

Electronic supplementary information

Complexation of Neutral 1,4-Dihalobutanes with Simple Pillar[5]arenes That is Dominated by Dispersion Forces

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Copies of ^1H NMR and ^{13}C NMR spectra of hosts.

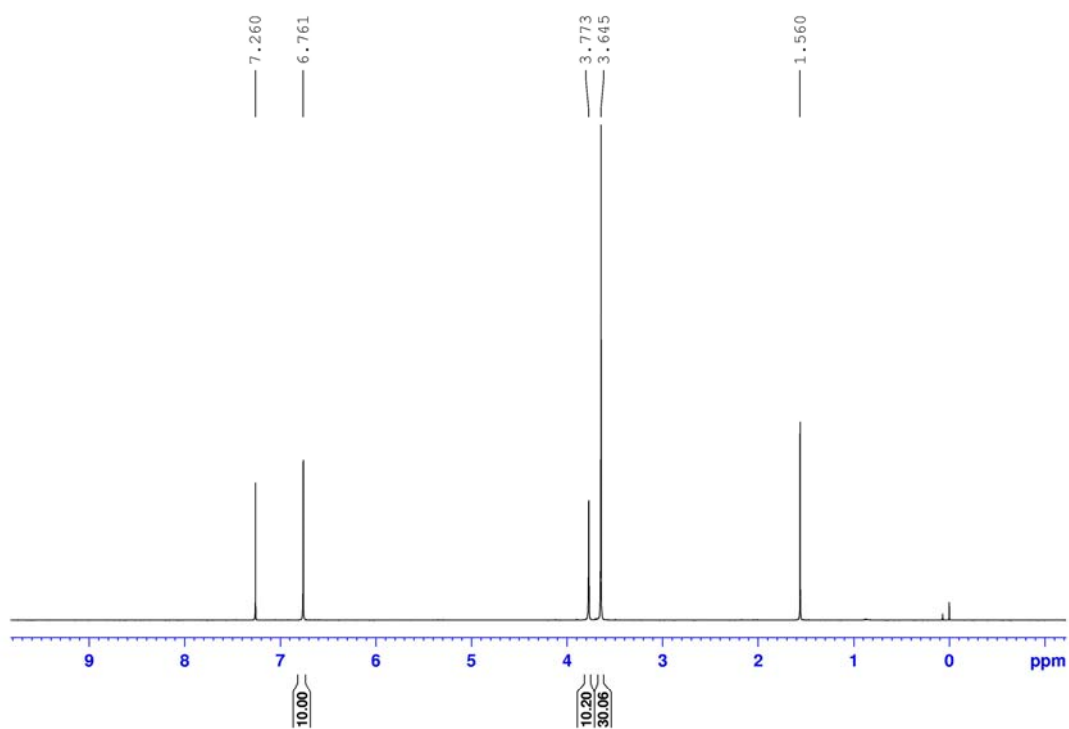


Figure S1. ^1H NMR spectrum (500 MHz) of MeP5A in CDCl_3 .

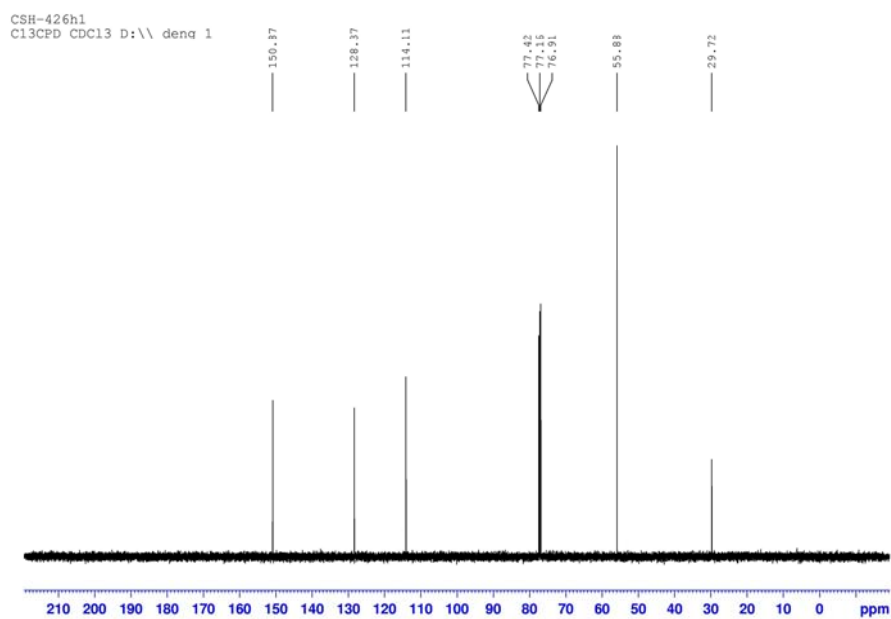


Figure S2. ^{13}C NMR spectrum (125 MHz) of MeP5A in CDCl_3 .

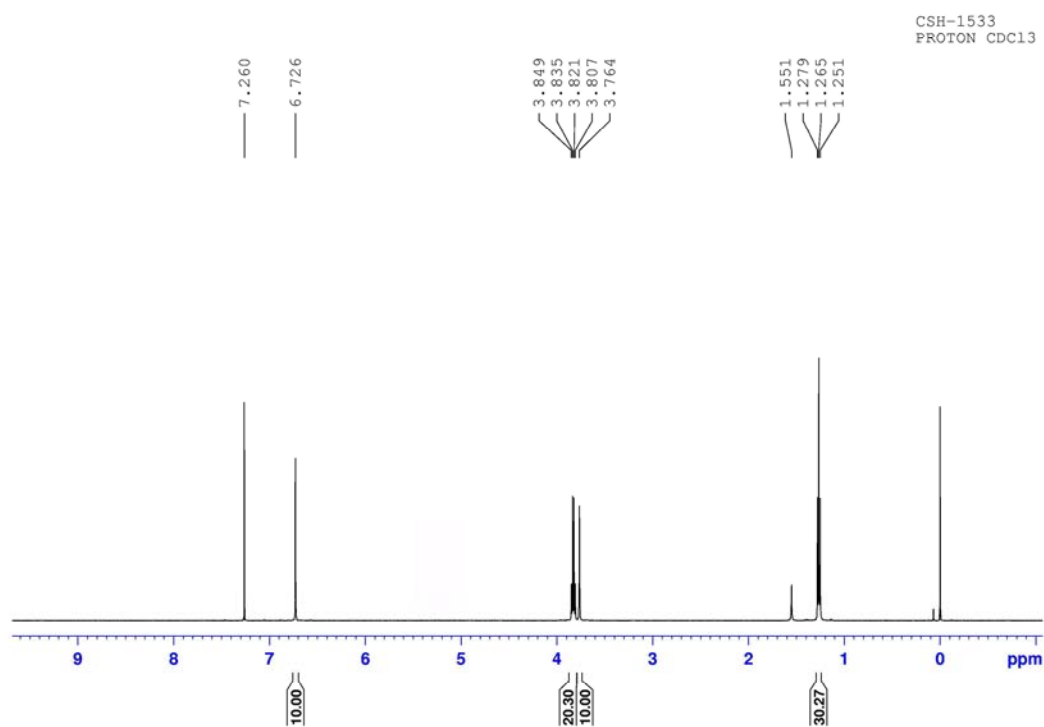


Figure S3. ¹H NMR spectrum (500 MHz) of EtP5A in CDCl₃.

