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Formation of [2]Rotaxanes by Encircling [20], [21] and [22]Crown Ethers Onto the Dibenzylammonium Dumbbell

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Electronic Supplementary Information

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1. Synthetic procedures and characterization data

Synthesis of 5: Dry THF (24 mL) was added to NaH (0.5 g, 12.5 mmol) under nitrogen atmosphere. The suspension was stirred for 5 minutes followed by the addition of pentaethylene glycol (**3**) (1 g, 4.2 mmol). After 2 hours of stirring, 1,4 - diiodobutane (1.56 g, 5.03 mmol) was added and the reaction mixture was heated at 80 ^{o}C for 48 hours. The reaction mixture was then cooled to room temperature. Cold water was added to the reaction mixture to quench the excess NaH, followed by extraction with $CHCl_3$. The organic layer was washed by brine, dried over anhydrous Na_2SO_4, and the solvent was then removed under vacuum leaving a residue which was purified by column chromatography (silica gel, acetone/hexane = 2:3) to give compound **5** (0.35 g, 29%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.85 (m, 1 H, C(H)=CH_2), 5.10-5.01 (m, 2 H, CH=C(H_2)), 3.71 (br, 2 H, OCH_2CH_2O), 3.68-3.63 (br, 14 H, OCH_2CH_2O), 3.61 (br, 4 H, OCH_2CH_2O), 3.51 (t, J = 6.9 Hz, 2 H, CH_2), 2.36 (m, 2 H, CH_2). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 135.1, 116.3, 72.5, 71.1, 70.8, 70.66, 70.60, 70.56, 70.52, 70.3, 70.1, 61.7, 34.1. HR MS (ESI): m/z Calcd for $C_{14}H_{28}O_6Na$ $[M+Na]^+$: 315.1778, found 315.1770.

Synthesis of 6: Dry THF (48 mL) was added to NaH (1.1 g, 27.5 mmol) under nitrogen atmosphere. The suspension was stirred for 5 minutes followed by the addition of pentaethylene glycol (**3**) (2 g, 8.39 mmol). After 2 hours of stirring, allyl bromide (1.52 g, 12.56 mmol) was added and stirred for 36 hours. Cold water was added to the reaction mixture to quench the excess NaH, followed by extraction with $CHCl_3$. The organic layer was washed by brine, dried over anhydrous Na_2SO_4, and the solvent was then removed under vacuum leaving a residue which was purified by column chromatography (silica gel, acetone/hexane = 2:3) to give compound **6** (0.56 g, 24%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.95 (m, 1 H, C(H)=CH_2), 5.29-5.24 (m, 1 H, CH=C(H_2)), 5.18-5.16 (m, 1 H, CH=C(H_2)), 4.03 (m, 2 H, CH_2), 3.73 (br, 2 H, OCH_2CH_2O), 3.66 (br, 14 H, OCH_2CH_2O), 3.60 (br, 4 H, OCH_2CH_2O). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 134.8, 117.1, 117.0, 72.5, 72.2, 70.63, 70.58, 70.56, 70.3, 69.4, 61.7. HR MS (ESI): m/z Calcd for $C_{13}H_{26}O_6Na$ $[M+Na]^+$: 301.1622, found 301.1623.

Synthesis of 2a: Compound **6** (0.5 g, 1.79 mmol) was dissolved in ethyl vinyl ether (10 mL). A solution of $Pd(OAc)_2$ (0.06 g, 0.089 mmol) and 1,10-phenanthroline (0.017 g, 0.098 mmol) in DCM (5 mL) was added to the solution containing compound **6** and stirred for 7 days. The mixture was filtered over celite and concentrated in vacuum leaving a residue which was purified by column chromatography (silica gel, acetone/hexane = 1:4) to give compound **2a** (0.16 g, 30%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.51 (dd, $^4J_{trans}$ = 14.5 Hz, $^4J_{cis}$ = 6.9 Hz, 1 H, OC(H)=CH_2), 5.95 (m, 1 H, C(H)=CH_2), 5.29-5.24 (m, 1 H, CH=C(H_2)), 5.18-5.16 (m, 1 H, CH=C(H_2)), 4.19-4.16 (dd, $^4J_{trans}$ = 14.5 Hz, ^{2}J = 1.9 Hz, 1 H, OCH=C(H_2)), 4.02-3.99 (br, 3 H, CH_2 & OCH=C(H_2)), 3.84 (m, 2 H, OCH_2CH_2O), 3.74 (m, 2 H, OCH_2CH_2O), 3.66-3.64 (br, 14 H, OCH_2CH_2O), 3.60 (m, 2 H, OCH_2CH_2O). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 151.7, 134.7, 117.0, 86.6, 72.2, 70.7, 70.63, 70.61, 70.58, 69.6, 69.4, 67.2. HR MS (ESI): m/z Calcd for $C_{15}H_{28}O_6Na$ $[M+Na]^+$: 327.1778, found 327.1794.

Synthesis of 2b: Dry THF (30 mL) was added to NaH (2.5 g, 62.95 mmol) under nitrogen atmosphere. The suspension was stirred for 5 minutes followed by the addition of pentaethylene glycol (**3**) (1.5 g, 6.3 mmol).

After 2 hours of stirring, allyl bromide (3.05 g, 25.18 mmol) was added and stirred for 36 hours. Cold water was added to the reaction mixture to quench the excess NaH, followed by extraction with $CHCl_3$. The organic layer was washed by brine, dried over anhydrous Na_2SO_4, and the solvent was then removed under vacuum leaving a residue which was purified by column chromatography (silica gel, acetone/hexane = 1:4) to give compound **2b** (1.01 g, 51%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.94 (m, 2 H, C(H)=CH_2), 5.28-5.24 (m, 2 H, CH=C(H_2)), 5.18-5.15 (m, 2 H, CH=C(H_2)), 4.01 (m, 4 H, CH_2), 3.66-3.64 (br, 16 H, OCH_2CH_2O), 3.60 (m, 4 H, OCH_2CH_2O). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 134.7, 116.9, 72.1, 70.54, 70.51, 69.3. HR MS (ESI): m/z Calcd for $C_{16}H_{30}O_6Na$ $[M+Na]^+$: 341.1935, found 341.1950.

Synthesis of 2c: Dry THF (10 mL) was added to NaH (0.14 g, 3.48 mmol) under nitrogen atmosphere. The suspension was stirred for 5 minutes followed by the addition of compound **5** (0.51 g, 1.74 mmol). After 2 hours of stirring, allyl bromide (0.42 g, 3.48 mmol) was added and stirred for 36 hours. Cold water was added to the reaction mixture to quench the excess NaH, followed by extraction with $CHCl_3$. The organic layer was washed by brine, dried over anhydrous Na_2SO_4, and the solvent was then removed under vacuum leaving a residue which was purified by column chromatography (silica gel, acetone/hexane = 1:4) to give compound **2c** (0.43 g, 75%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.91-5.83 (m, 1 H, C(H)=CH_2), 5.81-5.73 (m, 1 H, C(H)=CH_2), 5.25-5.21 (m, 1 H, CH=C(H_2)), 5.14-5.12 (m, 1 H, CH=C(H_2)), 5.06-5.02 (m, 1 H, CH=C(H_2)), 4.99-4.97 (m, 1 H, CH=C(H_2)), 3.98 (m, 2 H, CH_2), 3.62-3.59 (br, 16 H, OCH_2CH_2O), 3.56 (m, 4 H, OCH_2CH_2O), 3.48 (t, J = 6.9 Hz, 2 H, CH_2), 2.32 (m, 2 H, CH_2). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 135.0, 134.7, 116.9, 116.2, 72.1, 70.55, 70.51, 70.49, 70.47, 70.0, 69.3, 34.0. HR MS (ESI): m/z Calcd for $C_{17}H_{32}O_6Na$ $[M+Na]^+$: 355.2091, found 355.2107.

Synthesis of 2d: Dry THF (22 mL) was added to NaH (1.71 g, 42.80 mmol) under nitrogen atmosphere. The suspension was stirred for 5 minutes followed by the addition of pentaethylene glycol (**3**) (1.02 g, 4.28 mmol). After 2 hours of stirring, 1,4 - diiodobutane (5.30 g, 17.12 mmol) was added and the reaction mixture was heated at 80 ^{o}C for 48 hours. The reaction mixture was then cooled to room temperature. Cold water was added to the reaction mixture to quench the excess NaH, followed by extraction with $CHCl_3$. The organic layer was washed by brine, dried over anhydrous Na_2SO_4, and the solvent was then removed under vacuum leaving a residue which was purified by column chromatography (silica gel, acetone/hexane = 1:4) to give compound **2d** (0.66 g, 45%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.84 (m, 2 H, C(H)=CH_2), 5.09-5.01 (m, 4 H, CH=C(H_2)), 3.64-3.62 (br, 16 H, OCH_2CH_2O), 3.59 (m, 4 H, OCH_2CH_2O), 3.51 (t, J = 6.9 Hz, 4 H, CH_2), 2.35 (m, 2 H, CH_2). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 135.1, 116.2, 70.66, 70.61, 70.59, 70.58, 70.1, 34.1. HR MS (ESI): m/z Calcd for $C_{18}H_{34}O_6Na$ $[M+Na]^+$: 369.2248, found 369.2263.

Synthesis of 2e: Dry THF (10 mL) was added to NaH (0.185 g, 4.61 mmol) under nitrogen atmosphere. The suspension was stirred for 5 minutes followed by the addition of compound **5** (0.45 g, 1.54 mmol). After 2 hours of stirring, 5-bromo-1-pentene (0.92 g, 6.15 mmol) was added and stirred for 36 hours. Cold water was added to the reaction mixture to quench the excess NaH, followed by extraction with $CHCl_3$. The organic layer was washed by brine, dried over anhydrous Na_2SO_4, and the solvent was then removed under vacuum leaving a residue which was purified by column chromatography (silica gel, acetone/hexane = 1:4) to give compound **2e** (0.29 g, 54%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.84 (m, 2 H, C(H)=CH_2), 5.09-4.93 (m & br, 4 H, CH=C(H_2)), 3.64-3.61 (br, 16 H, OCH_2CH_2O), 3.57 (m, 4 H, OCH_2CH_2O), 3.51 (t, J = 6.9 Hz, 2 H, CH_2), 3.45 (t, J = 6.3 Hz, 2 H, CH_2), 2.35 (m, 2 H, CH_2), 2.12 (m, 2 H, CH_2), 1.66 (p, 2 H, CH_2). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 138.2, 135.1, 116.3, 114.6, 70.67, 70.63, 70.58, 70.57, 70.1, 70.0, 34.1, 30.2, 28.7. HR MS (ESI): m/z Calcd for $C_{19}H_{36}O_6Na$ $[M+Na]^+$: 383.2404, found 383.2417.

Synthesis of 2f: Dry THF (35 mL) was added to NaH (2.83 g, 70.8 mmol) under nitrogen atmosphere. The suspension was stirred for 5 minutes followed by the addition of hexaethylene glycol (**4**) (2 g, 7.08 mmol). After 2 hours of stirring, allyl bromide (3.5 g, 28.33 mmol) was added and stirred for 36 hours. Cold water was added to the reaction mixture to quench the excess NaH, followed by extraction with $CHCl_3$. The organic layer was washed by brine, dried over anhydrous Na_2SO_4, and the solvent was then removed under vacuum leaving a residue which was purified by column chromatography (silica gel, acetone/hexane = 1:4) to give compound **2f** (1.7 g, 67%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.91 (m, 2 H, C(H)=CH_2), 5.25-5.21 (m, 2 H, CH=C(H_2)), 5.14-5.12 (m, 2 H, CH=C(H_2)), 3.98 (m, 4 H, CH_2), 3.62-3.61 (br, 20 H, OCH_2CH_2O), 3.57 (m, 4 H, OCH_2CH_2O). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 134.7, 116.9, 72.1, 70.52, 70.49, 69.3. HR MS (ESI): m/z Calcd for $C_{18}H_{34}O_7Na$ $[M+Na]^+$: 385.2197, found 385.2208.

Synthesis of 10e: Compound **2e** (0.16 g, 0.44 mmol) was dissolved in dry DCM (440 mL) under nitrogen atmosphere. 2nd Generation Grubbs catalyst (0.037 g, 0.04 mmol) was added and the resulting mixture was refluxed for 60 hours. The reaction mixture was cooled followed by quenching with ethyl vinyl ether. The excess solvent was removed in vacuum and the residue was subjected to column chromatography (silica gel, MeOH/$CHCl_3$ = 1:19 (v/v)) to give the desired product **10e** (0.11 g, 75%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.50 (m, 2 H, C(H)=C(H)), 3.69 – 3.56 (br, 20 H, OCH_2CH_2O), 3.53 – 3.45 (m, 4 H, CH_2), 2.38 – 2.08 (m, 4 H, CH_2), 1.66 (m, 2 H, CH_2). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 131.3, 127.8, 71.0, 70.99, 70.94, 70.81, 70.80, 70.74, 70.72, 70.6, 70.27, 70.23, 33.0, 29.2, 29.0. HR MS (ESI): m/z Calcd for $C_{17}H_{32}O_6Na$ $[M+Na]^+$: 355.2091, found 355.2092.

Synthesis of 11e: Compound **10e** (0.19 g, 0.57 mmol) was dissolved in dry THF (12 mL) under nitrogen atmosphere. A pinch of Pd/C (cat) was added and the resulting suspension was stirred under an atmosphere of hydrogen for 16 hours. The reaction mixture was filtered through celite to remove Pd/C and concentrated in

vacuum. The crude residue was purified by column chromatography (silica gel, MeOH/$CHCl_3$ = 1:19 (v/v)) to give the desired product **11e** (0.17 g, 89%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 3.70 (br, 4 H, OCH_2CH_2O), 3.68 (br, 8 H, OCH_2CH_2O), 3.64 (m, 4 H, OCH_2CH_2O), 3.58 (m, 4 H, OCH_2CH_2O), 3.48 (t, J = 6.3 Hz, 4 H, CH2), 1.62–1.54 (br, 4 H, CH_2), 1.43-1.30 (br, 6 H, CH_2). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 71.08, 71.06, 70.87, 70.80, 70.7, 70.4, 29.5, 28.9, 26.2. HR MS (ESI): m/z Calcd for $C_{17}H_{34}O_6Na$ $[M+Na]^+$: 357.2248, found 357.2257.

Synthesis of 10f: Compound **2f** (0.15 g, 0.41 mmol) was dissolved in dry DCM (410 mL) under nitrogen atmosphere. 2nd Generation Grubbs catalyst (0.035 g, 0.041 mmol) was added and the resulting mixture was refluxed for 60 hours. The reaction mixture was cooled followed by quenching with ethyl vinyl ether. The excess solvent was removed in vacuum and the residue was subjected to column chromatography (silica gel, MeOH/$CHCl_3$ = 1:19 (v/v)) to give the desired product **10f** (0.08 g, 58%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 5.82 (m, 2 H, C(H)=C(H)), 4.05 (m, 4 H, CH_2), 3.68-3.65 (br, 20 H, OCH_2CH_2O), 3.61 (m, 4 H, OCH_2CH_2O). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 129.3, 70.95, 70.92, 70.79, 70.76, 69.4. HR MS (ESI): m/z Calcd for $C_{16}H_{30}O_7Na$ $[M+Na]^+$: 357.1884, found 357.1892.

Synthesis of 11f: Compound **10f** (0.18 g, 0.53 mmol) was dissolved in dry THF (12 mL) under nitrogen atmosphere. A pinch of Pd/C (cat) was added and the resulting suspension was stirred under an atmosphere of hydrogen for 16 hours. The reaction mixture was filtered through celite to remove Pd/C and concentrated in vacuum. The crude residue was purified by column chromatography (silica gel, MeOH/$CHCl_3$ = 1:19 (v/v)) to give the desired product **11f** (0.16 g, 91%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 3.69 (br, 8 H, OCH_2CH_2O), 3.67-3.64 (br, 12 H, OCH_2CH_2O), 3.59 (m, 4 H, OCH_2CH_2O), 3.51 (t, J = 5.6 Hz, 4 H, CH_2), 1.66 (p, J = 3.1 Hz, 4 H, CH_2). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 70.99, 70.97, 70.92, 70.82, 70.79, 70.75, 70.2, 26.4. HR MS (ESI): m/z Calcd for $C_{16}H_{32}O_7Na$ $[M+Na]^+$: 359.2040, found 359.2045.

Synthesis of [2]R_{20C6}, 8b·PF_6: Compound **1·PF_6** (0.06 g, 0.174 mmol) and compound **2b** (0.11 g, 0.349 mmol) were dissolved in the mixed solvent (40 mL, $CHCl_3$/CH_3CN = 3:1 (v/v)). The solution was stirred for 24 hours and the solvent was then removed under vacuum without heating and then the residue (**7b·PF_6**) was dissolved in dry DCM (350 mL, 0.001 M) under nitrogen atmosphere. 2nd Generation Grubbs catalyst (0.03 g, 0.034 mmol) was added and the resulting mixture was refluxed for 60 hours. The reaction mixture was cooled followed by quenching with ethyl vinyl ether. The excess solvent was removed in vacuum and the residue was subjected to column chromatography (silica gel, MeOH/$CHCl_3$ = 1:9 (v/v)) to give the desired product **8b·PF_6** (0.07 g, 64%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.02 (br, 2 H, NH_2^+), 7.47-7.38 (br, 10 H, ph), 5.81 (br, 2 H, C(H)=C(H)), 4.48 (m, 4 H, $C(H_2)NH_2^+$), 3.97 (br, 4 H, CH_2), 3.75 (br, 4 H, OCH_2CH_2O), 3.63 (br, 4 H, OCH_2CH_2O), 3.50 (br, 4 H, OCH_2CH_2O), 3.38-3.35 (br, 8 H, OCH_2CH_2O). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 132.8, 132.1, 130.5, 129.5, 128.9, 128.7, 71.3, 70.79, 70.73, 70.6, 70.4, 69.9, 50.8. HR MS (ESI): m/z Calcd for $C_{28}H_{42}NO_6$ $[M-PF_6]^+$: 488.3007, found 488.3012.

Synthesis of [2]R_{21C6}, 8c·PF_6: Compound **1·PF_6** (0.07 g, 0.204 mmol) and compound **2c** (0.135 g, 0.407 mmol) were dissolved in the mixed solvent (40 mL, $CHCl_3/CH_3CN$ = 3:1 (v/v)). The solution was stirred for 24 hours and the solvent was then removed under vacuum without heating and then the residue (**7c·PF_6**) was dissolved in dry DCM (410 mL, 0.001 M) under nitrogen atmosphere. 2nd Generation Grubbs catalyst (0.035 g, 0.04 mmol) was added and the resulting mixture was refluxed for 60 hours. The reaction mixture was cooled followed by quenching with ethyl vinyl ether. The excess solvent was removed in vacuum and the residue was subjected to column chromatography (silica gel, MeOH/$CHCl_3$ = 1:9 (v/v)) to give the desired product **8c·PF_6** (0.115 g, 87%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.89 (br, 2 H, NH_2^+), 7.44-7.41 (br, 10 H, ph), 5.82 (m, 2 H, C(H)=C(H)), 4.48-4.37 (m, 4 H, $C(H_2)NH_2^+$), 4.00 (br, 2 H, CH_2), 3.72 (br, 4 H, OCH_2CH_2O), 3.68-3.65 (br, 4 H, OCH_2CH_2O), 3.61 (t, J = 5 Hz, 2 H, OCH_2CH_2O), 3.57 (m, 2 H, OCH_2CH_2O), 3.41 (m, 2 H, OCH_2CH_2O), 3.34 (m, 2 H, OCH_2CH_2O), 3.18 (m, 2 H, OCH_2CH_2O), 3.13 (m, 2 H, OCH_2CH_2O), 3.09 (m, 2 H, CH2), 2.46 (m, 2 H, CH_2). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 136.5, 131.9, 130.1, 129.6, 128.9, 127.8, 72.5, 71.97, 71.88, 71.5, 71.27, 71.21, 70.7, 70.43, 70.39, 70.30, 70.24, 70.21, 51.3, 33.6. HR MS (ESI): m/z Calcd for $C_{29}H_{44}NO_6$ $[M\text{-}PF_6]^+$: 502.3163, found 502.3164.

Synthesis of [2]R_{22C6}, 8d·PF_6: Compound **1·PF_6** (0.075 g, 0.218 mmol) and compound **2d** (0.151 g, 0.436 mmol) were dissolved in the mixed solvent (40 mL, $CHCl_3/CH_3CN$ = 3:1 (v/v)). The solution was stirred for 24 hours and the solvent was then removed under vacuum without heating and then the residue (**7d·PF_6**) was dissolved in dry DCM (440 mL, 0.001 M) under nitrogen atmosphere. 2nd Generation Grubbs catalyst (0.038 g, 0.04 mmol) was added and the resulting mixture was refluxed for 60 hours. The reaction mixture was cooled followed by quenching with ethyl vinyl ether. The excess solvent was removed in vacuum and the residue was subjected to column chromatography (silica gel, MeOH/$CHCl_3$ = 1:9 (v/v)) to give the desired product **8d·PF_6** (0.10 g, 70%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.70 (br, 2 H, NH_2^+), 7.45-7.40 (br, 10 H, ph), 5.55 (br, 2 H, C(H)=C(H)), 4.46 (br, 4 H, $C(H_2)NH_2^+$), 3.71-3.59 (br, 12 H, OCH_2CH_2O), 3.49-3.45 (br, 4 H, OCH_2CH_2O), 3.24 (br, 4 H, OCH_2CH_2O), 3.08-3.03 (br, 4 H, CH_2), 2.38 (br, 4 H, CH_2). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 131.7, 130.5, 130.1, 129.91, 129.88, 129.5, 128.9, 71.84, 71.79, 71.73, 71.6, 71.2, 71.1, 70.5, 70.4, 70.33, 70.28, 52.4, 52.1, 32.9. HR MS (ESI): m/z Calcd for $C_{30}H_{46}NO_6$ $[M\text{-}PF_6]^+$: 516.3320, found 516.3331.

Synthesis of [2]R_{20C6H2}, 9b·PF_6: Compound **8b·PF_6** (0.07 g, 0.11 mmol) was dissolved in dry THF (12 mL) under nitrogen atmosphere. A pinch of Pd/C (cat) was added and the resulting suspension was stirred under an atmosphere of hydrogen for 16 hours. The reaction mixture was filtered through celite to remove Pd/C and concentrated in vacuum. The crude residue was purified by column chromatography (silica gel, MeOH/$CHCl_3$ = 1:9 (v/v)) to give the desired product **9b·PF_6** (0.047 g, 67%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.37 (br, 2 H, NH_2^+), 7.52-7.38 (br, 10 H, ph), 4.61 (br, 4 H, $C(H_2)NH_2^+$), 3.78-3.70 (br, 12 H, OCH_2CH_2O), 3.60 (br, 4 H, OCH_2CH_2O), 3.48 (br, 4 H, OCH_2CH_2O), 3.18 (br, 4 H, CH_2), 1.11 (br, 4 H, CH_2). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 132.3, 131.0, 129.5, 128.7, 71.6, 70.9, 70.8, 70.2, 70.1, 50.9, 25.0. HR MS (ESI): m/z Calcd for $C_{28}H_{44}NO_6$ $[M\text{-}PF_6]^+$: 490.3163, found 490.3167.

Synthesis of [2]R_{21C6H2}, 9c·PF_6: Compound **8c·PF_6** (0.07 g, 0.108 mmol) was dissolved in dry THF (12 mL) under nitrogen atmosphere. A pinch of Pd/C (cat) was added and the resulting suspension was stirred under an atmosphere of hydrogen for 16 hours. The reaction mixture was filtered through celite to remove Pd/C and concentrated in vacuum. The crude residue was purified by column chromatography (silica gel, MeOH/$CHCl_3$ = 1:9 (v/v)) to give the desired product **9c·PF_6** (0.05 g, 72%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.02 (br, 2 H, NH_2^+), 7.47-7.39 (br, 10 H, ph), 4.57 (m, 4 H, $C(H_2)NH_2^+$), 3.73-3.68 (br, 8 H, OCH_2CH_2O), 3.56-3.55 (br, 8 H, OCH_2CH_2O), 3.28 (m, 4 H, OCH_2CH_2O), 3.03 (s, 4 H, CH_2), 1.70-1.62 (br, 6 H, CH_2). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 131.7, 130.3, 129.6, 128.8, 72.1, 72.0, 71.2, 71.1, 70.19, 70.14, 51.4, 30.0, 25.3. HR MS (ESI): m/z Calcd for $C_{29}H_{46}NO_6$ $[M\text{-}PF_6]^+$: 504.3320, found 504.3334.

Synthesis of [2]R_{22C6H2}, 9d·PF_6: Compound **8d·PF_6** (0.1 g, 0.15 mmol) was dissolved in dry THF (12 mL) under nitrogen atmosphere. A pinch of Pd/C (cat) was added and the resulting suspension was stirred under an atmosphere of hydrogen for 16 hours. The reaction mixture was filtered through celite to remove Pd/C and concentrated in vacuum. The crude residue was purified by column chromatography (silica gel, MeOH/$CHCl_3$ = 1:9 (v/v)) to give the desired product **9d·PF_6** (0.081 g, 81%). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.95 (br, 2 H, NH_2^+), 7.48-7.43 (br, 10 H, ph), 4.48 (m, 4 H, $C(H_2)NH_2^+$), 3.75-3.72 (br, 8 H, OCH_2CH_2O), 3.59-3.55 (br, 8 H, OCH_2CH_2O), 3.21 (m, 4 H, OCH_2CH_2O), 2.95 (s, 4 H, CH_2), 1.63 (br, 4 H, CH_2), 1.50 (br, 4 H, CH_2). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 131.3, 130.1, 129.7, 128.9, 72.1, 71.1, 71.0, 70.8, 70.3, 70.2, 51.9, 29.4, 25.4. HR MS (ESI): m/z Calcd for $C_{30}H_{48}NO_6$ $[M\text{-}PF_6]^+$: 518.3476, found 518.3484.

2. Stacked partial ^{1}H NMR spectra of 7a·PF$_6$

Mixing of **2a** with **1·PF$_6$** in $CHCl_3/CH_3CN$ (3:1) leads to decomposition of the **2a** presumably due to the acid (-NH_2^+-) promoted oligomerization of vinyl ether units in **2a**.

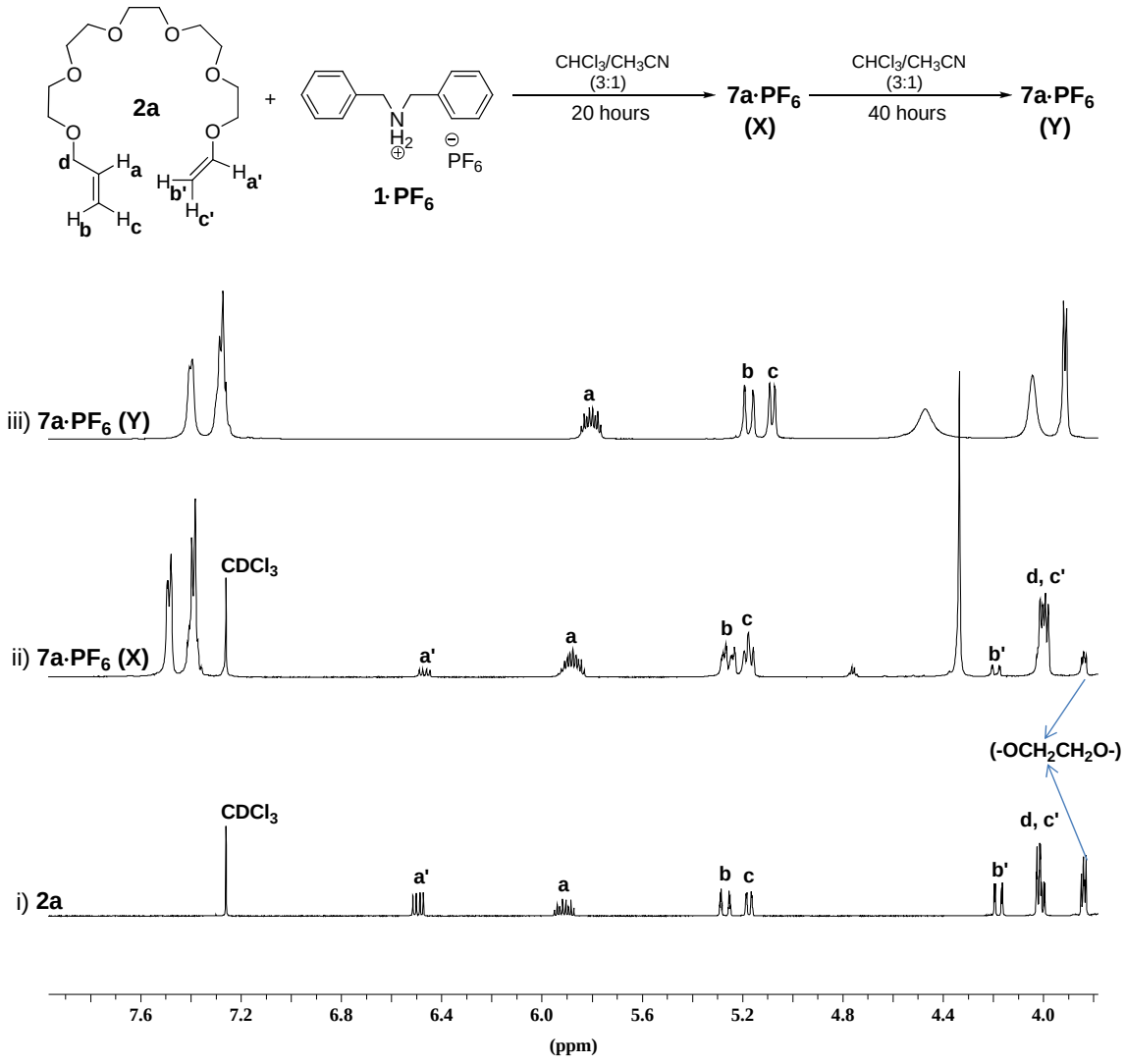


Figure S1. Partial ^{1}H NMR spectra (500 MHz, $CDCl_3$) of i) **2a**, ii) **7a·PF$_6$** (**X**), obtained after 20 hours of stirring of **1·PF$_6$** with two equivalents of **2a** at room temperature in a mixed solvent system (MSS, $CHCl_3$:CH_3CN = 3:1). The reduced intensity of **a'**, **b'** and **c'** compared to **a**, **b** and **c** suggests decomposition of vinyl ether moiety of **2a** in presence of **1·PF$_6$**. iii) **7a·PF$_6$** (**Y**), obtained when the same mixture was continued to stir at room temperature for another 40 hours. The absence of peaks corresponding to vinyl ether moiety indicates complete decomposition of the electron rich vinyl ether of **2a** due to -NH_2^+- moiety of **1·PF$_6$**, rendering **7a·PF$_6$** unfit for ring closing metathesis.

