hz
FIDRES                    0.366798 Hz
AQ                        1.3631988 sec
RG                               203
DW                          20.800 usec
DE                           6.50 usec
TE                           294.4 K
D1                     1.00000000 sec
D11                    0.03000000 sec
TD0                                1

======== CHANNEL f1 ========
NUC1                            13C
P1                            9.68 usec
PL1                          -0.60 dB
PL1W                 41.24164963 W
SFO1                100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                    waltz16
NUC2                            1H
PCPD2                        90.00 usec
PL2                           0.00 dB
PL12                         15.17 dB
PL13                         15.92 dB
PL2W                  8.31434441 W
PL12W                 0.25282964 W
PL13W                 0.21272963 W
SFO2                400.1316005 MHz
SI                           32768
SF                  100.6127645 MHz
WDW                             EM
SSB                              0
LB                            1.00 Hz
GB                               0
PC                            1.40
```