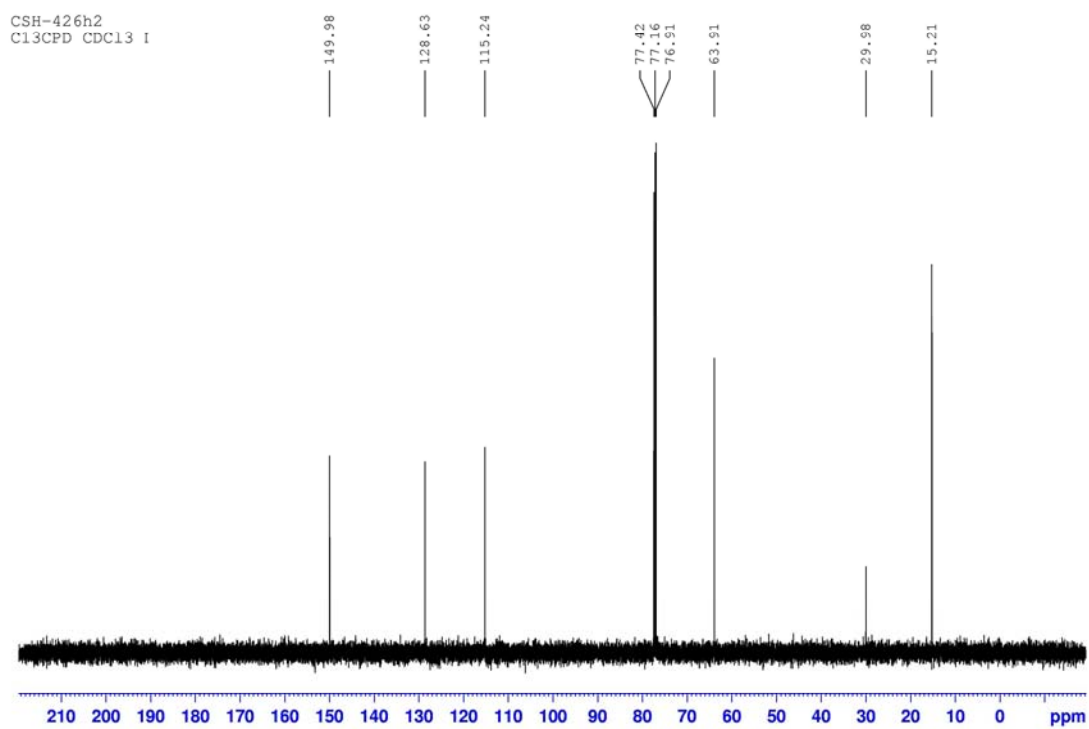


Figure S4. ¹³C NMR spectrum (125 MHz) of EtP5A in CDCl₃.

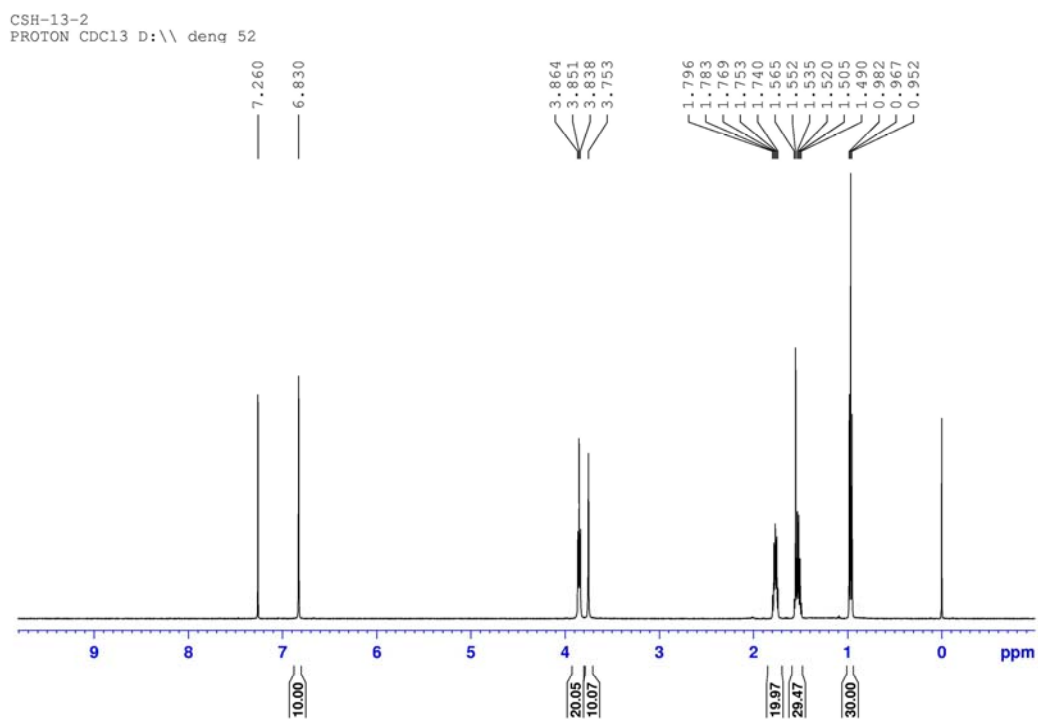


Figure S5. ^1H NMR spectrum (500 MHz) of BuP5A in CDCl_3 .

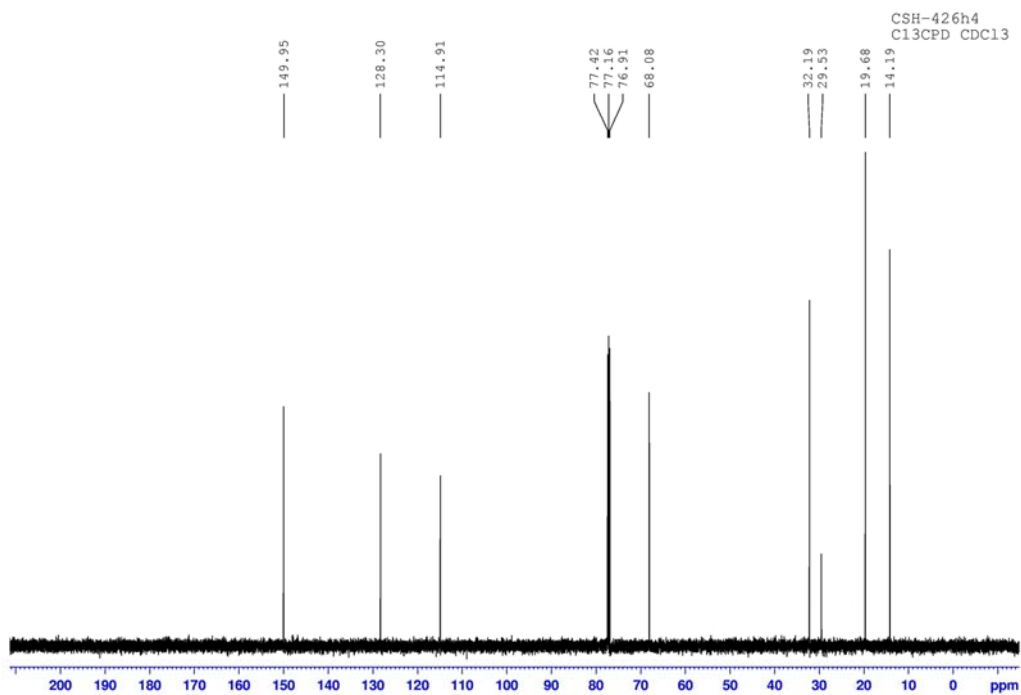


Figure S6. ^{13}C NMR spectrum (125 MHz) of BuP5A in CDCl_3 .