3. HR ESI mass spectra of [2]rotaxanes

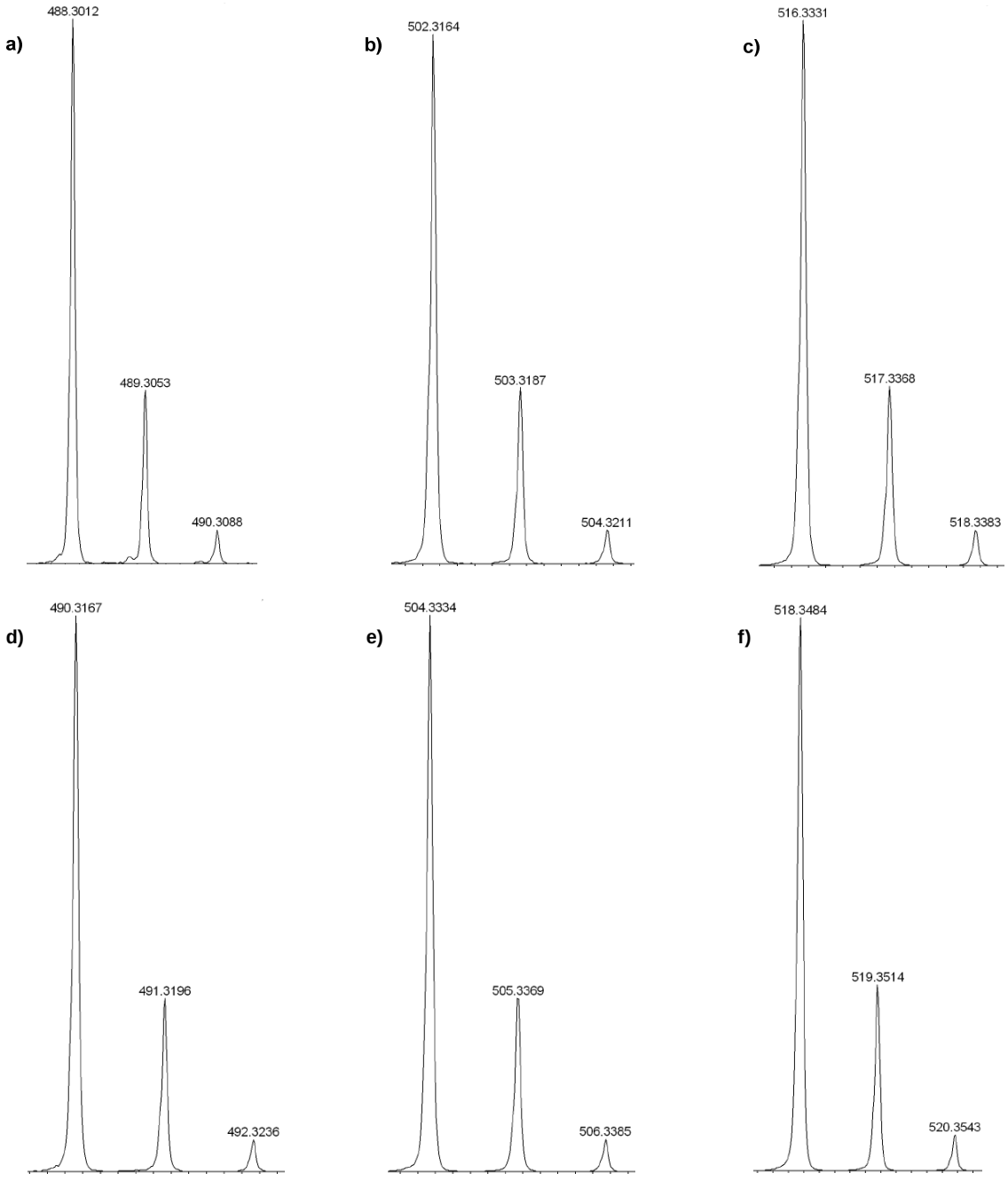


Figure S2. HR ESI mass spectra of a) **8b**·PF_6; b) **8c**·PF_6; c) **8d**·PF_6; d) **9b**·PF_6; e) **9c**·PF_6 and f) **9d**·PF_6 show the purity of the obtained [2]rotaxanes.

4. Characterization of 8f·PF_6

4.1 Stacked partial ^{1}H NMR spectra in different solvents

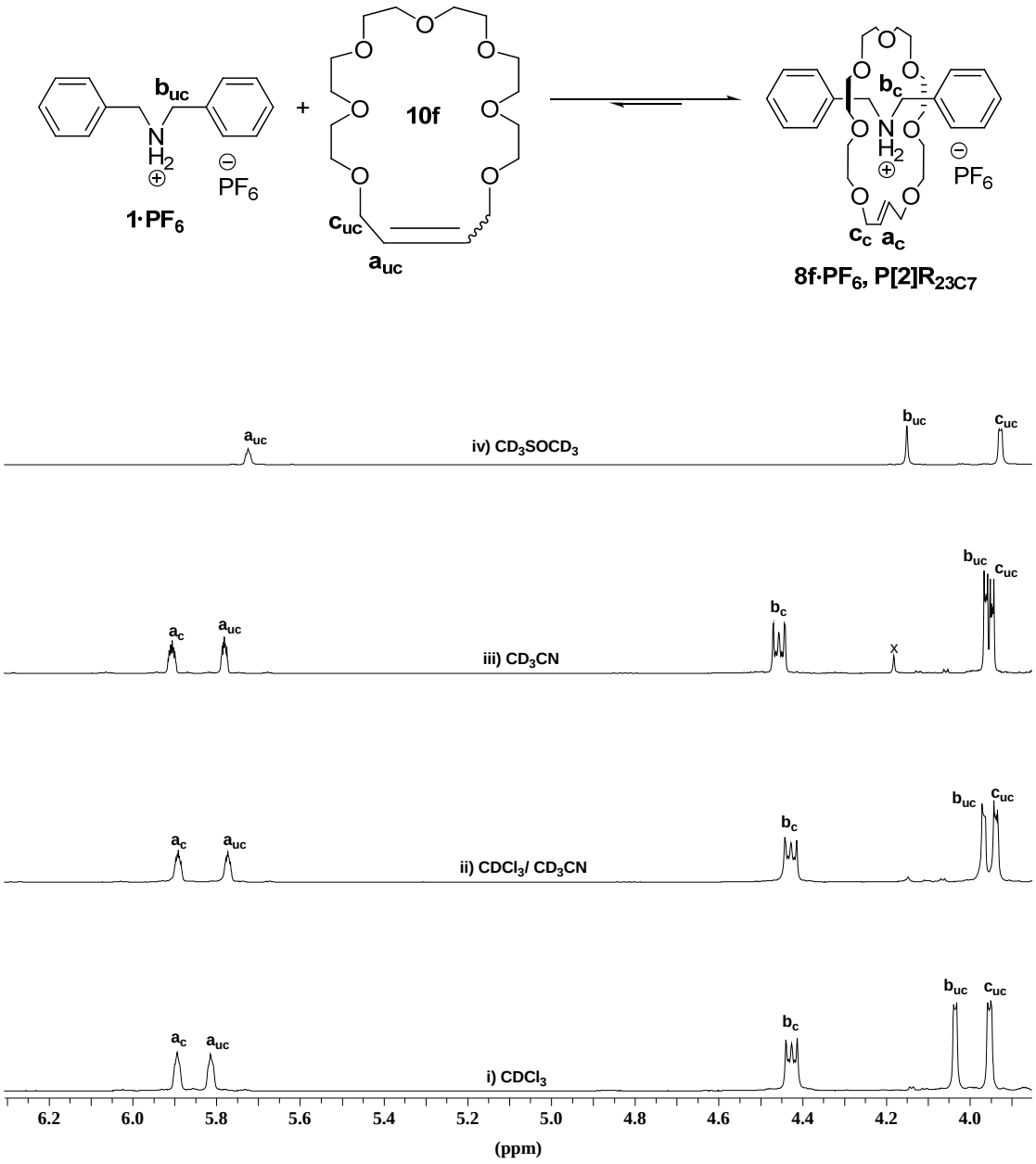


Figure S3. Stacked partial ^{1}H NMR spectra (500 MHz) of the above equilibrium reaction mixture at room temperature in different solvent systems (i - iv). The peaks of interest ($\mathbf{a_c}$, $\mathbf{a_{uc}}$, $\mathbf{b_c}$, $\mathbf{b_{uc}}$) were zoomed to appreciate the effect of solvent system on the position of the equilibrium. The absence of peaks corresponding to complexed species in CD_3SOCD_3 confirmed that **8f·PF_6** is a pseudorotaxane species at room temperature.

4.2 ESI-MS

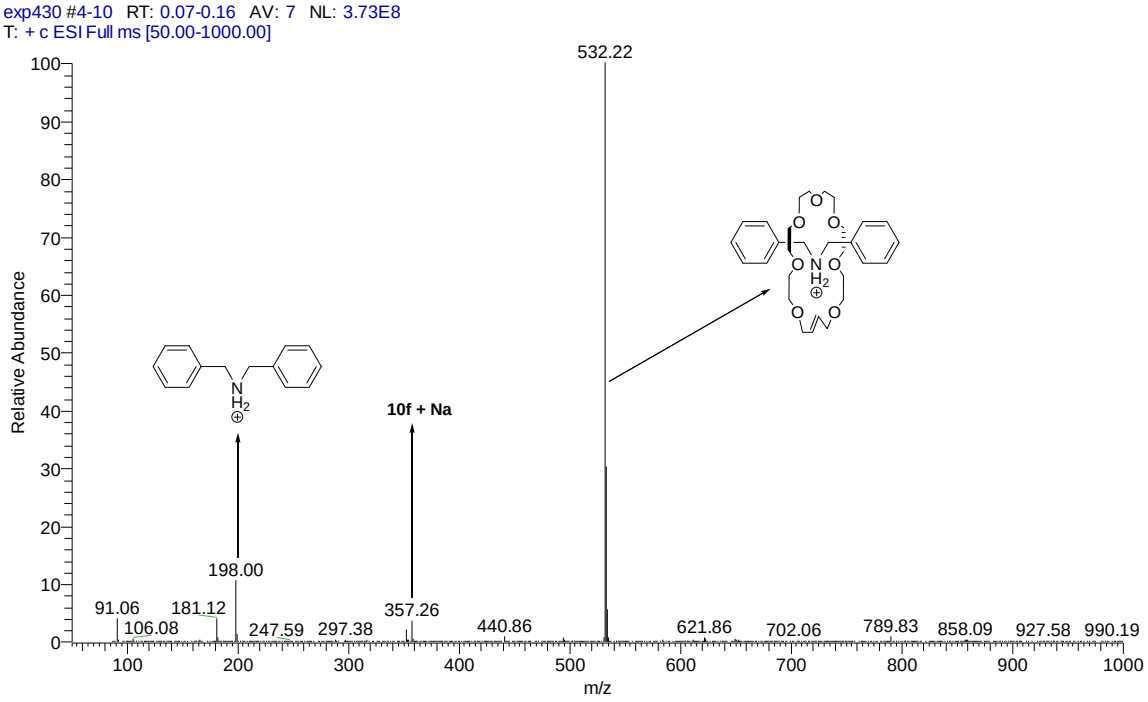


Figure S4. ESI mass spectrum of the equilibrium reaction mixture in $CHCl_3$ displayed base peak at m/z 532.2 which corresponds to **8f**·$\mathbf{PF_6}$ without its counterion. Very weak intensity peaks for **10f** (357.2) and **1**·$\mathbf{PF_6}$ (198) was also observed indicating **8f**·$\mathbf{PF_6}$ to be a strong pseudorotaxane complex at room temperature.

5. Characterization of 9f·PF_6

5.1 Stacked partial ^{1}H NMR spectra in different solvents

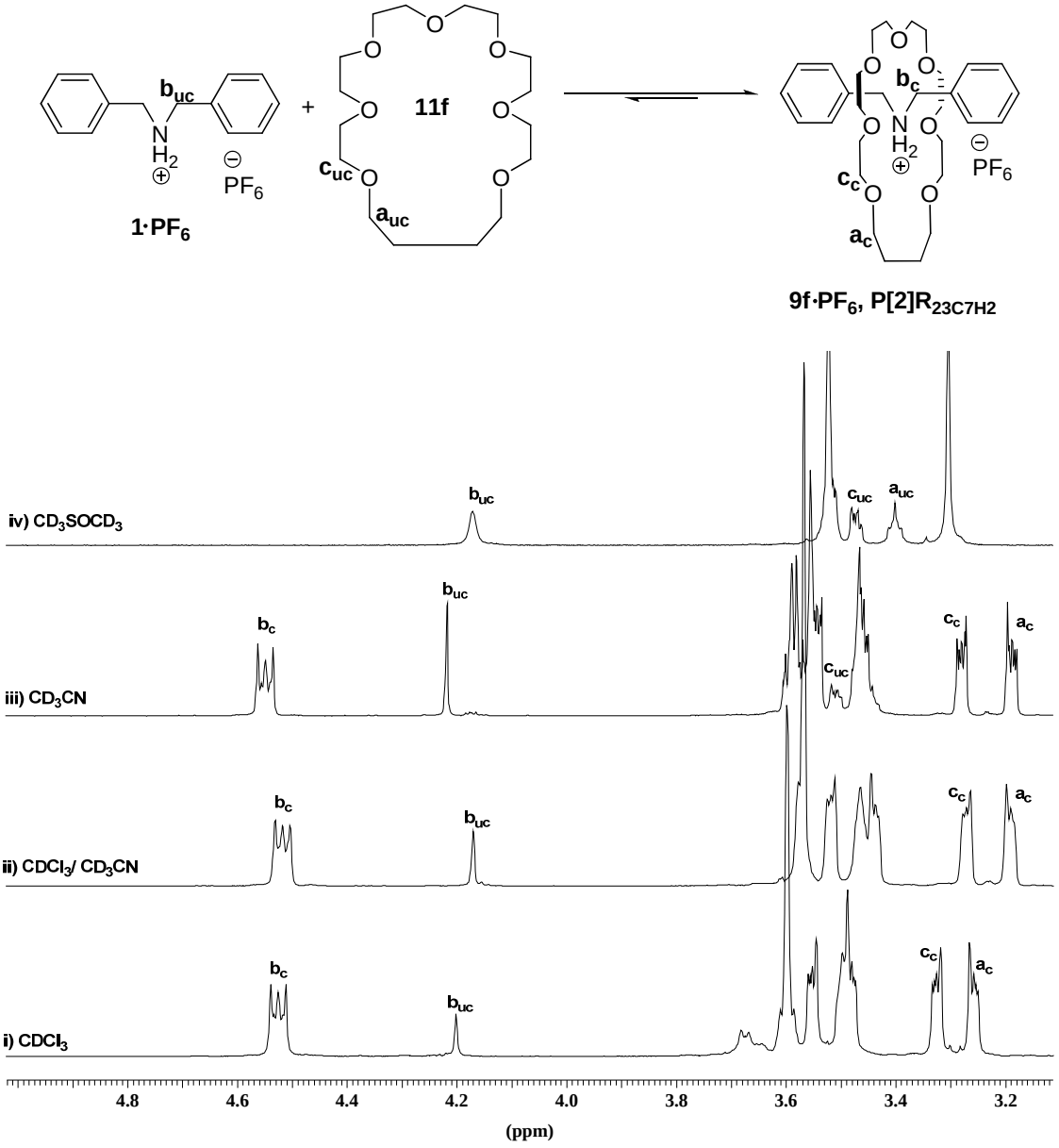


Figure S5. Stacked partial ^{1}H NMR spectra (500 MHz) of the above equilibrium reaction mixture at room temperature in different solvent systems (i - iv). The peaks of interest ($\mathbf{a_c}$, $\mathbf{a_{uc}}$, $\mathbf{b_c}$, $\mathbf{b_{uc}}$) were zoomed to appreciate the effect of solvent system in determining the position of the equilibrium. Given that the peaks $\mathbf{b_c}$ and $\mathbf{b_{uc}}$ are distinctly separated in all the solvents, we have chosen the relative intensity of $\mathbf{b_c}$ and $\mathbf{b_{uc}}$ as the basis for determining and comparing the association constant of **9f·PF_6** in different solvent systems. The initial concentrations of both **1·PF_6** and **11f** are 1.45 x 10^{-2} M. The absence of peaks corresponding to complexed species in CD_3SOCD_3 confirmed that **9f·PF_6** is a pseudorotaxane species at room temperature.

5.2 Determination of association constants in different solvents

Table S1. Effect of solvent system on the association constants K_a of the pseudorotaxane species **9f·PF$_6$** at 300 K.

Entry	Solvent	K_a [M^{-1}][a]	ΔG [kcal mol^{-1}][b]
1	$CDCl_3$	4.6×10^3	- 5.03
2	$CDCl_3/CD_3CN$ (1:1)	1.9×10^3	- 4.50
3	CD_3CN	5.8×10^2	- 3.79
4	CD_3SOCD_3	0	-

[a] Association constants K_a were evaluated from the relative intensity of the peaks $\mathbf{b_c}$ and $\mathbf{b_{uc}}$ in each solvent system using the expression K_a = [**9f·PF$_6$**]/([**1·PF$_6$**][**11f**]). [b] The free energies of complexation in all the four solvent systems were calculated by applying the equation $\Delta G = -RT\ln K_a$.

5.3 ESI-MS

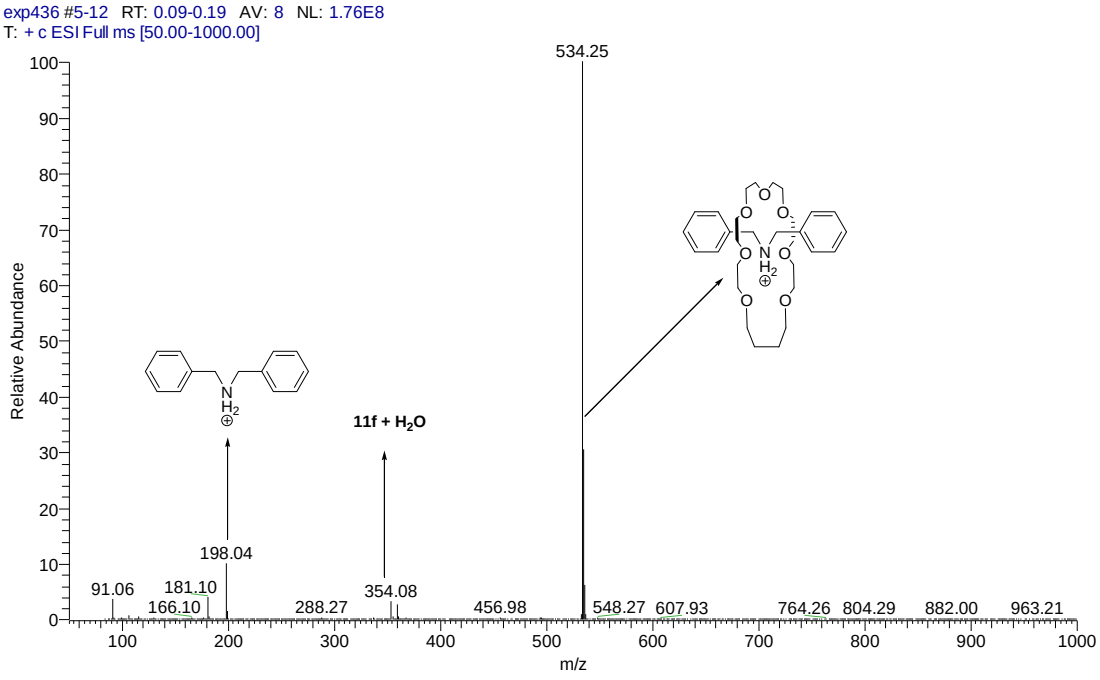


Figure S6. ESI mass spectrum of the equilibrium reaction mixture in $CHCl_3$ displayed base peak at m/z 534.2 which corresponds to **9f·PF$_6$** without its counterion. Weak intensity peaks for **11f** (354) and **1·PF$_6$** (198) was also observed indicating **9f·PF$_6$** to be a strong pseudorotaxane complex at room temperature.

6. Characterization of 9e·PF_6

6.1 Stacked partial ^{1}H NMR spectra in different solvents

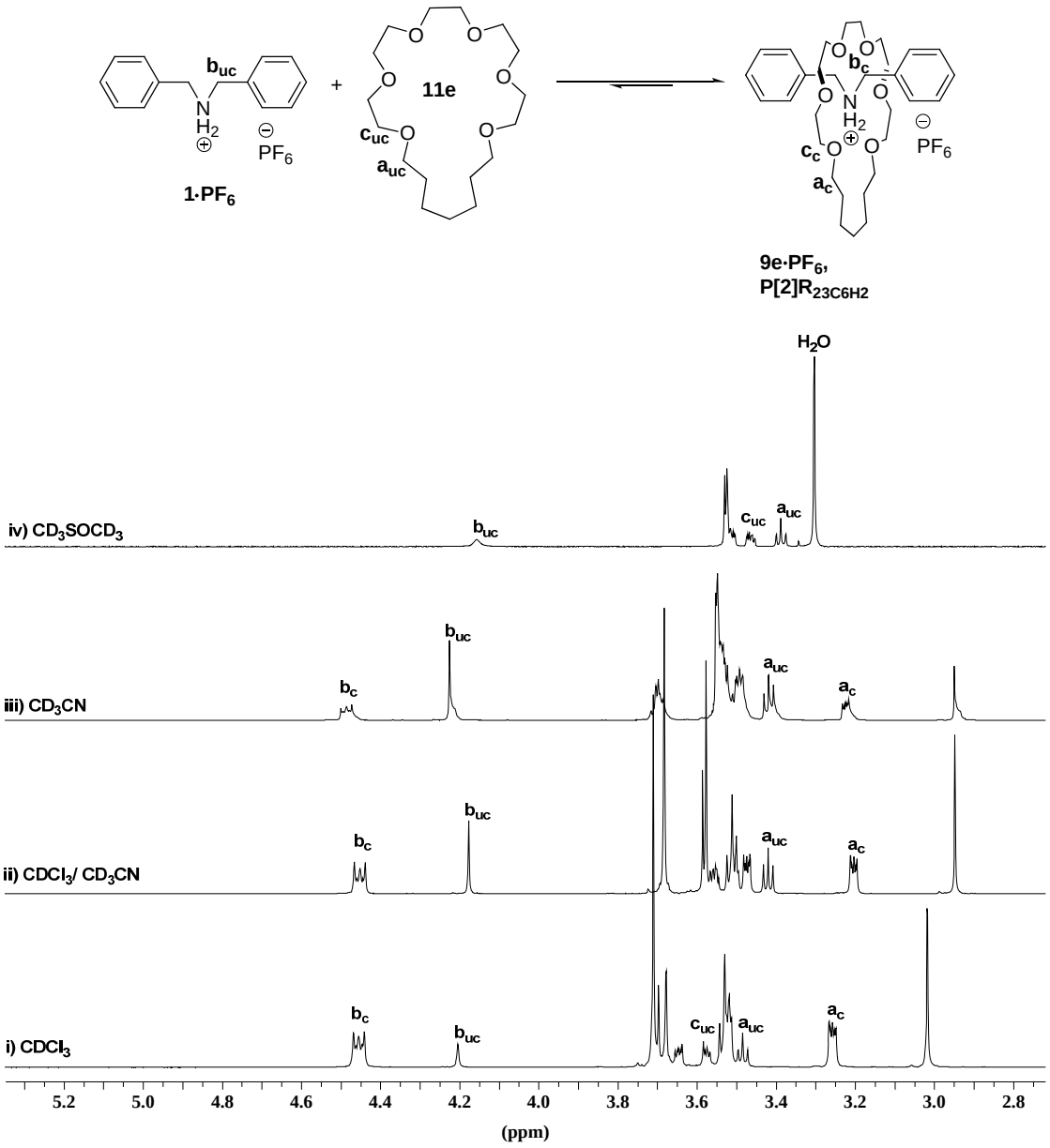


Figure S7. Stacked partial ^{1}H NMR spectra (500 MHz) of the above equilibrium reaction mixture at room temperature in different solvent systems (i - iv). The peaks of interest ($\mathbf{a_c}$, $\mathbf{a_{uc}}$, $\mathbf{b_c}$, $\mathbf{b_{uc}}$) were zoomed to appreciate the effect of solvent system in determining the position of the equilibrium. Given that the peaks $\mathbf{b_c}$ and $\mathbf{b_{uc}}$ are distinctly separated in all the solvents, we have chosen the relative intensity of $\mathbf{b_c}$ and $\mathbf{b_{uc}}$ as the basis for determining and comparing the association constant of **9e·PF_6** in different solvent systems. The initial concentrations of both **1·PF_6** and **11e** are 1.45 x 10^{-2} M. The absence of peaks corresponding to complexed species in CD_3SOCD_3 confirmed that **9e·PF_6** is a pseudorotaxane species at room temperature.

6.2 Determination of association constants in different solvents

Table S2. Effect of solvent system on the association constants K_a of the pseudorotaxane species **9e·PF$_6$** at 300 K.

Entry	Solvent	K_a [M^{-1}][a]	ΔG [kcal mol^{-1}][b]
1	$CDCl_3$	2.8×10^3	- 4.74
2	$CDCl_3/CD_3CN$ (1:1)	5.7×10^2	- 3.78
3	CD_3CN	1.0×10^2	- 2.76
4	CD_3SOCD_3	0	-

[a] Association constants K_a were evaluated from the relative intensity of the peaks **b$_c$** and **b$_{uc}$** for each solvent system using the expression K_a = [**9e·PF$_6$**]/([**1·PF$_6$**][**11e**]). [b] The free energies of complexation in all the four solvent systems were calculated by applying the equation $\Delta G = -RT\ln K_a$.

6.3 ESI-MS

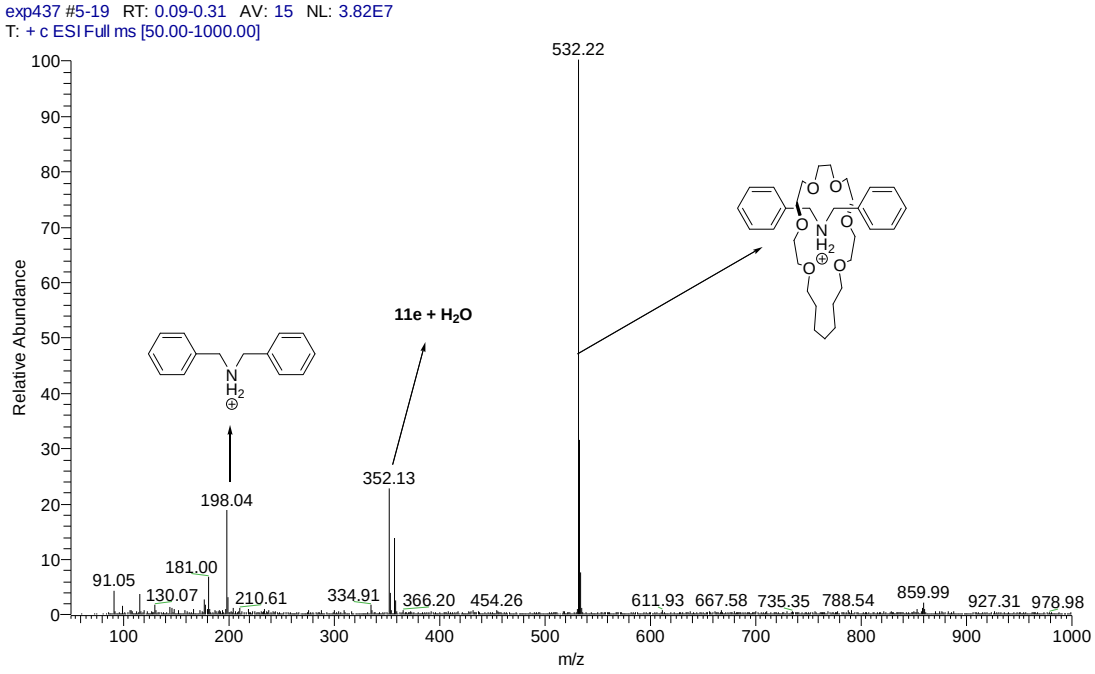
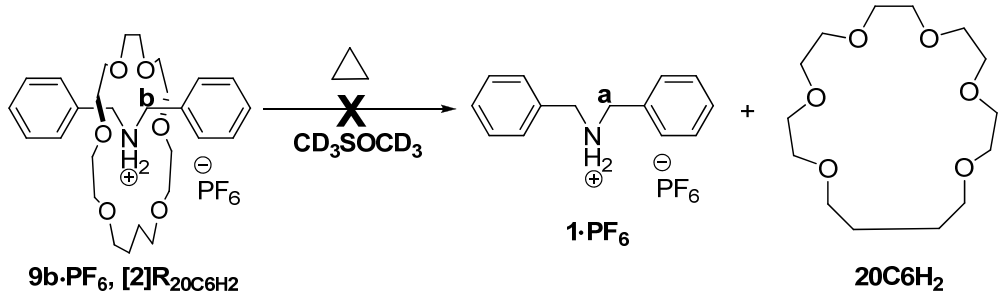


Figure S8. ESI mass spectrum of the equilibrium reaction mixture in $CHCl_3$ displayed base peak at m/z 532.2 which corresponds to **9e·PF$_6$** without its counterion. Weak intensity peaks for **11e** (352.1) and **1·PF$_6$** (198) was also observed indicating **9e·PF$_6$** to be a strong pseudorotaxane complex at room temperature.