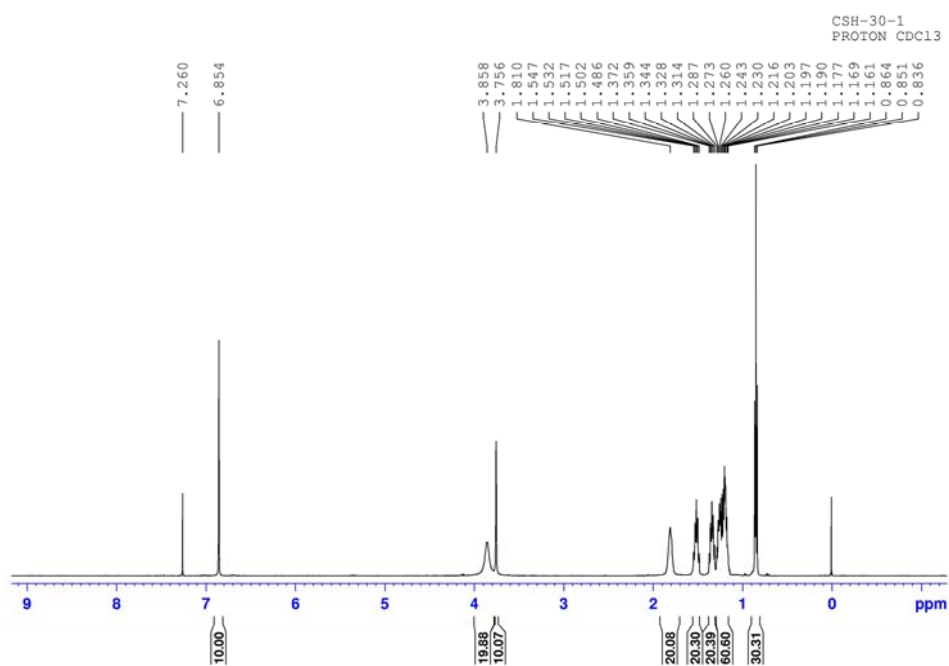


Figure S7. ¹H NMR spectrum (500 MHz) of OctP5A in CDCl₃.

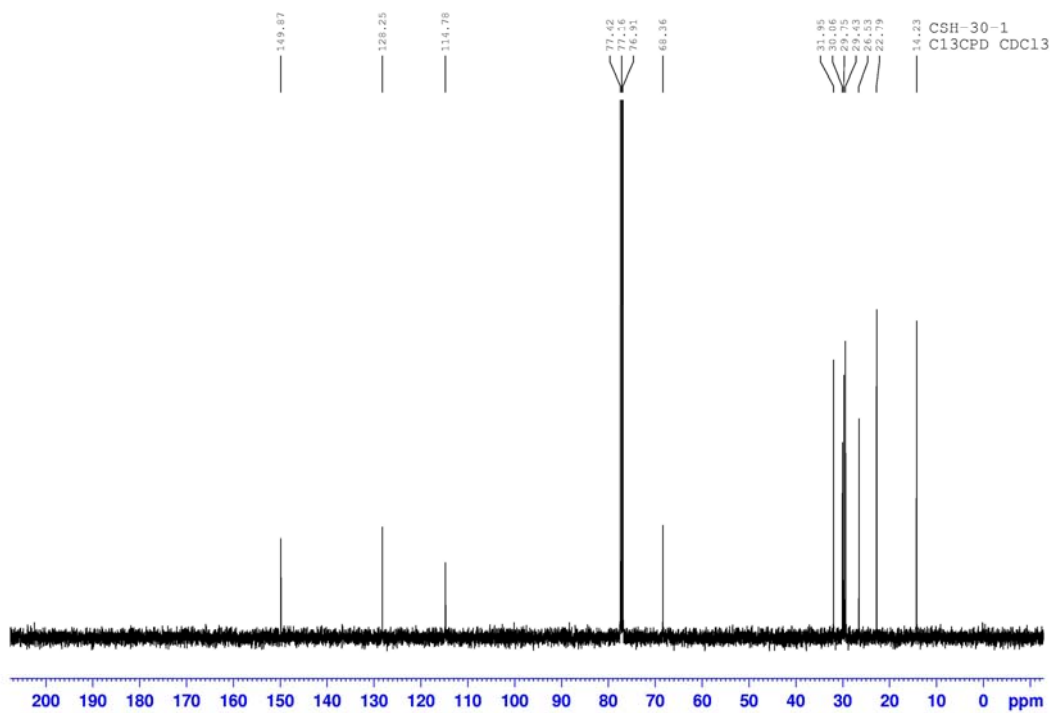


Figure S8. ¹³C NMR spectrum (125 MHz) of OctP5A in CDCl₃.

^1H NMR spectra of guests in the absence and presence of AIP5As.

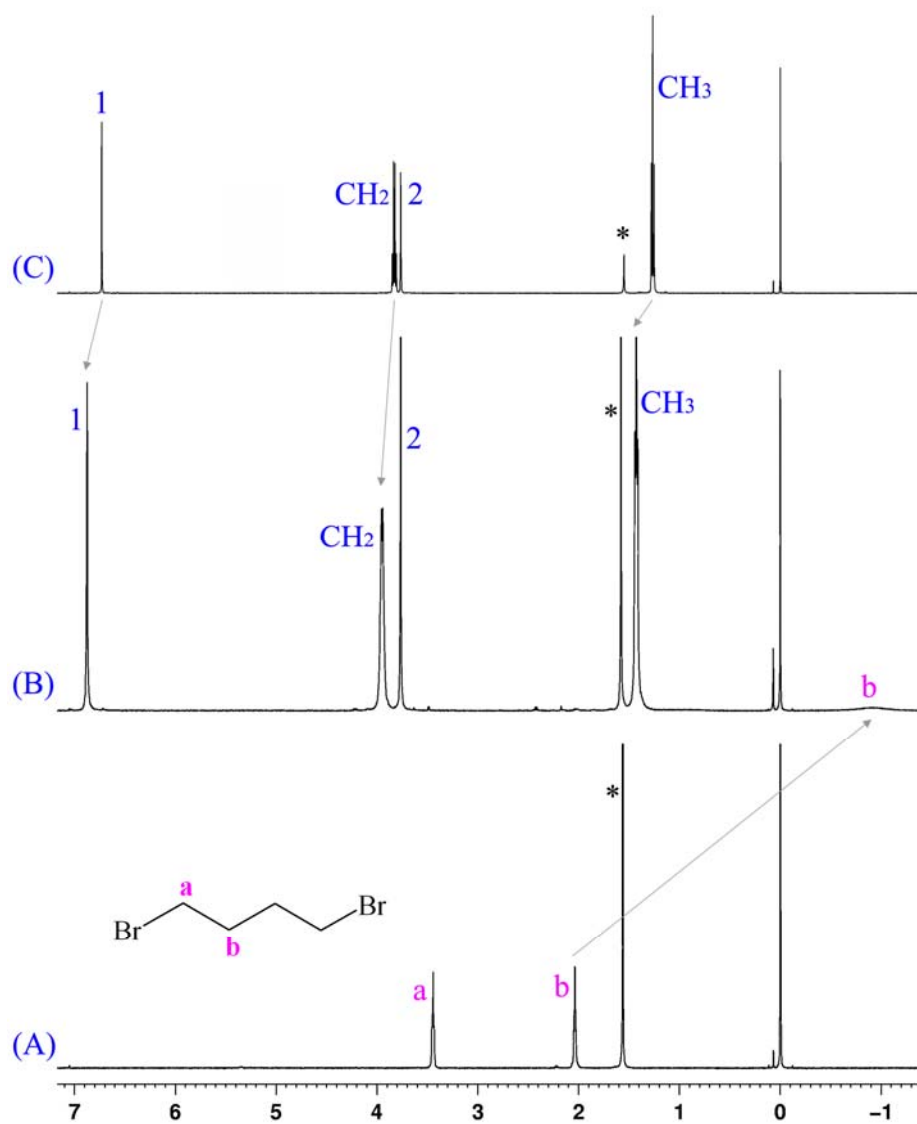


Figure S9. ^1H NMR spectra (500 MHz) of (a) DBrBu, (b) DBrBu + EtP5A, and (c) EtP5A in CD_3Cl at 4.2–5.0 mM. The peaks marked with an asterisk are due to water.

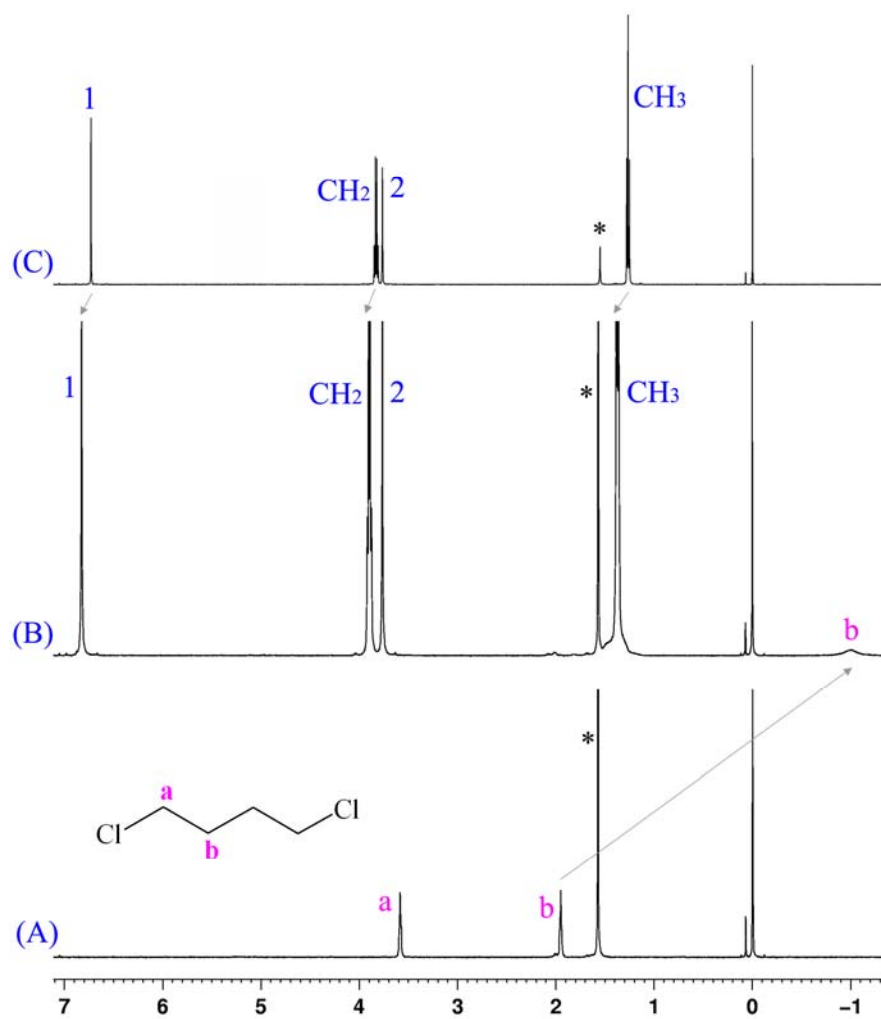


Figure S10. ¹H NMR spectra (500 MHz) of (a) DCIBu, (b) DCIBu + EtP5A, and (c) EtP5A in CD₃Cl at 4.4–5.2 mM. The peaks marked with an asterisk are due to water.

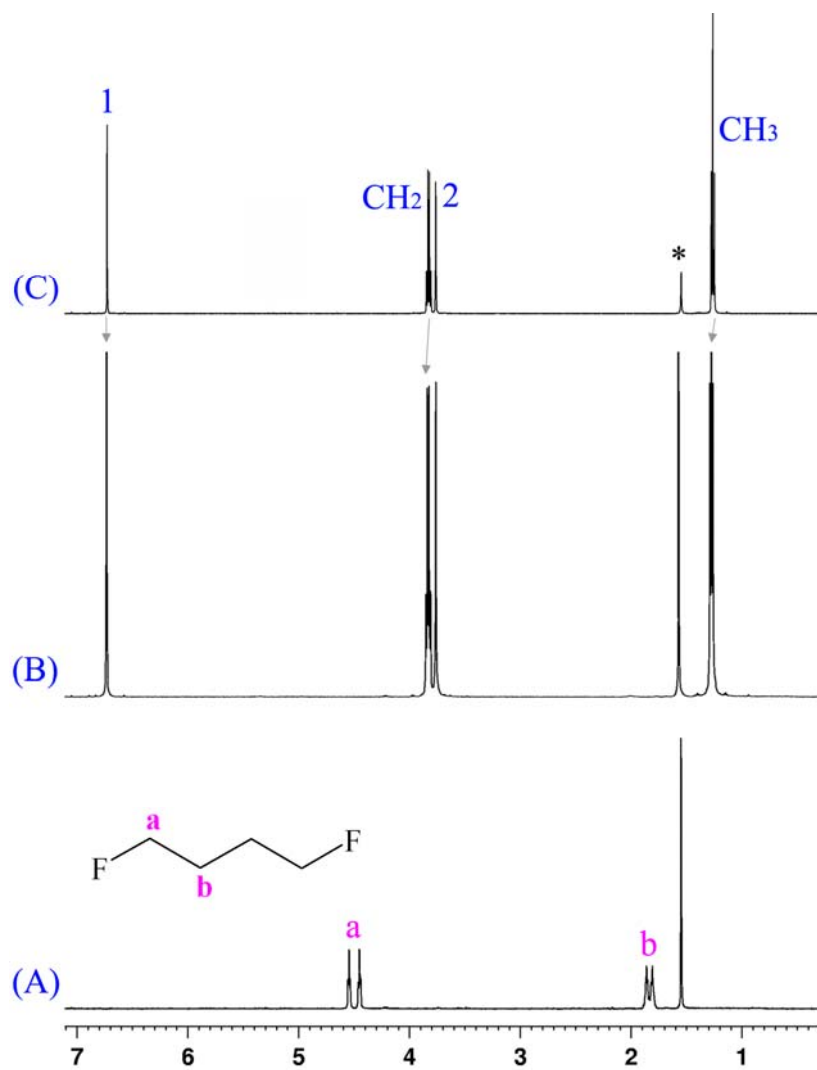


Figure S11. ^1H NMR spectra (500 MHz) of (a) DFBu, (b) DFBu + EtP5A, and (c) EtP5A in CD_3Cl at 4.3–5.2 mM. The peaks marked with an asterisk are due to water.