7. Variable temperature ^{1}H NMR spectra in DMSO-d6 of

7.1 [2]Rotaxane 9b·PF$_6$



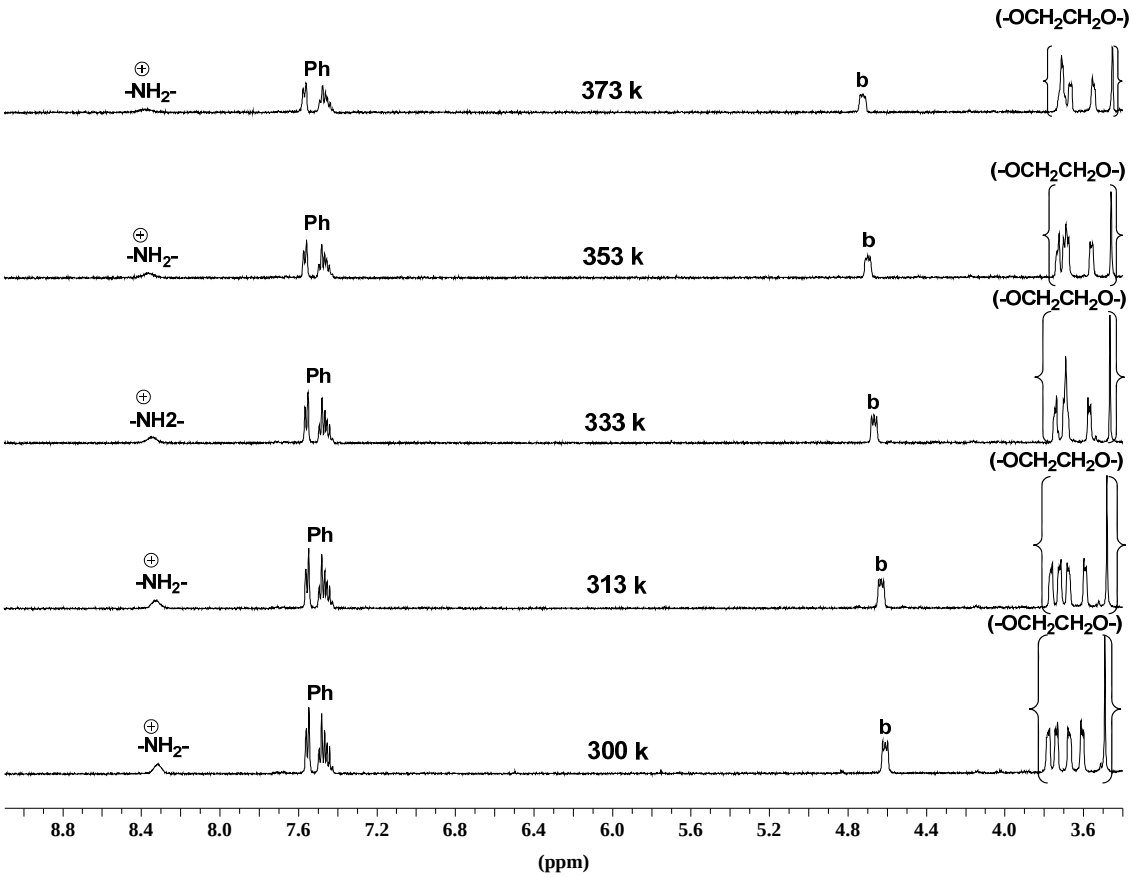


Figure S9. Stacked partial ^{1}H NMR spectra (500 MHz, CD_3SOCD_3) of **9b·PF$_6$** at different temperatures. The peaks of interest (**b** & -**NH$_2^+$**-) were zoomed to appreciate the effect of thermal energy in dethreading of [20]C6H$_2$ crown ether from **9b·PF$_6$**. Contrary to Figure 8 for **9d·PF$_6$**, we did not observe any dethreading of [20]C6H$_2$ on increasing the temperature to even 373 K, implying that **9b·PF$_6$** is a kinetically stable [2]rotaxane not only at room temperature but also at 373 K.

7.2 [2]Rotaxane 9c·PF_6

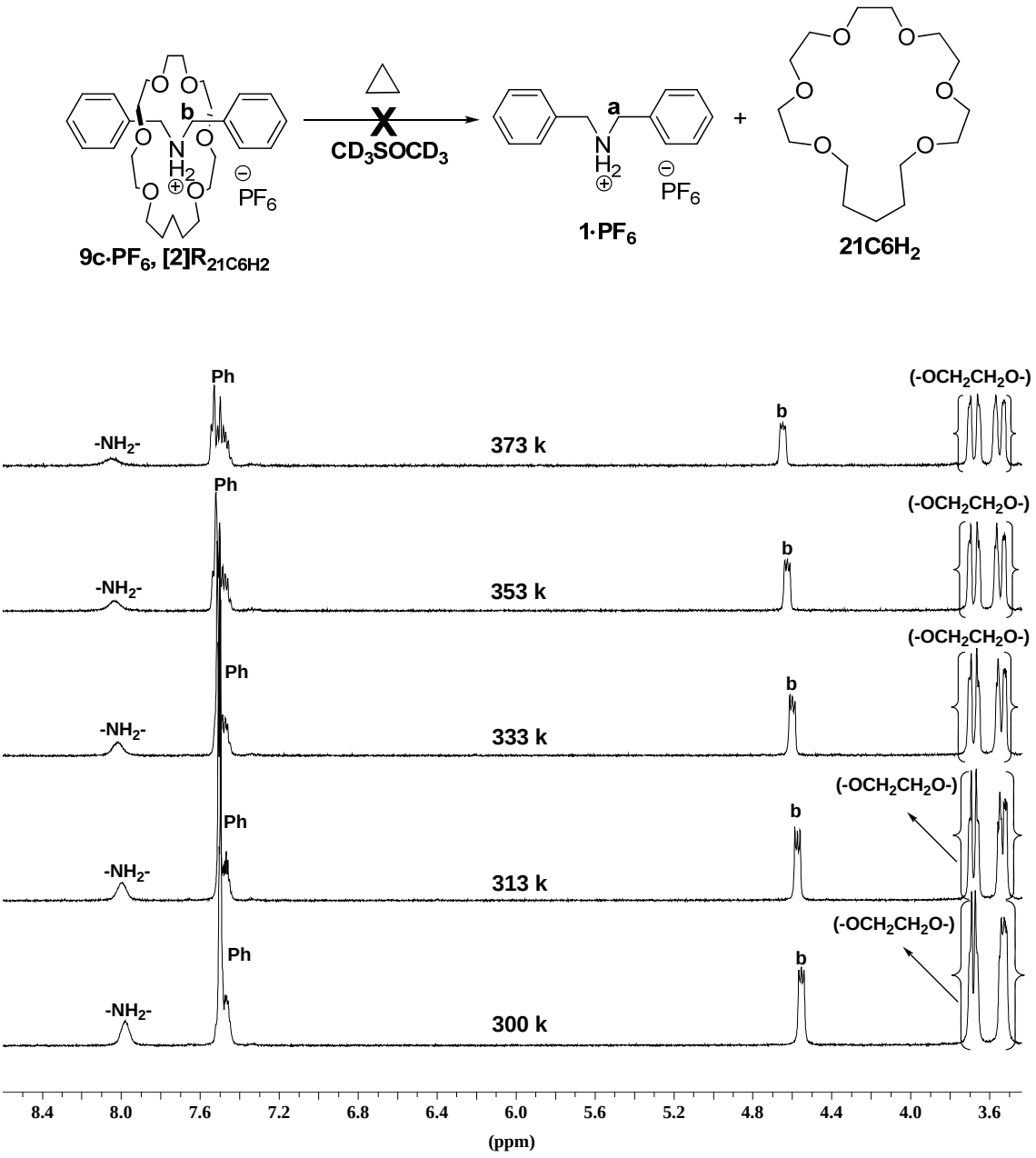
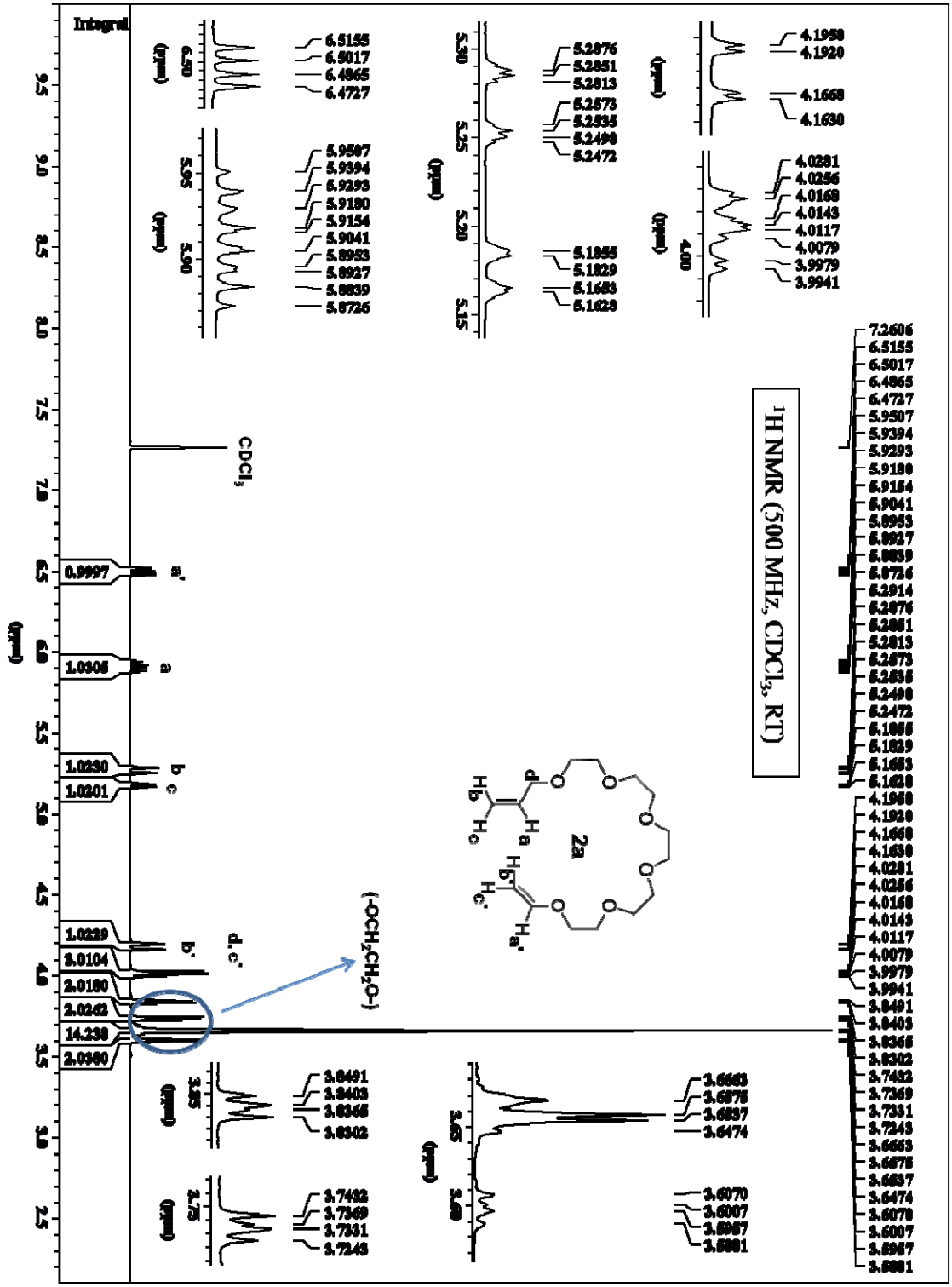
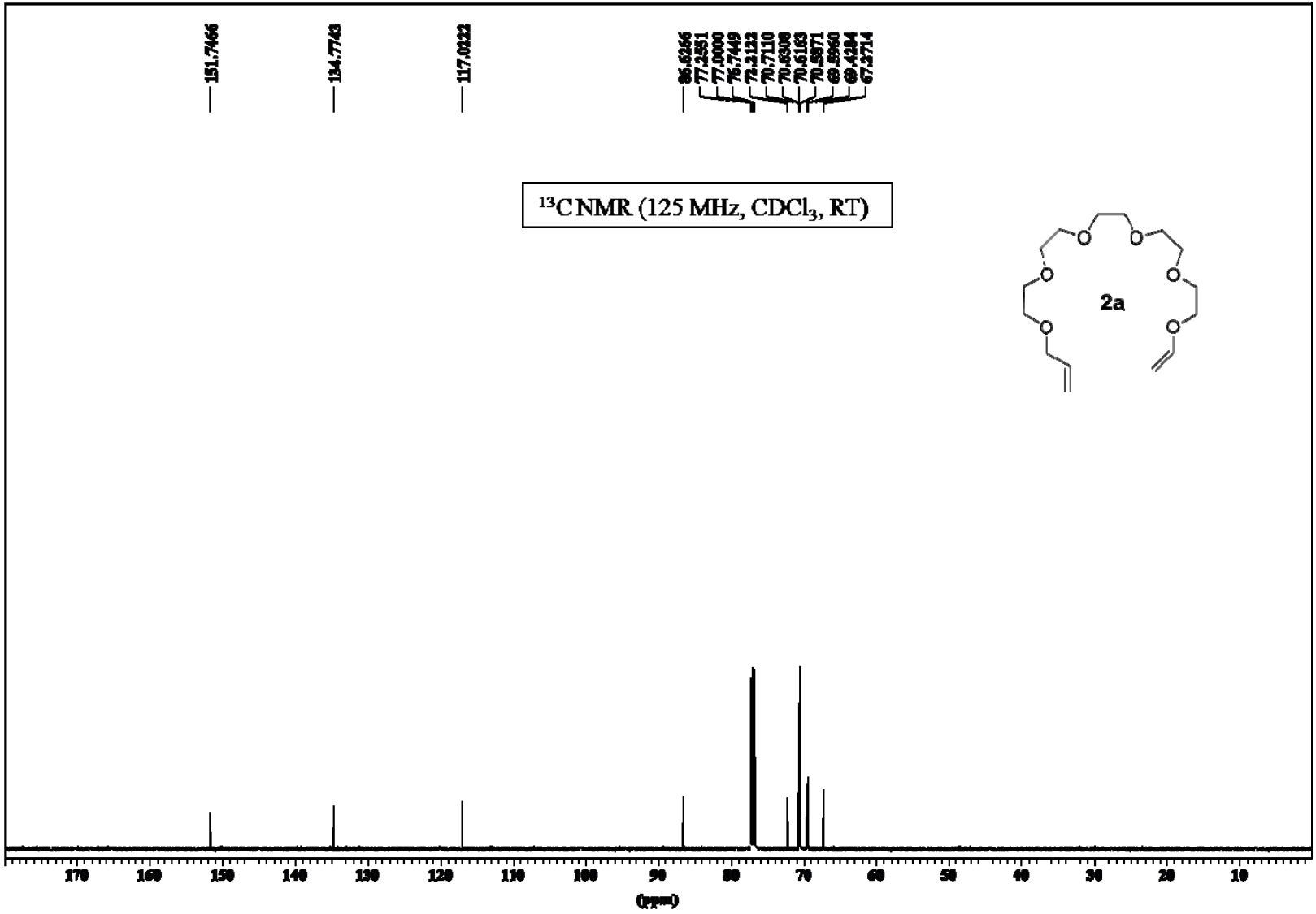
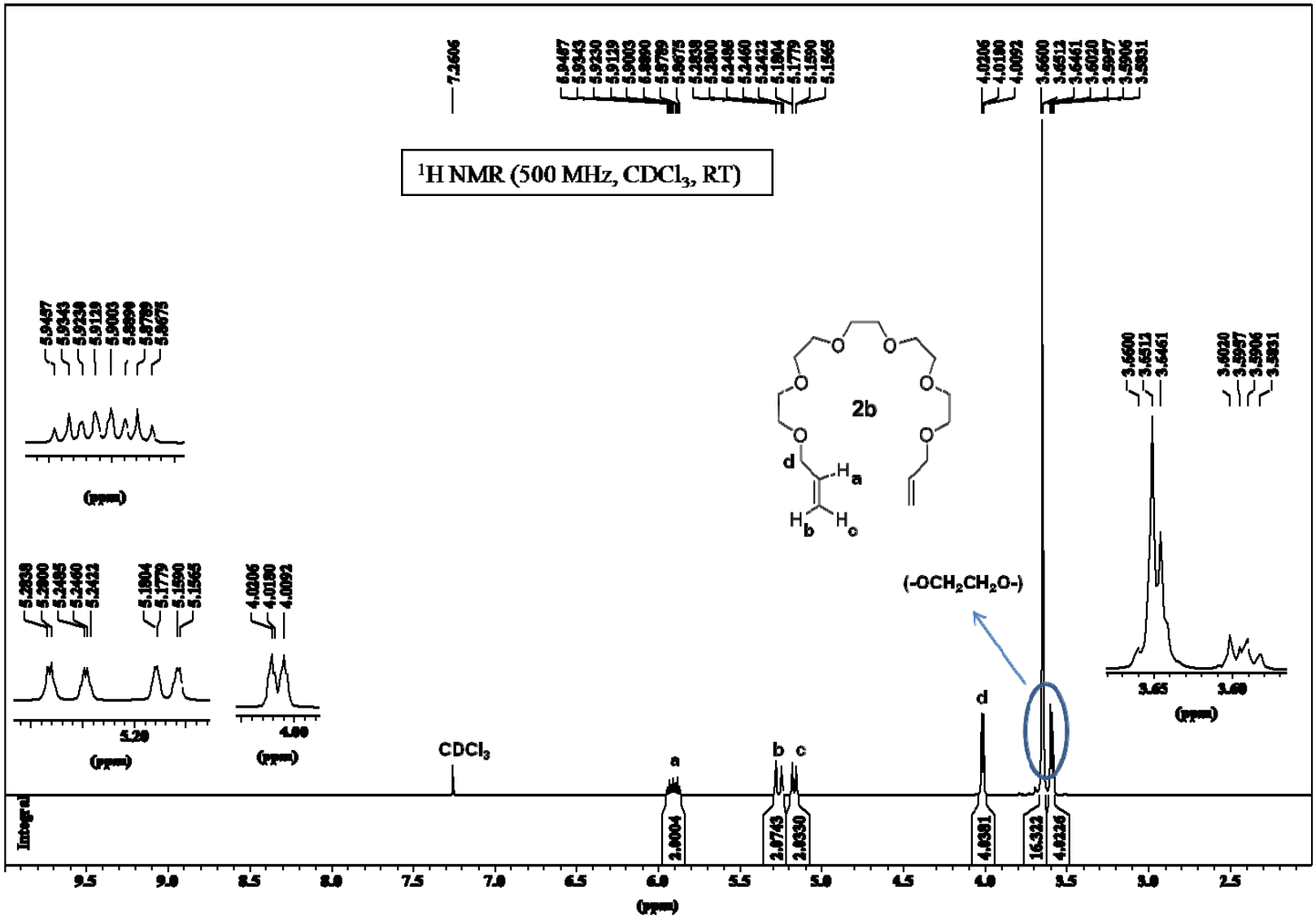


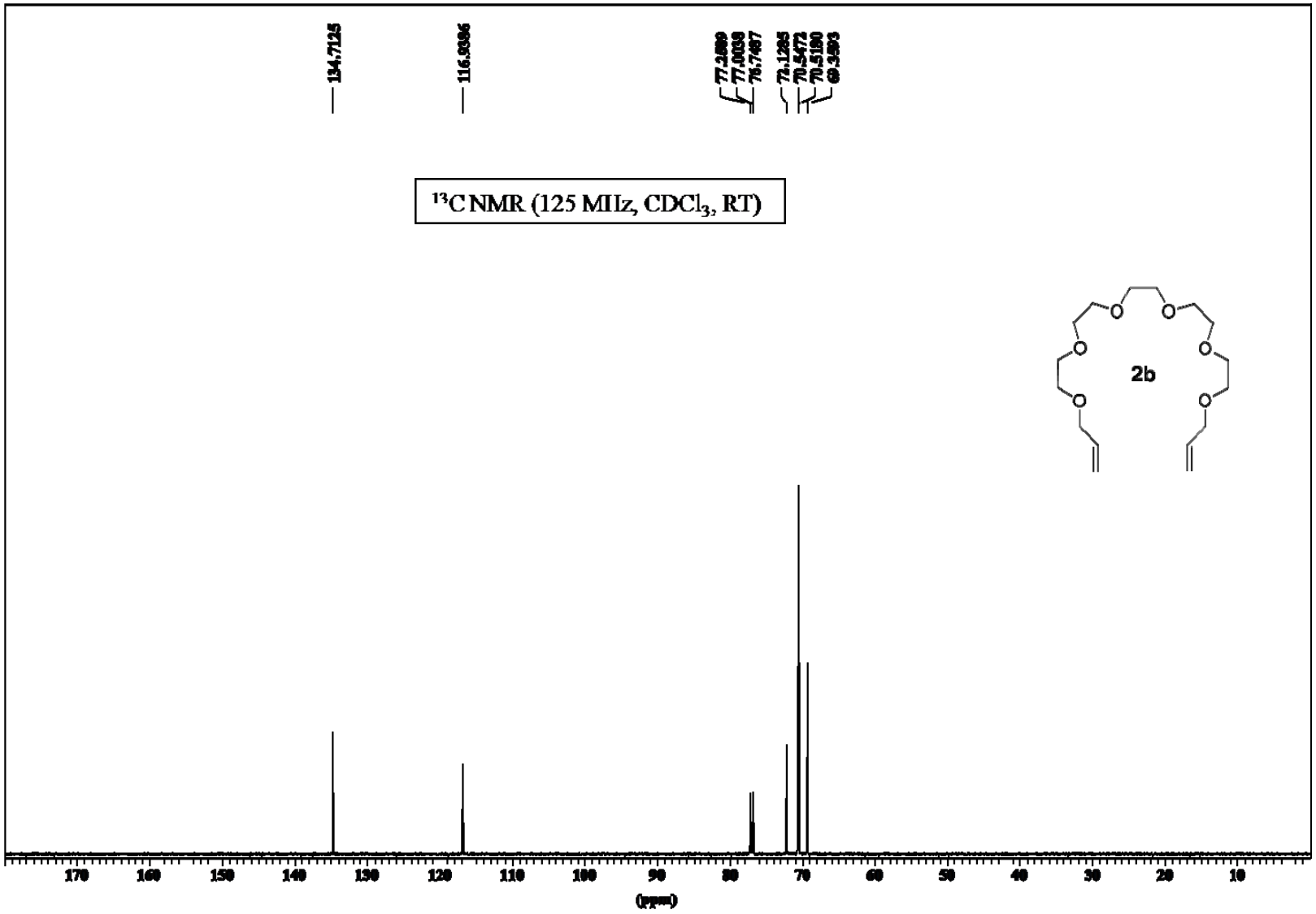
Figure S10. Stacked partial ^{1}H NMR spectra (500 MHz, CD_3SOCD_3) of **9c·PF_6** at different temperatures. The peaks of interest (**b** & -NH_2^+-) were zoomed to appreciate the effect of thermal energy in dethreading of [21]C6H_2 crown ether from **9c·PF_6**. Contrary to Figure 8 for **9d·PF_6**, we did not observe any dethreading of [21]C6H_2 on increasing the temperature to even 373 K, implying that **9c·PF_6** like **9b·PF_6** is a kinetically stable [2]rotaxane not only at room temperature but also at 373 K.

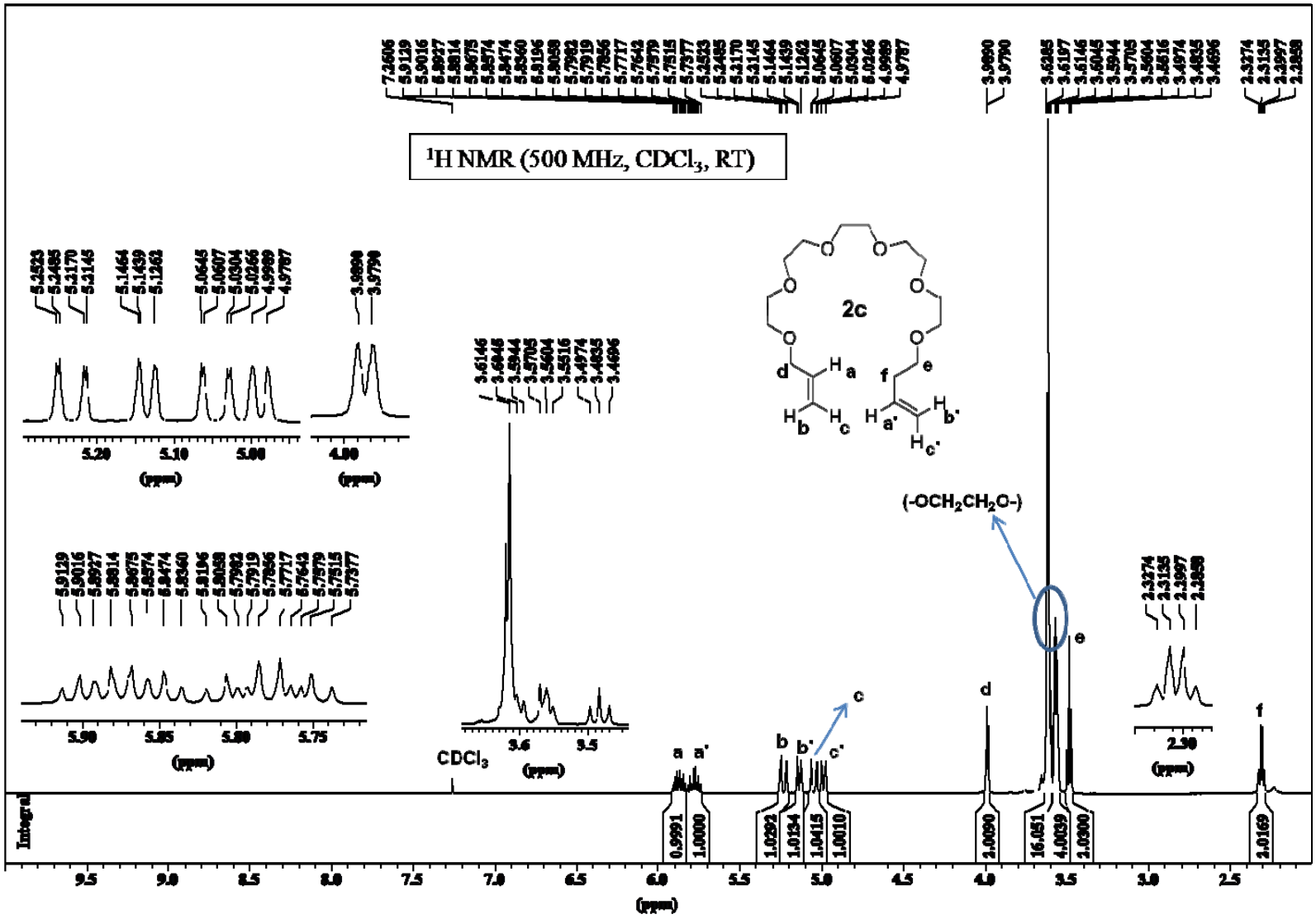
8. ^{1}H NMR and ^{13}C NMR spectra of key compounds