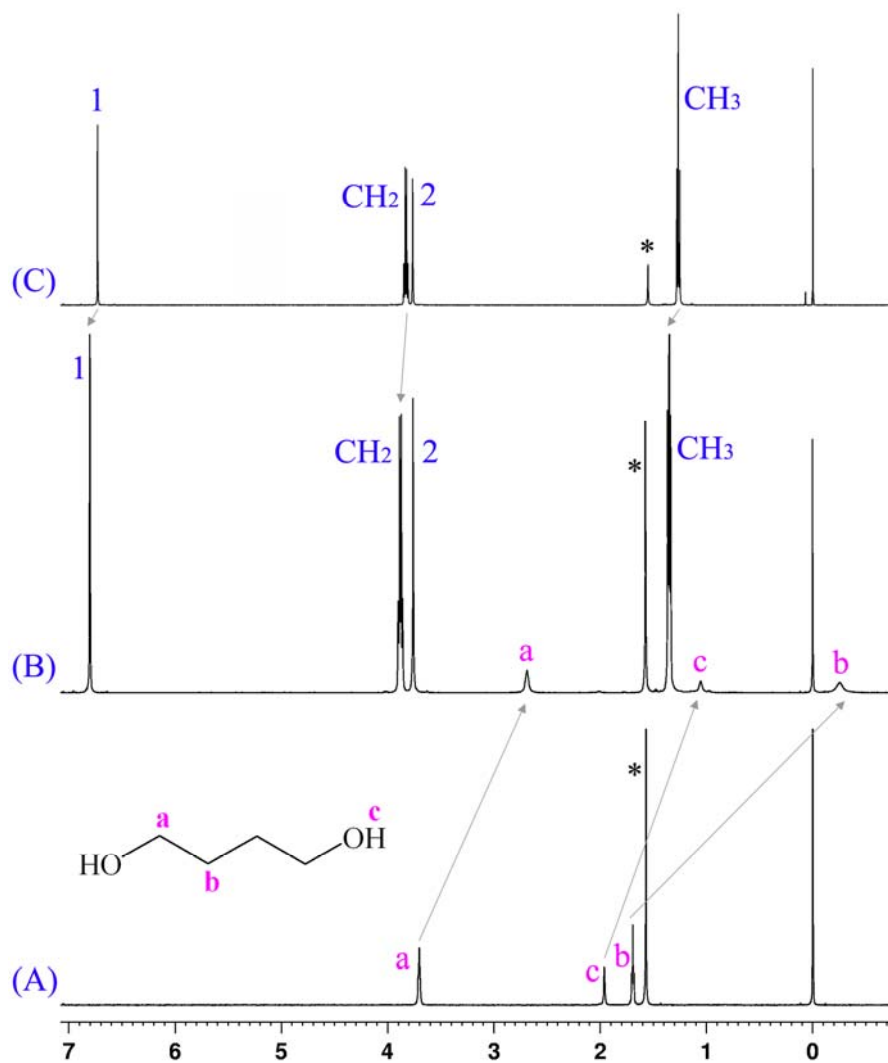


Figure S12. ^1H NMR spectra (500 MHz) of (a) DOHBU, (b) DOHBU + EtP5A, and (c) EtP5A in CD_3Cl at 4.1–4.7 mM. The peaks marked with an asterisk are due to water.

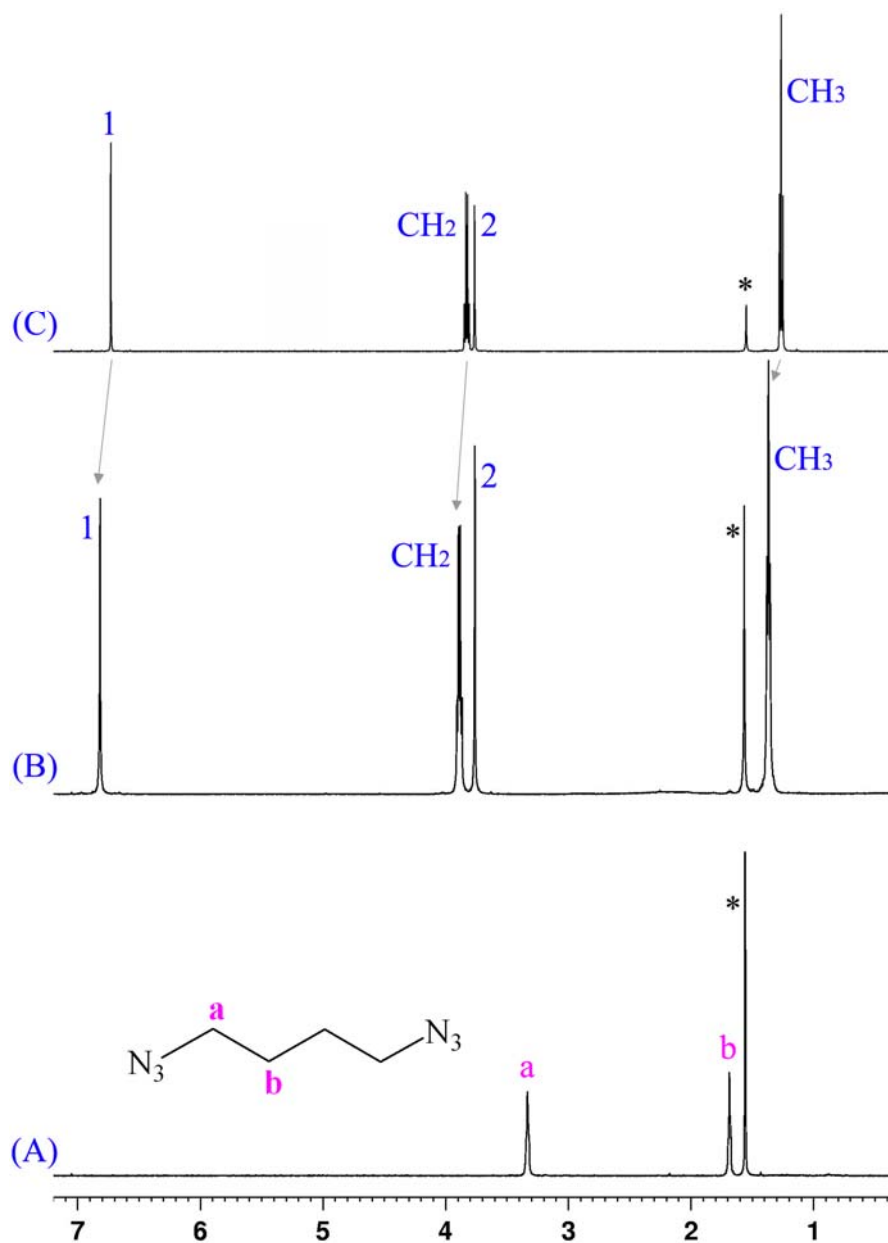


Figure S13. ¹H NMR spectra (500 MHz) of (a) DN₃Bu, (b) DN₃Bu + EtP5A, and (c) EtP5A in CD₃Cl at 4.0–4.6 mM. The peaks marked with an asterisk are due to water.