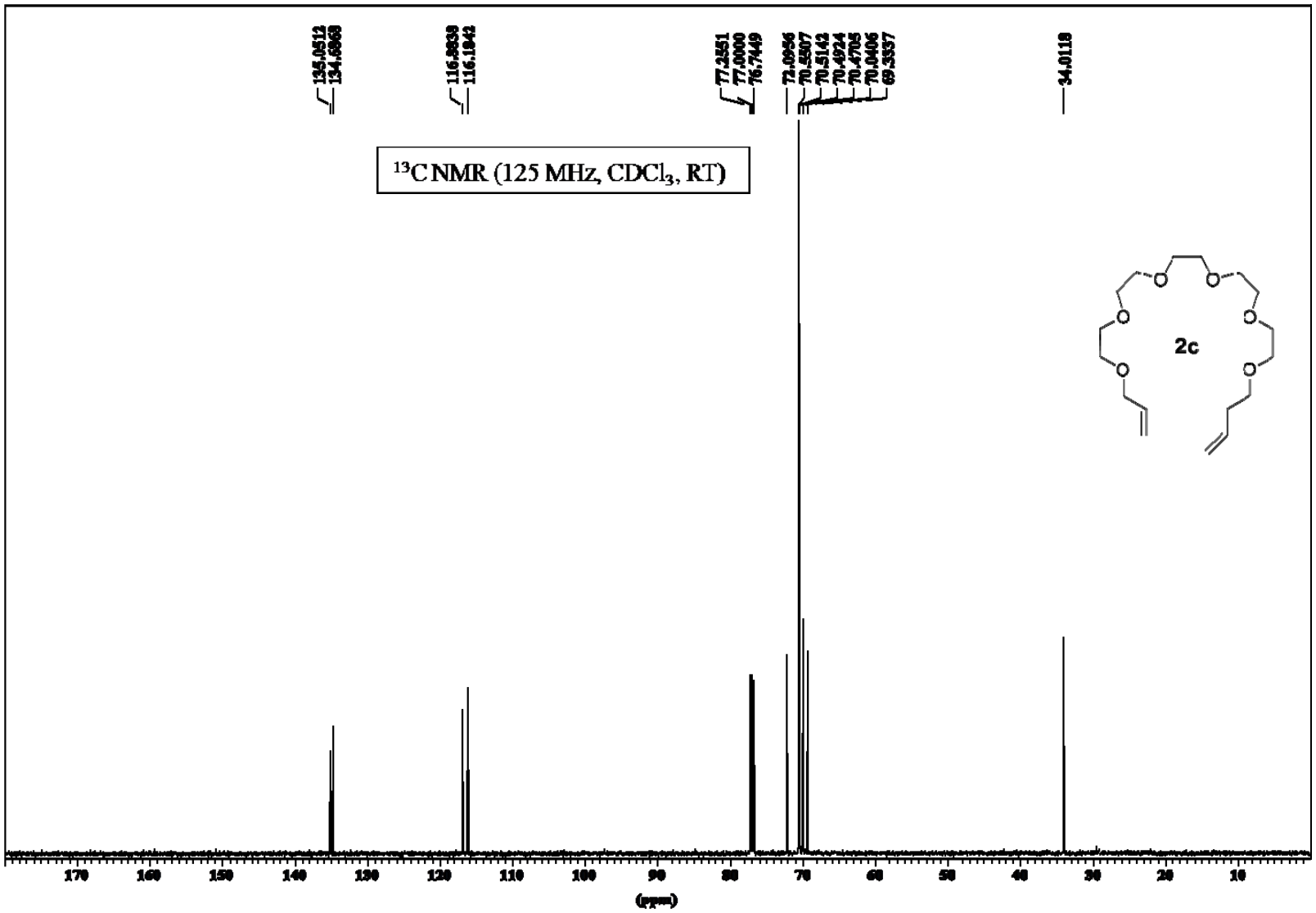


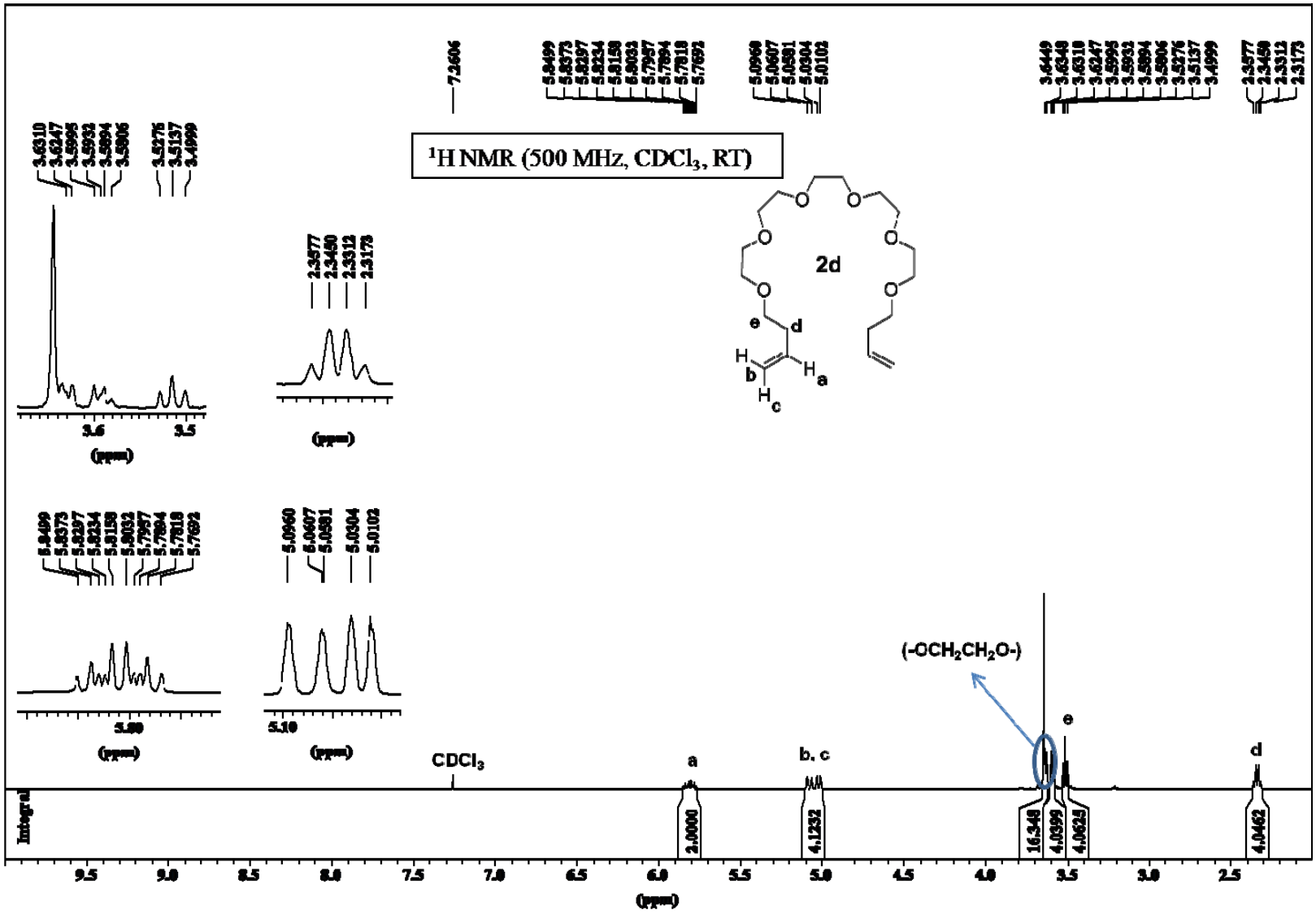


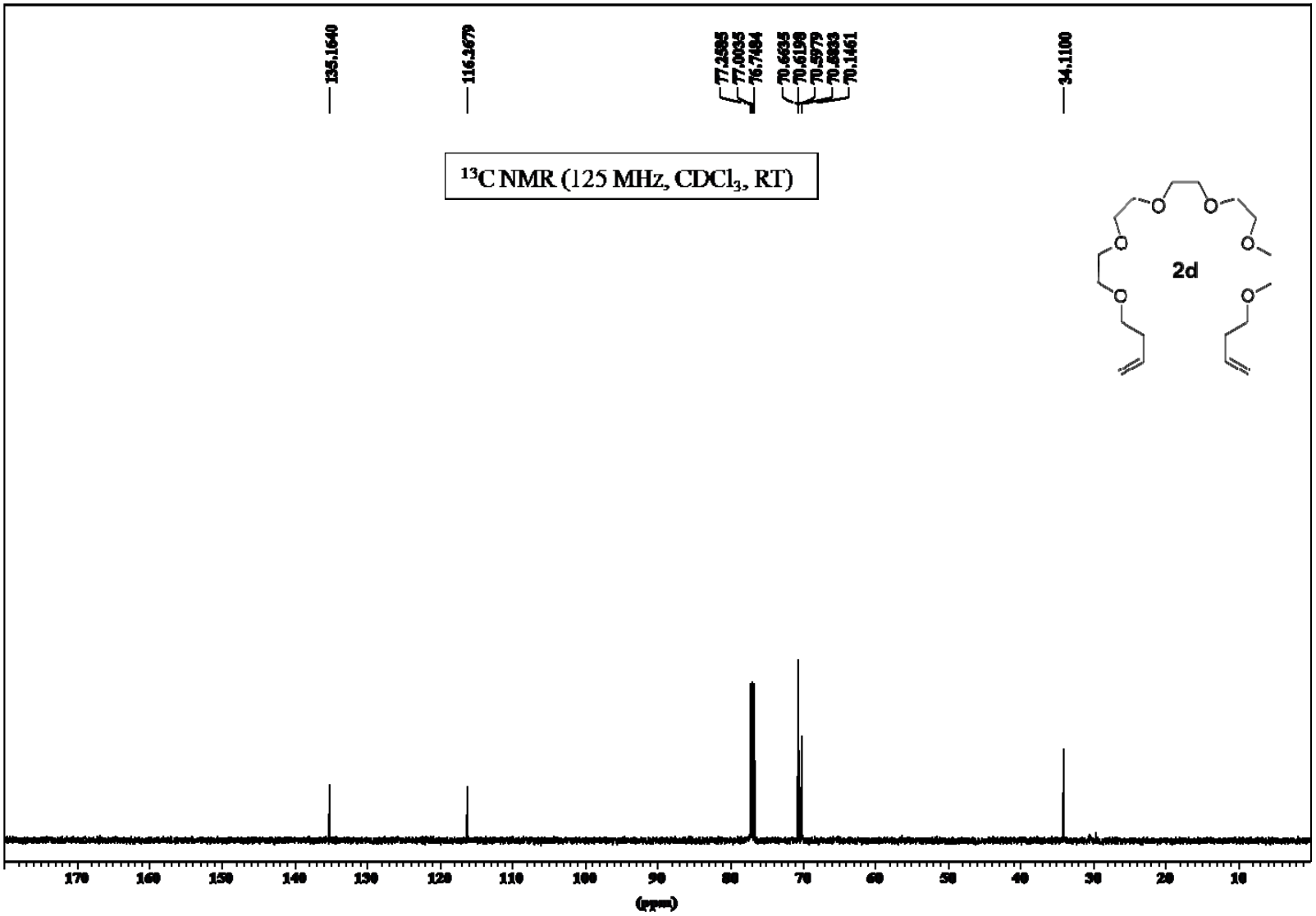


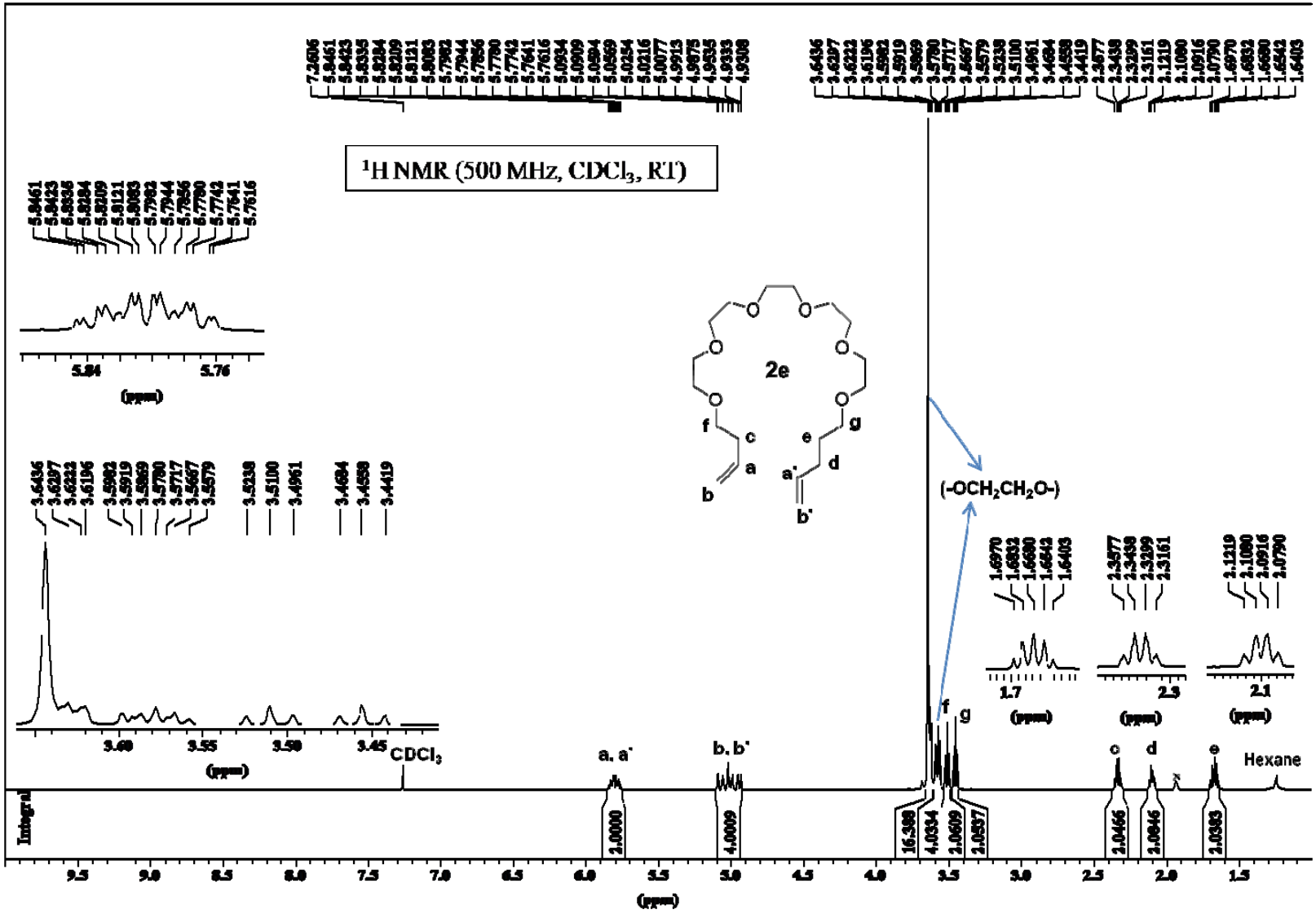


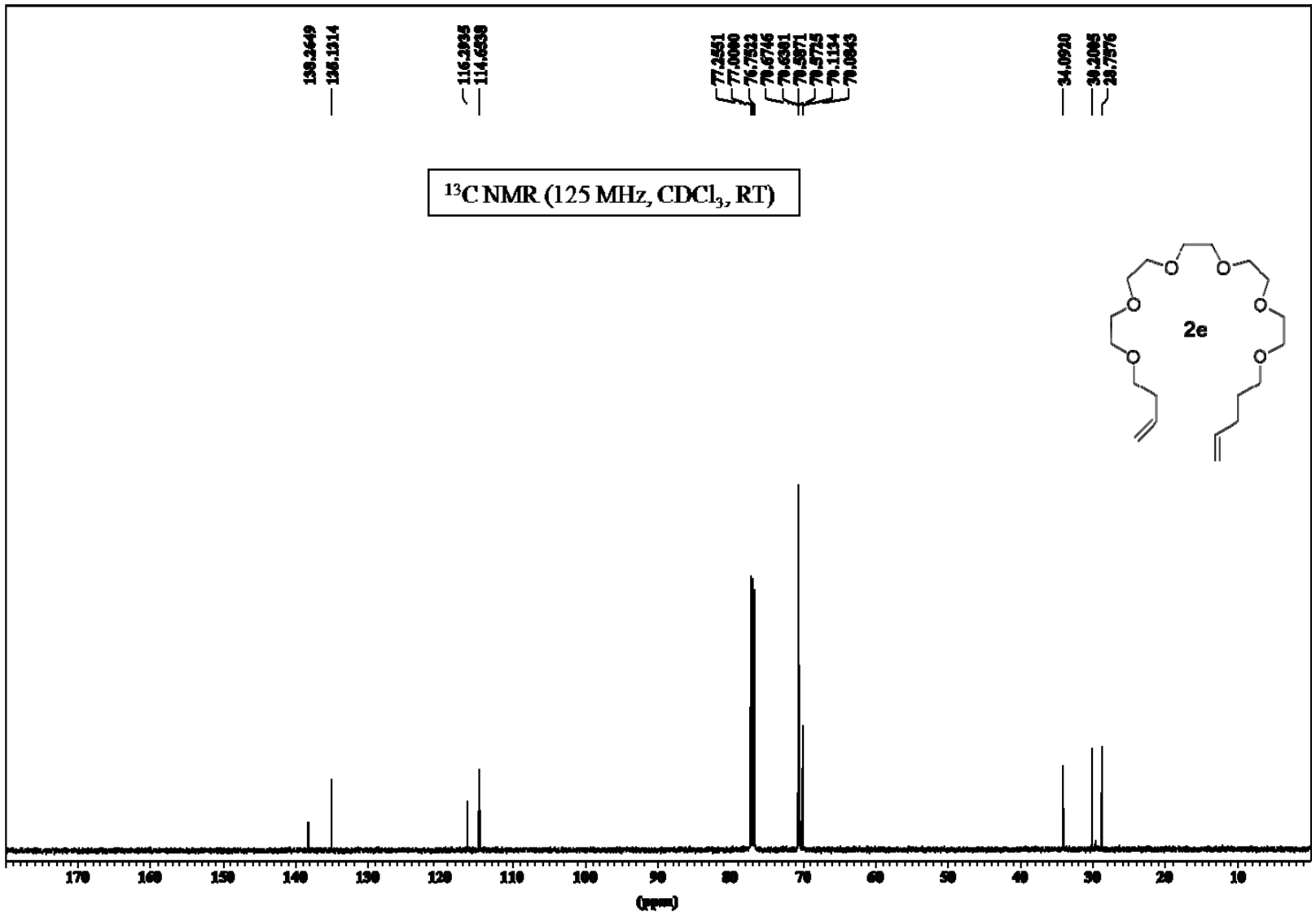


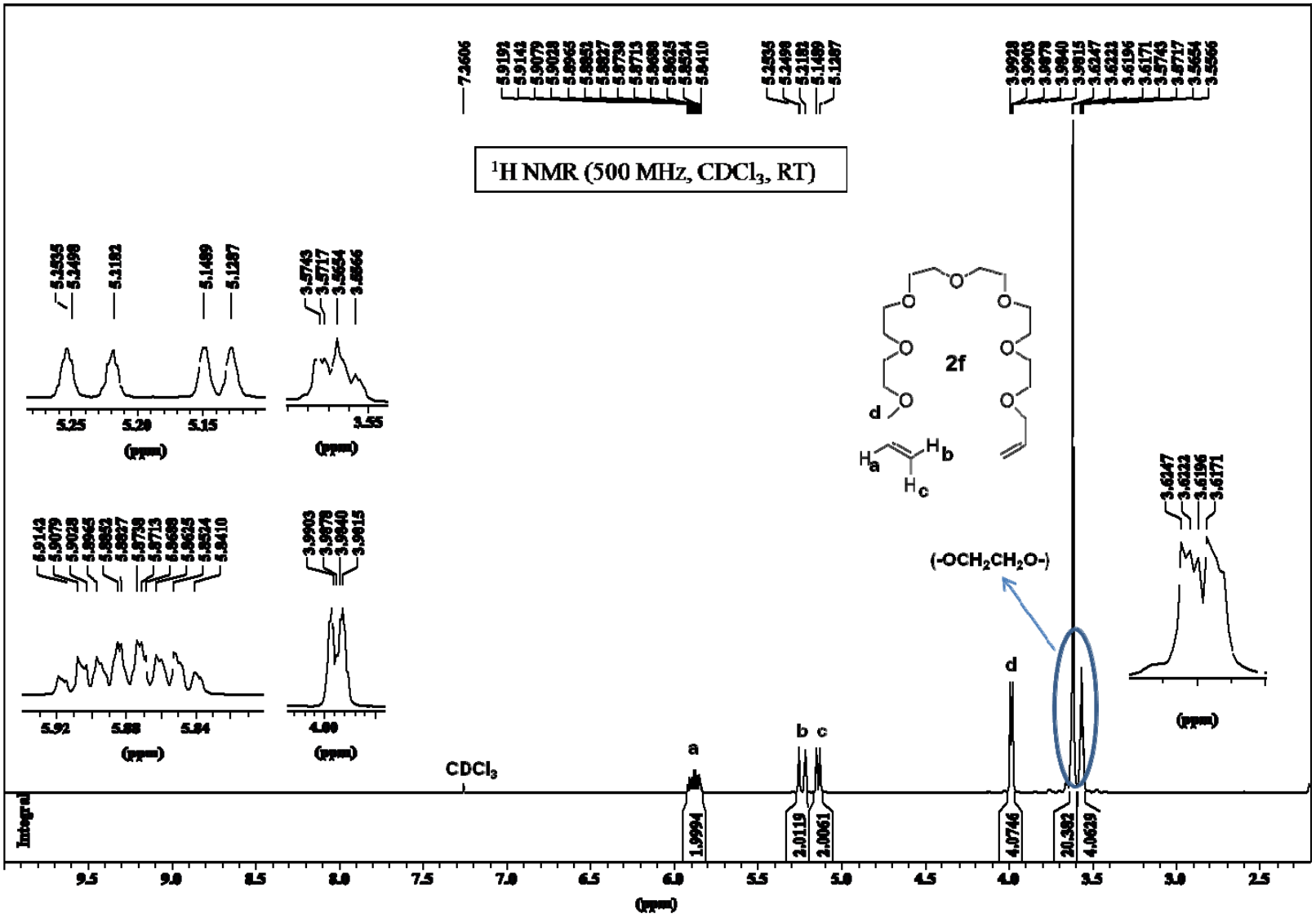


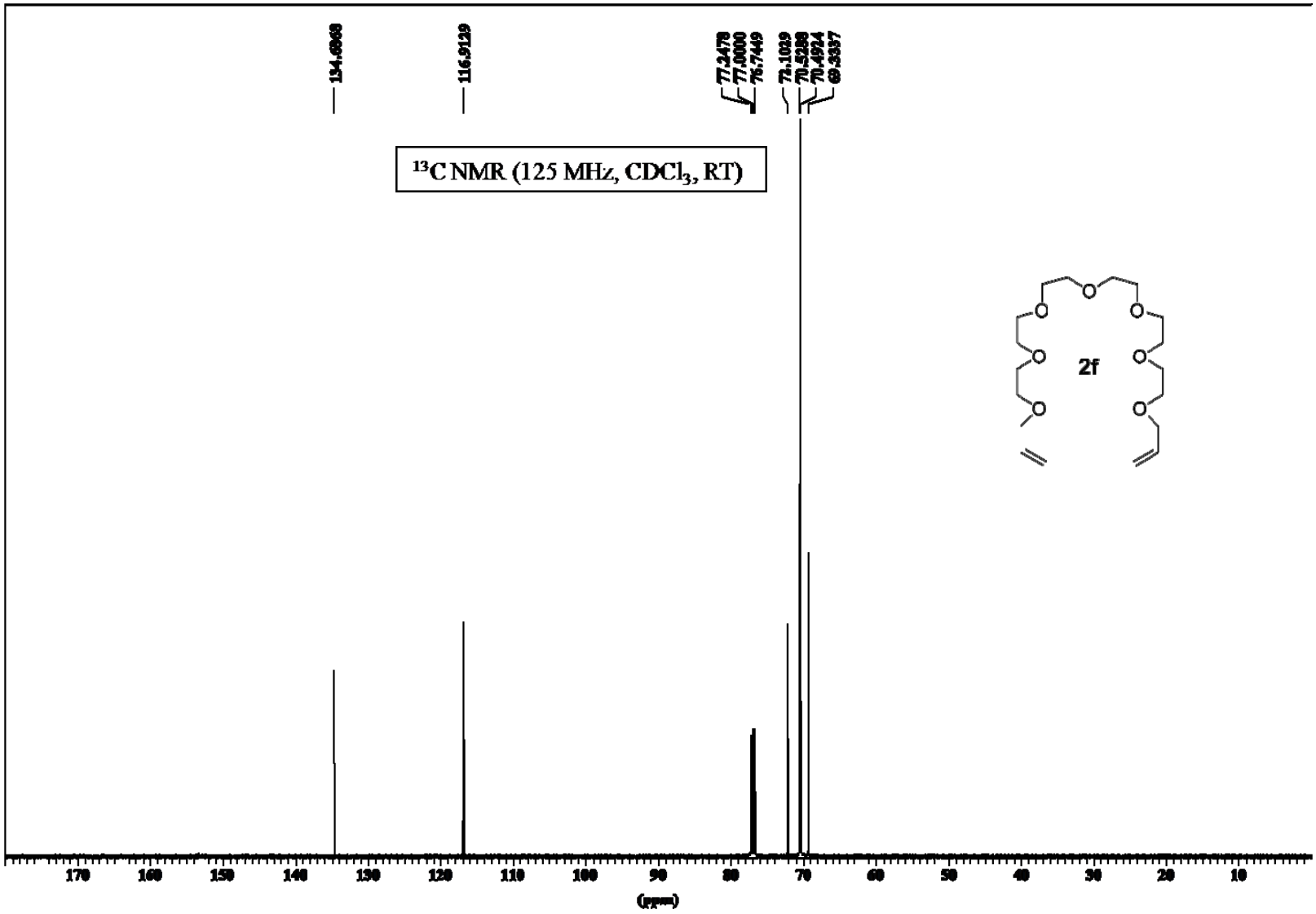


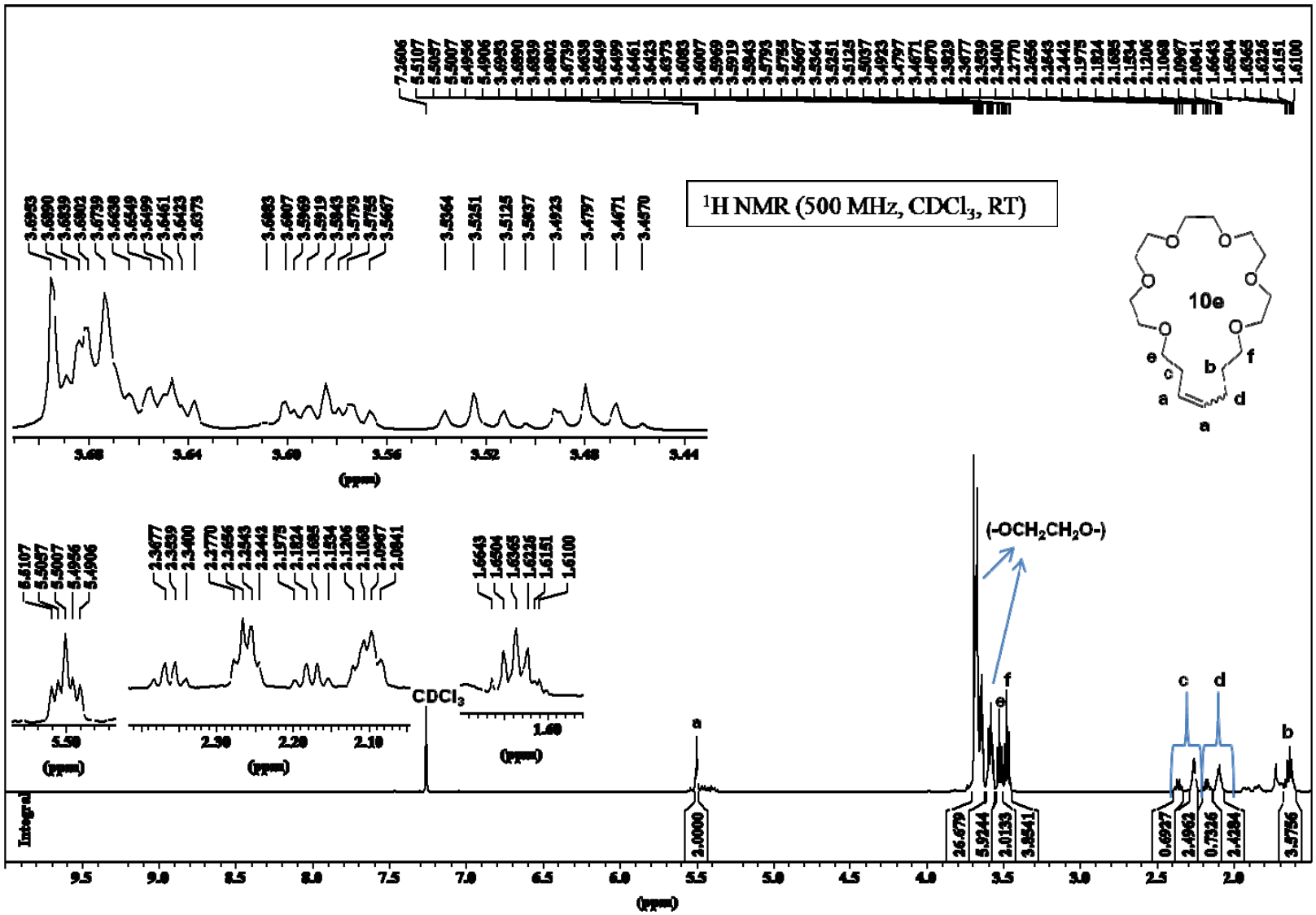


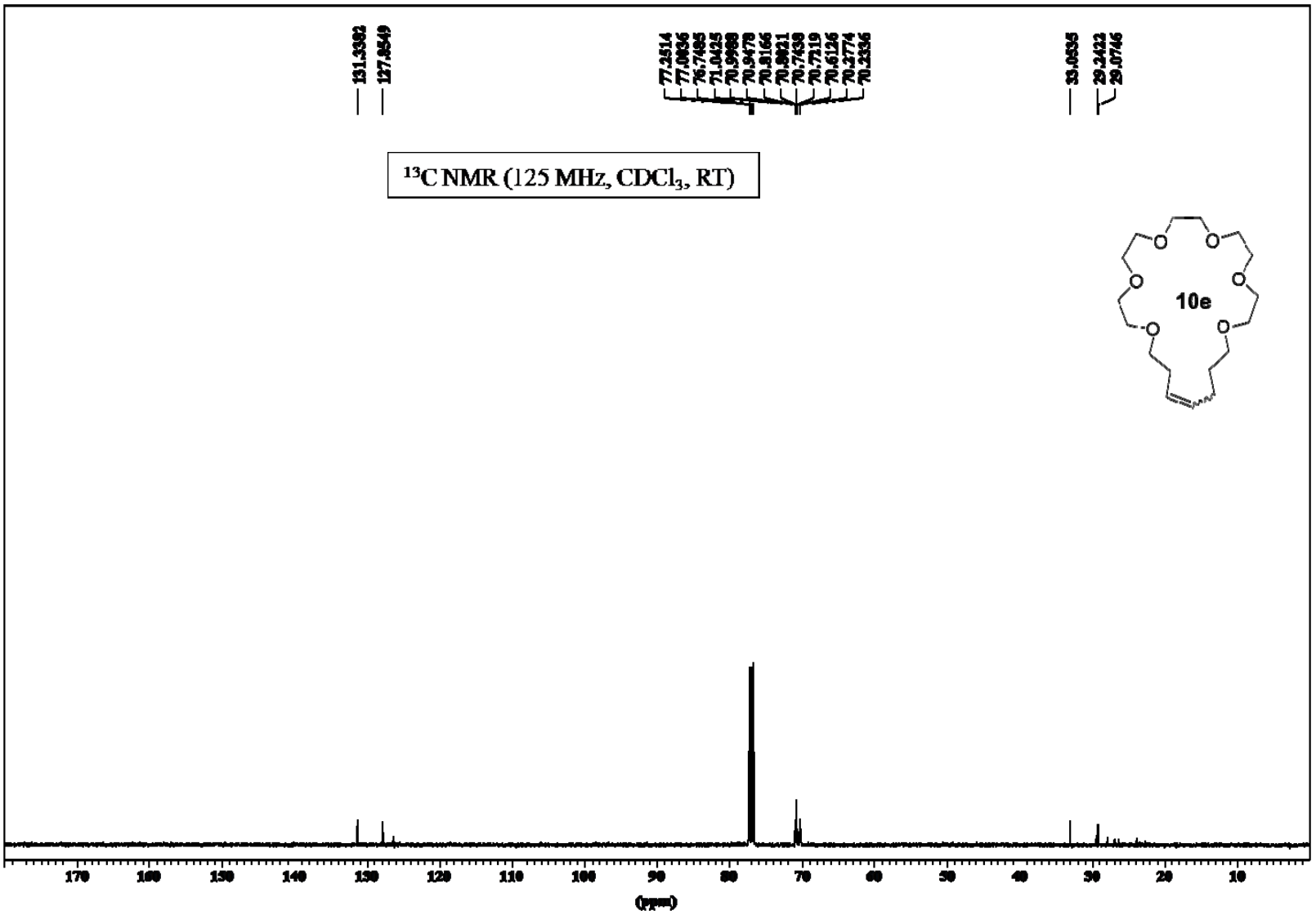


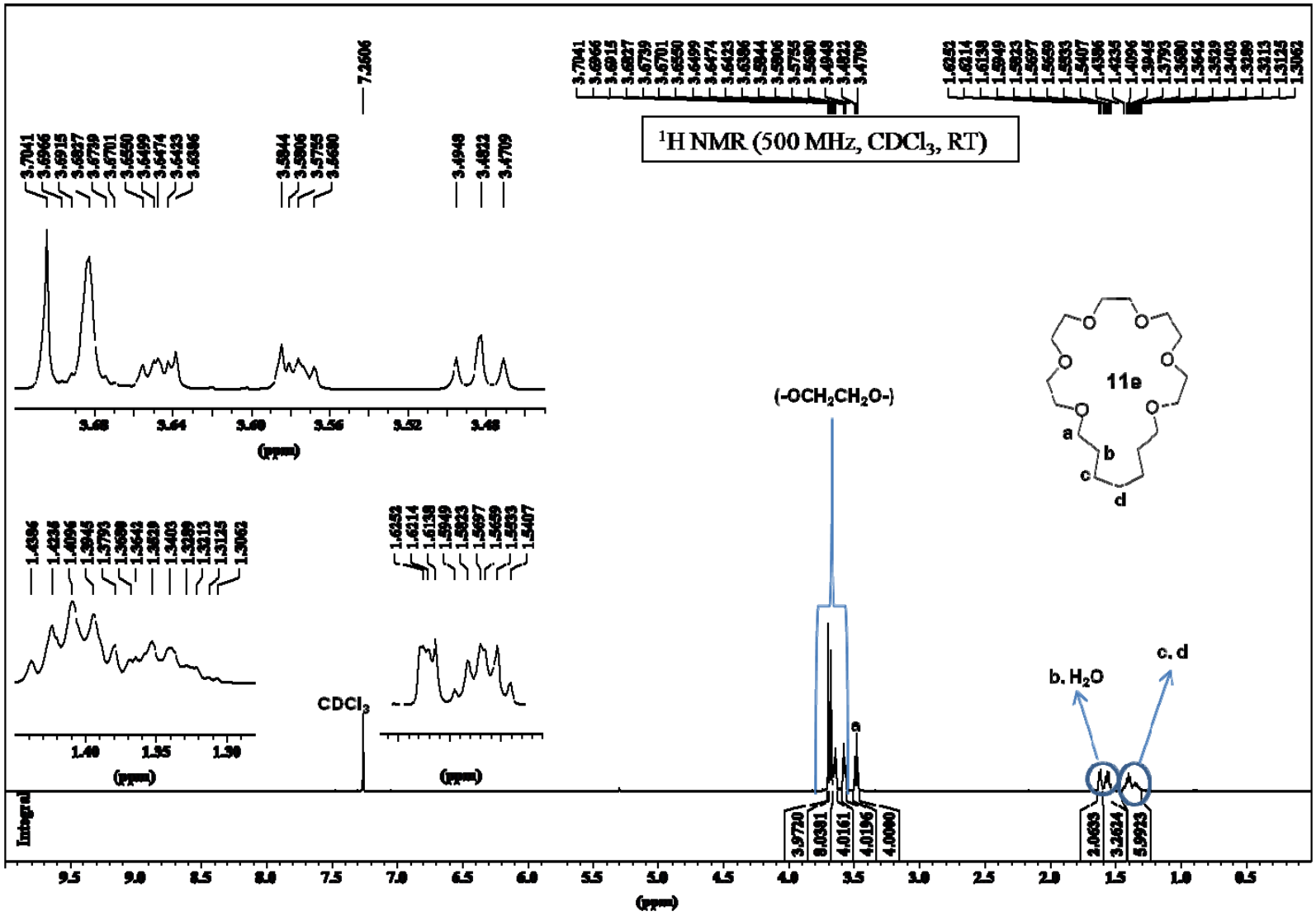


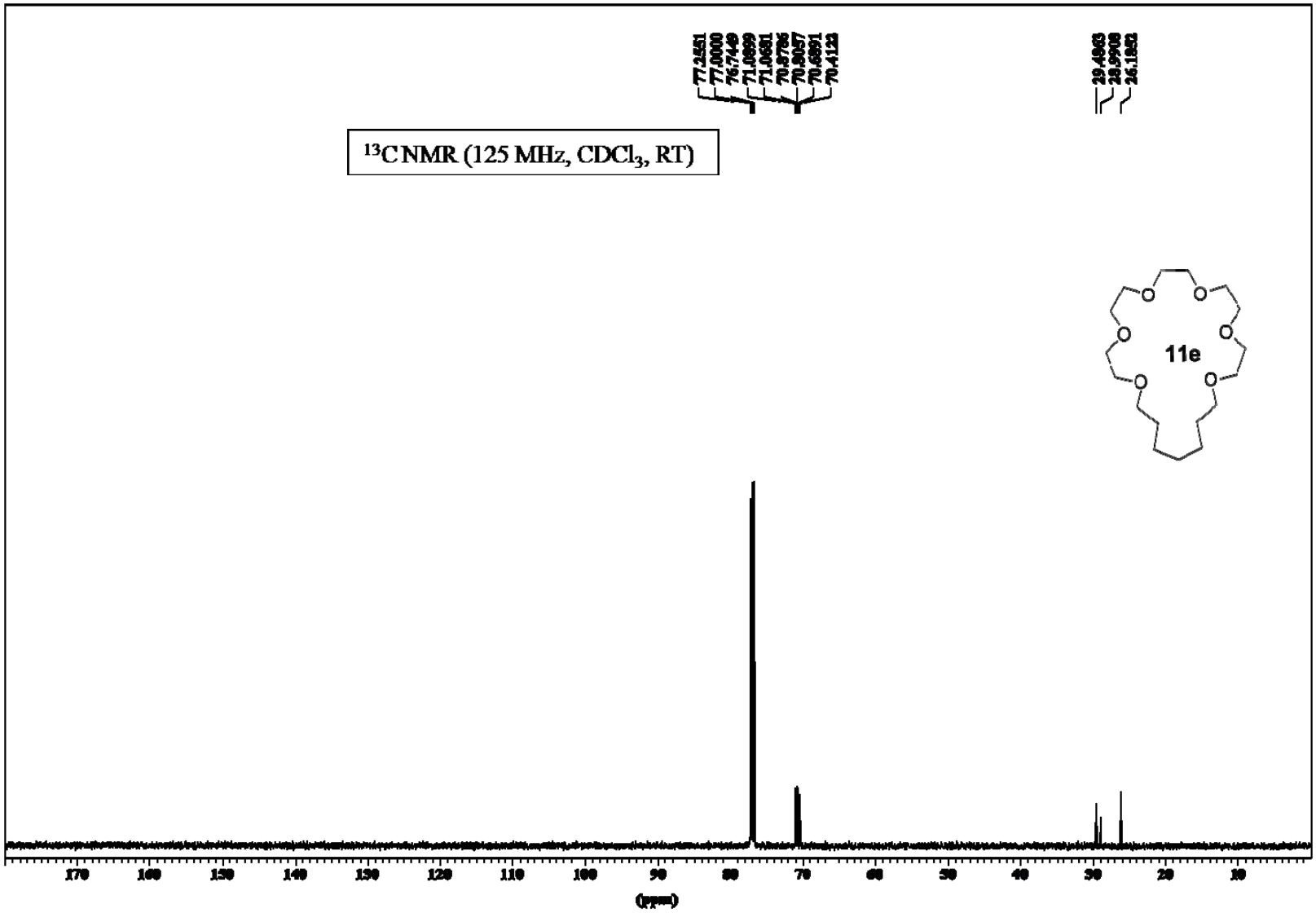


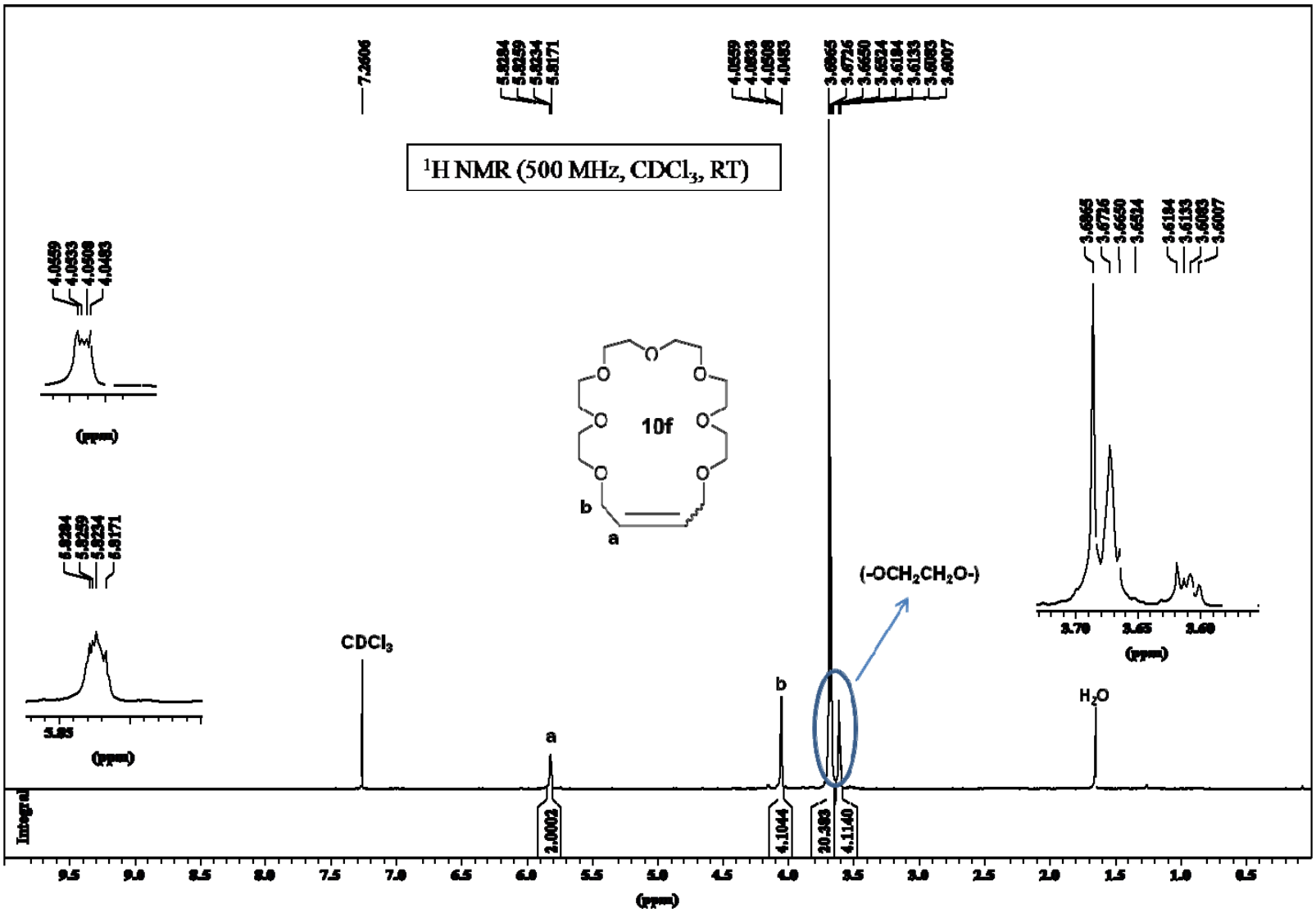


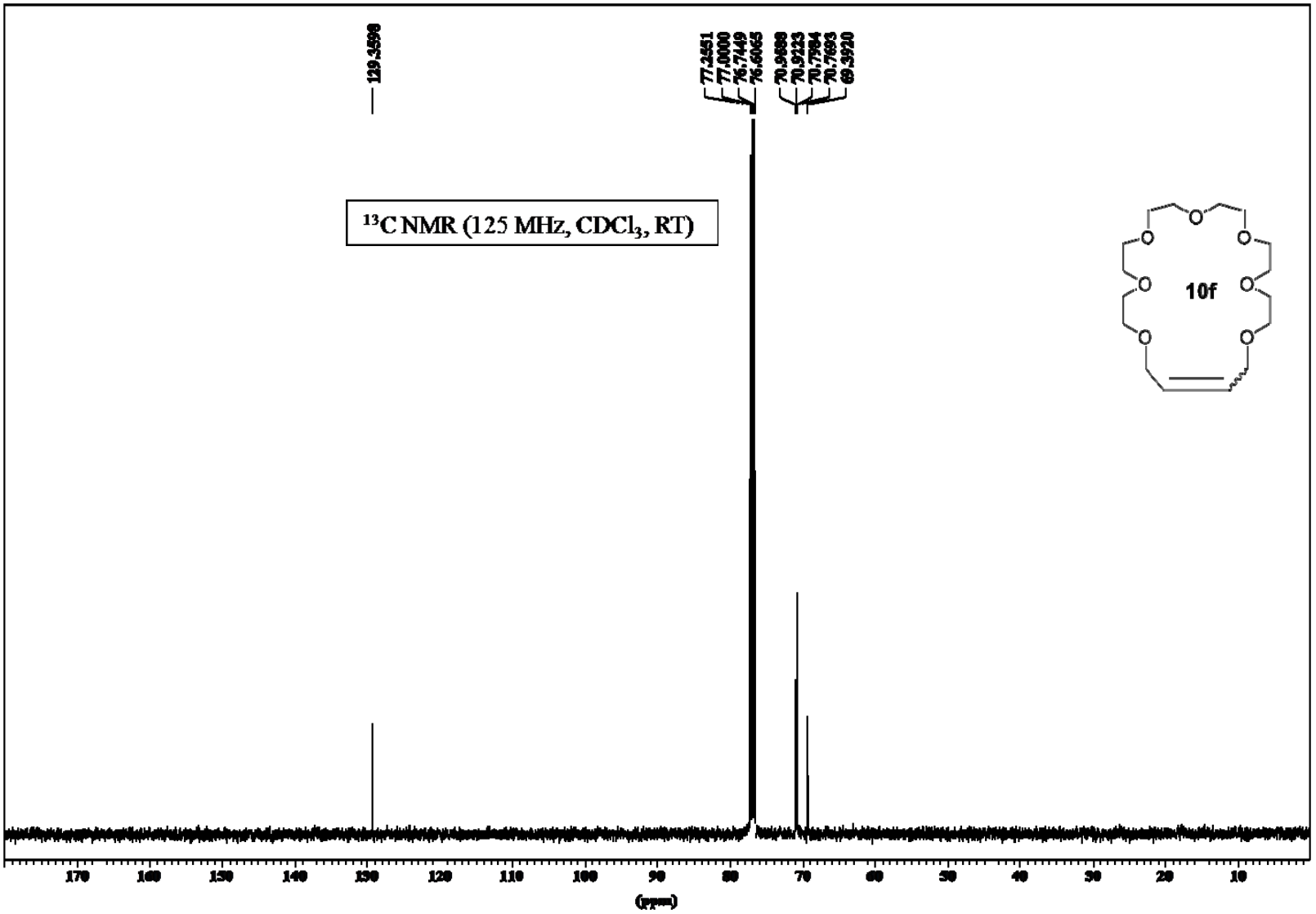


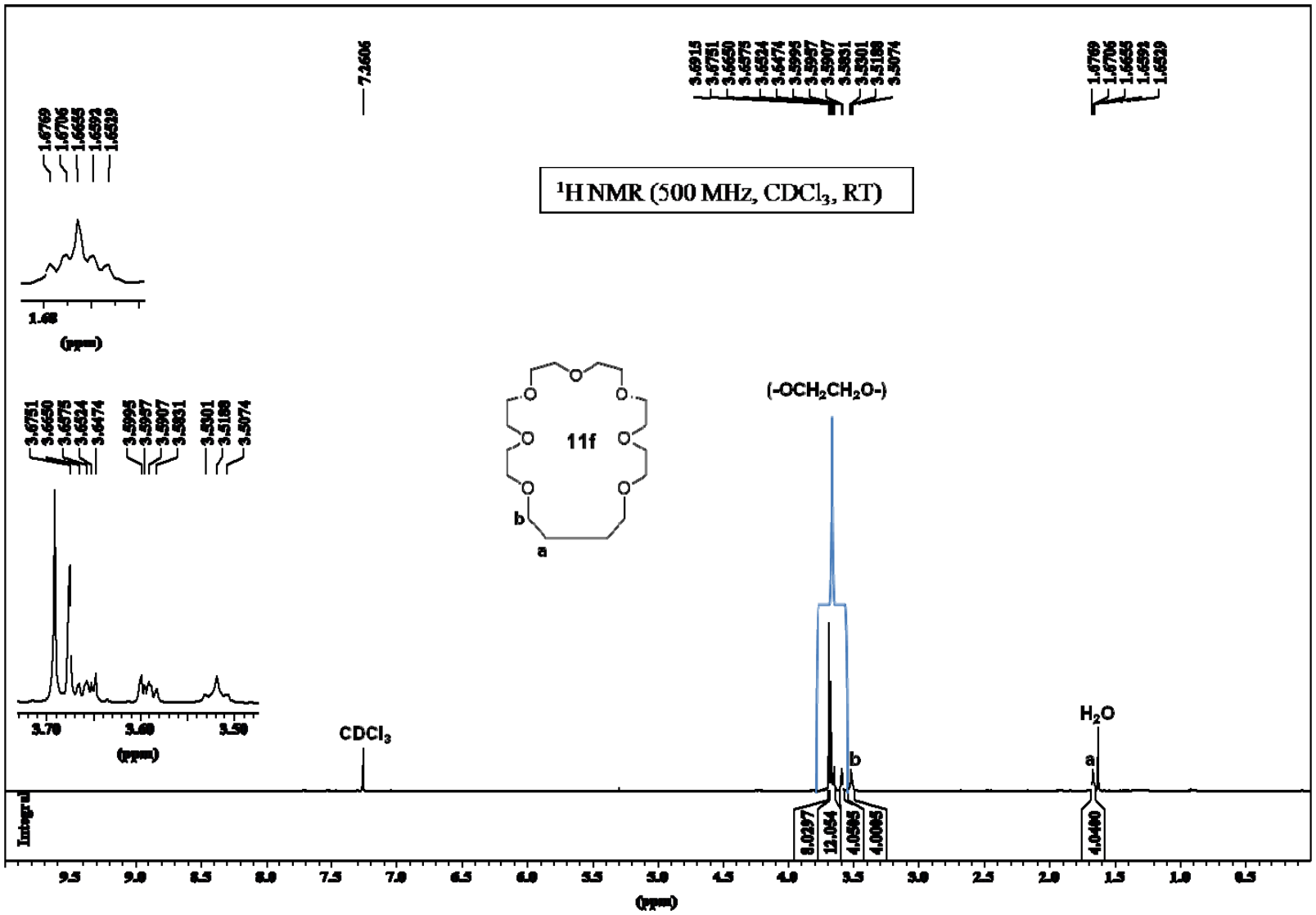


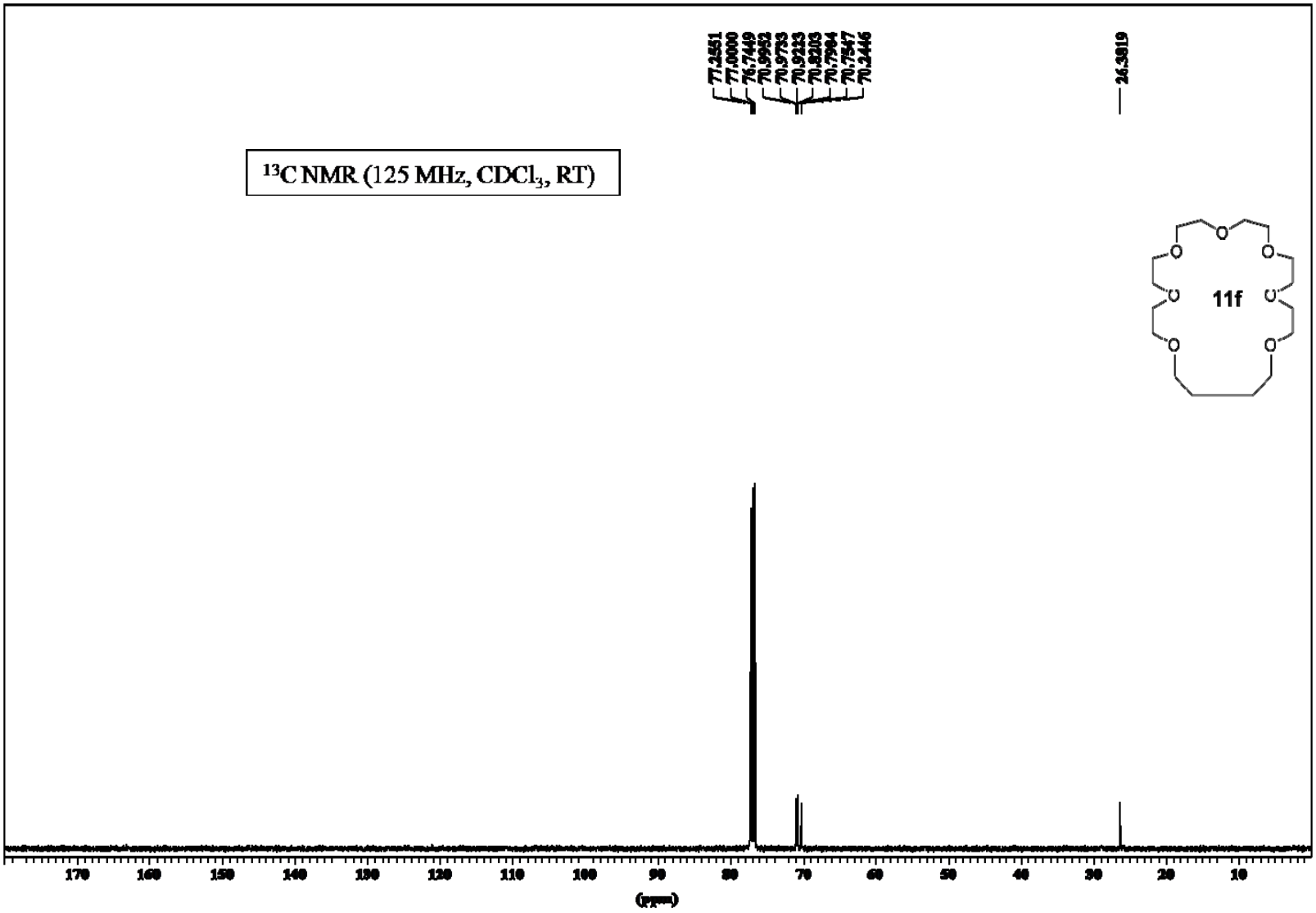


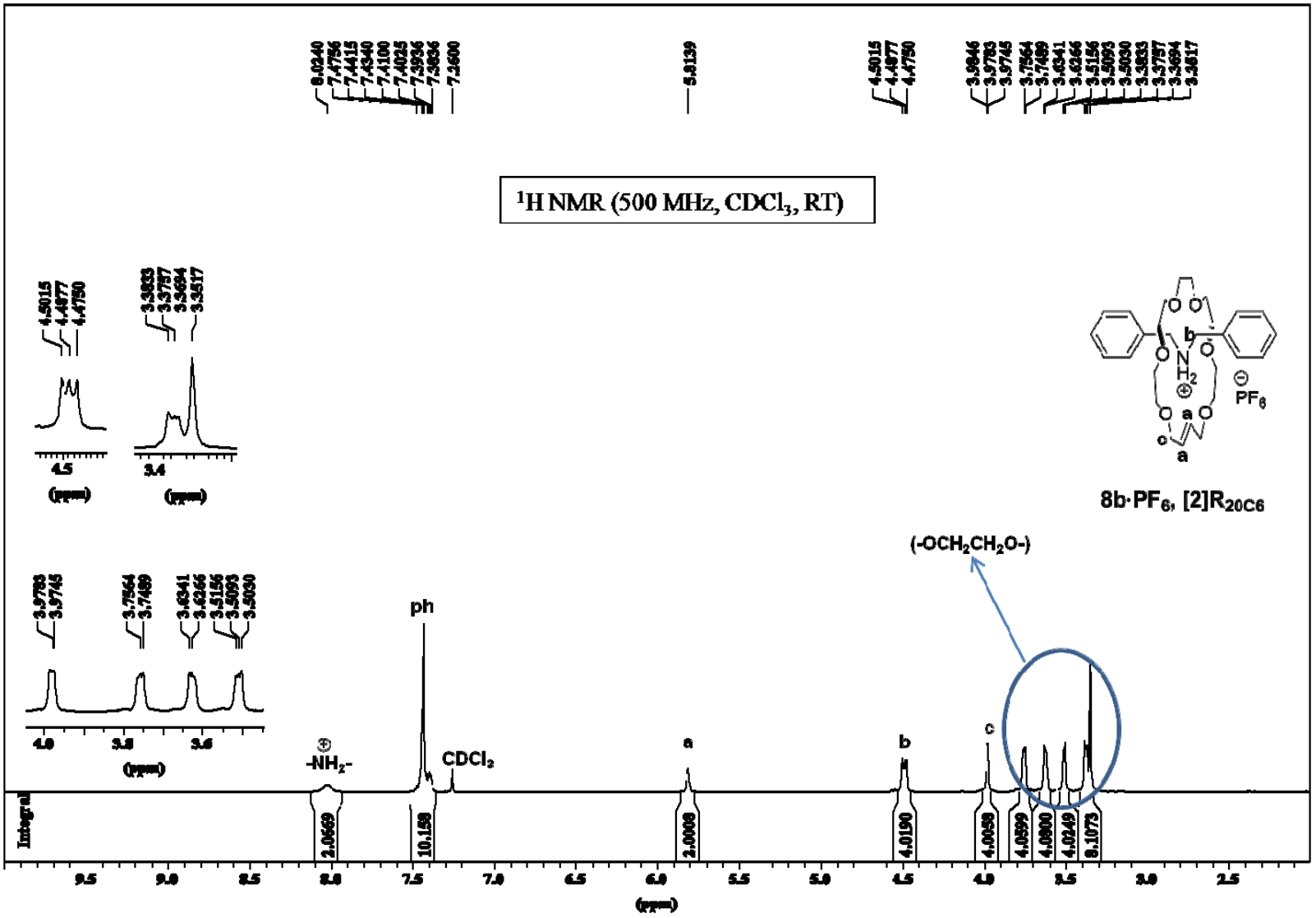


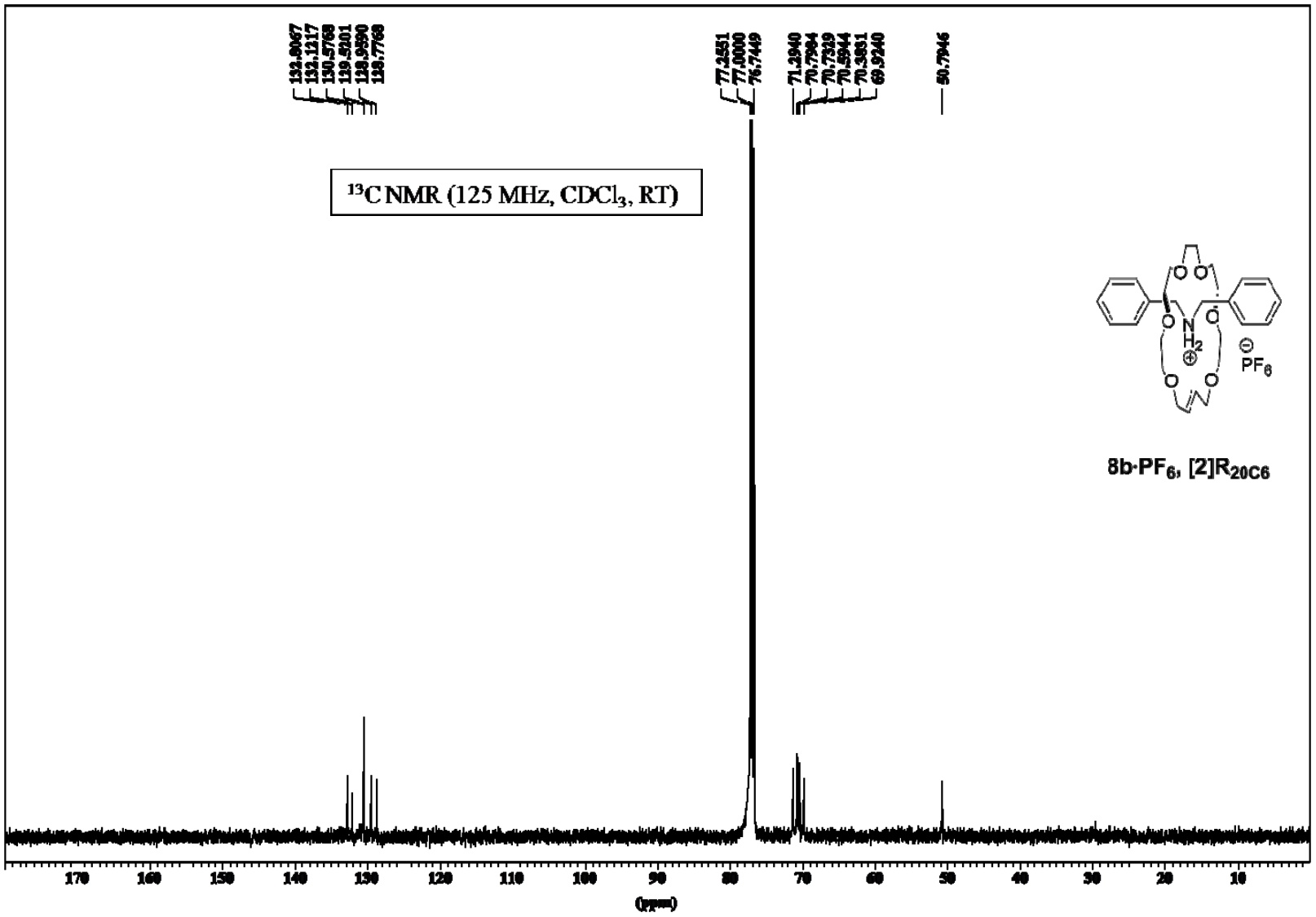


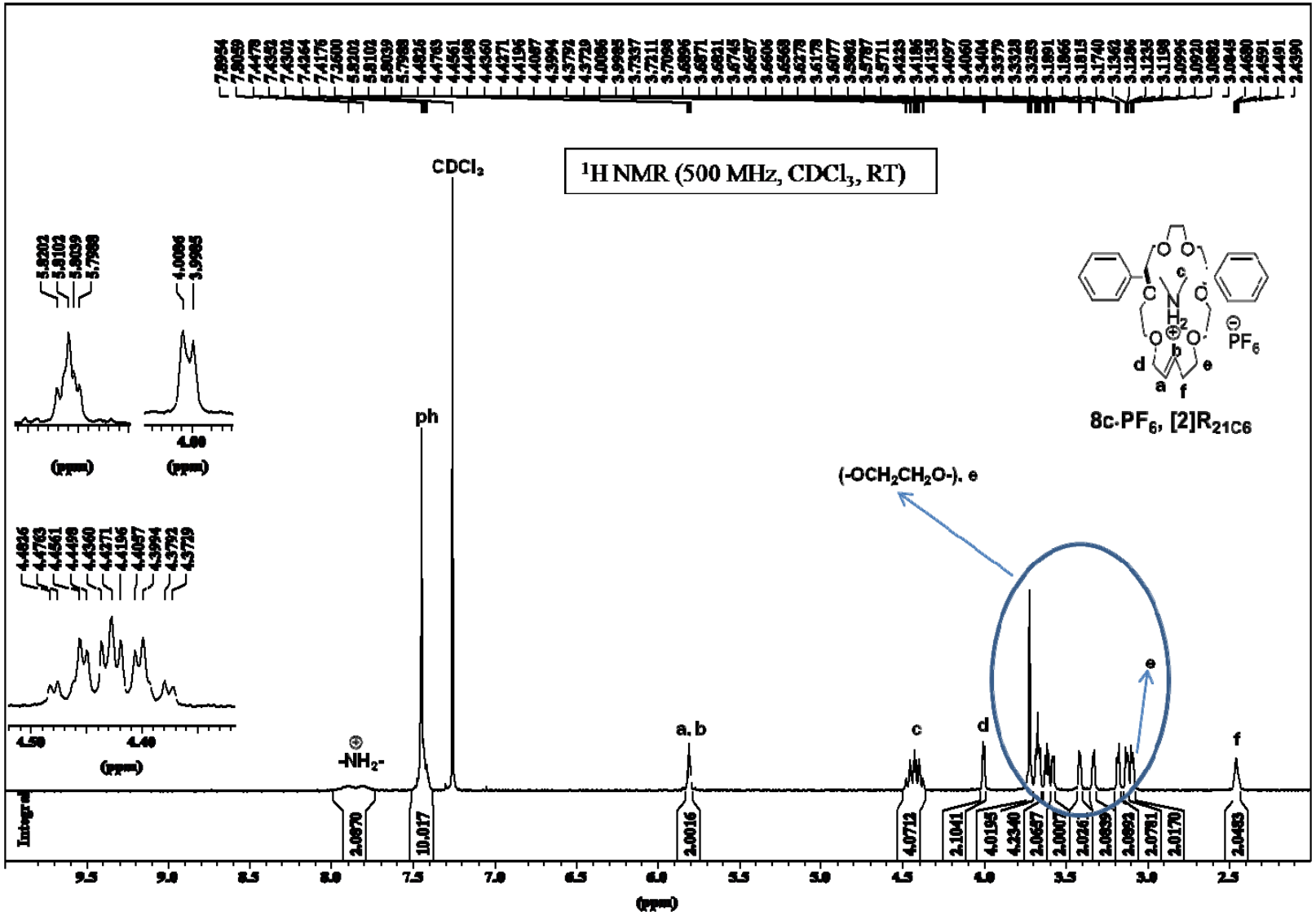


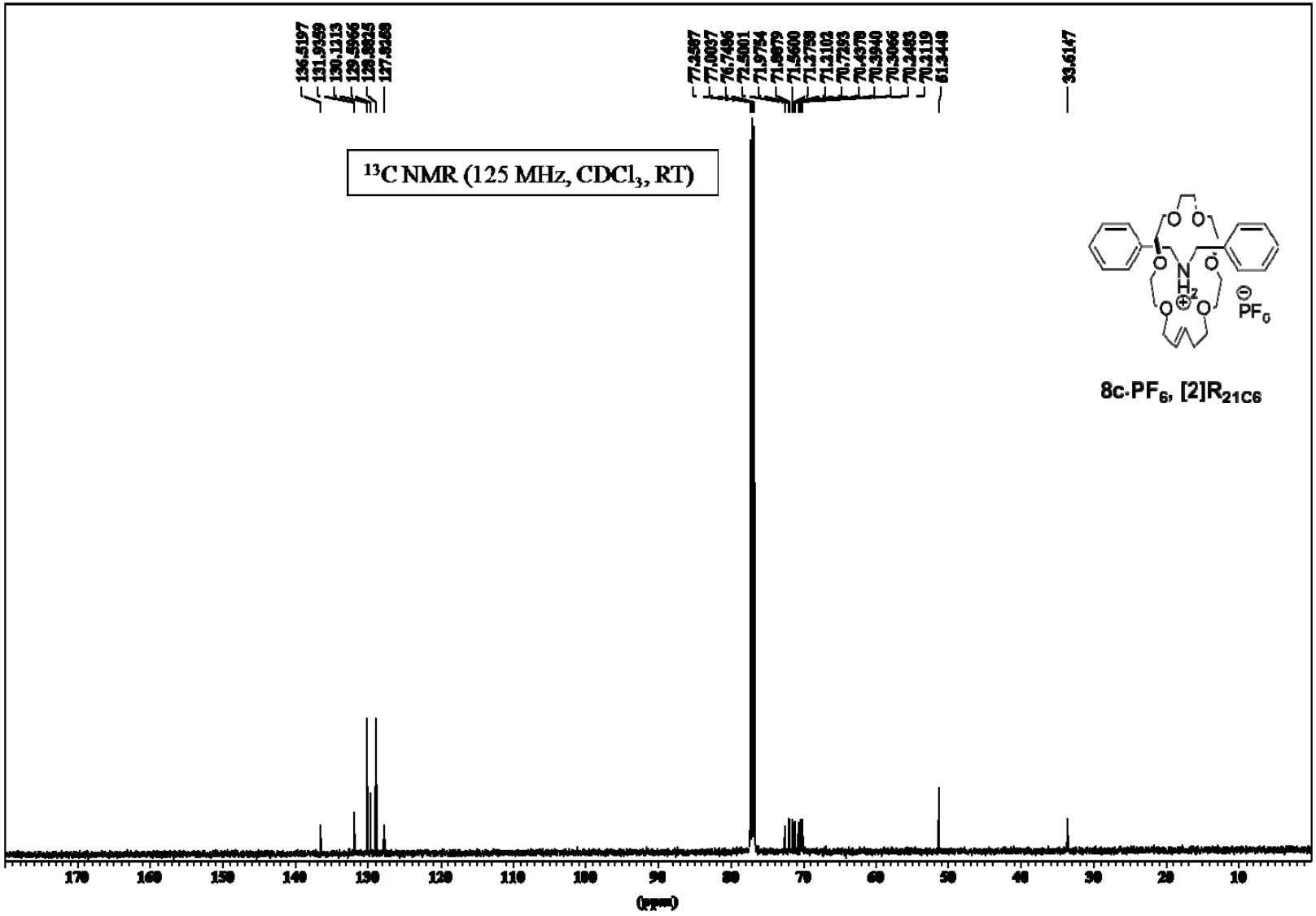


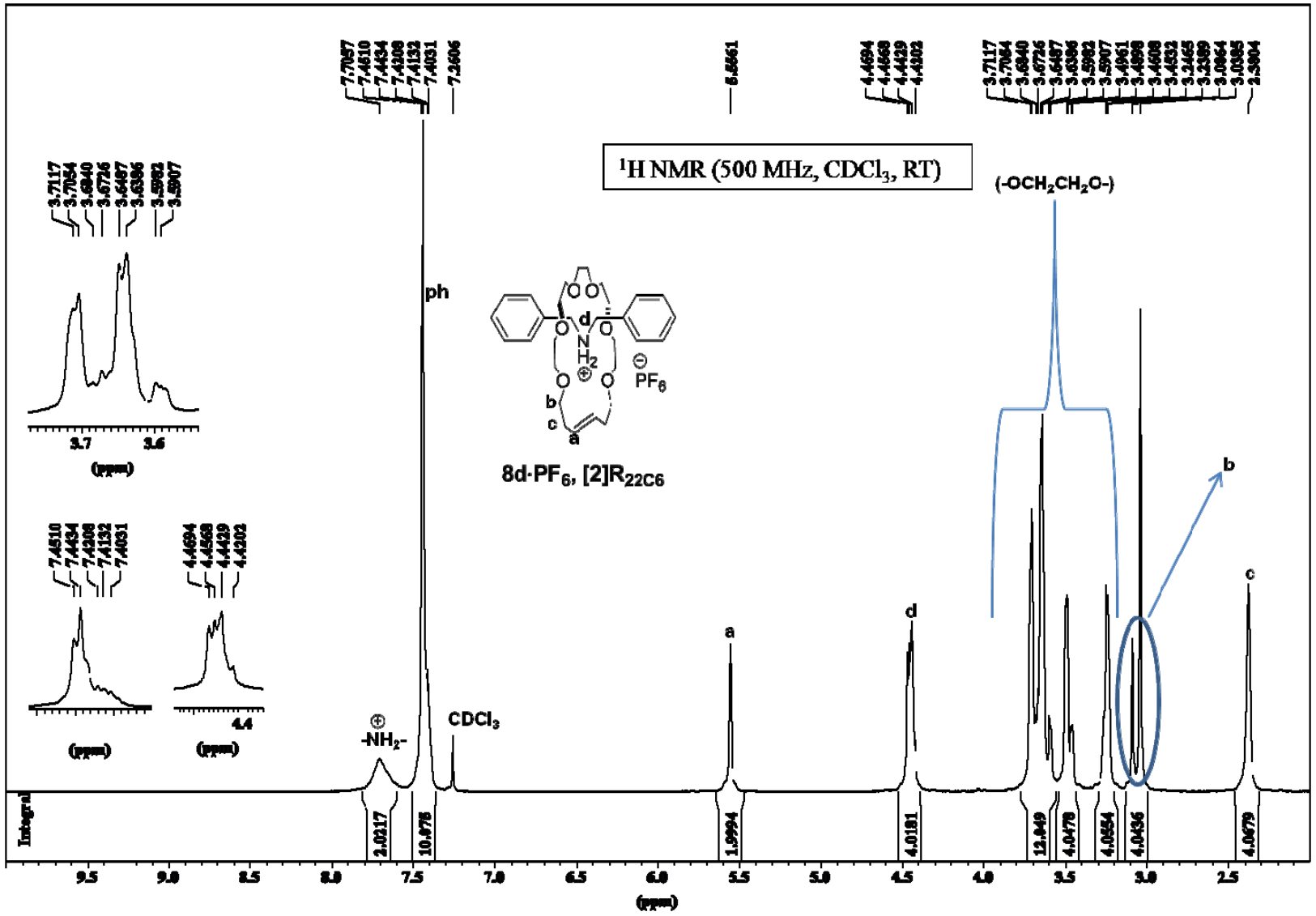


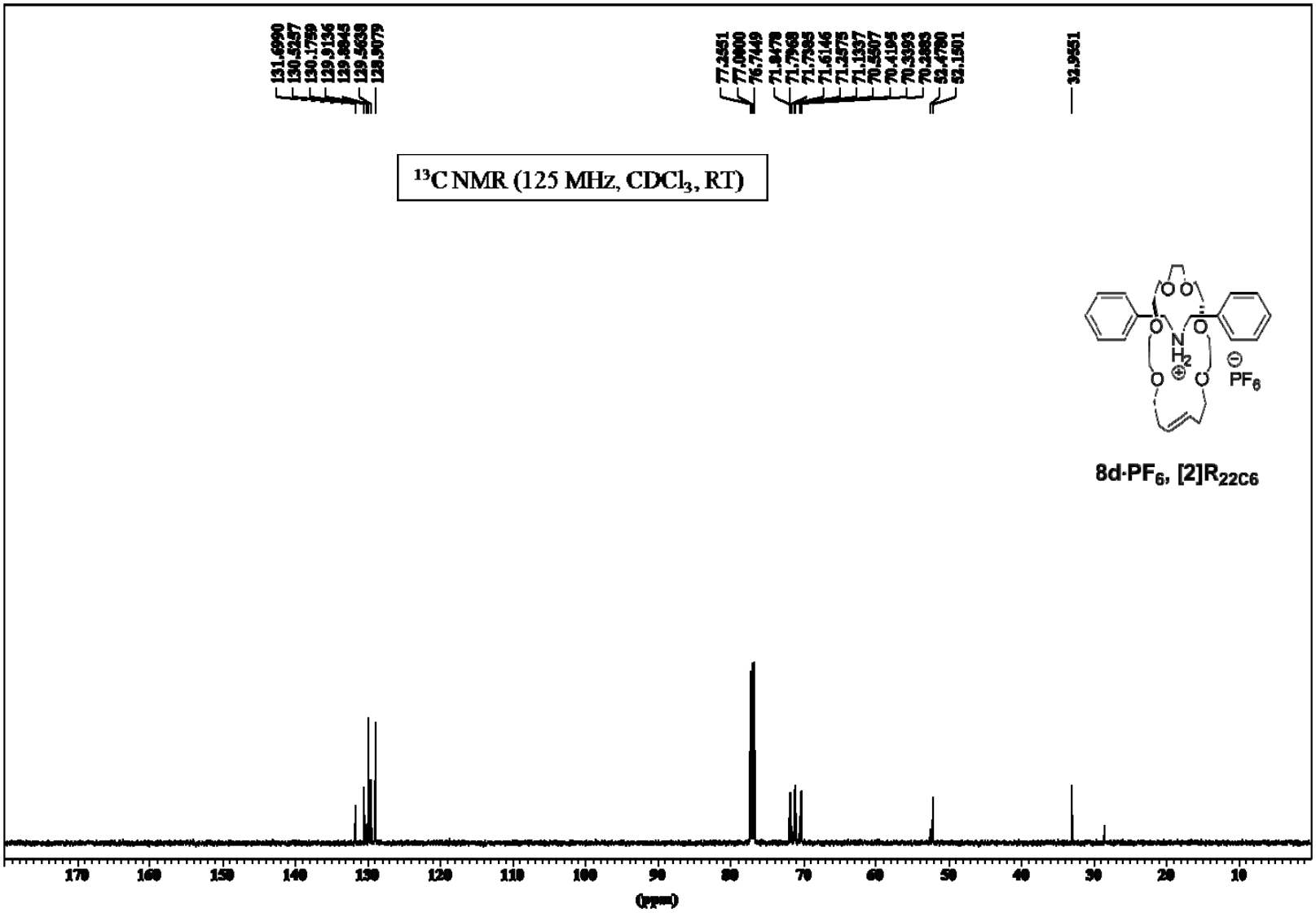


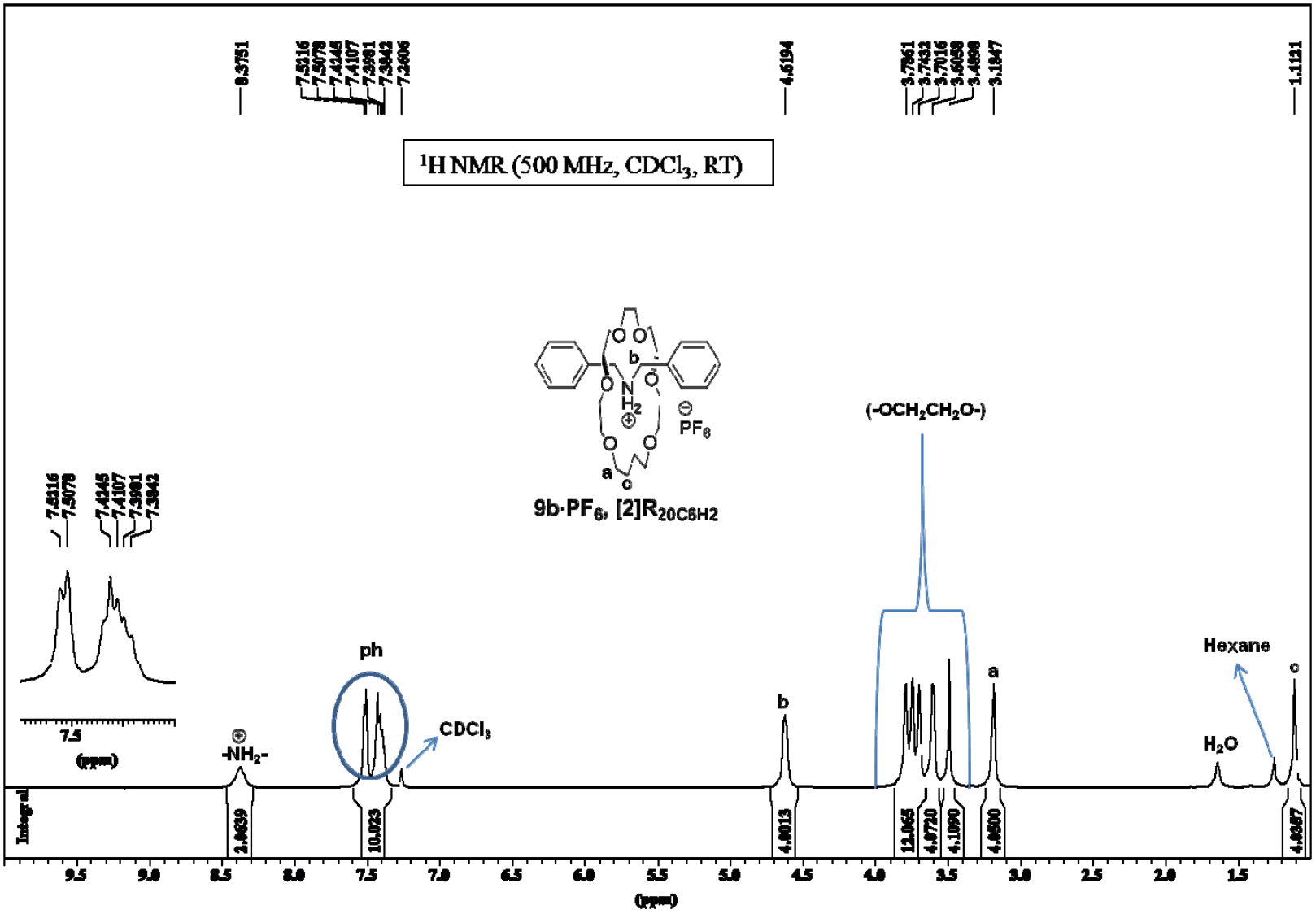


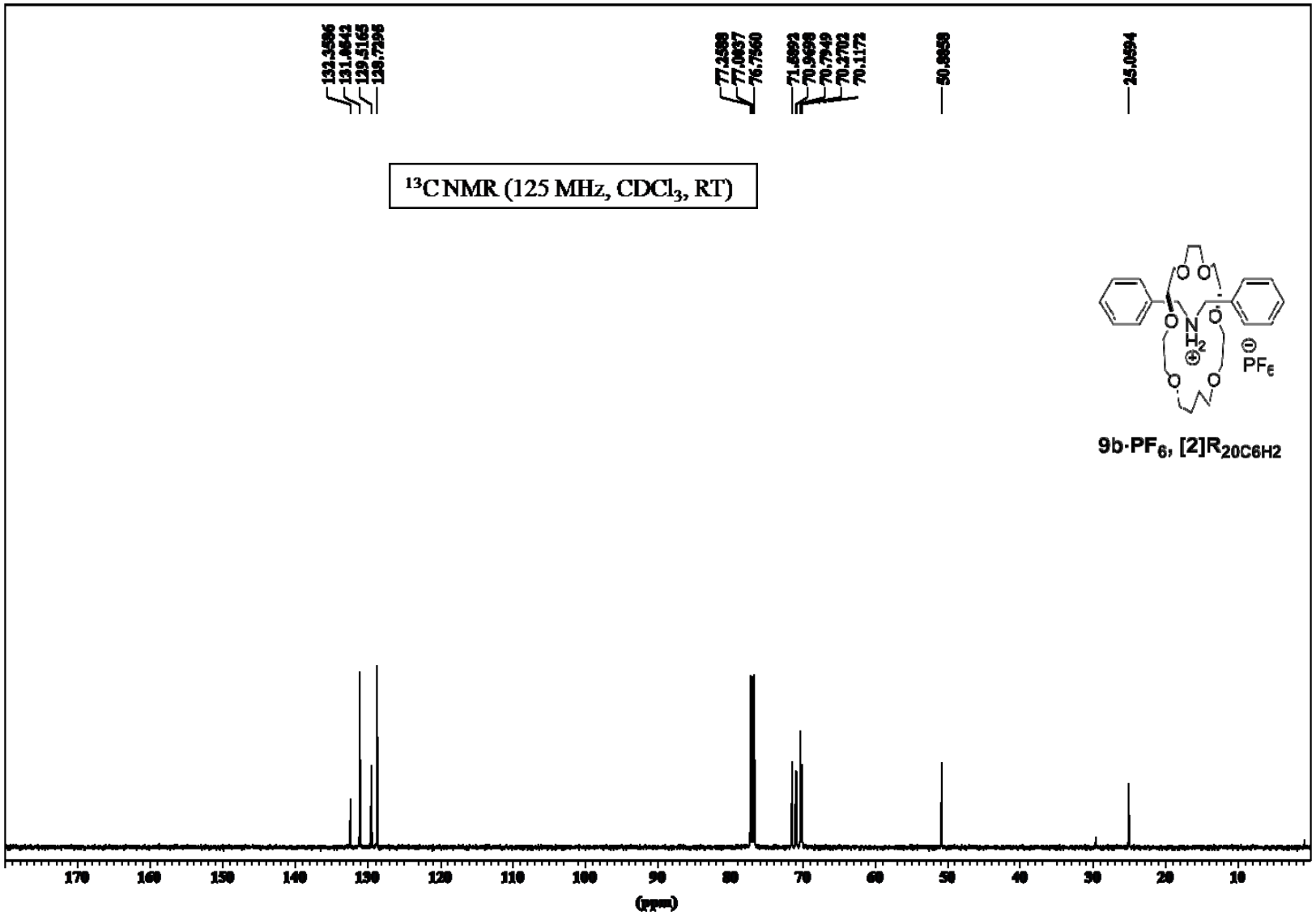


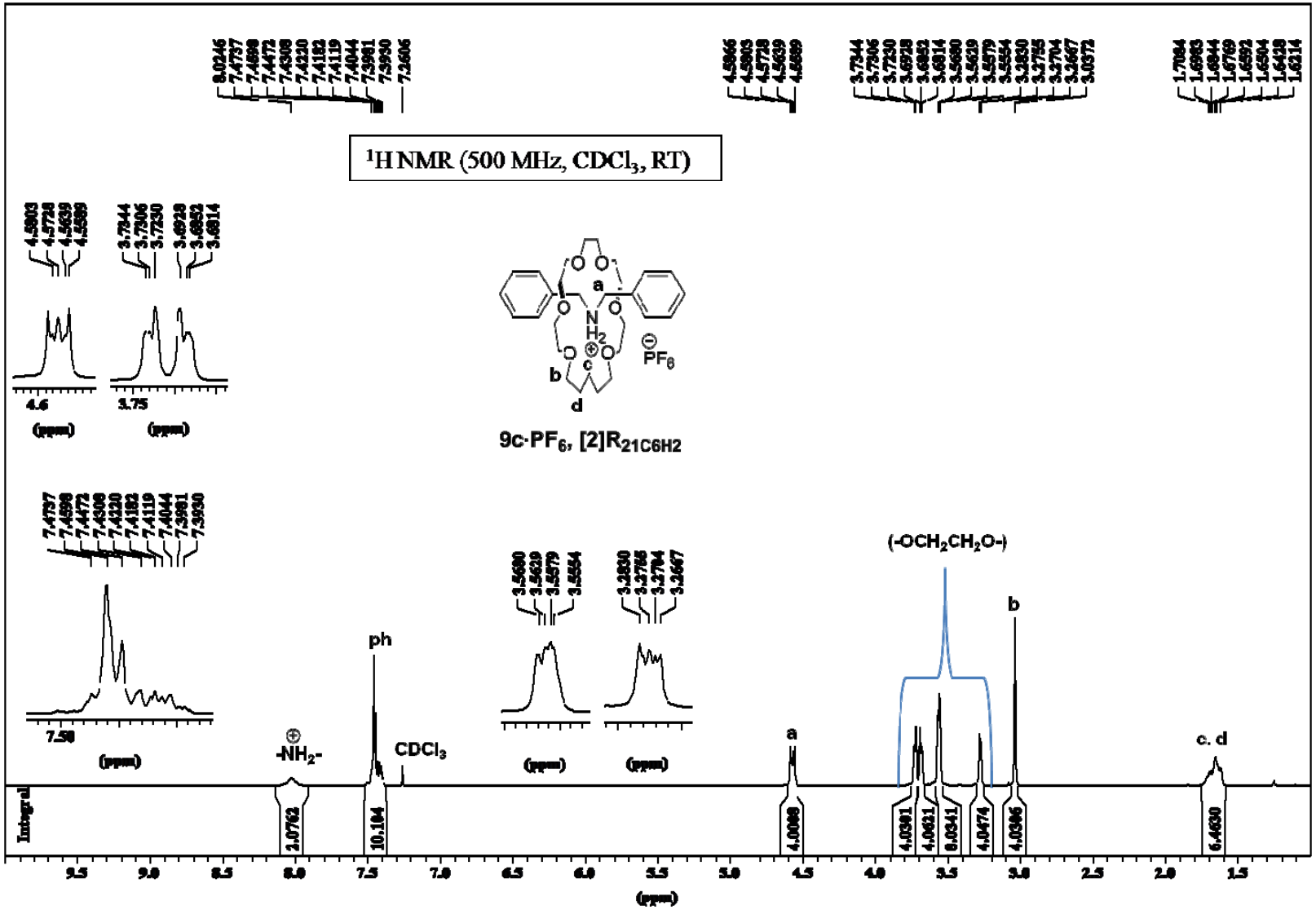


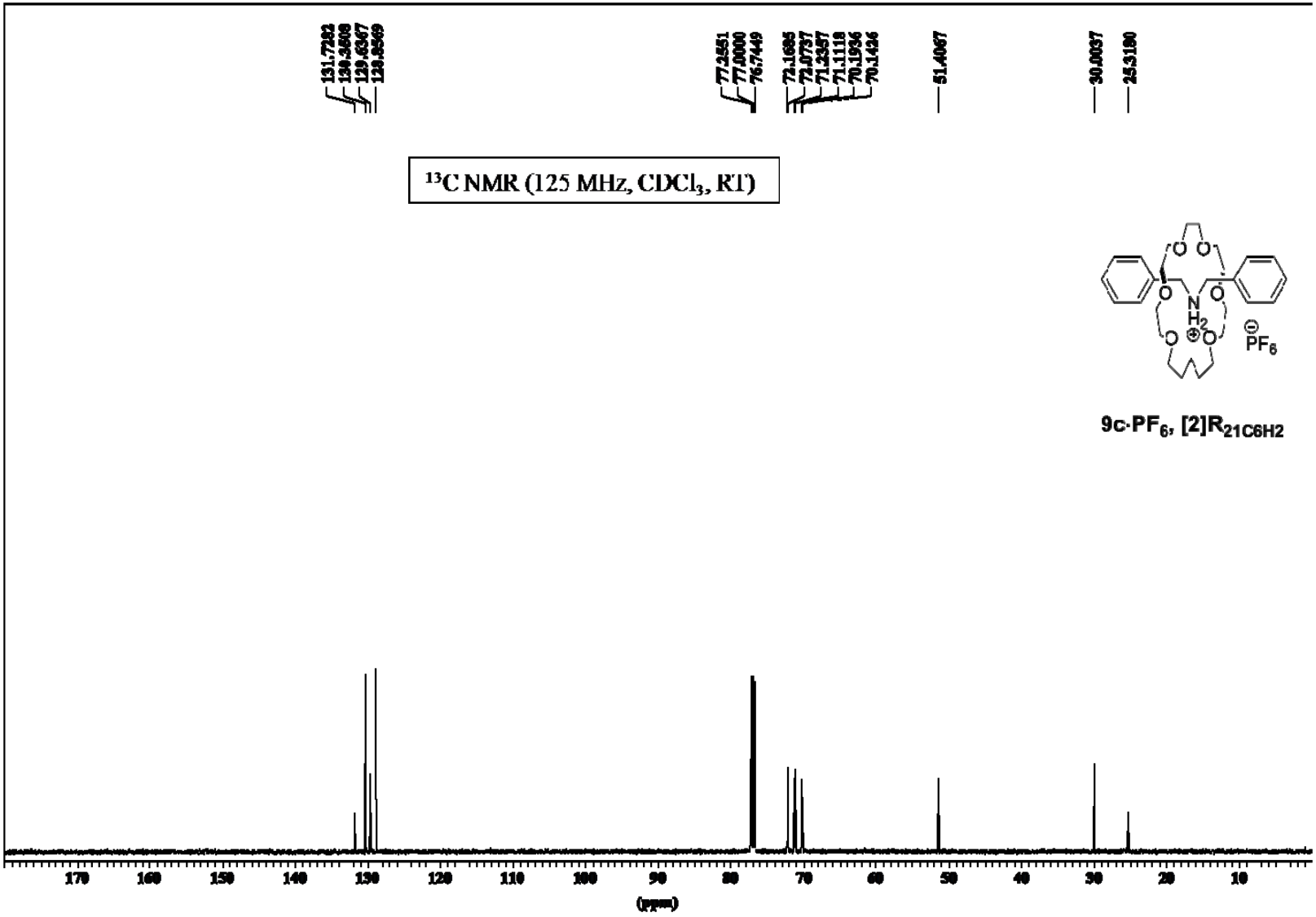


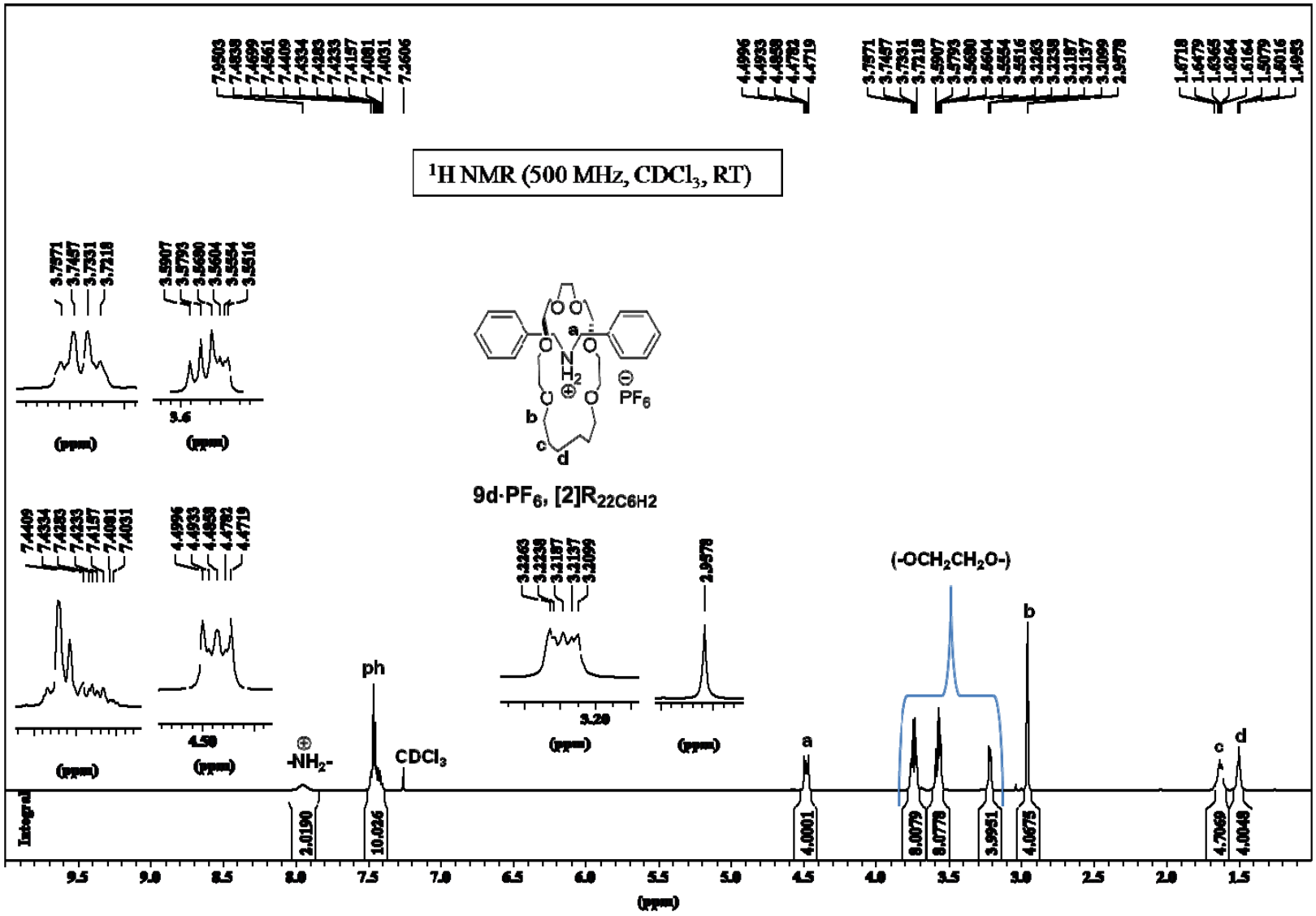


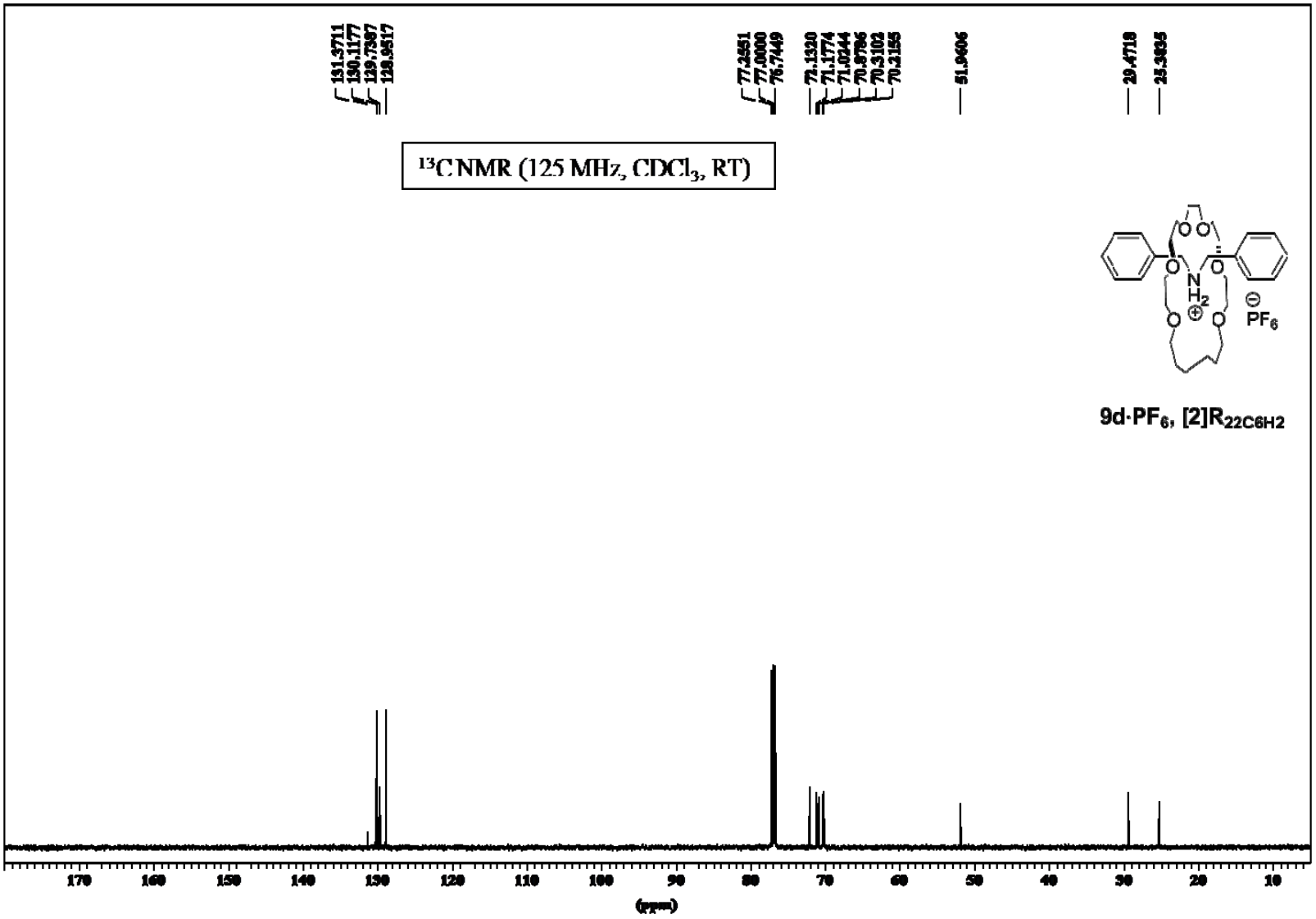




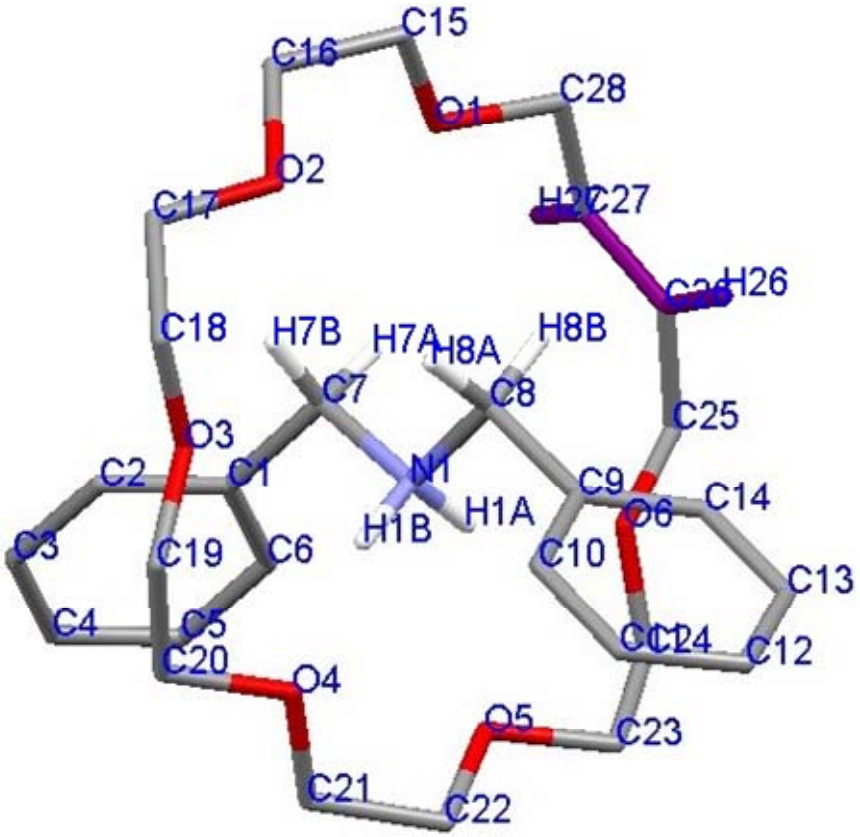








9. Detailed X-ray crystallographic analysis data



8b·PF$_6$

Table S3. Bond lengths [Å] and angles [°].

O(1)-C(15)	1.419(5)
O(1)-C(28)	1.427(5)
O(2)-C(16)	1.420(5)
O(2)-C(17)	1.425(5)
O(3)-C(19)	1.423(5)
O(3)-C(18)	1.426(4)
O(4)-C(20)	1.429(5)
O(4)-C(21)	1.434(4)
O(5)-C(23)	1.411(5)
O(5)-C(22)	1.413(4)
O(6)-C(25)	1.416(5)

O(6)-C(24)	1.416(5)
N(1)-C(7)	1.494(4)
N(1)-C(8)	1.501(4)
C(1)-C(6)	1.372(5)
C(1)-C(2)	1.378(5)
C(1)-C(7)	1.504(4)
C(2)-C(3)	1.380(6)
C(3)-C(4)	1.346(6)
C(4)-C(5)	1.373(5)
C(5)-C(6)	1.394(5)
C(8)-C(9)	1.502(4)
C(9)-C(10)	1.365(5)
C(9)-C(14)	1.367(5)
C(10)-C(11)	1.391(6)
C(11)-C(12)	1.370(6)
C(12)-C(13)	1.343(6)
C(13)-C(14)	1.387(6)
C(15)-C(16)	1.501(6)
C(17)-C(18)	1.483(6)
C(19)-C(20)	1.498(6)
C(21)-C(22)	1.485(6)
C(23)-C(24)	1.467(8)
C(25)-C(26)	1.494(6)
C(26)-C(27)	1.312(6)
C(27)-C(28)	1.497(6)
P(1)-F(2)	1.571(6)
P(1)-F(1)	1.596(6)
P(1)-F(6)	1.598(5)
P(1)-F(3)	1.605(7)
P(1)-F(5)	1.606(7)
P(1)-F(4)	1.623(6)
P(2)-F(10)	1.582(6)
P(2)-F(7)	1.588(5)
P(2)-F(12)	1.597(5)
P(2)-F(9)	1.603(7)
P(2)-F(11)	1.607(7)
P(2)-F(8)	1.619(6)

C(15)-O(1)-C(28)	113.3(3)
C(16)-O(2)-C(17)	112.9(3)
C(19)-O(3)-C(18)	112.1(3)
C(20)-O(4)-C(21)	112.9(3)
C(23)-O(5)-C(22)	112.8(3)
C(25)-O(6)-C(24)	114.2(3)
C(7)-N(1)-C(8)	115.4(2)
C(6)-C(1)-C(2)	118.1(3)
C(6)-C(1)-C(7)	121.4(3)
C(2)-C(1)-C(7)	120.4(3)
C(1)-C(2)-C(3)	120.4(4)
C(4)-C(3)-C(2)	121.5(4)
C(3)-C(4)-C(5)	119.2(4)
C(4)-C(5)-C(6)	119.8(4)
C(1)-C(6)-C(5)	120.9(3)
N(1)-C(7)-C(1)	112.1(2)
N(1)-C(8)-C(9)	110.0(2)
C(10)-C(9)-C(14)	116.9(3)
C(10)-C(9)-C(8)	121.5(3)
C(14)-C(9)-C(8)	121.5(3)
C(9)-C(10)-C(11)	121.2(4)
C(12)-C(11)-C(10)	120.6(4)
C(13)-C(12)-C(11)	118.6(4)
C(12)-C(13)-C(14)	120.6(4)
C(9)-C(14)-C(13)	122.1(4)
O(1)-C(15)-C(16)	110.0(3)
O(2)-C(16)-C(15)	110.3(3)
O(2)-C(17)-C(18)	111.8(3)
O(3)-C(18)-C(17)	111.1(3)
O(3)-C(19)-C(20)	110.3(3)
O(4)-C(20)-C(19)	109.8(3)
O(4)-C(21)-C(22)	110.5(3)
O(5)-C(22)-C(21)	108.5(3)
O(5)-C(23)-C(24)	109.1(5)
O(6)-C(24)-C(23)	109.6(4)
O(6)-C(25)-C(26)	106.2(3)

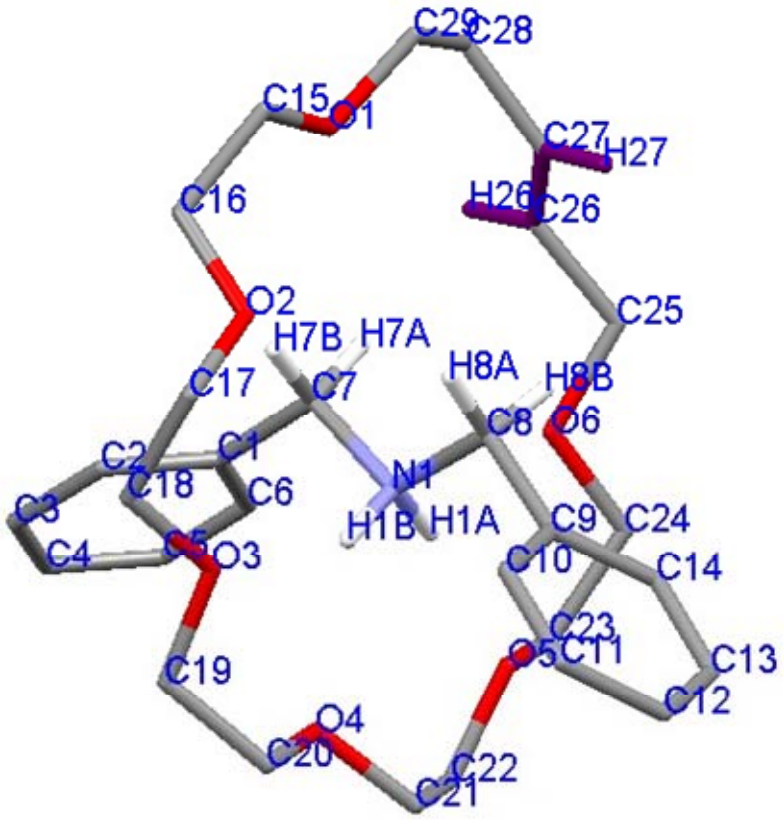
C(27)-C(26)-C(25)	125.1(4)
C(26)-C(27)-C(28)	124.7(4)
O(1)-C(28)-C(27)	105.7(3)
F(2)-P(1)-F(1)	90.5(4)
F(2)-P(1)-F(6)	92.1(4)
F(1)-P(1)-F(6)	90.2(4)
F(2)-P(1)-F(3)	91.6(5)
F(1)-P(1)-F(3)	176.7(6)
F(6)-P(1)-F(3)	87.2(7)
F(2)-P(1)-F(5)	90.0(5)
F(1)-P(1)-F(5)	92.9(7)
F(6)-P(1)-F(5)	176.2(6)
F(3)-P(1)-F(5)	89.6(8)
F(2)-P(1)-F(4)	179.2(5)
F(1)-P(1)-F(4)	89.6(4)
F(6)-P(1)-F(4)	88.7(4)
F(3)-P(1)-F(4)	88.3(5)