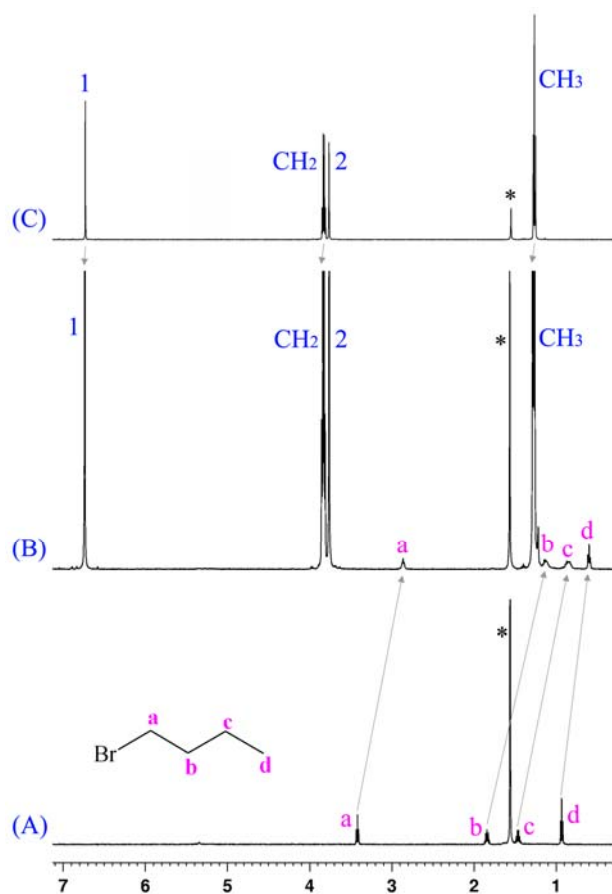


Figure S14. ^1H NMR spectra (500 MHz) of (a) BrBu, (b) BrBu + EtP5A, and (c) EtP5A in CD_3Cl at 4.0–4.6 mM. The peaks marked with an asterisk are due to water.

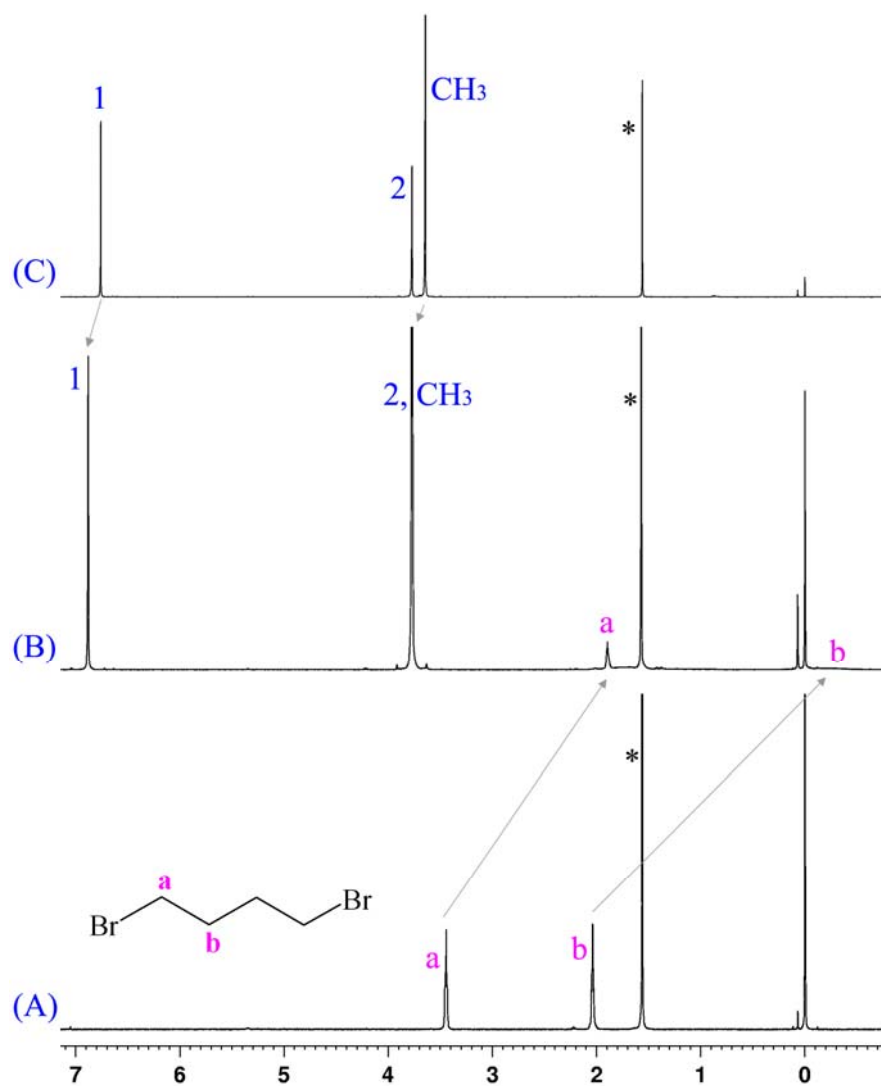


Figure S15. ¹H NMR spectra (500 MHz) of (a) DBrBu, (b) DBrBu + MeP5A, and (c) MeP5A in CD₃Cl at 4.2–4.8 mM. The peaks marked with an asterisk are due to water.

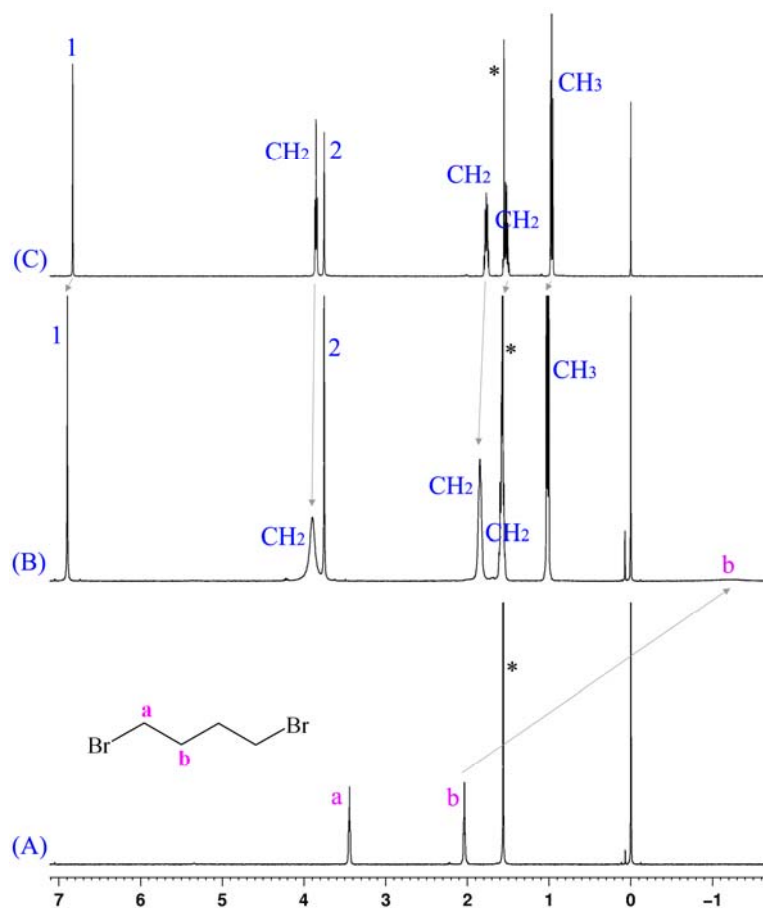


Figure S16. ¹H NMR spectra (500 MHz) of (a) DBrBu, (b) DBrBu + BuP5A, and (c) BuP5A in CD₃Cl at 4.2–5.0 mM. The peaks marked with an asterisk are due to water.

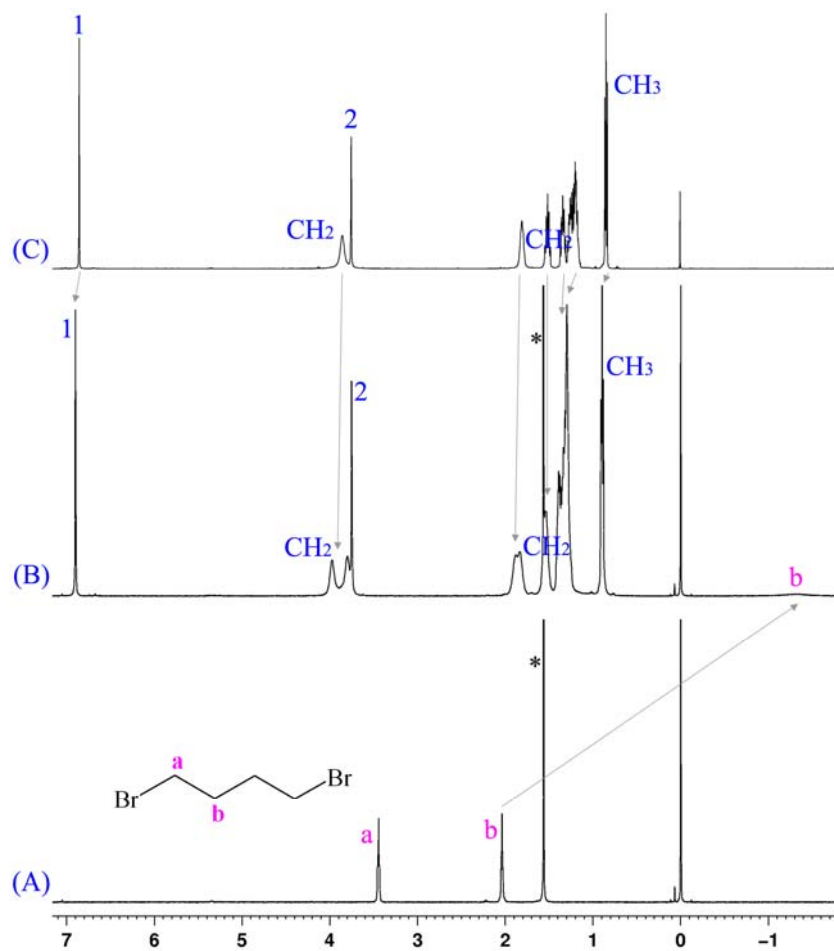


Figure S17. ^1H NMR spectra (500 MHz) of (a) DBrBu, (b) DBrBu + OctP5A, and (c) OctP5A in CD_3Cl at 4.2–5.1 mM. The peaks marked with an asterisk are due to water.

Crystal structures of DIBu \subset BuP5A, DFBu \subset BuP5A and DCIBu \subset OctP5A complexes.

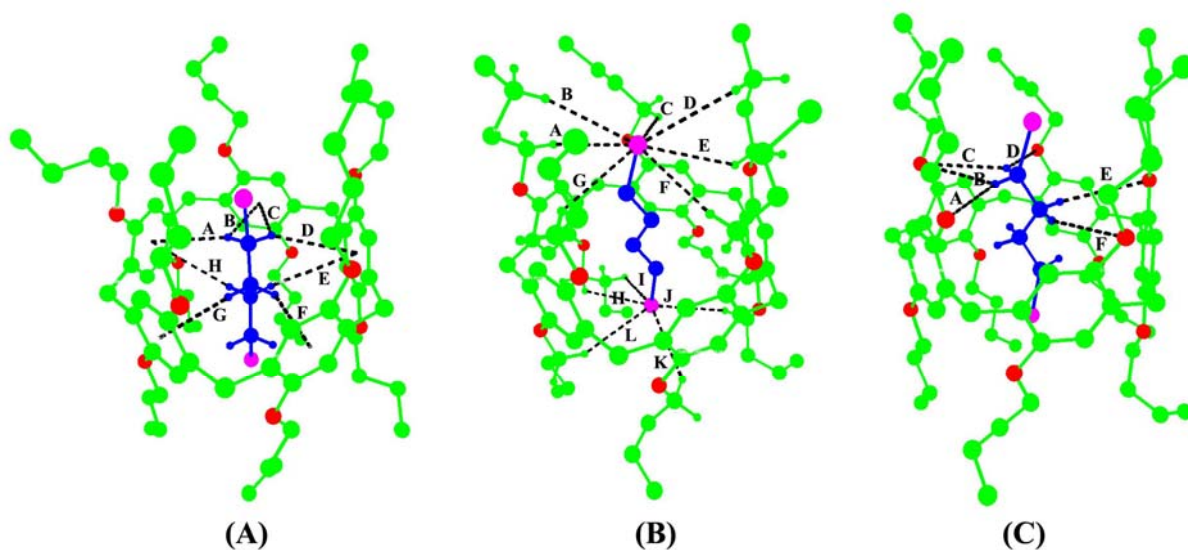


Figure S18. Crystal structure of the interpenetrated complex DIBu \subset BuP5A. Hydrogens of the host have been omitted for clarity. BuP5A is green, DIBu is blue, oxygens are red, and iodines are magenta. Dashes represent C–H \cdots π interactions or C–H \cdots I/O hydrogen bonds.

(A) C–H \cdots π parameters: H \cdots ring centre distances (Å), C–H \cdots ring angles (deg) A, 3.15, 156; B, 3.44, 111; C, 3.33, 118; D, 3.18, 144; E, 3.45, 135; F, 2.75, 144; G, 2.81, 176; H, 3.40, 150.

(B) C–H \cdots I hydrogen-bond parameters: H \cdots I distances (Å), C–H \cdots I angles (deg) A, 3.25, 162; B, 3.19, 167; C, 3.47, 135; D, 3.44, 135; E, 3.41, 138; F, 3.34, 168; G, 3.33, 139; H, 3.25, 130; I, 3.43, 123; J, 3.21, 142; K, 3.35, 138; L, 3.29, 154.

(C) C–H \cdots O hydrogen-bond parameters: H \cdots O distances (Å), C–H \cdots O angles (deg) A, 3.09, 155; B, 3.49, 102; C, 3.19, 133; D, 3.30, 135; E, 3.32, 144; F, 3.47, 162.

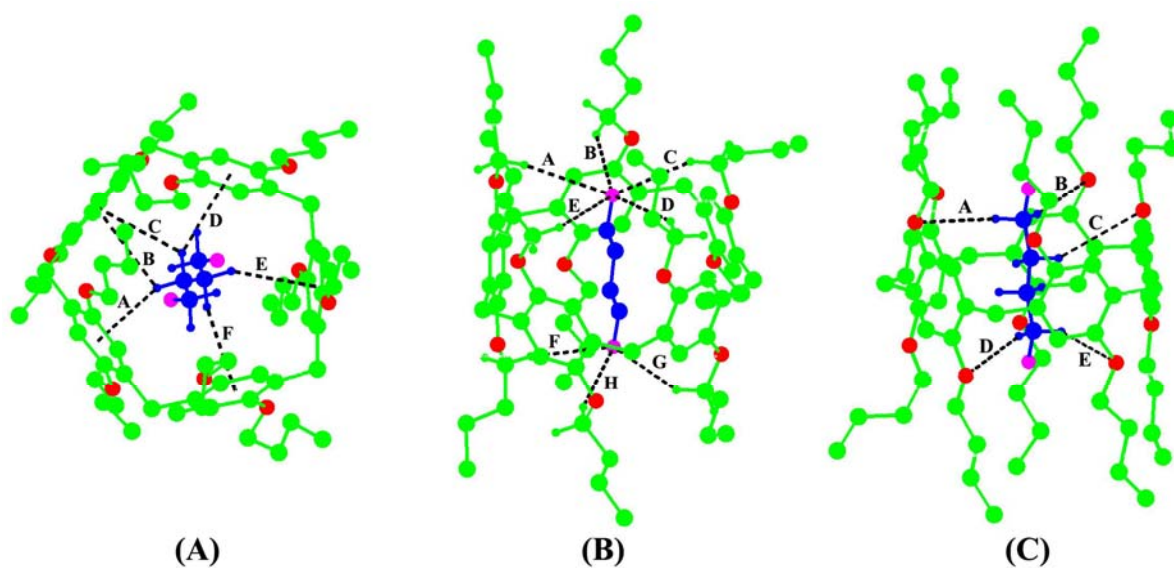


Figure S19. Crystal structure of the interpenetrated complex DFBu \subset BuP5A. Hydrogens of the host have been omitted for clarity. BuP5A is green, DFBu is blue, oxygens are red, and fluorines are magenta. Dashes represent C–H \cdots π interactions or C–H \cdots F/O hydrogen bonds.