F(5)-P(1)-F(4)	89.2(5)
F(10)-P(2)-F(7)	90.8(4)
F(10)-P(2)-F(12)	89.9(3)
F(7)-P(2)-F(12)	90.3(3)
F(10)-P(2)-F(9)	90.4(5)
F(7)-P(2)-F(9)	176.7(8)
F(12)-P(2)-F(9)	92.7(8)
F(10)-P(2)-F(11)	91.8(4)
F(7)-P(2)-F(11)	87.2(8)
F(12)-P(2)-F(11)	177.0(7)
F(9)-P(2)-F(11)	89.7(10)
F(10)-P(2)-F(8)	179.3(4)
F(7)-P(2)-F(8)	88.8(4)
F(12)-P(2)-F(8)	89.6(4)
F(9)-P(2)-F(8)	90.0(5)
F(11)-P(2)-F(8)	88.7(5)

Symmetry transformations used to generate equivalent atoms:

Table S4. Hydrogen bonds [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1B)...O(3)	0.92	2.61	3.107(3)	114.7
N(1)-H(1B)...O(4)	0.92	1.89	2.797(3)	166.8
N(1)-H(1A)...O(5)	0.92	2.19	2.863(3)	129.7
N(1)-H(1A)...O(6)	0.92	2.11	2.949(3)	150.6

Symmetry transformations used to generate equivalent atoms:



8c·PF$_6$

Table S5. Bond lengths [Å] and angles [°].

O(1)-C(15)	1.425(3)
O(1)-C(29)	1.431(3)

O(2)-C(16)	1.423(3)
O(2)-C(17)	1.436(3)
O(3)-C(18)	1.419(3)
O(3)-C(19)	1.422(3)
O(4)-C(20)	1.423(2)
O(4)-C(21)	1.427(2)
O(5)-C(23)	1.430(2)
O(5)-C(22)	1.432(2)
O(6)-C(25)	1.427(3)
O(6)-C(24)	1.427(3)
N(1)-C(7)	1.492(2)
N(1)-C(8)	1.504(2)
C(1)-C(2)	1.390(3)
C(1)-C(6)	1.392(3)
C(1)-C(7)	1.509(3)
C(2)-C(3)	1.391(3)
C(3)-C(4)	1.383(3)
C(4)-C(5)	1.392(3)
C(5)-C(6)	1.379(3)
C(8)-C(9)	1.506(3)
C(9)-C(14)	1.392(3)
C(9)-C(10)	1.393(3)
C(10)-C(11)	1.385(3)
C(11)-C(12)	1.385(3)
C(12)-C(13)	1.380(3)
C(13)-C(14)	1.387(3)
C(15)-C(16)	1.501(4)
C(17)-C(18)	1.478(4)
C(19)-C(20)	1.497(3)
C(21)-C(22)	1.493(3)
C(23)-C(24)	1.497(3)
C(25)-C(26)	1.496(3)
C(26)-C(27)	1.311(3)
C(27)-C(28)	1.494(3)
C(28)-C(29)	1.510(3)
P(1)-F(6)	1.5965(14)

P(1)-F(3)	1.5978(14)
P(1)-F(2)	1.6002(14)
P(1)-F(1)	1.6008(14)
P(1)-F(5)	1.6036(14)
P(1)-F(4)	1.6058(14)

C(15)-O(1)-C(29)	111.48(17)
C(16)-O(2)-C(17)	117.08(18)
C(18)-O(3)-C(19)	112.31(17)
C(20)-O(4)-C(21)	111.57(15)
C(23)-O(5)-C(22)	111.59(15)
C(25)-O(6)-C(24)	111.11(16)
C(7)-N(1)-C(8)	114.37(15)
C(2)-C(1)-C(6)	118.76(19)
C(2)-C(1)-C(7)	121.06(18)
C(6)-C(1)-C(7)	120.16(18)
C(1)-C(2)-C(3)	120.5(2)
C(4)-C(3)-C(2)	120.0(2)
C(3)-C(4)-C(5)	119.8(2)
C(6)-C(5)-C(4)	119.9(2)
C(5)-C(6)-C(1)	121.0(2)
N(1)-C(7)-C(1)	111.71(16)
N(1)-C(8)-C(9)	110.37(15)
C(14)-C(9)-C(10)	118.53(19)
C(14)-C(9)-C(8)	121.36(18)
C(10)-C(9)-C(8)	120.09(18)
C(11)-C(10)-C(9)	121.1(2)
C(12)-C(11)-C(10)	119.7(2)
C(13)-C(12)-C(11)	119.8(2)
C(12)-C(13)-C(14)	120.5(2)
C(13)-C(14)-C(9)	120.3(2)
O(1)-C(15)-C(16)	110.85(18)
O(2)-C(16)-C(15)	110.12(19)
O(2)-C(17)-C(18)	114.4(2)
O(3)-C(18)-C(17)	109.8(2)
O(3)-C(19)-C(20)	109.50(17)
O(4)-C(20)-C(19)	108.71(17)

O(4)-C(21)-C(22)	109.38(16)
O(5)-C(22)-C(21)	109.03(16)
O(5)-C(23)-C(24)	108.45(17)
O(6)-C(24)-C(23)	108.82(17)
O(6)-C(25)-C(26)	109.18(18)
C(27)-C(26)-C(25)	123.8(2)
C(26)-C(27)-C(28)	124.9(2)
C(27)-C(28)-C(29)	114.65(19)
O(1)-C(29)-C(28)	110.50(18)
F(6)-P(1)-F(3)	90.37(8)
F(6)-P(1)-F(2)	90.01(8)
F(3)-P(1)-F(2)	90.64(8)
F(6)-P(1)-F(1)	90.11(8)
F(3)-P(1)-F(1)	179.11(9)
F(2)-P(1)-F(1)	90.11(8)
F(6)-P(1)-F(5)	179.78(9)
F(3)-P(1)-F(5)	89.42(8)
F(2)-P(1)-F(5)	89.93(8)
F(1)-P(1)-F(5)	90.10(8)
F(6)-P(1)-F(4)	90.07(8)
F(3)-P(1)-F(4)	89.58(8)
F(2)-P(1)-F(4)	179.77(9)
F(1)-P(1)-F(4)	89.67(8)
F(5)-P(1)-F(4)	89.99(8)

Symmetry transformations used to generate equivalent atoms:

Table S6. Hydrogen bonds [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1B)...O(4)	0.92	2.22	2.868(2)	126.8
N(1)-H(1B)...O(3)	0.92	2.18	3.009(2)	150.1
N(1)-H(1A)...O(6)	0.92	2.60	3.178(2)	121.5
N(1)-H(1A)...O(5)	0.92	2.02	2.919(2)	163.8

Symmetry transformations used to generate equivalent atoms:

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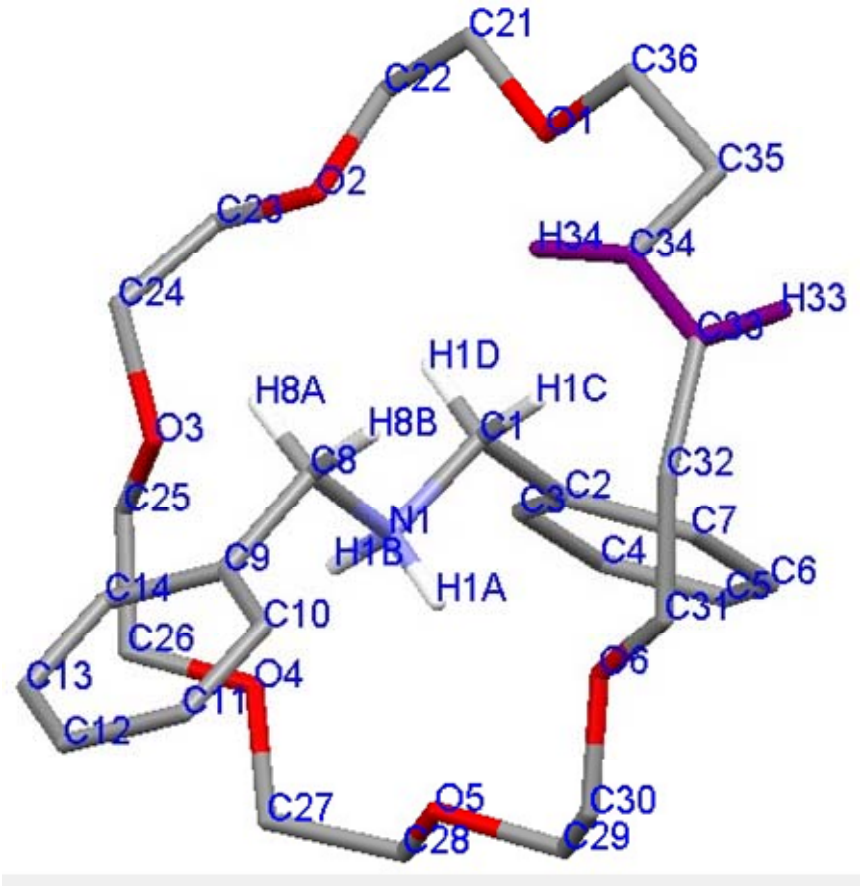

8d·PF$_6$

Table S7. Bond lengths [Å] and angles [°].