(A) C–H \cdots π parameters: H \cdots ring centre distances (Å), C–H \cdots ring angles (deg) A, 3.17, 150; B, 3.44, 112; C, 3.42, 114; D, 3.14, 144; E, 3.07, 153; F, 2.98, 160.

(B) C–H \cdots F hydrogen-bond parameters: H \cdots F distances (Å), C–H \cdots F angles (deg) A, 3.21, 164; B, 2.90, 145; C, 2.87, 153; D, 3.39, 151; E, 3.48, 138; F, 2.92, 141; G, 3.02, 135; H, 3.11, 159.

(C) C–H \cdots O hydrogen-bond parameters: H \cdots O distances (Å), C–H \cdots O angles (deg) A, 3.39, 128; B, 3.19, 168; C, 3.38, 151; D, 3.35, 167; E, 3.11, 140.

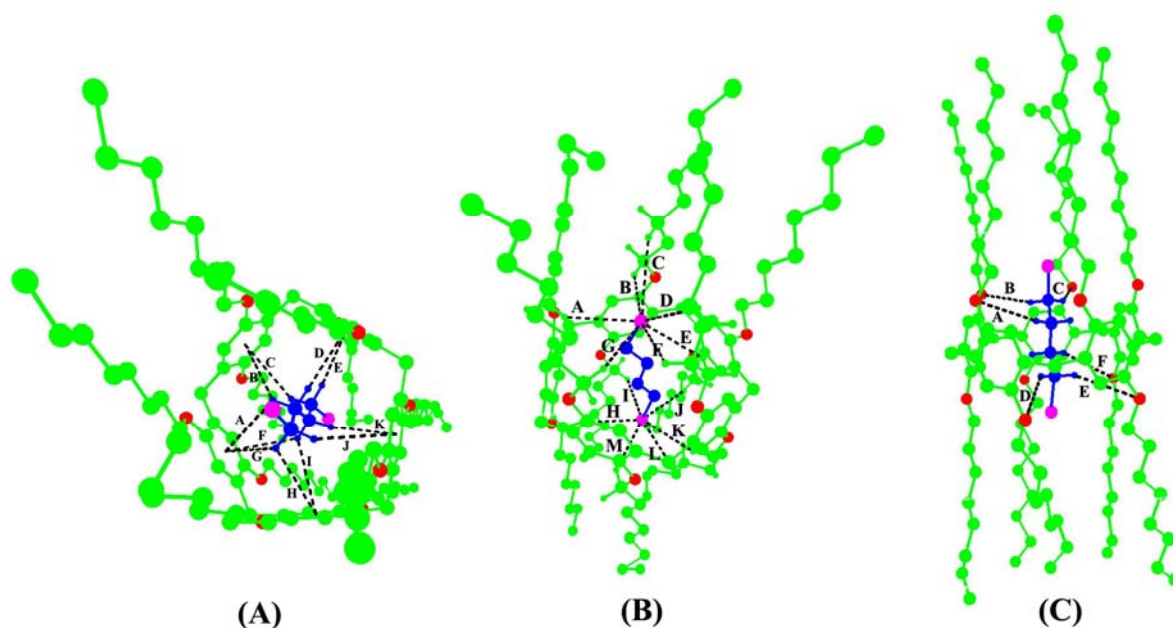


Figure S20. Crystal structure of the interpenetrated complex DCIBu ⊂ OctP5A. Hydrogens of the host have been omitted for clarity. OctP5A is green, DCIBu is blue, oxygens are red, and chlorines are magenta. Dashes represent C-H...π interactions or C-H...Cl/O hydrogen bonds.

(A) C-H...π parameters: H...ring centre distances (Å), C-H...ring angles (deg) A, 3.42, 116; B, 3.38, 137; C, 3.13, 138; D, 3.28, 144; E, 2.89, 166; F, 3.17, 132; G, 3.45, 130; H, 3.42, 115; I, 3.40, 122; J, 3.42, 142; K, 2.91, 160.

(B) C-H...Cl hydrogen-bond parameters: H...I distances (Å), C-H...Cl angles (deg) A, 3.23, 137; B, 2.84, 147; C, 3.45, 135; D, 3.30, 129; E, 3.03, 132; F, 3.26, 116; G, 3.02, 161; H, 3.11, 163; I, 2.94, 149; J, 3.22, 165; K, 3.05, 160; L, 3.49, 117; M, 3.02, 138.

(C) C-H...O hydrogen-bond parameters: H...O distances (Å), C-H...O angles (deg) A, 3.17, 164; B, 2.89, 165; C, 2.86, 140; D, 3.32, 139; E, 3.23, 159; F, 3.37, 155.

Job plots.

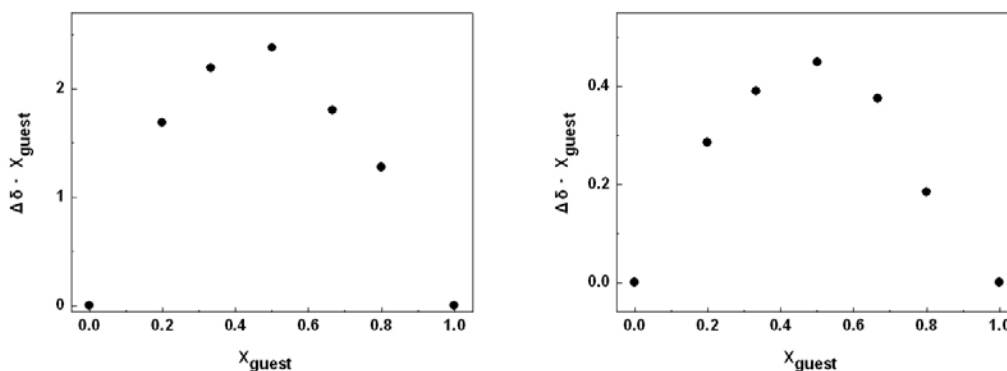


Figure S21. Left: Job plot showing the 1 : 1 stoichiometry of the complex between DOHBU and EtP5A in CDCl_3 by plotting the $\Delta\delta$ in chemical shift of the guest's methylene proton H_b observed by ^1H NMR spectroscopy against the mole fraction of guest (X_{guest}). ($[\text{host}] + [\text{guest}] = 12.0 \text{ mM}$). Right: Job plot showing the 1 : 1 stoichiometry of the complex between BrBu and EtP5A in CDCl_3 by plotting the $\Delta\delta$ in chemical shift of the guest's methyl proton observed by ^1H NMR spectroscopy against the mole fraction of guest (X_{guest}). ($[\text{host}] + [\text{guest}] = 16.0 \text{ mM}$).