O(1)-C(36)	1.408(9)
O(1)-C(21)	1.464(9)
O(2)-C(23)	1.408(13)
O(2)-C(22)	1.662(10)
O(3)-C(25)	1.424(10)
O(3)-C(24)	1.441(11)
O(4)-C(27)	1.413(9)
O(4)-C(26)	1.425(10)
O(5)-C(28)	1.425(11)
O(5)-C(29)	1.432(11)
O(6)-C(30)	1.448(12)
O(6)-C(31)	1.469(8)
C(21)-C(22)	1.490(12)

C(23)-C(24)	1.480(13)
C(25)-C(26)	1.494(14)
C(27)-C(28)	1.485(13)
C(29)-C(30)	1.489(14)
C(31)-C(32)	1.518(5)
C(32)-C(33)	1.546(5)
C(33)-C(34)	1.354(5)
C(34)-C(35)	1.505(5)
C(35)-C(36)	1.497(5)
O(11)-C(56)	1.416(16)
O(11)-C(41)	1.430(16)
O(12)-C(42)	1.403(13)
O(12)-C(43)	1.445(18)
O(13)-C(44)	1.393(15)
O(13)-C(45)	1.418(16)
O(14)-C(47)	1.445(9)
O(14)-C(46)	1.481(9)
O(15)-C(49)	1.243(13)
O(15)-C(48)	1.455(9)
O(16)-C(50)	1.320(15)
O(16)-C(51)	1.459(18)
C(41)-C(42)	1.488(9)
C(43)-C(44)	1.496(10)
C(45)-C(46)	1.464(10)
C(47)-C(48)	1.552(9)
C(49)-C(50)	1.504(9)
C(51)-C(52)	1.504(5)
C(52)-C(53)	1.516(5)
C(53)-C(54)	1.322(5)
C(54)-C(55)	1.500(5)
C(55)-C(56)	1.499(5)
P(1)-F(1)	1.594(3)
P(1)-F(2)	1.595(3)
P(1)-F(6)	1.600(3)
P(1)-F(5)	1.603(3)
P(1)-F(4)	1.606(3)
P(1)-F(3)	1.607(3)

N(1)-C(1)	1.494(5)
N(1)-C(8)	1.503(4)
C(1)-C(2)	1.506(5)
C(2)-C(3)	1.379(6)
C(2)-C(7)	1.389(6)
C(3)-C(4)	1.389(6)
C(4)-C(5)	1.375(7)
C(5)-C(6)	1.372(7)
C(6)-C(7)	1.384(7)
C(8)-C(9)	1.503(5)
C(9)-C(10)	1.380(5)
C(9)-C(14)	1.391(5)
C(10)-C(11)	1.381(6)
C(11)-C(12)	1.378(6)
C(12)-C(13)	1.370(6)
C(13)-C(14)	1.381(6)

C(36)-O(1)-C(21)	109.4(6)
C(23)-O(2)-C(22)	91.4(7)
C(25)-O(3)-C(24)	114.0(6)
C(27)-O(4)-C(26)	111.5(6)
C(28)-O(5)-C(29)	111.1(6)
C(30)-O(6)-C(31)	119.6(7)
O(1)-C(21)-C(22)	113.5(7)
C(21)-C(22)-O(2)	92.2(6)
O(2)-C(23)-C(24)	109.9(9)
O(3)-C(24)-C(23)	114.0(7)
O(3)-C(25)-C(26)	109.5(7)
O(4)-C(26)-C(25)	109.3(7)
O(4)-C(27)-C(28)	109.4(7)
O(5)-C(28)-C(27)	109.9(7)
O(5)-C(29)-C(30)	109.4(8)
O(6)-C(30)-C(29)	111.1(8)
O(6)-C(31)-C(32)	118.3(9)
C(31)-C(32)-C(33)	148.8(11)
C(34)-C(33)-C(32)	134.2(8)

C(33)-C(34)-C(35)	112.2(7)
C(36)-C(35)-C(34)	105.3(6)
O(1)-C(36)-C(35)	108.6(6)
C(56)-O(11)-C(41)	110.4(9)
C(42)-O(12)-C(43)	111.6(9)
C(44)-O(13)-C(45)	112.1(10)
C(47)-O(14)-C(46)	109.6(8)
C(49)-O(15)-C(48)	109.6(9)
C(50)-O(16)-C(51)	111.7(10)
O(11)-C(41)-C(42)	111.3(11)
O(12)-C(42)-C(41)	110.7(11)
O(12)-C(43)-C(44)	108.6(14)
O(13)-C(44)-C(43)	111.0(12)
O(13)-C(45)-C(46)	119.1(13)
C(45)-C(46)-O(14)	106.8(11)
O(14)-C(47)-C(48)	91.3(6)
O(15)-C(48)-C(47)	93.5(6)
O(15)-C(49)-C(50)	100.1(10)
O(16)-C(50)-C(49)	110.5(11)
O(16)-C(51)-C(52)	115.7(12)
C(51)-C(52)-C(53)	137.6(12)
C(54)-C(53)-C(52)	126.6(12)
C(53)-C(54)-C(55)	130.5(12)
C(56)-C(55)-C(54)	115.7(11)
O(11)-C(56)-C(55)	111.5(12)
F(1)-P(1)-F(2)	179.63(16)
F(1)-P(1)-F(6)	90.13(15)
F(2)-P(1)-F(6)	89.97(15)
F(1)-P(1)-F(5)	89.97(15)
F(2)-P(1)-F(5)	89.94(15)
F(6)-P(1)-F(5)	179.87(17)
F(1)-P(1)-F(4)	90.12(15)
F(2)-P(1)-F(4)	89.52(15)
F(6)-P(1)-F(4)	89.96(14)
F(5)-P(1)-F(4)	90.12(14)
F(1)-P(1)-F(3)	90.17(15)

F(2)-P(1)-F(3)	90.18(15)
F(6)-P(1)-F(3)	90.18(14)
F(5)-P(1)-F(3)	89.73(14)
F(4)-P(1)-F(3)	179.67(16)
C(1)-N(1)-C(8)	111.7(3)
N(1)-C(1)-C(2)	112.7(3)
C(3)-C(2)-C(7)	118.5(4)
C(3)-C(2)-C(1)	121.1(4)
C(7)-C(2)-C(1)	120.3(4)
C(2)-C(3)-C(4)	120.6(4)
C(5)-C(4)-C(3)	120.6(4)
C(6)-C(5)-C(4)	119.2(4)
C(5)-C(6)-C(7)	120.6(5)
C(6)-C(7)-C(2)	120.6(4)
N(1)-C(8)-C(9)	112.1(3)
C(10)-C(9)-C(14)	119.3(4)
C(10)-C(9)-C(8)	120.4(3)
C(14)-C(9)-C(8)	120.4(4)
C(9)-C(10)-C(11)	120.7(4)
C(12)-C(11)-C(10)	119.9(4)
C(13)-C(12)-C(11)	119.6(4)
C(12)-C(13)-C(14)	121.1(4)
C(13)-C(14)-C(9)	119.5(4)

Symmetry transformations used to generate equivalent atoms:

Table S8. Hydrogen bonds [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1B)...O(3)	0.92	2.47	3.073(6)	123.0
N(1)-H(1B)...O(11)	0.92	2.22	3.072(9)	154.2
N(1)-H(1B)...O(12)	0.92	2.16	2.793(9)	125.3
N(1)-H(1A)...O(14)	0.92	2.65	3.119(8)	112.6
N(1)-H(1A)...O(6)	0.92	2.28	3.102(6)	149.3
N(1)-H(1A)...O(5)	0.92	2.25	2.923(6)	129.7
N(1)-H(1A)...O(13)	0.92	1.83	2.737(8)	167.5

Symmetry transformations used to generate equivalent atoms:

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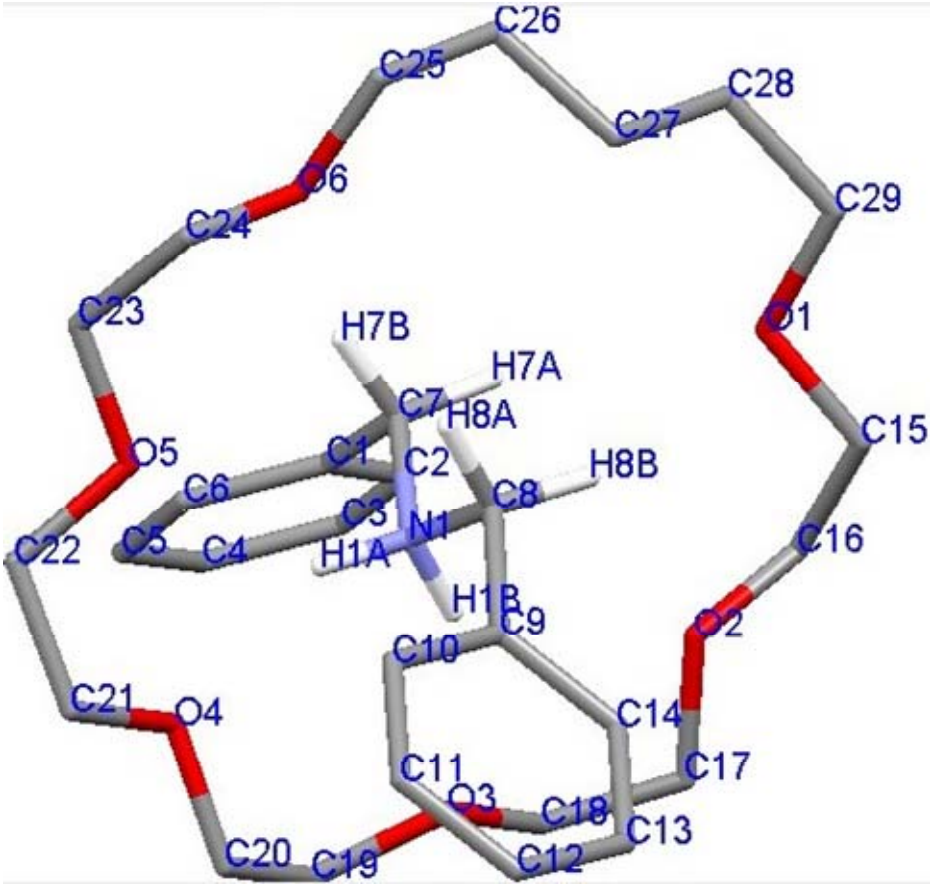

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Table S9. Bond lengths [Å] and angles [°].

N(1)-C(8)	1.491(3)
N(1)-C(7)	1.493(3)
O(1)-C(15)	1.412(3)
O(1)-C(29)	1.426(3)
O(2)-C(16)	1.413(3)
O(2)-C(17)	1.433(3)
O(3)-C(18)	1.410(3)
O(3)-C(19)	1.426(3)
O(4)-C(20)	1.423(3)
O(4)-C(21)	1.424(3)
O(5)-C(23)	1.426(3)
O(5)-C(22)	1.430(3)
O(6)-C(24)	1.419(3)

O(6)-C(25)	1.428(3)
C(1)-C(2)	1.380(4)
C(1)-C(6)	1.384(3)
C(1)-C(7)	1.504(3)
C(2)-C(3)	1.388(4)
C(3)-C(4)	1.363(5)
C(4)-C(5)	1.369(5)
C(5)-C(6)	1.389(4)
C(8)-C(9)	1.502(3)
C(9)-C(10)	1.385(3)
C(9)-C(14)	1.395(3)
C(10)-C(11)	1.385(4)
C(11)-C(12)	1.376(4)
C(12)-C(13)	1.381(4)
C(13)-C(14)	1.382(4)
C(15)-C(16)	1.489(4)
C(17)-C(18)	1.463(4)
C(19)-C(20)	1.495(4)
C(21)-C(22)	1.488(4)
C(23)-C(24)	1.492(4)
C(25)-C(26)	1.509(4)
C(26)-C(27)	1.508(4)
C(27)-C(28)	1.518(4)
C(28)-C(29)	1.500(4)
P(1)-F(5)	1.554(2)
P(1)-F(6)	1.561(3)
P(1)-F(1)	1.562(2)
P(1)-F(3)	1.572(2)
P(1)-F(4)	1.581(2)
P(1)-F(2)	1.589(2)

C(8)-N(1)-C(7)	114.97(17)
C(15)-O(1)-C(29)	111.80(19)
C(16)-O(2)-C(17)	114.7(2)
C(18)-O(3)-C(19)	113.4(2)
C(20)-O(4)-C(21)	112.83(19)

C(23)-O(5)-C(22)	111.86(18)
C(24)-O(6)-C(25)	111.44(19)
C(2)-C(1)-C(6)	118.2(2)
C(2)-C(1)-C(7)	120.5(2)
C(6)-C(1)-C(7)	121.3(2)
C(1)-C(2)-C(3)	121.1(3)
C(4)-C(3)-C(2)	119.7(3)
C(3)-C(4)-C(5)	120.4(3)
C(4)-C(5)-C(6)	119.9(3)
C(1)-C(6)-C(5)	120.7(3)
N(1)-C(7)-C(1)	110.84(18)
N(1)-C(8)-C(9)	111.38(17)
C(10)-C(9)-C(14)	118.7(2)
C(10)-C(9)-C(8)	121.1(2)
C(14)-C(9)-C(8)	120.2(2)
C(9)-C(10)-C(11)	120.5(2)
C(12)-C(11)-C(10)	120.3(3)
C(11)-C(12)-C(13)	120.0(2)
C(12)-C(13)-C(14)	119.8(2)
C(13)-C(14)-C(9)	120.7(2)
O(1)-C(15)-C(16)	111.5(2)
O(2)-C(16)-C(15)	111.6(2)
O(2)-C(17)-C(18)	113.9(2)
O(3)-C(18)-C(17)	110.6(3)
O(3)-C(19)-C(20)	109.8(2)
O(4)-C(20)-C(19)	109.7(2)
O(4)-C(21)-C(22)	109.4(2)
O(5)-C(22)-C(21)	110.1(2)
O(5)-C(23)-C(24)	112.0(2)
O(6)-C(24)-C(23)	112.1(2)
O(6)-C(25)-C(26)	110.1(2)
C(27)-C(26)-C(25)	114.4(2)
C(26)-C(27)-C(28)	114.6(2)
C(29)-C(28)-C(27)	113.7(2)
O(1)-C(29)-C(28)	109.5(2)
F(5)-P(1)-F(6)	179.08(18)

F(5)-P(1)-F(1)	90.33(19)
F(6)-P(1)-F(1)	89.92(18)
F(5)-P(1)-F(3)	91.41(17)
F(6)-P(1)-F(3)	88.34(17)
F(1)-P(1)-F(3)	178.26(18)
F(5)-P(1)-F(4)	89.09(13)
F(6)-P(1)-F(4)	91.79(18)
F(1)-P(1)-F(4)	89.90(14)
F(3)-P(1)-F(4)	90.01(12)
F(5)-P(1)-F(2)	89.39(15)
F(6)-P(1)-F(2)	89.73(18)
F(1)-P(1)-F(2)	90.67(14)
F(3)-P(1)-F(2)	89.47(12)
F(4)-P(1)-F(2)	178.38(15)

Symmetry transformations used to generate equivalent atoms:

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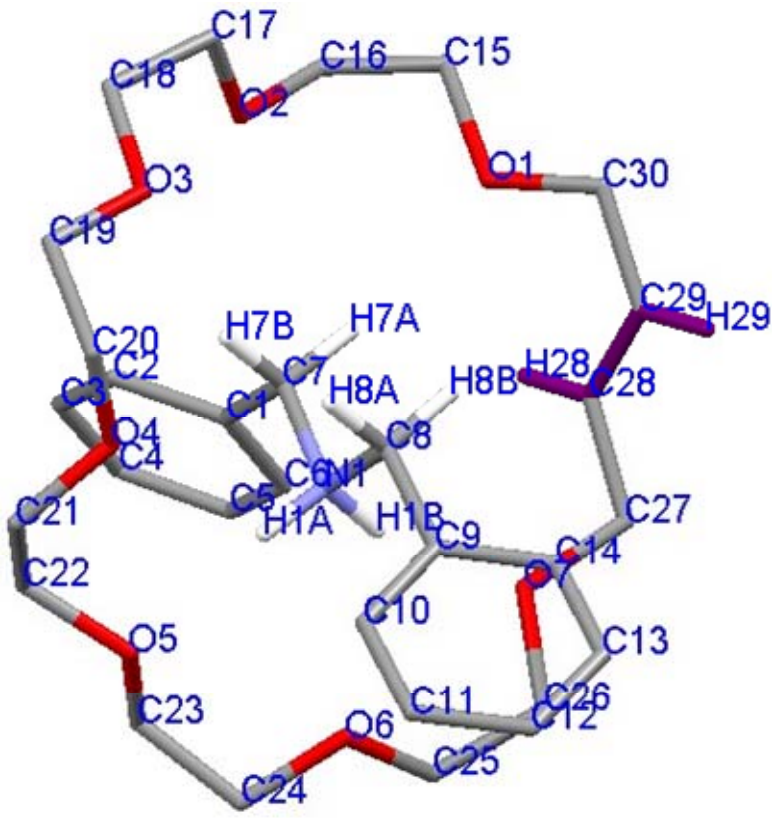

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Table S10. Bond lengths [Å] and angles [°].

O(1)-C(30)	1.419(3)
O(1)-C(15)	1.423(3)
O(2)-C(17)	1.422(3)
O(2)-C(16)	1.431(3)
O(3)-C(18)	1.421(3)
O(3)-C(19)	1.424(3)
O(4)-C(21)	1.428(3)
O(4)-C(20)	1.431(3)
O(5)-C(23)	1.425(3)
O(5)-C(22)	1.436(3)
O(6)-C(25)	1.383(3)
O(6)-C(24)	1.413(3)
O(7)-C(26)	1.421(3)
O(7)-C(27)	1.430(3)

N(1)-C(8)	1.497(3)
N(1)-C(7)	1.502(3)
C(1)-C(2)	1.391(3)
C(1)-C(6)	1.398(3)
C(1)-C(7)	1.506(3)
C(2)-C(3)	1.386(3)
C(3)-C(4)	1.381(3)
C(4)-C(5)	1.391(3)
C(5)-C(6)	1.386(3)
C(8)-C(9)	1.508(3)
C(9)-C(14)	1.391(3)
C(9)-C(10)	1.392(3)
C(10)-C(11)	1.386(3)
C(11)-C(12)	1.387(4)
C(12)-C(13)	1.374(4)
C(13)-C(14)	1.389(3)
C(15)-C(16)	1.508(3)
C(17)-C(18)	1.500(3)
C(19)-C(20)	1.506(3)
C(21)-C(22)	1.489(3)
C(23)-C(24)	1.494(4)
C(25)-C(26)	1.496(4)
C(27)-C(28)	1.484(3)
C(28)-C(29)	1.325(3)
C(29)-C(30)	1.491(3)
P(1)-F(5)	1.5942(14)
P(1)-F(6)	1.5970(15)
P(1)-F(3)	1.6003(15)
P(1)-F(1)	1.6025(15)
P(1)-F(4)	1.6057(16)
P(1)-F(2)	1.6065(15)

C(30)-O(1)-C(15)	112.71(18)
C(17)-O(2)-C(16)	113.52(18)
C(18)-O(3)-C(19)	111.81(17)
C(21)-O(4)-C(20)	113.06(17)
C(23)-O(5)-C(22)	112.02(18)

C(25)-O(6)-C(24)	114.80(19)
C(26)-O(7)-C(27)	114.05(17)
C(8)-N(1)-C(7)	110.82(16)
C(2)-C(1)-C(6)	118.6(2)
C(2)-C(1)-C(7)	119.6(2)
C(6)-C(1)-C(7)	121.77(19)
C(3)-C(2)-C(1)	121.2(2)
C(4)-C(3)-C(2)	119.8(2)
C(3)-C(4)-C(5)	119.9(2)
C(6)-C(5)-C(4)	120.3(2)
C(5)-C(6)-C(1)	120.3(2)
N(1)-C(7)-C(1)	113.22(17)
N(1)-C(8)-C(9)	112.96(17)
C(14)-C(9)-C(10)	119.0(2)
C(14)-C(9)-C(8)	119.7(2)
C(10)-C(9)-C(8)	121.2(2)
C(11)-C(10)-C(9)	120.4(2)
C(10)-C(11)-C(12)	120.0(2)
C(13)-C(12)-C(11)	120.1(2)
C(12)-C(13)-C(14)	120.2(2)
C(13)-C(14)-C(9)	120.4(2)
O(1)-C(15)-C(16)	108.35(19)
O(2)-C(16)-C(15)	114.12(19)
O(2)-C(17)-C(18)	108.45(19)
O(3)-C(18)-C(17)	109.37(19)
O(3)-C(19)-C(20)	108.74(18)
O(4)-C(20)-C(19)	112.29(19)
O(4)-C(21)-C(22)	109.25(18)
O(5)-C(22)-C(21)	109.01(19)
O(5)-C(23)-C(24)	109.2(2)
O(6)-C(24)-C(23)	110.6(2)
O(6)-C(25)-C(26)	109.7(2)
O(7)-C(26)-C(25)	109.87(19)
O(7)-C(27)-C(28)	107.28(17)
C(29)-C(28)-C(27)	125.2(2)
C(28)-C(29)-C(30)	124.2(2)
O(1)-C(30)-C(29)	109.57(19)

F(5)-P(1)-F(6)	179.89(11)
F(5)-P(1)-F(3)	90.19(8)
F(6)-P(1)-F(3)	89.79(8)
F(5)-P(1)-F(1)	89.74(8)
F(6)-P(1)-F(1)	90.28(8)
F(3)-P(1)-F(1)	179.84(10)
F(5)-P(1)-F(4)	89.85(8)
F(6)-P(1)-F(4)	90.27(9)
F(3)-P(1)-F(4)	90.06(8)
F(1)-P(1)-F(4)	89.80(9)
F(5)-P(1)-F(2)	89.62(8)
F(6)-P(1)-F(2)	90.26(9)
F(3)-P(1)-F(2)	89.96(8)
F(1)-P(1)-F(2)	90.19(8)
F(4)-P(1)-F(2)	179.47(9)

Symmetry transformations used to generate equivalent atoms:

Table S11. Hydrogen bonds [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1B)...O(6)	0.92	2.41	2.969(2)	119.2
N(1)-H(1B)...O(7)	0.92	1.99	2.874(2)	161.0
N(1)-H(1A)...O(4)	0.92	2.50	3.017(2)	116.3
N(1)-H(1A)...O(5)	0.92	2.05	2.954(2)	166.1

Symmetry transformations used to generate equivalent atoms:

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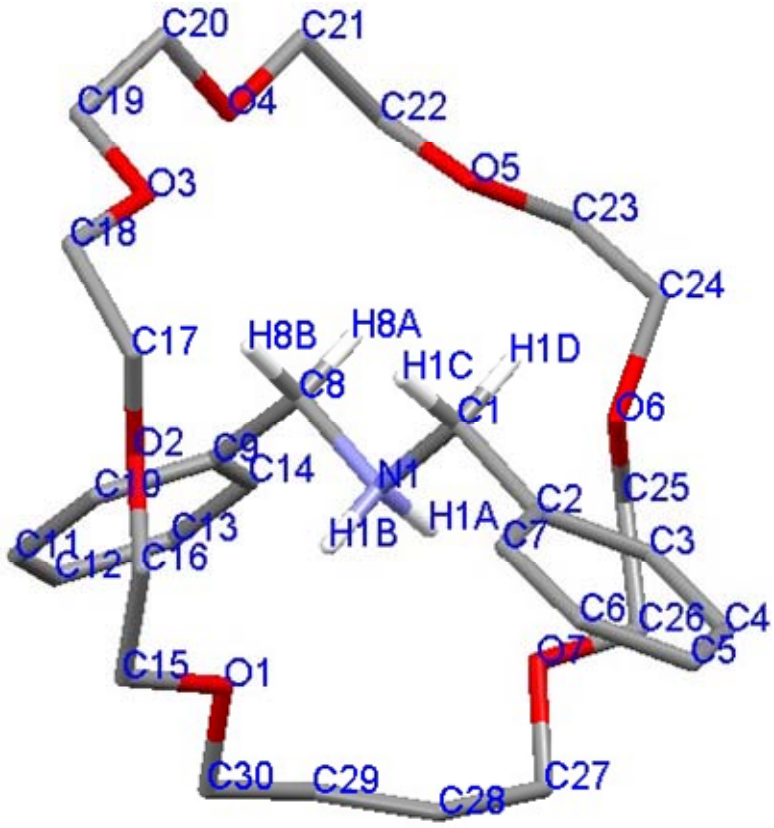

9f ·PF_6

Table S12. Bond lengths [Å] and angles [°].

O(1)-C(15)	1.424(3)
O(1)-C(30)	1.430(3)
O(2)-C(16)	1.419(3)
O(2)-C(17)	1.421(3)
O(3)-C(18)	1.412(3)
O(3)-C(19)	1.425(3)
O(4)-C(21)	1.421(3)
O(4)-C(20)	1.423(3)
O(5)-C(22)	1.421(3)
O(5)-C(23)	1.426(3)
O(6)-C(24)	1.428(3)
O(6)-C(25)	1.435(3)
O(7)-C(26)	1.418(3)
O(7)-C(27)	1.435(3)

O(8)-C(45)	1.419(3)
O(8)-C(60)	1.430(3)
O(9)-C(46)	1.424(3)
O(9)-C(47)	1.425(3)
O(10)-C(49)	1.423(3)
O(10)-C(48)	1.433(3)
O(11)-C(50)	1.427(3)
O(11)-C(51)	1.438(3)
O(12)-C(53)	1.429(3)
O(12)-C(52)	1.437(3)
O(13)-C(55)	1.430(3)
O(13)-C(54)	1.435(3)
O(14)-C(57)	1.419(3)
O(14)-C(56)	1.419(3)
N(1)-C(1)	1.503(3)
N(1)-C(8)	1.503(3)
N(2)-C(38)	1.501(3)
N(2)-C(31)	1.506(3)
C(1)-C(2)	1.499(4)
C(2)-C(7)	1.389(4)
C(2)-C(3)	1.400(3)
C(3)-C(4)	1.393(4)
C(4)-C(5)	1.386(4)
C(5)-C(6)	1.380(4)
C(6)-C(7)	1.387(4)
C(8)-C(9)	1.513(3)
C(9)-C(10)	1.388(3)
C(9)-C(14)	1.392(3)
C(10)-C(11)	1.395(3)
C(11)-C(12)	1.391(4)
C(12)-C(13)	1.389(4)
C(13)-C(14)	1.380(4)
C(15)-C(16)	1.509(4)
C(17)-C(18)	1.514(4)
C(19)-C(20)	1.497(4)
C(21)-C(22)	1.491(4)
C(23)-C(24)	1.510(4)

C(25)-C(26)	1.492(4)
C(27)-C(28)	1.497(4)
C(28)-C(29)	1.509(4)
C(29)-C(30)	1.526(4)
C(31)-C(32)	1.511(3)
C(32)-C(33)	1.393(4)
C(32)-C(37)	1.394(4)
C(33)-C(34)	1.387(4)
C(34)-C(35)	1.389(4)
C(35)-C(36)	1.385(4)
C(36)-C(37)	1.395(4)
C(38)-C(39)	1.517(3)
C(39)-C(40)	1.391(4)
C(39)-C(44)	1.402(3)
C(40)-C(41)	1.390(4)
C(41)-C(42)	1.388(4)
C(42)-C(43)	1.381(4)
C(43)-C(44)	1.385(4)
C(45)-C(46)	1.499(4)
C(47)-C(48)	1.493(4)
C(49)-C(50)	1.508(4)
C(51)-C(52)	1.496(4)
C(53)-C(54)	1.487(4)
C(55)-C(56)	1.505(4)
C(57)-C(58)	1.511(4)
C(58)-C(59)	1.526(3)
C(59)-C(60)	1.512(4)
P(1)-F(5)	1.6030(17)
P(1)-F(4)	1.6030(16)
P(1)-F(2)	1.6064(16)
P(1)-F(1)	1.6078(16)
P(1)-F(6)	1.6084(15)
P(1)-F(3)	1.6088(16)
P(2)-F(10)	1.6000(17)
P(2)-F(8)	1.6016(17)
P(2)-F(12)	1.6030(17)
P(2)-F(11)	1.6045(16)

P(2)-F(7)	1.6058(16)
P(2)-F(9)	1.6066(16)

C(15)-O(1)-C(30)	110.4(2)
C(16)-O(2)-C(17)	111.1(2)
C(18)-O(3)-C(19)	110.9(2)
C(21)-O(4)-C(20)	110.63(19)
C(22)-O(5)-C(23)	111.58(18)
C(24)-O(6)-C(25)	112.7(2)
C(26)-O(7)-C(27)	111.0(2)
C(45)-O(8)-C(60)	110.77(19)
C(46)-O(9)-C(47)	112.16(18)
C(49)-O(10)-C(48)	113.11(19)
C(50)-O(11)-C(51)	111.3(2)
C(53)-O(12)-C(52)	111.1(2)
C(55)-O(13)-C(54)	114.0(2)
C(57)-O(14)-C(56)	113.6(2)
C(1)-N(1)-C(8)	110.84(18)
C(38)-N(2)-C(31)	111.23(18)
C(2)-C(1)-N(1)	112.91(19)
C(7)-C(2)-C(3)	119.2(2)
C(7)-C(2)-C(1)	120.5(2)
C(3)-C(2)-C(1)	120.2(2)
C(4)-C(3)-C(2)	119.6(2)
C(5)-C(4)-C(3)	120.3(2)
C(6)-C(5)-C(4)	120.3(3)
C(5)-C(6)-C(7)	119.6(2)
C(6)-C(7)-C(2)	121.0(2)
N(1)-C(8)-C(9)	111.62(19)
C(10)-C(9)-C(14)	119.3(2)
C(10)-C(9)-C(8)	120.0(2)
C(14)-C(9)-C(8)	120.7(2)
C(9)-C(10)-C(11)	120.2(2)
C(12)-C(11)-C(10)	120.1(2)
C(13)-C(12)-C(11)	119.6(2)
C(14)-C(13)-C(12)	120.2(2)

C(13)-C(14)-C(9)	120.7(2)
O(1)-C(15)-C(16)	111.1(2)
O(2)-C(16)-C(15)	110.1(2)
O(2)-C(17)-C(18)	109.5(2)
O(3)-C(18)-C(17)	110.7(2)
O(3)-C(19)-C(20)	110.8(2)
O(4)-C(20)-C(19)	110.3(2)
O(4)-C(21)-C(22)	110.6(2)
O(5)-C(22)-C(21)	110.0(2)
O(5)-C(23)-C(24)	109.2(2)
O(6)-C(24)-C(23)	114.3(2)
O(6)-C(25)-C(26)	108.8(2)
O(7)-C(26)-C(25)	109.2(2)
O(7)-C(27)-C(28)	108.1(2)
C(27)-C(28)-C(29)	113.0(2)
C(28)-C(29)-C(30)	113.8(2)
O(1)-C(30)-C(29)	108.8(2)
N(2)-C(31)-C(32)	111.63(19)
C(33)-C(32)-C(37)	119.4(2)
C(33)-C(32)-C(31)	119.9(2)
C(37)-C(32)-C(31)	120.7(2)
C(34)-C(33)-C(32)	120.0(2)
C(33)-C(34)-C(35)	120.6(2)
C(36)-C(35)-C(34)	119.7(2)
C(35)-C(36)-C(37)	120.0(2)
C(32)-C(37)-C(36)	120.3(2)
N(2)-C(38)-C(39)	111.73(19)
C(40)-C(39)-C(44)	119.4(2)
C(40)-C(39)-C(38)	120.1(2)
C(44)-C(39)-C(38)	120.5(2)
C(41)-C(40)-C(39)	120.1(2)
C(42)-C(41)-C(40)	120.3(2)
C(43)-C(42)-C(41)	119.7(2)
C(42)-C(43)-C(44)	120.7(2)
C(43)-C(44)-C(39)	119.8(2)
O(8)-C(45)-C(46)	109.6(2)
O(9)-C(46)-C(45)	109.7(2)

O(9)-C(47)-C(48)	109.67(19)
O(10)-C(48)-C(47)	114.6(2)
O(10)-C(49)-C(50)	108.6(2)
O(11)-C(50)-C(49)	108.8(2)
O(11)-C(51)-C(52)	109.5(2)
O(12)-C(52)-C(51)	109.7(2)
O(12)-C(53)-C(54)	109.3(2)
O(13)-C(54)-C(53)	108.4(2)
O(13)-C(55)-C(56)	111.0(2)
O(14)-C(56)-C(55)	107.5(2)
O(14)-C(57)-C(58)	107.7(2)
C(57)-C(58)-C(59)	112.6(2)
C(60)-C(59)-C(58)	114.0(2)
O(8)-C(60)-C(59)	109.0(2)
F(5)-P(1)-F(4)	179.56(10)
F(5)-P(1)-F(2)	90.22(9)
F(4)-P(1)-F(2)	89.67(9)
F(5)-P(1)-F(1)	90.14(9)
F(4)-P(1)-F(1)	89.97(9)
F(2)-P(1)-F(1)	179.64(10)
F(5)-P(1)-F(6)	89.98(9)
F(4)-P(1)-F(6)	89.59(8)
F(2)-P(1)-F(6)	90.08(8)
F(1)-P(1)-F(6)	89.95(8)
F(5)-P(1)-F(3)	90.46(9)
F(4)-P(1)-F(3)	89.96(9)
F(2)-P(1)-F(3)	90.01(9)
F(1)-P(1)-F(3)	89.95(9)
F(6)-P(1)-F(3)	179.54(10)
F(10)-P(2)-F(8)	90.02(9)
F(10)-P(2)-F(12)	90.35(10)
F(8)-P(2)-F(12)	89.89(9)
F(10)-P(2)-F(11)	179.88(11)
F(8)-P(2)-F(11)	90.10(9)
F(12)-P(2)-F(11)	89.62(9)
F(10)-P(2)-F(7)	89.92(9)

F(8)-P(2)-F(7)	179.91(12)
F(12)-P(2)-F(7)	90.04(9)
F(11)-P(2)-F(7)	89.97(9)
F(10)-P(2)-F(9)	90.38(10)
F(8)-P(2)-F(9)	90.14(9)
F(12)-P(2)-F(9)	179.27(10)
F(11)-P(2)-F(9)	89.65(9)
F(7)-P(2)-F(9)	89.93(9)

Symmetry transformations used to generate equivalent atoms: